PREDESIGN INVESTIGATION WORK PLAN

Operable Unit 1/Operable Unit 2 Portion of the Anniston PCB Site

Submitted by

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June 2022

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ACRONYMS AND ABBREVIATIONS

°C	degrees Celsius
APCO	Alabama Power Corporation
ASCII	American Standard Code for Information Interchange
ASTM	ASTM International
AWQC	ambient water quality criteria
BBL	Blasland, Bouck & Lee, Inc.
bgs	below ground surface
CD	Consent Decree
DGPS	differential global positioning system
DQO	data quality objective
DU	decision unit
EM	electromagnetic
EU	exposure unit
FEMA	Federal Emergency Management Agency
FS	feasibility study
GAC	granular activated carbon
gpm	gallons per minute
GPR	ground-penetrating radar
GPS	global positioning system
HASP	health and safety plan
HDPE	high-density polyethene
ID	identification
IDW	investigation-derived waste
IM	interim measure
ISM	Incremental Sampling Methodology
ITRC	Interstate Technology and Regulatory Council
JSA	Job Safety Analysis
LiDAR	light detection and ranging
µg/L	microgram(s) per liter
mg/kg	milligram(s) per kilogram

NAD83	North American Datum of 1983
NAVD88	National American Vertical Datum of 1988
NTC	non-time-critical
NTCRA	non-time-critical removal action
NTU	nephelometric turbidity unit
OU	operable unit
PAH	polycyclic aromatic hydrocarbon
PCB	polychlorinated biphenyl
PCDD/DF	polychlorinated dibenzo-p-dioxin and dibenzofuran
PDI	predesign investigation
PDIWP	Predesign Investigation Work Plan
PPE	personal protective equipment
PPIN	property identification number
P/S	Pharmacia LLC/Solutia Inc.
PTW	principal threat waste
QA/QC	quality assurance/quality control
RA	remedial action
RCRA	Resource Conservation and Recovery Act
RD	remedial design
RD/RA	remedial design/remedial action
RDWP	Remedial Design Work Plan
RF	radio frequency
RFI	RCRA Facility Investigation
RI	remedial investigation
ROD	Record of Decision
RSD	relative standard deviation
SHPO	State Historic Preservation Office
SOP	standard operating procedure
SOW	Statement of Work
SPT	standard penetration test
TEQ	toxic equivalent

TOC	total organic carbon
TS	treatability study
UAS	unmanned aerial system
UFP-QAPP	Uniform Federal Policy Quality Assurance Project Plan
USACE	United States Army Corps of Engineers
USCS	Unified Soil Classification System
USEPA	United States Environmental Protection Agency
USGS	United States Geological Survey
UST	underground storage tank

1. INTRODUCTION

This Predesign Investigation Work Plan (PDIWP) has been developed for the Operable Unit 1/Operable Unit 2 (OU-1/OU-2) portion of the Anniston PCB Site (the Site; Figure 1-1). The PDIWP is a deliverable under the OU-1/OU-2 Remedial Design/Remedial Action (RD/RA) Consent Decree (CD: United States Environmental Protection Agency [USEPA] 2021a) and was developed on behalf of Pharmacia LLC/Solutia Inc. (P/S). This PDIWP presents the field, laboratory, and desktop support activities that will be conducted to support the remedial design (RD) for the OU-1/OU-2 remedies identified in the CD and is consistent with the respective components of the OU-1/OU-2 Record of Decision (ROD; USEPA 2017).

This PDIWP is available as an electronic deliverable at PDIWP. Other publicly available technical documents for OU-1/OU-2 and the Site are available at Anniston PCB Site File Exchange.

1.1 Purpose of this Predesign Investigation Work Plan

As described in the OU-1/OU-2 Remedial Design Work Plan (OU-1/OU-2 RDWP; P/S 2021a), this PDIWP provides an evaluation and summary of existing data and a description of data gaps to be addressed as the second technical deliverable under the CD. Additionally, the means and methods of sampling and collecting other data (e.g., topographic surveys) to address identified data gaps as part of implementing the PDIWP are included. The PDIWP is based on technical requirements identified in the CD, including Section 3.3(a) of the Statement of Work (SOW), Appendix B to the CD.

1.2 Remedial Action Summary

The remedy USEPA selected for OU-1/OU-2 includes multiple components for soil, groundwater, and sediment. The ROD identified eight categories or types of remedies based on the range of remedial alternatives presented in the *OU-1/OU-2 Feasibility Study Report, Anniston PCB Site* (OU-1/OU-2 FS; Ramboll Environ 2016), and P/S and USEPA agreed that P/S would implement the selected remedy for seven of these categories. The seven categories of remedial alternatives for OU-1/OU-2 identified in the CD and the selected remedy for each category are as follows:

- **Residential soil:** The selected remedy for residential soil is removal and on-site or off-site disposal of surface soil (i.e., soil from ground surface to 12 inches below ground surface [bgs]) with polychlorinated biphenyl (PCB) concentrations greater than or equal to 1 milligram per kilogram (mg/kg) but less than 10 mg/kg and removal and off-site disposal of surface and subsurface soil (i.e., soil deeper than 12 inches bgs) with PCB concentrations greater than or equal to 10 mg/kg. The selected remedy also includes long-term soil management for (1) properties with residual PCBs greater than or equal to 1 mg/kg, (2) properties with PCB concentrations in soil greater than or equal to 1 mg/kg that have been designated as "unsuitable for removal," and (3) properties that potentially have residual PCBs beneath a structure (i.e., a building, shed, or paved area) that limits exposure.

- **Special use properties:** For high-activity special use properties, the selected remedy includes removal and on-site or off-site disposal of surface soil with PCB concentrations greater than or equal to 1 mg/kg but less than 10 mg/kg and removal and off-site disposal of subsurface soil with PCB concentrations greater than or equal to 10 mg/kg. For low-activity special use properties, the selected remedy includes removal and on-site or off-site disposal of surface soil with PCB concentrations greater than or equal to 1 mg/kg but less than 10 mg/kg and removal and off-site disposal of surface soil with PCB concentrations greater than or equal to 10 mg/kg and subsurface soil with PCB concentrations greater than or equal to 97 mg/kg. The selected remedy also includes long-term soil management for (1) properties where residual PCBs are at concentrations greater than or equal to 1 mg/kg, (2) properties with PCB concentrations in soil greater than or equal to 1 mg/kg that have been designated as "unsuitable for removal," and (3) properties that potentially have residual PCBs beneath a structure (i.e., a building, shed, or paved area) that limits exposure.
- **Interim measure (IM) areas:** The IM component of the OU-1/OU-2 remedy includes maintaining the existing IMs and excavating soil around the Northside Area, Eastside Area, and the Eastside Drainageway (including the drainage ditch to the north and PB-RR-37 area) IMs to meet the surface soil remedial goal for PCBs of 21 mg/kg and subsurface soil remedial goal for PCBs of 97 mg/kg. Excavated soil will be disposed of off-site. The OU-1/OU-2 remedy also includes assessing the potential for principal threat waste (PTW) to be present in the portion of OU-1/OU-2 that is located just east of OU-3. The presence of PTW will be characterized by groundwater with PCBs at concentrations greater than the State maximum contaminant level and the Federal Safe Drinking Water Act maximum contaminant level. If PTW is found to be present, the specific approach to address the PTW material will be developed in coordination with USEPA during the RD.
- **Dredge spoil piles:** The remedy includes removal and off-site disposal of four dredge spoil piles located near or adjacent to Snow Creek. The CD includes predesign investigation (PDI) sampling for dredge spoil pile SC-2, which was not characterized during the remedial investigation (RI) to assess whether PCB concentrations are greater than or equal to the threshold concentration of 3 mg/kg for dredge spoil pile removal in OU-1/OU-2 under the selected remedy. The surface soil located directly beneath the dredge spoil piles will also be evaluated for conformance with the surface soil remedial goal of 21 mg/kg. The subsurface soil will be evaluated using the remedial goal of 97 mg/kg. Long-term soil management for areas underlying dredge spoil piles with residual PCBs greater than or equal to 1 mg/kg is also part of the remedy.
- **Nonresidential soil:** For nonresidential soil, the remedy includes removal and off-site disposal of surface soil with PCB concentrations greater than or equal to 21 mg/kg, removal and off-site disposal for subsurface soil with PCB concentrations greater than or equal to 97 mg/kg, and long-term soil management for residual soil with PCB concentrations greater than or equal to 1 mg/kg. The remedy also includes the removal and off-site disposal of nonresidential soil from several small areas where non-PCB

constituents are present at concentrations greater than their respective remedial goals from the OU-1/OU-2 ROD.

- **Soil and groundwater at T-11:** For the T-11 area, the remedy includes removal and off-site disposal of soil from the upper 18-inch horizon across the broader T-11 area and the removal and off-site disposal of soil at depth directly surrounding groundwater well T-11; placement of a low-permeability cap over the broader T-11 area; the extraction, treatment, and discharge of groundwater from new groundwater wells installed in the T-11 area; and land use controls (e.g., deed restrictions or environmental covenant) that prohibit future groundwater use.
- **Sediment and creek bank soil:** For sediment and creek bank soil, the remedy includes stabilization of select creek bank areas and the removal and off-site disposal of sediment with PCB concentrations greater than or equal to 3 mg/kg. The remedy also includes the removal and off-site disposal of several sediment deposits containing non-PCB constituents at concentrations greater than their respective remedial goals from the OU-1/OU-2 ROD. The remedy for creek bank areas along Snow Creek includes stabilization of the specific reaches of the creek identified in the OU-1/OU-2 ROD.

Additional details for the remedy selected for each of the categories identified above are provided in Section 2.2 of the OU-1/OU-2 RDWP (P/S 2021a).

1.3 Predesign Investigation Objectives

The objectives of this PDIWP include collecting data that, in combination with existing data collected for OU-1/OU-2, will be used to develop the RD for implementing the OU-1/OU-2 remedy. These PDI objectives are consistent with the requirements of the supporting documents in Section 6.7 of the SOW and include the following:

- Presentation and evaluation of existing data and the identification of data gaps
- Field sampling plan, including the media to be sampled; number of samples to be collected, including locations, aerial extent, and depths; and contaminants to be analyzed
- Applicable cross references between the planned sampling activities and the Uniform Federal Policy Quality Assurance Project Plan (UFP-QAPP)

1.4 Organization of the PDIWP

This PDIWP includes nine sections, including this introduction, and five appendices:

- Section 2 presents existing data and identifies data gaps for OU-1/OU-2 floodplain and creek bank soil, groundwater, and Snow Creek sediment.
- Sections 3 and 4 describe the field and laboratory activities that will be conducted as part of the PDI to support OU-1/OU-2 RD development.
- Section 5 presents an overview of the access process and a summary of properties where access is needed to implement the PDI.

- Section 6 describes the health and safety plan (HASP).
- Section 7 describes the UFP-QAPP.
- Section 8 presents what will be included in the PDI evaluation report and the PDI schedule.
- Section 9 includes the references for documents cited in this PDIWP.
- Appendix A to this PDIWP is the UFP-QAPP.
- Appendices B and C provide the details of the treatability studies (TSs) for groundwater and sediment, respectively.
- Appendices D and E provide the details of the confined space entry plans for the Highway 202 culverts and the Quintard Mall culvert, respectively.

To document compliance with the SOW, Table 1-1 presents the SOW requirements and identifies the location in this PDIWP where the associated information is presented.

2. SUMMARY OF EXISTING DATA, DATA NEEDS, AND DATA GAPS

PDIWP development included reviewing existing data from the OU-1/OU-2 RI and FS and other historical reports and conducting a data gap analysis to identify the additional information needed to complete the OU-1/OU-2 RD as defined in the ROD. The PDI has been designed to collect the necessary and relevant information needed to successfully complete the OU-1/OU-2 RD. For each of the major components of the OU-1/OU-2 RD (i.e., floodplain and creek bank soil, groundwater, and Snow Creek sediment), this section presents a description of the existing data, data needs, and data gaps the PDI addresses. The associated scope of work and investigative methods for these data gaps are provided in Section 3.

2.1 Summary of Existing Data

The OU-1/OU-2 portion of the Site has been extensively sampled over the past 20-plus years, and the results of these investigations are presented in a collection of reports, including the OU-1/OU-2 RI (Environ 2015). Summaries of the existing data for floodplain and creek bank soil, groundwater, sediment, and surface water are provided in Sections 2.1.1 through 2.1.4, respectively.

2.1.1 Floodplain and Creek Bank Soil

The multiple categories of floodplain and creek bank soil identified in the OU-1/OU-2 ROD that have been part of the sampling that has been conducted over the past 20-plus years are presented in the OU-1/OU-2 RI and the *NTC Removal Action Completion Report for Residential Properties in OU-1/OU-2* (Completion Report; Golder 2015) and its associated annual addenda. The individual data for the residential properties and high-activity special use properties are not presented in this PDIWP due to their voluminous nature and because they have been presented in previous documents (OU-1/OU-2 RI and Completion Report). Additionally, all required removals have already been completed for the residential properties and high-activity special use areas where access is available and the properties are suitable for removal.

Summaries for the existing data for the other categories of floodplain soil are provided below, including low-activity special use areas, IM areas, dredge spoil piles, the low-permeability cover for the T-11 area, and select creek banks with stability-related concerns.

2.1.1.1 Low-Activity Special Use Areas

A significant amount of sampling was conducted for the low-activity special use areas under the *Stipulation of the Parties Clarifying Partial Consent Decree between USEPA, Solutia Inc., and Pharmacia Corporation* (Stipulation; USEPA 2006), and the results are reported in the Completion Report (Golder 2015). Soil sampling results for the low-activity portions of the special use properties are presented on Figures 2-1a through 2-1l of this PDIWP, and low-activity use areas targeted for remediation are identified on these figures. The low-activity area located in the southeast corner of exposure unit 14 (EU14) North (Figure 2-1j) no longer requires remediation because an electric utility company (Alabama Power Corporation [APCO]) has constructed a transformer facility on this property since the OU-1/OU-2 ROD was issued. The work conducted for the property was described in a construction support work plan (P/S 2021b), and additional clarification regarding transition of the property from a low-activity special use property to a

nonresidential industrial use property was provided by P/S in response (P/S 2021c) to comments from USEPA on the construction support work plan (USEPA 2021b). This land use condition transition will be confirmed during the PDI.

2.1.1.2 Interim Measure Areas

Each OU-1/OU-2 IM (Figure 2-2) was evaluated for effectiveness in the OU-1/OU-2 FS (Ramboll Environ 2016) and the OU-1/OU-2 ROD (USEPA 2017). Most of the IMs were deemed as being effective, with small areas identified where additional remedial efforts are needed to ensure long-term effectiveness. These remedial areas were identified in the OU-1/OU-2 ROD for portions of several IMs (Figures 2-3a through 2-3f). The remedial areas include portions of the Northside Properties (Figure 2-3a), portions of the Eastside Properties (Figures 2-3b through 2-3d), and portions of the Eastside Drainage Way (Figure 2-3e). An overview of the Hall Street IM is shown on Figure 2-3f. The sampling results for each of the IMs identified on Figure 2-2 and the construction-related reporting details are included in Appendix A of the OU-1/OU-2 RI (Environ 2016).

Surface soil remediation has already been conducted for one of the IM areas in the Northside Properties located along the western side of Clydesdale Ave just north of the railroad tracks (Figure 2-3a). The subsurface soil PCB concentrations will be evaluated for this IM as part of the PDI.

Results from a recent inspection of the Hall Street IM indicated that portions of the surface soil cover were disturbed. The soil disturbance occurred through the staging of construction equipment on the surface of the IM by a utility construction contractor. While the overall IM appears intact, P/S are working with the landowner (Oxford Water and Sewer Board) to inspect the IM as part of the PDI. This inspection will evaluate whether portions of the Hall Street IM cover soils require augmentation to meet the IM design requirements. The OU-1/OU-2 RD will identify physical controls to limit future unauthorized vehicle access to the Hall Street IM area.

2.1.1.3 Dredge Spoil Piles

Dredging that was previously conducted in Snow Creek by the City of Anniston resulted in a series of dredge spoil areas placed along the nearby banks of the creek. Information regarding these dredge spoil piles was presented in the OU-1/OU-2 RI (Environ 2015). Several of the dredge spoil piles were previously removed while four remain (Figure 2-4). The available analytical testing results for these dredge spoil piles are shown on Figure 2-5a for dredge spoil pile SC-1 and on Figure 2-5b for dredge spoil piles SC-7 and SC-8.

2.1.1.4 Nonresidential Soil Areas

A significant number of nonresidential soil samples have been collected for OU-1/OU-2 over the past 20-plus years. PCB results for the surface samples are shown on Figure 2-6, and the subsurface PCB results are shown on Figure 2-7. The results of this sampling were evaluated on an EU basis, and these EUs are shown on Figure 2-8. Summary-level statistics for PCBs in surface soil from the OU-1/OU-2 RI and FS are presented in Table 2-1 on an EU basis and include the number of samples collected, the mean of the detected samples, the percentage of the EU that is nonresidential versus residential or special use properties, and the spatially weighted mean PCB concentration

that accounts for exposure potentially occurring in residential/special use portions of the EU. The subsurface PCB results from the OU-1/OU-2 RI are presented in Table 2-2.

In addition to PCBs, the OU-1/OU-2 ROD identified the need for remediation at specific locations for three non-PCB constituents:

- Polycyclic aromatic hydrocarbons (PAHs) as benzo(a)pyrene equivalents
- Chromium
- Polychlorinated dibenzo-p-dioxin and dibenzofurans (PCDD/DFs, as 2,3,7,8-tetrachlorodibenzo-p-dioxin equivalent [toxic equivalent or TEQ])

The results of soil sampling conducted for these three constituents are presented on Figures 2-9, 2-10, and 2-11, respectively.

2.1.1.5 T-11 Area Soil

The T-11 area is located adjacent to Snow Creek and just downstream of its confluence with the 11th Street Ditch. This area is highlighted in dark-blue shading on Figure 2-12, and the figure includes PCB concentrations for surface and subsurface soil. A low-permeability cover will be placed over this footprint following removal of an 18-inch layer of soil over the T-11 area and the deeper excavation of soil potentially acting as PTW directly surrounding groundwater well T-11.

2.1.1.6 PB-RR-37 Area

PCB concentrations in surface soil within the PB-RR-37 area are characterized by two soil samples (PB-RR-37 and PB-RR-40) that USEPA collected in March 2000 (Figure 2-3e). The samples have identical location coordinates and were reported to have been collected from the interval of 0 to 3 inches below ground surface (bgs). Sample PB-RR-37 had a reported total PCB concentration of 3,700 mg/kg, and PB-RR-40 had a total PCB concentration of 2.3 mg/kg. Because the reported concentrations of these co-located samples varied by more than 3 orders of magnitude, additional soil sampling in this area is proposed to evaluate the representative PCB concentrations in surface soil and subsurface soil relative to the respective remedial goals of 21 mg/kg and 97 mg/kg. Subsurface soil sampling at PB-RR-37 will also be conducted to the elevation of the groundwater table and if PCBs are detected in soil at the groundwater interface, the potential installation of a groundwater well at this location will be discussed with USEPA.

2.1.1.7 Select Creek Bank Areas with Stability Concerns

Creek banks along Snow Creek were inventoried and assessed in the OU-1/OU-2 RI and further evaluated in the OU-1/OU-2 FS. The results of the evaluation indicated that long stretches of Snow Creek were bordered by hardened structures such as concrete sidewalls or riprap. The other creek bank areas were inspected during the OU-1/OU-2 RI, and three general locations were identified in the OU-1/OU-2 FS as having concerns for creek bank stability based on a combination of bank conditions and elevated PCB concentrations in floodplain soil proximate to the creek banks. Two of the locations are in the EU5 portion of Snow Creek, including just downstream of the confluence of Snow Creek and the 11th Street Ditch and at the downstream end where EU5 intersects EU10

(Figure 2-13a). The third creek bank stability area is located downstream of Highway 202 (Figure 2-13b).

2.1.2 Groundwater

Groundwater investigations were conducted in OU-1/OU-2 prior to the RI as part of previous activities conducted at and adjacent to OU-3 and under the non-time-critical (NTC) residential removal program. Groundwater was also investigated as part of the RI. Details of investigations and their results relevant to the groundwater RD for the Eastside Properties and T-11 area are provided in the following subsections. Section 2.1.1.6 above discussed existing data for the PB-RR-37 area, where the need for groundwater RD activities will be determined based on findings of the PDI.

2.1.2.1 Eastside Properties

As discussed in the OU-1/OU-2 ROD, PCB concentrations in soil in portions of the Eastside Properties have been detected at concentrations over 500 mg/kg, indicating the potential presence of PTW. Between 1997 and 2012, IMs were implemented to reduce potential exposure to and migration of PCB-impacted soil. The IMs generally included soil caps where total PCB concentrations were less than 500 mg/kg and geomembrane caps where PCB concentrations were over 500 mg/kg. Existing soil characterization data for the Eastside Properties shown on Figures 2-14a through 2-14c were used to evaluate potential locations for a new groundwater monitoring well that met the following criteria: (1) an area with low or no PCB impacts in shallow soil to minimize potential drag-down of PCBs from soil to groundwater during well installation and (2) an area close to and downgradient of areas within the Eastside Properties with high PCB concentrations in soil.

2.1.2.2 T-11 Area

Groundwater samples collected from monitoring well T-11 (Figure 2-12) as part of the OU-1/OU-2 RI in 2008 exhibited total PCB concentrations in unfiltered samples ranging from 3.3 micrograms per liter (µg/L) (Aroclors) to 17.3 µg/L (homologs), with a nondetect result (Aroclors) and a concentration of 10.4 µg/L (homologs) reported following redevelopment of the monitoring well that was completed due to high turbidity noted in the initial samples (Environ 2015). Additional samples collected from T-11 in 2012 exhibited total PCB concentrations in the unfiltered sample between 12 µg/L based on Aroclors and 30 µg/L based on the sum of PCB homologs (Environ 2015). As noted in the ROD, the distribution of PCBs in the floodplain area surrounding T-11 appeared to be unique for the Site, characterized by the presence of the lower-chlorinated PCB compounds (e.g., Aroclor 1221 and monochlorobiphenyl and dichlorobiphenyl homolog groups).

Existing hydrogeologic data related to T-11 groundwater includes information on the dominant soil type observed during well installation, groundwater elevation data over time, and aquifer slug testing data. The soils encountered at T-11 consisted of an “orange to red, stiff to soft clay” that were present from ground surface to the termination depth of 21 feet bgs (Environ 2015). Based on the proximity of T-11 to the surface water of Snow Creek, the well was installed to monitor an interval of “minor moisture” observed from 6 to 10 feet bgs. The well is constructed with a 5-foot screen placed from 8.5 to 13.5 feet bgs. The water table at T-11 was observed at approximately 7 feet bgs following installation. Recent depth-to-water measurements have been consistent with

the initial observation, corresponding to a static water elevation of approximately 687.7 feet above mean sea level. The hydraulic conductivity reported for T-11 based on slug testing was 5.93 feet per day, corresponding to 2 x 10^{-3} centimeters per second (Environ 2015).

2.1.3 Snow Creek Sediment

Multiple sediment sampling programs provided data that were presented in the OU-1/OU-2 RI and used in the OU-1/OU-2 FS to evaluate sediment conditions in the OU-1/OU-2 portion of Snow Creek. Sediment samples were collected during the following sampling programs from locations shown on Figure 2-15:

- The Off-Site Resource Conservation and Recovery Act (RCRA) Facility Investigation (RFI) Program (Blasland, Bouck & Lee, Inc., [BBL] 1999)
- The Field Sampling Plan for Non-Residential Properties of the Operable Unit 1/Operable Unit 2 Area of the Anniston PCB Site (BBL 2004) and associated addenda (BBL 2005 and 2006)
- The USEPA-led sediment sampling in 2000 (these data were provided to P/S via electronic data transfer [i.e., sediment samples collected at seven locations])
- West 9th Street Creek sediment sampling data that provides background results for locations upstream of the OU-1/OU-2 portion of Snow Creek (Solutia 2003)

Sediment data generated during these investigations served as the basis for evaluating the remedial alternatives presented in the OU-1/OU-2 FS. Most of the sediment samples and geomorphological information for Snow Creek were collected in 1999 as part of the Off-Site RFI program. The initial steps for this program included a detailed geomorphological mapping of the creek to identify the sediment deposits in the creek bed. The geomorphological information included mapping the sediment deposit locations, dimensions, and material types based on visual observations and geomorphological characterization (e.g., channel deposit, aggrading bar, bank deposit, terrace deposit, sand bar). The creek mapping results, which showed that some portions of the creek bed had no sediment due to localized high surface water velocity conditions, were used to develop the sampling strategy to characterize the Snow Creek sediment deposits for the Off-Site RFI. The data for that sampling program were reported in the *Off-Site RCRA Facility Investigation Report* (BBL 2000). The RFI investigation for Snow Creek included collecting 73 sediment samples from 34 individual locations within the OU-1/OU-2 portion of Snow Creek downstream of the creek's confluence with the 11th Street Ditch. Each sample was analyzed for PCBs, total organic carbon (TOC), and grain-size distribution. At the request of the Alabama Department of Environmental Management, approximately 10% of the samples with a bias toward the fine-grained sediment were analyzed for the following metals: arsenic, barium, beryllium, cadmium, chromium, cobalt, lead, manganese, mercury, nickel, and vanadium. An additional round of sediment sampling in the OU-1/OU-2 portion of Snow Creek was conducted under the Nonresidential Properties field sampling plan and associated addenda (BBL 2005, 2006). During this program, three sediment samples were collected from locations chosen to reflect low, medium, and high PCB concentrations. The primary goal of this sampling program was to generate data to evaluate, in addition to PCBs and TOC, the wider list of constituents over the range of PCB concentrations

typically present in Snow Creek. Data from this sampling program were reported in the OU-1/OU-2 RI (Environ 2015).

The primary constituent of concern in sediment is PCB (Ramboll Environ 2016). PCB concentrations in sediment are shown on Figure 2-16 based on the downstream distance (in miles) between the sample collection location and the confluence of Choccolocco Creek (i.e., the downstream end of OU-4) and Lake Logan Martin. Figure 2-16 also presents PCB results for samples that were collected upstream of the OU-1/OU-2 portion of Snow Creek to provide background context. Results for non-PCB constituents in sediment are respectively shown on Figures 2-17 through 2-24 for barium, chromium, cobalt, lead, manganese, mercury, nickel, and vanadium.

The existing sediment sampling data indicate that the highest total PCB concentrations (60 mg/kg) in Snow Creek sediment are generally found in the upstream portions of the creek between the 11th Street Ditch and Highway 202, including the culverts that convey flow under the highway. The data also indicate that sediment deposits in a small portion of Snow Creek near a railroad bridge located just downstream of Highway 202 have a mean sediment PCB concentration of 12 mg/kg. The sediment in the portion of Snow Creek between the railroad bridge and Highway 78 had an average PCB concentration of approximately 1.9 mg/kg. None of the prior sampling efforts included sediment sampling within the culvert beneath Quintard Mall, which is immediately upstream of Highway 78 (construction of Quintard Mall and the associated culvert was underway during the 1999 RFI). The culvert beneath Quintard Mall will be probed and sampled as part of the PDI (Section 3.3.2).

The OU-1/OU-2 FS identified two other sediment deposits as candidate remedial areas. These two areas had non-PCB constituents at concentrations above the remedial goals and PCB concentrations below the remedial goals. These sediment deposits were in the downstream portion of Snow Creek (i.e., in the vicinity of Snow Creek railroad crossing along South Noble Street [Figures 2-16 through 2-24]). One sediment deposit contained manganese above the remedial goal, and the other contained chromium, lead, manganese, and nickel above the remedial goals.

The existing investigation data indicated that Snow Creek sediments were predominantly coarse grained; only 26% of the deposits were characterized as containing more than trace amounts of silt and clay.

Snow Creek is a high-gradient stream, with a typical slope of 20 to 30 feet per mile, flowing through an urbanized corridor of Anniston and Oxford. The mean flow within Snow Creek increases from approximately 5 cubic feet per second at the confluence with the 11th Street Ditch to 28 cubic feet per second at the confluence with Choccolocco Creek. The steep basin terrain produces sharp peak flows. The estimated 10-year and 100-year recurrence interval floods for Snow Creek at the point it discharges to Choccolocco Creek are 4,030 and 6,900 cubic feet per second, respectively (Federal Emergency Management Agency [FEMA] 1993), with in-channel velocities frequently more than 5 feet per second during these large storm events.

There are no surface water gauging stations on Snow Creek, so a localized determination of the frequency of flood events cannot be calculated directly from Snow Creek data. There are two

gauging stations maintained by the United States Geological Survey (USGS) on Choccolocco Creek—one near Boiling Spring, Alabama (Station Number 04203310), and one approximately 30 miles downstream at Jackson Shoal near Lincoln, Alabama (Station Number 0240440). Data from these gauging stations were used to evaluate regional flood frequency. The USGS data from 1961 to 2019 at Jackson Shoal were used to calculate the 2-year storm flow of 11,500 cubic feet per second at Jackson Shoals near the downstream end of OU-4. The station near Boiling Spring was operational later, and data from 2003 to 2019 were used to calculate a 2-year storm flow of 3,350 cubic feet per second. The 2-year storm flow was exceeded 11 times at Jackson Shoals over the past 20 years. At Boiling Springs, the 2-year storm flow was exceeded 14 times over the period of record. These data indicate that storm flows that have occurred in the region appear to be in line with typical conditions over the period since the RFI sediment sampling program was conducted.

2.1.4 Snow Creek Surface Water

Surface water samples were collected during two sampling programs that provided data that were presented in the OU-1/OU-2 RI and used in the OU-1/OU-2 FS to evaluate surface water conditions in the OU-1/OU-2 portion of Snow Creek. The first sampling was conducted as part of the RCRA Program, and the second was conducted under the Nonresidential Properties FSP and associated addenda. Under the RCRA program, samples were collected from four locations that were selected to reflect the potential downstream movement of PCBs and sediment particulates from Snow Creek (OU- 1/OU-2) to Choccolocco Creek (OU-4). Two of the sampling stations were located approximately 2,000 feet (14th Street) and 3,000 feet (16th Street) upstream of the 11th Street Ditch–Snow Creek confluence. The other sampling stations were located downstream of OU-1/OU-2 at Snow Street and Oxford Lake Park. A total of 6 base-flow and 19 high-flow events were sampled at the downstream sampling stations. A single round of base-flow samples was collected at each of the upstream locations.

The second surface water sampling program was conducted under the Nonresidential Properties FSP and associated addenda in 2007. This program included collecting four whole-water samples during three different high-flow events. The samples were collected using an automated in-water ISCO sampler that was triggered by a flow gauge mounted on the bank near the sampling location. The sampling station was in the downstream area of Snow Creek (Oxford Lake Park). This location is just downstream of OU-1/OU-2 and was chosen to be representative of conditions at the downstream end of the OUs.

The surface water data for Snow Creek indicate that during base-flow conditions the downstream PCB contributions from Snow Creek to OU-4 are small and that total suspended solids and PCB transport in Snow Creek are extremely responsive to high-flow events. Most annual sediment transport occurs infrequently and over short periods during high-flow events. Given the low concentration of solids generally present in the creek during periods of low flow, the surface water PCB concentrations are expected to be quite low. This is important as base-flow conditions for the creek occur approximately 90% of the time.

2.2 Identification of Data Needs and Data Gaps

During the review of existing data from the OU-1/OU-2 RI, the OU-1/OU-2 FS, and other historical reports, data needs and data gaps were identified for floodplain and creek bank soil, groundwater, Snow Creek sediment, and site-wide evaluations for utilities, structures, and topo-bathymetry that need to be addressed to complete the OU-1/OU-2 RD as defined in the ROD. Descriptions of these data needs and data gaps are provided in the following subsections.

2.2.1 Floodplain and Creek Bank Soil

The existing floodplain soil and creek bank data were reviewed relative to the data needed to prepare an RD that meets the requirements of the OU-1/OU-2 ROD to identify data gaps. Data gaps were not identified for residential soil because all removal actions have been conducted for residential properties in OU-1/OU-2 where access has been granted and the properties have been identified as suitable for removal. No data gaps were identified for the high-activity special use areas because these areas have been sampled and removal actions completed where required under the Non-Time-Critical Removal Action (NTCRA) Agreement.

Comparing the data needed to complete the RD with the existing data did reveal data gaps for several other categories of floodplain soil and the creek bank areas identified in the OU-1/OU-2 ROD. The results of this comparison are presented below for the low-activity special use areas, IM areas, dredge spoil piles, nonresidential soils, and creek bank areas.

- **Low-activity special use areas:** PCB concentration data for subsurface soil in the low-activity special use area are needed to compare with the remedial goal of 97 mg/kg. While these data could be collected as confirmation samples during remedial action (RA) construction, most of these low-activity areas are easy to access, and the data can be efficiently collected during the PDI and used to scope the required excavation depths as part of the OU-1/OU-2 RD. Land use conditions at the low-activity special use areas will be reviewed during the PDI prior to completing sampling to confirm they are consistent with the Stipulation's definition of a low-activity special use area and are suitable for removal (e.g., are not heavily vegetated or wooded; like low-activity special use area with property identification number [PPIN] 20717). Documentation of current land use will be collected for all low-activity special use areas identified in the OU-1/OU-2 ROD. This information will be used to confirm that the land uses initially identified as part of the investigations conducted under the Stipulation have not changed. For example, if a low-activity area has transitioned to industrial use, relevant information (ownership, zoning, the removal or addition of built-structures, etc.) will be gathered to document this change in land use. If no changes to the properties have taken place since the initial surveys were conducted under the Stipulation, the plan is to conduct the sampling as described in Section 3.1.2.1. Any changes in land use will be discussed with USEPA and may result in modifying the sampling program. Consistent with the OU-1/OU-2 ROD, subsurface soil sampling will be conducted only for low-activity special use areas that are suitable for removal. For example, overgrown and paved areas will not be sampled.

- **IM areas:** PCB concentrations in subsurface soil for the specific IM areas identified in the OU-1/OU-2 ROD are needed to compare with the remedial goal of 97 mg/kg. These IM areas are accessible, and the data can be efficiently collected as part of the PDI. The Hall Street IM area is also accessible for conducting physical inspections of the cover integrity as part of the PDI. PCB concentrations in surface and subsurface soil for the drainage ditch located west and east of the PB-RR-37 location will be collected for comparison with the surface remedial goals for surface and subsurface soil of 21 mg/kg and 97 mg/kg, respectively. Surface soil at the downstream end of the eastern portion of this ditch that have the potential to erode and migrate to Snow Creek will also be compared to the remedial goal for sediment of 3 mg/kg.
- **Dredge spoil piles:** PCB concentration data are needed to assess whether remediation of dredge spoil pile SC-2 is required. Dredge spoil SC-1 and dredge spoil pile SC-2 are located sufficiently away from Snow Creek that soils located at the outside edge of the piles are not potential sources to Snow Creek. Soil PCB concentrations beneath the dredge spoil piles required to be removed will be assessed through analysis of post-removal samples collected during the RA construction phase of the project. The dredge spoil piles are in remote areas with difficult physical access. Collecting samples beneath the piles during RA construction will limit the disturbance of existing vegetation because the equipment needed to sample beneath these piles is like the equipment that will be used for their excavation. Since dredge spoil pile SC-8 is located adjacent to and within the borders of Snow Creek, this dredge spoil pile is being addressed as part of the Snow Creek sediment RD.
- **Nonresidential soil:** PCB concentrations in subsurface soil for the nonresidential soil areas identified in the OU-1/OU-2 ROD as candidate removal areas are needed to compare with the remedial goal of 97 mg/kg. For the PB-RR-37 area, surface soil PCB concentrations will be confirmed as an initial activity and, pending the results, the subsurface soil may be analyzed. Additionally, PCB concentrations at PB-RR-37 will be profiled from the ground surface to the groundwater table to evaluate the potential of PCBs to be leaching groundwater. Surface and subsurface soil from the ditch upstream and downstream of PB-RR-37 will also be evaluated relative to the applicable remedial goals. The surface soil PCB results from samples collected from the portion of the ditch closest to Snow Creek will also be assessed relative to potential erosion into Snow Creek. Geotechnical data are also needed to design the excavation of PTW soil at the T-11 area and for the broader T-11 area that will be capped with clean backfill and a low-permeability cover. These data will assist in assessing the potential for differential settlement of the post-cap surface if groundwater treatment equipment is constructed on the T-11 side of Snow Creek. Geotechnical data are also needed to design long-term access to the T-11 area that may include a bridge or pipe bridge.
- **Creek bank areas:** Geotechnical data are needed to assess creek bank stabilization options along Snow Creek at and immediately downstream of its confluence with the 11th Street Ditch.

The PDI field sampling procedures to address these data gaps are included in Section 3.1.

2.2.2 Groundwater

The review of existing groundwater data (Section 2.1.2) revealed several data needs and data gaps relevant to designing the groundwater-related components of the OU-1/OU-2 RD. The following subsections describe these data needs and the data gaps that need to be addressed to assess the potential for PCBs to leach from soil to groundwater at the Eastside Properties and design a treatment remedy for the T-11 area.

At the PB-RR-37 area, soil sampling will first be conducted to evaluate the potential for PCB-leaching from soil to groundwater as discussed in Section 3.1.2.1. Following receipt of soil sampling results, P/S will consult with USEPA on the need for groundwater investigation at the PB-RR-37 area. Depending on laboratory testing results, possible outcomes for this area may include no further investigation or installation of a groundwater monitoring well.

2.2.2.1 Eastside Properties

The data gap at the Eastside Properties to be addressed by the PDI is evaluating whether PCBs have leached from soil into groundwater at concentrations exceeding the remedial goal of 0.5 µg/L. PDI activities to address this data gap will include installing two new groundwater piezometers and installing and sampling two new groundwater monitoring wells (Section 3.2.2.1). Following receipt of groundwater monitoring results and interpretation, P/S will consult with USEPA on the need for and extent of additional PDI activities. Depending on laboratory testing results, possible outcomes may include no further investigation, confirmation resampling, or additional investigation to support the OU-1/OU-2 RD.

2.2.2.2 T-11 Area

Data gaps related to groundwater extraction and treatment at the T-11 area include current concentrations of total and dissolved PCBs as well as geochemical parameters, hydraulic properties of the groundwater-bearing unit (i.e., local gradient, hydraulic conductivity, well yield), the extent of aquifer response to pumping, and performance of carbon adsorption for removal of PCBs from groundwater. A range of PDI activities and a TS are planned to address these data gaps as discussed in Sections 3.2 and 4.1, respectively. Additional information on the groundwater TS is provided in Appendix B.

2.2.3 Snow Creek Sediment

Most of the existing sediment data for the OU-1/OU-2 portion of Snow Creek were generated as part of the 1999 RFI investigation. Due to the dynamic nature of the creek system, there is the potential for sediment deposits to have moved or for new sediment deposits to have formed over the past 20-plus years. In addition, the culvert beneath Quintard Mall and other portions of Snow Creek where property access was not granted were not characterized during the 1999 RFI. The culvert beneath Quintard Mall will be probed and sampled as part of the PDI as described in Section 3.3.2.

The sediment PDI will be conducted to address the following data gaps:

- Additional chemical and physical data are needed to provide updated information to refine the sediment remedial extents and volumes and to establish off-site disposal requirements.
- Treatability testing is needed to evaluate potential options for dewatering sediment that will be removed from the creek.

The PDI field sampling procedures and TS bench-scale testing procedures to address these data gaps are included in Section 3.3 and Section 4.2, respectively. Additional information on the sediment TS is provided in Appendix C.

2.2.4 OU-1/OU-2-Wide Evaluations

Data needs and data gaps were identified for floodplain and creek bank soil, groundwater, and Snow Creek sediment, as well as site-wide evaluations for utilities, structures, and topo-bathymetry that need to be addressed to complete the OU-1/OU-2 RD as defined in the ROD. Descriptions of these data needs and data gaps are provided in the following subsections.

As part of the OU-1/OU-2 RD process, existing conditions of utilities, structures (e.g., bridges, roadways, outfalls, and culverts), surface elevations, and the presence of cultural and archeological resources need to be evaluated for inclusion on design drawings and for any preventative measures that need to be implemented to protect the integrity of the utilities and structures.

The PDI will be conducted to address the following data gaps:

- Information is needed regarding structures immediately adjacent to target remedial areas to confirm that the remediation work will not affect the integrity of the structures.
- Current project base mapping is needed to support the development of the RD and contract drawings.
- Updated topography and bathymetry data are needed to support the design for the soil and sediment remediation and for use in hydraulic modeling of potential flooding impacts associated with potential remediation within and near the creek and the Highway 202 culvert area.

The PDI field sampling procedures to address these data gaps are included in Section 3.4.

2.2.5 Snow Creek Surface Water

The USEPA-selected remedy includes monitoring surface water at the upstream and downstream ends of OU-1/OU-2. Surface water sampling will be conducted as part of the PDI to establish a baseline for comparing post-remedy conditions. Since the ROD specified remedial goals for surface water based on the USEPA's chronic aquatic life freshwater ambient water quality criteria (AWQC), the monitoring will be conducted during base-flow conditions that are present 90% of the time.

As described in Section 3.5.2, the baseline surface water sampling program will include collecting up to 8 grab samples (1 sample quarterly for 2 years) from 1 upstream and 1 downstream location. Each grab sample will include collecting samples to measure total (unfiltered) and dissolved (filtered) concentrations in surface water. In addition, the baseline surface water sampling program will include collecting up to 4 passive samples (twice per year for 2 years) from each upstream and downstream location. Details for the baseline PDI surface water sampling are described in Section 3.5.

3. PREDESIGN INVESTIGATIONS FIELD SAMPLING PLAN

The PDI has been designed to collect necessary and relevant information to successfully complete the OU-1/OU-2 RD as defined in the ROD. Descriptions of the rationale and objectives for all PDI activities are included below, and the field sampling procedures to be followed to complete the PDI are described in the following sections. All PDI field work will be conducted in accordance with the UFP-QAPP (Appendix A).

3.1 Floodplain and Creek Bank Soil

PDI activities related to the soil RD will include floodplain subsurface soil sampling for the low-activity special use areas, the IM areas, and the nonresidential floodplain soil identified in the OU-1/OU-2 ROD. The PDI will also include sampling dredge spoil pile SC-2. Geotechnical sampling will also be conducted in the T-11 area. The following subsections present the investigation rationale and objectives and the scope of work and investigative methods to be employed.

3.1.1 Rationale and Objectives

The PDI field work for soil includes sampling and analyzing soil for dredge spoil pile SC-2, low-activity special use areas, IM areas, and nonresidential floodplain areas. The field work will also include geotechnical testing of soil borings from the T-11 area and in creek bank areas. Sampling residential or high-activity special use areas is not included in this PDIWP because these areas were sufficiently characterized during the OU-1/OU-2 RI and remediated where necessary based on the NTCRA requirements. The assessment of soil relative to PTW potentially being present at PB-RR-37 and the Eastside Properties is discussed in Section 3.2.2 of this PDIWP.

PDI efforts are focused on the following:

- For the dredge spoil piles, sampling will be focused on SC-2 to evaluate PCB concentrations relative to the remedial goal of 3 mg/kg.
- For confirmed low-activity special use areas, subsurface soil will be sampled at the same surface soil locations associated with the Stipulation (USEPA 2006) for comparison to the OU-1/OU-2 ROD requirement of 97 mg/kg. Sample locations will only include locations where surface soil concentrations were found to be greater than or equal to 1 mg/kg.
- For IM areas (excluding the Hall Street IM area and the Former Miller property), subsurface soil will be sampled relative to the subsurface remedial goal of 97 mg/kg. For the Hall Street IM area, a visual inspection of this area will be completed to evaluate whether portions of the IM cover soils require augmentation to meet the IM design requirements. The remedy of the Former Miller property portion of the Eastside Drainageway area includes removing the upper foot of soil in the identified areas, placing a 1-foot layer of clean backfill, and stabilizing the area with vegetation. Subsurface soil sampling will not be conducted for this IM area because PCB-containing soil is isolated at depth by design.

- For the nonresidential areas, subsurface soil will be sampled for the target remedial footprints identified in the OU-1/OU-2 ROD for comparison to the subsurface remedial goal of 97 mg/kg.
- For the PB-RR-37 area, historical results of co-located soil samples (PB-RR-37 and PB-RR-40) exhibited PCB concentrations differing by more than 3 orders of magnitude (3,700 mg/kg and 2.3 mg/kg, respectively). These variable concentrations suggest the potential for heterogeneity in surface soil PCB concentrations. Surface soil confirmation sampling will be conducted to assess PCB concentrations relative to the OU-1/OU-2 ROD requirement of 21 mg/kg. If PCB concentrations in surface soil are greater than or equal to 21 mg/kg, then the subsurface soil samples collected for this area will be analyzed and compared with the remedial goal of 97 mg/kg. In addition to the samples collected for the PB-RR-37 area, a discrete soil boring will be conducted at the PB-RR-37 location to develop a vertical profile of PCB concentrations from ground the surface to the elevation of the groundwater table. If PCBs are detected in soil at the groundwater interface, the potential installation of a groundwater well at this location will be discussed with USEPA. Additionally, soil sampling will be conducted to evaluate PCB concentrations in surface and subsurface soils within the drainage ditch upstream and downstream of the PB-RR-37 area.
- For the T-11 area, a geotechnical characterization will be completed to support the development of the RD for excavation and low-permeability capping in this area. The geotechnical investigation will also be used to evaluate potential bridge or pipe bridge options to access the T-11 area during and following engineering design aspects of the low-permeability cap, the groundwater treatment plant, and bridge foundations. Results of the geotechnical investigations along the banks of Snow Creek in the T-11 area will also be used, along with physical inspection results, to develop the RD for the select creek bank areas identified in the OU-1/OU-2 ROD.

The following are specific data quality objectives (DQOs) for the OU-1/OU-2 PDI for soil:

- SO-DQO 1: Refine the current understanding of PCBs for dredge spoil pile SC-2 to evaluate whether PCB concentrations exceed the remedial goal of 3 mg/kg. Once the dredge spoil piles identified for remediation are removed, surface soil and subsurface soil beneath the dredge spoil piles will be sampled and assessed for PCBs based on the ROD requirements of 21 mg/kg for surface soil and 97 mg/kg for subsurface soil.
- SO-DQO 2: Refine the current understanding of subsurface soil PCB concentrations in floodplain soil areas, including confirmed low-activity special use areas, certain IM areas, and the nonresidential areas, for comparison with the OU-1/OU-2 ROD remedial goal of 97 mg/kg.
- SO-DQO 3: Gather geotechnical characterization data from the T-11 area to support the detailed design of a low-permeability cap and creek bank stabilization measures.
- SO-DQO 4: Evaluate whether representative soil PCB concentrations at the PB-RR-37 area, including soil along the drainage ditch upstream and downstream of this area, are above the ROD requirements of 21 mg/kg for surface soil and 97 mg/kg for subsurface

soil. For locations adjacent to Snow Creek, concentrations will be compared to the OU-1/OU-2 ROD remedial goal of 3 mg/kg and evaluated relative to their ability to erode and migrate into Snow Creek. If PCBs are detected in subsurface soil proximate to the groundwater table in the PB-RR-37, discussions will be held with USEPA relative to the potential installation of a groundwater monitoring well to assess if groundwater PCB concentrations in this area exceed the remedial goal of 0.5 µg/L.

- SO-DQO 5: Evaluate whether portions of the Hall Street IM cover soils require augmentation to meet the IM design requirements.

Details of the proposed PDI activities to achieve SO-DQO 1 through SO-DQO 5 are provided in Section 3.1.2.

3.1.2 Scope of Work and Investigative Methods

Soil sampling and geotechnical borings will be completed as part of the PDI of floodplain and creek bank soil. The scope of work and field sampling procedures to be used to address data needs and data gaps identified in Section 2.2.1 are described below.

3.1.2.1 Soil Sampling

Composite soil sampling procedures will be used to sample dredge soil pile SC-2 and low-activity special use areas. A combination of composite sampling and Incremental Sampling Methodology (ISM) will be used to sample subsurface soil for the IM and nonresidential areas. The locations sampled in the low-activity special use areas will be reoccupied for the PDI, and composite subsurface samples will be collected using the NTCRA approach. The general procedures are outlined below, and additional details can be found in Attachment A-5 (Surface and Subsurface Soil Sampling), Attachment A-9 (Procedures for Compositing or Homogenizing Soil or Sediment Samples), and Attachment A-12 (Incremental Sampling Methodology Procedures) of the UFP-QAPP (Appendix A).

Dredge Spoil Pile SC-2

A single 5-point composite soil sample will be collected from dredge spoil pile SC-2 (Figure 3-1). The five sample points will be evenly distributed over the surface of the dredge spoil pile and collected from the 0–1-foot horizon.

Soil samples will be collected in accordance with Attachment A-5 (Surface and Subsurface Soil Sampling) of the UFP-QAPP (Appendix A). These general procedures will be followed for collecting the soil borings:

1. The sample location will be located, identified, and marked with a stake, flag, or similar marker.
2. Any debris or vegetation present will be cleared from the boring location to expose the ground surface.
3. Borings will be advanced using a hand auger (or equivalent method) to a depth of 1 foot.

4. The material collected from the five locations will be combined and homogenized into a bucket with a Teflon® liner or a stainless steel bowl or equivalent to form the composite sample for each dredge spoil pile in accordance with Attachment A-9 (Procedures for Compositing or Homogenizing Soil or Sediment Samples) of the UFP-QAPP. Quality assurance/quality control (QA/QC) samples will be collected and submitted to the laboratory in accordance with the UFP-QAPP. Sample container sizes, holding times, preservation requirements, and other information regarding the analytical program, specific methods, and QA/QC procedures are provided in the UFP-QAPP.
5. Composited material will be transferred into laboratory-supplied bottleware for shipment to the analytical laboratory. The bottleware will be labeled and temporarily stored on ice (4 ± 2 degrees Celsius [°C]) to await transport or preparation for shipment to the laboratory as directed in the UFP-QAPP. A minimum of 100 grams of material will be submitted to the analytical laboratory for analysis; sample mass requirements for PCB Aroclors by USEPA SW-846 8082 are listed in the UFP-QAPP.
6. Nondedicated sampling equipment, if used, will be decontaminated following completion of sampling of dredge spoil pile SC-2.

Low-Activity Special Use Areas

Land use conditions at the low-activity special use areas will be reviewed during the PDI prior to completing sampling activities to confirm they are consistent with the Stipulation's definition of a low-activity special use area and are suitable for removal (e.g., are not overgrown). Documentation of current land use will be collected for all low-activity special use areas, and the findings and any revisions to the proposed sampling plan will be discussed with USEPA. Subsurface soil sampling will be conducted only for confirmed low-activity special use areas that are suitable for removal.

Five-point composite subsurface soil samples will be collected in confirmed low-activity special use areas from the same locations that were used for surface soil sampling under the Stipulation where surface soil concentrations were found to be greater than or equal to 1 mg/kg (Figures 3-2a-l). Composite samples will be collected from three soil depth intervals of 1–2 feet bgs, 2–3 feet bgs, and 3–4 feet bgs. Composite samples collected from the soil depths of 2–3 feet bgs and 3–4 feet bgs will be archived at the analytical laboratory pending analysis of the composite sample collected from 1–2 feet bgs. The subsequent depth interval sample will be analyzed if PCB concentrations detected in the analyzed interval are above the PCB remedial goal for subsurface soils (97 mg/kg). For example, if PCB concentrations in the 1–2-foot-bgs composite sample interval are detected above the remedial goal for subsurface soil (97 mg/kg), the composite sample collected from the 2–3-foot depth interval will be analyzed.

The low-activity areas that will be sampled, pending confirmation of land use condition, are presented on Figures 3-2a through 3-2l except for the low-activity area located in the southeast corner of EU14 North that has been developed for industrial purposes after the OU-1/OU-2 ROD was issued. Soil samples will be collected in accordance with Attachment A-5 (Surface and Subsurface Soil Sampling) of the UFP-QAPP (Appendix A).

These general procedures will be followed for collecting the soil borings:

1. The sample location will be located, identified, and marked with a stake, flag, or similar marker.
2. Any debris or vegetation present will be cleared from the boring location to expose the ground surface.
3. Borings will be advanced using a hand auger, Geoprobe®, or equivalent method to depths of 1–2 feet, 2–3 feet, and 3–4 feet bgs.
4. For each depth, the material collected from the five locations will be combined and homogenized into a bucket with a Teflon® liner or a stainless steel bowl or equivalent to form the composite sample for each low-activity special use area in accordance with Attachment A-9 (Procedures for Compositing or Homogenizing Soil or Sediment Samples) of the UFP-QAPP. QA/QC samples will be collected and submitted in accordance with the UFP-QAPP. Sample container sizes, holding times, preservation requirements, and other information regarding the analytical program, specific methods, and QA/QC procedures are provided in the UFP-QAPP (Appendix A).
5. Composited material will be transferred into laboratory-supplied bottleware or zip-top bags for shipment to the analytical laboratory. The bottleware or zip-top bags will be labeled and temporarily stored on ice (4 ± 2°C) to await transport or preparation for shipment to the laboratory as directed in the UFP-QAPP. A minimum of 100 grams of material will be submitted to the analytical laboratory for analysis; sample mass requirements for PCB Aroclors by USEPA SW-846 8082A and PCB homologs by USEPA Method 680 are listed in the UFP-QAPP.
6. Nondedicated sampling equipment, if used, will be decontaminated between sampling in the low-activity special use areas.

Interim Measures and Nonresidential Areas

Soil samples will be used to develop the remediation depths for the targeted IM and nonresidential areas relative to the PCB remedial goal for subsurface soils of 97 mg/kg. The IM areas identified for sampling, remediation, and assessment are shown on Figures 3-3a through 3-3f. Composite sampling will also be conducted for surface soils (0–1 foot) in the area surrounding location PB-RR-37/PB-RR-40 (Figure 3-3e) to assess whether PCB concentrations in that area are greater than or equal to 21 mg/kg. The subsurface soil sampling efforts also include the IM located along the western side of Clydesdale Avenue just north of the railroad tracks (Figure 3-3a) where surface soil remediation has already been completed. The physical integrity of the Hall Street IM (Figure 3-3f) will also be assessed as part of the PDI.

The nonresidential areas selected for remediation in the OU-1/OU-2 ROD and included in the SOW are shown on Figure 3-4a through 3-4h. The figures show the remediation footprints where the surface soil will be excavated and the associated basis (PCBs versus non-PCB constituents). The PDI will be conducted for the PCB remediation footprints to evaluate conditions compared to the subsurface soil remedial goal of 97 mg/kg. One of the nonresidential remediation footprints shown on Figure 3-4a no longer requires remediation because the property was remediated under the NTCRA program after the OU-1/OU-2 ROD was issued. This property is in EU7 along its

border with EU5 (Figure 3-4a), and remediation involved the removal of surface and subsurface soil.

Five-point composite subsurface soil samples will be collected in IM areas and nonresidential areas where the size of the contiguous area to be sampled is equal to or less than 0.5 acres. All of the IM areas evaluated under this PDI are less than 0.5 acres and will be sampled by collecting one 5-point composite per 0.25-acre area (Table 3-1). Composite samples will be collected as described above from three soil depth intervals of 1–2 feet bgs, 2–3 feet bgs, and 3–4 feet bgs. Composite samples collected from 2–3 feet bgs and 3–4 feet bgs will be archived at the analytical laboratory pending analysis of the composite sample collected from 1–2 feet bgs. The subsequent depth interval sample will be analyzed if PCB concentrations detected in the analyzed interval are above the PCB remedial goal for subsurface soil (97 mg/kg).

Proposed sample locations are shown on Figures 3-3a-f for IM areas and Figures 3-4a-h for nonresidential areas. This includes collecting 5-point composite soil samples from the drainage ditches located upstream and downstream of PB-RR-37 (Figure 3-3e). Composite samples will be collected as described above from four sample depths: 0–1 foot bgs, 1–2 feet bgs, 2–3 feet bgs, and 3–4 feet bgs. Additionally, during collection of the 5-point composite samples, individual split samples (aliquots) for the two locations closest to Snow Creek will be collected from 0–1 foot bgs and analyzed for comparison to 3 mg/kg. The composite PCB results for samples collected from this ditch will be compared to the nonresidential remedial goal of 21 mg/kg. Samples collected from the soil depths of 1–2 feet and below will be archived at the analytical laboratory pending analysis of samples collected from 0–1 feet bgs.

The subsurface soil in nonresidential areas that are larger than 0.5 acres will be sampled using ISM in accordance with the Interstate Technology Regulatory Council (ITRC) *Technical Guidance: Incremental Sampling Methodology Update (ISM-2)* guidance document (ITRC 2020) and Attachment A-12 (Incremental Sampling Methodology Procedures) of the UFP-QAPP (Appendix A). ISM is a probabilistic sampling method used to evaluate the representative concentration of a constituent in an area. It is designed to provide an unbiased and statistically valid estimate of the mean concentration of analyzed constituents within each sample area of the IM areas. ISM was developed to decrease the effect of soil sample variability due to heterogeneity and provides a reasonably unbiased estimate of mean concentration in a defined soil volume, referred to as a decision unit (DU) (ITRC 2020). Each IM and nonresidential area will be sampled as three DUs based on sample depth: 1–2 feet bgs, 2–3 feet bgs, and 3–4 feet bgs.

The following general procedures will be followed for collecting all ISM samples:

1. The horizontal boundaries of the nonresidential area will be located using a handheld global positioning system (GPS) unit. The footprint of the nonresidential areas will be subdivided into a stratified grid of 30 cells as listed in Table 3-1, and 1 increment will be collected within each grid cell. A total of 30 increments will be collected per DU and composited into an increment sample as described in Step 5 below. The locations of each increment will be recorded with a handheld GPS.

2. Surface debris (e.g., wood, paper, sod, gravel, grass, and trash) will be removed in the immediate vicinity of the increment location prior to sampling. The increment location will be clearly marked.
3. Soil cores will be collected using a direct-push drill rig operated by a licensed drilling subcontractor. Larger pieces of material (cobbles, larger rocks) will not be included in samples.
4. At the time of sample collection, soil increments will be described using visual-manual procedures of ASTM International (ASTM) Standard D2488, which is based on the Unified Soil Classification System (USCS). Color, moisture content, visual grain-size distribution, and other pertinent characteristics will be noted. Information in the field notebook will include location, date, time, personnel and equipment present, down time, materials used, samples collected, and other observations or information that is relevant to the data collection objectives.
5. A representative subsample of approximately equal mass (at least 30 grams) from each increment will be collected using the plug subsampling technique (i.e., soil material from each core will be collected using a plug-type sampler [e.g., TerraCore®] equally spaced from one another along the length of the core) in accordance with ITRC ISM-2 guidance, depending on field conditions. If challenges are observed in the field using the plug subsampling technique, one of the following collection techniques found to be most suitable for field conditions will be consistently implemented to the increments within any given DU:
    a. Core wedge subsampling: Each soil core will be split into one-third to one-half wedges along the length of the core.
    b. Core slice subsampling: A 4-inch perpendicular slice will be randomly selected from each individual core.

    The representative subsamples from the 30 increments will be combined and homogenized into a bucket with a Teflon® liner or a stainless steel bowl or equivalent to form the complete ISM sample for each DU. Approximately 900 grams of material will be submitted per ISM sample to the laboratory.
6. Field replicates: The ISM-2 guidance document recommends that field triplicate ISM samples (i.e., the original increment sample plus two replicate increment samples) be collected. Field triplicates will be collected as indicated in Table 3-1. The collection of these field replicates allows for the calculation of the relative standard deviation (RSD),[1] which measures the precision or reproducibility of the sampling method and the heterogeneity of the analyte distribution throughout the DUs. The greater the RSD the less confidence there is in the estimate of the mean analyte concentration for that DU.

[1] RSD is expressed as a percent and is obtained by multiplying the standard deviation of the triplicate ISM sample concentrations by 100 and dividing this product by the average concentration of the triplicate ISM samples.

a. Field replicates will be collected as described in Steps 3 through 5 above for the original ISM increment sample. Replicate increment locations are unique from the original increment locations and are accessed using a different sampling path in the DU used to access the original ISM increment sample locations. For example, if the original ISM increment sample was collected by following a path from north to south starting in the west and moving east to access each increment location, a replicate increment sample could be collected by following a path from south to north starting in the east and moving west to access each increment location.

b. The RSD of the field replicate ISM samples will be calculated and expressed as a percentage. If the calculated RSD for a DU exceeds 50% and the average concentration for a DU exceeds the subsurface soil remedial goal of 97 mg/kg then additional ISM sampling may be necessary, using a larger number of sample increments per DU to obtain a more representative measure of the analyte concentrations in the DU. If additional sampling is necessary, it will be addressed in a separate addendum to this PDIWP.

7. Field QA/QC samples: Duplicates and matrix spike/matrix spike duplicates will be collected at frequencies specified in the UFP-QAPP (Appendix A).
8. After samples are collected and homogenized, material will be transferred into zip-top bags to encapsulate the sample. The zip-top bags will be labeled and temporarily stored on ice (4 ± 2°C) to await transport or preparation for shipment to the laboratory as directed in the UFP-QAPP.
9. Nondedicated sampling equipment, if used, will be decontaminated between DUs.

In accordance with sample preparation methods required for ISM, upon receipt at the laboratory, the ISM soil samples for inorganic and nonvolatile analyses will be air dried, disaggregated and sieved to 2 millimeters using a disaggregator prior to passing material though a #10 mesh screen to eliminate coarser materials not considered part of the soil fraction, and subsampled using the two-dimensional slabcake subsampling technique as described in ITRC ISM-2 and standard operating procedures (SOPs) for soil processing (SOP NC-OP-44) and subsampling (SOP NC-OP-046) provided in Attachment B (Analytical Lab SOPs) of the UFP-QAPP (Appendix A).

Soil samples will be analyzed for PCB Aroclors by USEPA SW-846 8082. ISM samples collected from the soil depths of 2–3 feet bgs and 3–4 feet bgs will be archived at the analytical laboratory pending analysis of the ISM samples collected from 1–2 feet bgs. The subsequent depth interval DU will be analyzed if PCB concentrations detected in the analyzed interval are above the PCB remedial goal for surface soils (21 mg/kg; 0–1-foot-bgs DU only) or subsurface soils (97 mg/kg). For example, if PCB concentrations in ISM samples collected from the 1–2-foot-bgs DU are detected above the remedial goal for subsurface soils (97 mg/kg), the ISM samples collected from the 2–3-foot-bgs DU will be analyzed.

DU horizontal extents and grid spacing for ISM are shown on Figures 3-4a-h for nonresidential areas. The horizontal extents and grid spacing will be the same for all sampling depths.

Discrete Boring at PB-RR-37

Surface and subsurface soil samples will be collected from depth intervals from a discrete location proximate to PB-RR-37 (Figure 3-5). Samples will be collected at 1-foot depth intervals from ground surface until the soil–groundwater interface is reached based on field observations (approximately 15 to 25 feet bgs). The last depth interval will be located immediately above the observed groundwater level. If PCBs are detected in soil at the groundwater interface, the installation of a groundwater well at this location will be discussed with USEPA.

These general procedures will be followed for collecting the soil boring:

1. The sample location will be located, identified, and marked with a stake, flag, or similar marker.
2. Any debris or vegetation present will be cleared from the boring location to expose the ground surface.
3. Borings will be advanced using a Geoprobe® or equivalent method to depths of groundwater.
4. The material collected from each 1-foot depth interval will be homogenized into a bucket with a Teflon® liner or a stainless steel bowl or equivalent in accordance with Attachment A-9 (Procedures for Compositing or Homogenizing Soil or Sediment Samples). QA/QC samples will be collected and submitted in accordance with the UFP-QAPP. Sample container sizes, holding times, preservation requirements, and other information regarding the analytical program, specific methods, and QA/QC procedures are provided in the UFP-QAPP (Appendix A).
5. Homogenized material will be transferred into laboratory-supplied bottleware or zip-top bags for shipment to the analytical laboratory. The bottleware or zip-top bags will be labeled and temporarily stored on ice (4 ± 2 °C) to await transport or preparation for shipment to the laboratory as directed in the UFP-QAPP. A minimum of 100 grams of material will be submitted to the analytical laboratory for analysis; sample mass requirements for PCB Aroclors by USEPA SW-846 8082A and PCB homologs by USEPA Method 680 are listed in the UFP-QAPP.
6. Nondedicated sampling equipment, if used, will be decontaminated between sampling depth intervals.

3.1.2.2 Geotechnical Investigation

Four geotechnical borings will be advanced at locations shown on Figure 3-6 within the general areas where the RA excavation and bank stabilization will be completed. The geotechnical boring to be advanced closest to well T-11 will be installed at one of the piezometer locations (i.e., TPZ-1 through TPZ-3) proposed as part of the groundwater PDI (Section 3.2.2). Track-mounted drill rigs will be used to advance the geotechnical borings to collect standard penetration test (SPT) blow counts and split-spoon samples for analysis as discussed in the following subsections.

Access and Preclearing Geotechnical Borings
Track-mounted drill rigs will be used to advance the geotechnical borings. For borings advanced in vegetated areas, paths will be cleared of the vegetation and construction mats (e.g., MobiDeck™ portable rigid construction mats or equivalent) will be placed to construct access roadways to the boring locations.

Prior to advancing geotechnical borings or other intrusive subsurface work, locations will be hand-cleared or air-knifed by the drilling subcontractor to a depth of 5 feet as a precaution to avoid shallow utilities in addition to completion of other project health and safety requirements (e.g., calling Alabama 811 and other utility clearance provisions identified in the OU-1/OU-2 RD/RA HASP). If the geophysical survey or hand-clearing/air-knifing indicate the presence of a utility, subsurface work will be relocated to an area that is expected to be clear of utilities, and the new location will be manually cleared to a depth of 5 feet.

Geotechnical Borings
SPT blow counts and split-spoon sampling will be conducted in accordance with ASTM D1586 (Standard Test Method for Penetration Test and Split-Barrel Sampling of Soils) by the drilling subcontractor with oversight and sampling conducted by P/S representatives. Split-spoon samples will be collected continuously to boring termination at approximately 40 feet bgs or refusal, whichever is encountered first. Split-spoon samples from each boring will be collected from the geotechnical borings for the following geotechnical laboratory analyses:

- Natural Moisture Content (ASTM D2216)
- Particle-Size Analysis (ASTM D6913 and ASTM D7928)
- Atterberg Limits (ASTM D4318)
- Engineering Classification (ASTM D2487)

One thin-walled Shelby-tube sample from each of the borings will be collected from cohesive soil horizons encountered in the borings. Representative Shelby-tube samples will be submitted for the following geotechnical laboratory analyses:

- Unconsolidated Undrained Triaxial Compression Test (ASTM D2850)
- Consolidated Undrained Triaxial Compression Test (ASTM D4767)

SPT blow counts and split-spoon samples will be collected generally as follows:

1. The split-spoon samplers and other reusable tools will be decontaminated at each sample location in accordance with Attachment A-2 (Field Cleaning and Decontamination Procedures) of the UFP-QAPP (Appendix A).
2. At each boring location, a 2-inch-diameter split-barrel sampler will be advanced by hollow-stem auger, rotary drilling, or sonic drilling. If hollow-stem auger or rotary drilling methods are used, the split-barrel sampler will be advanced using a 2-inch-diameter roller bit while circulating a bentonite slurry to maintain sidewall stability and

remove drill cuttings. Borings will be advanced continuously in 2-foot intervals to boring termination at approximately 40 feet bgs or refusal whichever is encountered first in accordance with Attachment A-6 (Subsurface Drilling and Monitoring Well Installation Procedures) of the UFP-QAPP (Appendix A).

3. At each 2-foot sampling interval, the split-barrel sampler will first be seated 6 inches and then driven an additional 18 inches with blows from a 140-pound hammer and 30-inch drop height. The number of hammer blows (i.e., blow counts) required to drive the sampler each 6 inches will be recorded. The blow counts required to advance the sampler through the middle 12-inch interval represents the N-value, which, when properly interpreted, is an index of soil strength, relative density (for granular soils), and consistency (for fine-grained soils).
4. Each boring and split-barrel sample will be appropriately logged by the P/S representative at the time of investigation. Lithologic field logs will include a visual description of material encountered for each depth interval and denote the corresponding blow counts, corresponding N-values, and relevant observations made during the drilling process.
5. Representative portions of the soil samples obtained from the spilt-spoon samplers will be placed in sample bags, marked with the corresponding boring designation and depth interval, and retained for submittal for geotechnical laboratory analysis. Samples will be maintained under chain-of-custody protocol in accordance with Attachment A-1 (Field Sample Packing, Handling, Shipping, and Chain-of-Custody Procedures) of the UFP-QAPP (Appendix A).
6. Once drilling is complete, each borehole will be tremie grouted from the bottom of the borehole to surface. The grout mixture will consist of 5% bentonite powder and 95% Portland cement as described in Attachment A-6 (Subsurface Drilling and Monitoring Well Installation Procedures) of the UFP-QAPP (Appendix A).
7. Soil and liquid investigation-derived waste (IDW) generated during field activities will be containerized separately in new, 55-gallon, steel drums and managed as described in Section 3.1.2.3.

Thin-walled Shelby tube samples will be used to recover relatively undisturbed cohesive soil samples from cohesive soils of the clay layer observed underneath fill material generally as follows:

1. If cohesive soil is observed during borehole advancement, a Shelby tube will be lowered to the depth and slowly pressed 24 inches into the soil.
2. Before the Shelby tube is withdrawn from the borehole, the tube will be left in the borehole for 10 to 15 minutes to allow the sampled material to stabilize inside the tube.
3. An end cap will be placed at the bottom end of the Shelby tube immediately upon retrieval. Any cuttings or wash materials will be removed from the top of the Shelby tube, and recovery will be measured and recorded in field notes.

4. Shelby tubes will be sealed at the ends with wax caps and several wraps of electrical tape. The exterior of the Shelby tube will be cleaned and clearly labeled with the boring number, depth range of the Shelby tube, recovery in inches, and an arrow indicating the top of the Shelby tube.
5. Shelby tubes will be transported and shipped in a vertical orientation to the geotechnical laboratory under chain-of-custody protocol.
6. Soil and liquid IDW generated during field activities will be containerized separately in new, 55-gallon, steel drums and managed as described in Section 3.1.2.4.

3.1.2.3 Inspection of Hall Street IM Area Cover Soils

Results from a recent inspection of the Hall Street IM area indicated that portions of the surface soil isolation cover were disturbed. The soil disturbance occurred when a utility construction contractor staged construction equipment on the surface of the IM. The Hall Street IM will be inspected as part of the PDI. This inspection will evaluate whether portions of the IM cover require augmentation to meet the IM design requirements (i.e., presence of geotextile marker layer, 12-inch soil cover layer, and vegetative cover).

The PDI visual inspection of the condition of the cover soils will include the observations of the following to document the most recent conditions of the surface soil cover:

- Poor vegetation (e.g., bare or unvegetated areas)
- Whether mowing or seeding and fertilizing is required
- Differential settlement producing areas of ponding
- Significant surface erosion
- Exposure of underlying geosynthetic layers
- Significant surface soil cracking (approximately 1 inch or greater in width)
- Other disturbances such as fallen trees that may adversely affect cap performance

In areas of disturbance, small (less than 1-inch diameter) soil cores will be collected to confirm cover soil thickness around the perimeter and within the area of the observed disturbance. The number of soil cores collected will depend on the surface area of the observed disturbance. If the thickness of the cover soil is less than the design requirement, the area will be repaired during the RA by placing and compacting a layer of soil on top of the disturbed area.

3.1.2.4 Characterization and Management of Investigation-Derived Waste

Soil cuttings, decontamination fluids, personal protective equipment (PPE), and other disposable soil-collection materials (e.g., acetate liners) will be containerized in 55-gallon, open-top, steel drums for off-site disposal at an appropriate facility. Drums will be filled to approximately 75% of volume capacity.

IDW identified as potentially contaminated with hazardous materials will be collected at the point of generation and later stored in a designated and clearly marked IDW management area. Containers will also be clearly labeled to indicate the source of the IDW. The IDW storage area will be inspected to ensure that storage procedures are adequate to keep the IDW isolated and contained. Potentially contaminated IDW will be identified based on its origin and olfactory and visual evidence. The volume of waste will be minimized whenever applicable, and soil, sediment, liquid, and PPE IDW will be segregated and separately containerized. Laboratory testing may be required to determine the proper disposal requirements of IDW anticipated to be generated as part of the PDI activities described above:

- The PPE and plastic sheeting will be disposed of as nonhazardous waste unless it has been visibly contaminated. Spent decontamination liquids will be containerized in drums and tested to determine the proper disposal method.
- Excess sample material will require testing, and the handling of this material will depend on its origin and characteristics. Olfactory and visual observations, as well as field screening with a photoionization detector and/or a flame ionization detector will be used to determine if the soil contains potentially elevated levels of volatile contaminants and direct where confirmation samples will be collected for analysis of PCB Aroclors by USEPA SW-846 8082 and hazardous waste characterization (i.e., toxicity characteristic leaching procedure, ignitability via SW846 7.1.2 or USEPA Method 1020B, corrosivity via USEPA 9045D, and reactivity via USEPA 9014 and USEPA 9034) per the UFP-QAPP. Excess sample material will be collected at the point of generation and later composited (as dictated by field measurements) and stored in a designated and clearly marked IDW management area.

Field personnel will document IDW generation in a drum log. A nonhazardous, hazardous, or pending-analysis label will be filled out with the appropriate generator information (i.e., generator contact information and contents of drum) and affixed to the drum.

Additionally, drums will be labeled with the following information using a paint pen (or similar):

- Unique identification (ID)
- Generator/contact information
- Date of waste generation
- Contents of the drum (PPE, acetate liners, etc.)
- Origin of contents
- Site name and address
- Percent of drum filled

After the PDI is completed, the drums will be disposed of at a permitted, off-site facility depending on their waste characterization designation (i.e., hazardous or nonhazardous).

3.2 Groundwater

PDI activities related to the groundwater RD will be performed at the Eastside Properties and the T-11 area. The following subsections present the investigation rationale and objectives as well as the scope of work and investigative methods to be employed. The approach for investigating the potential presence of PTW at PB-RR-37 is also provided below.

3.2.1 Rationale and Objectives

At the Eastside Properties, an assessment will be conducted to evaluate whether PCBs in soil have leached into underlying groundwater resulting in dissolved-phase PCB concentrations exceeding the remedial goal of 0.5 µg/L. Groundwater conditions will be assessed by installing two groundwater monitoring wells and two piezometers, collecting soil samples at various depths down to the soil–groundwater interface in one of the monitoring wells, and submitting select samples for analysis of PCB Aroclors and homologs. Once the two monitoring wells and two piezometers have been developed to remove fine sediment, static water levels will be measured in the new monitoring wells, new piezometers, and existing nearby monitoring wells to determine the local hydraulic gradient. Unfiltered and filtered groundwater samples will be collected from the two new monitoring wells and submitted for analysis of PCB Aroclors and homologs.

At the PB-RR-37 area, representative PCB concentrations in soil will be measured as described in Section 3.1 to evaluate the presence of PCBs at depth. If subsurface soil sampling indicates the presence of PCBs at the soil–groundwater interface, the results will be discussed with USEPA regarding potential next steps. These next steps could include no further actions regarding groundwater at PB-RR-37 or the installation of a groundwater monitoring well. If a groundwater monitoring well is installed, it will be developed to remove fine sediment, and unfiltered and filtered groundwater samples will be collected and analyzed for PCB Aroclors and homologs.

PDI activities related to groundwater in the T-11 area will provide data to support the design of the groundwater extraction-and-treatment system. The investigation will include assessing groundwater chemistry, hydrogeologic properties of the groundwater-bearing unit, and granular activated carbon (GAC) treatment performance.

Specific DQOs for the OU-1/OU-2 groundwater PDI are as follows:

- GW-DQO 1: Evaluate whether representative groundwater PCB concentrations at the Eastside Properties (and potentially location PB-RR-37) exceed the remedial goal of 0.5 µg/L.
- GW-DQO 2: Gather hydrogeologic data at the T-11 area to support the detailed design of a groundwater extraction-and-treatment system.
- GW-DQO 3: Evaluate potentially efficient and cost-effective GAC media for PCB treatment.
- GW-DQO 4: Estimate the time for PCB breakthrough in a full-scale GAC system.

Details of the proposed PDI activities to achieve GW-DQO 1 and GW-DQO 2 are provided in Section 3.2.2, and TS activities to be completed as part of the groundwater PDI to achieve GW-DQO 3 and GW-DQO 4 are provided in Section 4.1.

3.2.2 Scope of Work and Investigative Methods

3.2.2.1 Eastside Properties Groundwater Assessment

The proposed locations of the two new groundwater monitoring wells and two new piezometers in the Eastside Properties area are shown on Figure 3-7. The locations of the groundwater wells and piezometers were selected considering two key criteria:

- Selecting locations with low or no known PCB impacts in shallow soil (i.e., soil sample locations with light blue symbols on Figure 3-7) to minimize the potential to drag down PCBs from soil to groundwater during well installation
- Selecting a location reasonably close to and downgradient of the area within the Eastside Properties where elevated soil PCB concentrations have been observed[2]

The proposed well locations were determined, in consultation with USEPA, by reviewing the groundwater flow direction and PCB soil data within the Eastside Properties to identify areas that are both downgradient of the area where elevated soil PCB concentrations in proximity to the groundwater table have been observed and have low or no known PCB impacts in shallow soil. The two proposed well locations are approximately 75 and 130 feet downgradient from the area of elevated PCB soil concentrations and are surrounded by soil showing little (≥1 to <10 mg/kg) or no (not detected to <1 mg/kg) PCB impacts. The locations are also within a reasonable groundwater travel distance from the area of elevated PCB soil concentrations as further discussed below.

Potentiometric surface elevations from May 2020, which are shown on Figure 3-8, indicate that groundwater within the Eastside Properties flows to the north-northwest (P/S 2021d) with a hydraulic gradient of approximately 0.04. The potentiometric surface shown on Figure 3-8 was developed using water levels from all shallow residuum wells across and near OU-3 of the Anniston PCB site. At the time, six wells existed within the Eastside Properties, but in 2021, wells OW-07 and OW-09 were plugged and abandoned, leaving wells OW-06A, T-03, T-20, and OWR-09S within the Eastside properties.

A falling-head slug test conducted in well OW-07, located within the Eastside Properties (Figure 3-8), indicated a hydraulic conductivity of 7.1 x 10^{-5} centimeters per second (0.2 feet per day;

[2] Multiple IMs were implemented to address PCB-containing soil within the Eastside Properties with a majority of the construction occurring in 1997 and 1998. The IMs at the Eastside Properties include multilayer soil covers (i.e., nonwoven geotextile marker layer, minimum of 12–14-inch-thick soil isolation cover, and vegetative cover), as well as a high-density polyethene (HDPE) membrane liner in select areas. The IMs implemented during 1997/1998 were approved by the Alabama Department of Environmental Management at that time. These IMs were subsequently included in the OU-1/OU-2 ROD as part of the selected remedy subject to additional investigation of groundwater to confirm that PTW is not present in the Eastside Properties area.

Solutia 2002). When an estimated effective porosity for the shallow residuum clay of approximately 0.2 is used, the seepage velocity is approximately 10 to 20 feet per year. The proposed monitoring well locations are approximately 75 and 130 feet north-northwest (downgradient) of sample location SED0420R5 with a total PCB concentration of 25,000 mg/kg, 95 and 150 feet north-northwest (downgradient) of sample location SED0400 with a total PCB concentration of 200,000 mg/kg, and approximately 30 feet northeast and 70 feet east-southeast of sample location MH-7 with a total PCB concentration of 6,200 mg/kg in the 0–6-inch depth interval.

The proposed well locations represent a groundwater travel time of less than 10 years from the area of elevated PCBs in soil. Soil sample SED0400, along with the immediately surrounding soil samples with elevated PCB concentrations, were collected nearly 27 years ago in early 1995, suggesting that the estimated travel time in the downgradient direction is reasonable to assess groundwater impacts at the Eastside Properties. The two proposed monitoring wells and two proposed groundwater piezometers will be drilled, installed, and developed following the procedures in Attachment A-9 (Subsurface Drilling and Monitoring Well Installation Procedures) of the UFP-QAPP (Appendix A). Sonic drilling with two temporary surface isolation casings is anticipated for installation of these wells. The use of dual telescoping temporary isolation casings is proposed to minimize the likelihood of PCB drag-down from surface soil. The outermost isolation casing (10-inch diameter) is proposed to be installed to a depth of 5 feet, and the inner isolation casing (8-inch diameter) is proposed to be installed to a depth of 10 feet.

In addition to the standard soil logging that is conducted during installation of all monitoring wells and piezometers (as specified in Section 2.0 of Attachment A-6, Subsurface Drilling and Monitoring Well Installation Procedures), soil samples for analysis of PCB Aroclors and homologs will be collected from one of the monitoring well boreholes at 1-foot depth intervals from the ground surface until the soil–groundwater interface is reached based on field observations (approximately 10–15 feet bgs), following a similar procedure to that specified in Section 3.1.2.1 for the collection of soil samples from the discrete boring at PB-RR-37. Collection of the soil samples from one of the monitoring well boreholes will follow these general procedures:

1. The material collected from each 1-foot depth interval will be homogenized into a bucket with a Teflon® liner or a stainless steel bowl or equivalent in accordance with Attachment A-9 (Procedures for Compositing or Homogenizing Soil or Sediment Samples). QA/QC samples will be collected and submitted in accordance with the UFP-QAPP. Sample container sizes, holding times, preservation requirements, and other information regarding the analytical program, specific methods, and QA/QC procedures are provided in the UFP-QAPP (Appendix A).
2. Homogenized material will be transferred into laboratory-supplied bottleware or zip-top bags for shipment to the analytical laboratory. The bottleware or zip-top bags will be labeled and temporarily stored on ice (4 ± 2 °C) to await transport or preparation for shipment to the laboratory as directed in the UFP-QAPP. A minimum of 100 grams of material will be submitted to the analytical laboratory for analysis; sample mass

requirements for PCB Aroclors by USEPA SW-846 8082A and PCB homologs by USEPA Method 680 are listed in the UFP-QAPP.

3. Nondedicated sampling equipment, if used, will be decontaminated in accordance with Attachment A-2 (Field Cleaning and Decontamination Procedures) between sampling depth intervals.
4. Once the static water level in the well borehole has been determined following well installation but prior to well development, the following soil samples will be collected and then submitted to the analytical laboratory: the sample from the 1-foot interval immediately above the static water level and a subset of the overlying samples (e.g., one sample for every 5 feet bgs down to the static water level).

Based on previously installed wells within and near the Eastside Properties, it is anticipated that these wells and piezometers will be approximately 35 to 40 feet deep. Well screen intervals of 10 feet are recommended for the two new monitoring wells and two new piezometers based on the nature of groundwater flow in the shallow residuum occurring within thin silt and sand seams interbedded in the clay matrix. A 10-foot well screen interval is also consistent with the other wells located within the Eastside Properties (i.e., OW-06A, OW-09S, T-3, and T-20).

After the two new monitoring wells and two new piezometers have been installed and developed, depth to water measurements will be collected until the water levels have returned to static conditions in each new well and piezometer. After the water level in each new well and piezometer have recovered to static conditions, a static water level survey of the two new monitoring wells, the two new piezometers, and the four existing Eastside Properties monitoring wells OW-06A, OW-09S, T-3, and T-20 will be conducted to determine the local hydraulic gradient. Top-of-casing elevation measurements will be collected at the new monitoring wells and piezometers by a professional land surveyor.

Once the static water level survey is completed, groundwater samples will be collected from the two new monitoring wells following the procedures in Attachment A-7 (Groundwater Sampling Procedure) of the UFP-QAPP (Appendix A). At each of the two monitoring wells, an unfiltered sample and a filtered sample will be collected using a 0.1-micron filter. In addition, QA/QC samples, including a duplicate sample and matrix spike/matrix spike duplicate samples, will be collected from one of the monitoring wells. All samples, including QA/QC samples, will be analyzed for PCB Aroclors and PCB homologs. Required sample volumes, preservation methods, analytical methods, reporting limits, and DQOs for analysis of PCB Aroclors and PCB homologs in groundwater samples are provided in the UFP-QAPP (Appendix A).

The data will be evaluated in consultation with USEPA to assess whether additional data collection is warranted. Once a determination is made through consultation with USEPA that no additional data are needed, the two piezometers and groundwater wells will be overdrilled, plugged, and abandoned following the procedures in Attachment A-8 (Monitoring Well/Borehole Abandonment Procedure) of the UFP-QAPP (Appendix A).

3.2.2.2 T-11 Area Hydrogeologic Assessment

The groundwater PDI at the T-11 area will use a three-phase approach to evaluate hydrogeologic conditions in the T-11 area. Each phase will be designed to gather specific data to address the GW-DQO for this area:

- Phase 1 will include redeveloping the T-11 well, sampling groundwater for laboratory analysis of PCBs and geochemical parameters to supplement existing characterization data, and confirming the hydraulic conductivity of the transmissive zone through aquifer slug testing.
- Phase 2 will involve installing groundwater observation wells to be used in subsequent aquifer pump testing and collecting soil samples for geotechnical analysis.
- Phase 3 will include conducting aquifer pump testing to assess aquifer response and well yield for designing the extraction wells. It is anticipated that a portion of the groundwater discharge from the aquifer pump tests will be collected for use in the groundwater TS described in Section 4.1 and Appendix B.

The PDI activities for each phase are described in the following subsections.

Phase 1: Well Redevelopment, Groundwater Sampling, and Slug Testing

For Phase 1 of the groundwater PDI at the T-11 area, well T-11 will be redeveloped, groundwater samples will be collected and sent for analysis of PCBs and geochemical parameters, and hydraulic conductivity of the groundwater-bearing unit will be confirmed through aquifer slug testing.

Well T-11 will be redeveloped to achieve two primary objectives: (1) ensure that samples collected from T-11 are representative of groundwater by minimizing the presence of very fine-grained soil particles in groundwater samples and (2) ensure that results obtained during subsequent aquifer testing are representative by maximizing the well yield. Historical sampling data indicate high turbidity (due to very fine-grained soil particles present in the groundwater) in samples collected from well T-11 (e.g., >1000 nephelometric turbidity units [NTUs] in samples collected in April 2008). The presence of such fine-grained soil may affect PCB concentrations in groundwater samples due to the potential for sorbed-phase PCBs to enter the well. Well redevelopment will follow the procedures specified in Section 12.0 of Attachment A-6 (Subsurface Drilling and Monitoring Well Installation Procedures) of the UFP-QAPP (Appendix A). Ideally, well redevelopment will continue until measured turbidity readings are less than 10 NTUs or have stabilized to within ±10% for values greater than 10 NTUs.

After the well has been developed and water level in the monitoring well has recovered to static conditions, groundwater samples will be collected following the specified procedures in Attachment A-7 (Groundwater Sampling Procedure) of the UFP-QAPP (Appendix A). An unfiltered sample, a filtered sample collected using a 0.1-micron filter, duplicate unfiltered and filtered samples, and matrix spike/matrix spike duplicate unfiltered and filtered samples will be collected in accordance with the UFP-QAPP (Appendix A). Filtered and unfiltered samples, including QA/QC samples, will be analyzed for the following:

- PCB Aroclors (USEPA SW-846 8082)
- PCB homologs (USEPA Method 680)
- TOC (or dissolved organic carbon for filtered sample; USEPA Method 9060A)
- Cations (calcium, magnesium, sodium, potassium, manganese, iron; USEPA Method 6010C)
- Anions (sulfate, chloride, nitrate; USEPA Method 300.0)
- Alkalinity (Standard Method 2320B)
- Hardness (Standard Method SM 2340B)
- Total suspended solids (or total dissolved solids for filtered sample; Standard Method 2540D and 2540C)

A filtered sample will be collected using a 0.1-micron filter to evaluate the dissolved fractions of organic carbon, cations, anions, and hardness. Required sample volumes, preservation methods, analytical methods, reporting limits, and laboratory DQOs for analysis of PCB Aroclors, PCB homologs, and the geochemical parameters specified above in groundwater samples are provided in the UFP-QAPP (Appendix A). Water quality parameters (i.e., pH, conductivity, temperature, dissolved oxygen, oxidation-reduction potential, and turbidity) will be recorded in the field notebook at the time of sampling. Additionally, dissolved iron (Fe^{2+}) will be measured using a field test kit (e.g., Hach Model IR-18C or equivalent) and recorded in the field notebook at the time of sampling.

Once the water level in the monitoring well has recovered to static conditions after well development, T-11 will be slug tested following the procedures in Attachment A-10 (Slug Test Procedure) of the UFP-QAPP (Appendix A). Slug testing at well T-11 will be performed to confirm the hydraulic conductivity for the well of 5.93 feet per day that was reported in the OU-1/OU-2 RI (Environ 2015). Once the field activities of Phase 1 are completed, the slug testing data will be analyzed using the AQTESOLV software to calculate the hydraulic conductivity for T-11.

Phase 2: Observation Well Installation

During Phase 2, three observations wells (also referred to as piezometers) will be installed around T-11 for use in Phase 3. The proposed locations of these three observation wells were established based on theoretical drawdown-versus-distance estimates developed using the reported hydraulic conductivity estimate of 5.93 feet per day (Environ 2015). The graphic below (Exhibit 1) shows the expected drawdown as calculated using the Theis equation as a function of distance from the extraction well at various time steps at a pumping rate of 1.5 gallons per minute (gpm). Specific yield of 0.3 (indicative of an unconfined sandy aquifer; Morris and Johnson 1967) was used in the analysis. The hydraulic gradient used in the analysis is based on the gradient from well T-11 to Snow Creek. Based on this analysis, drawdown in the well is not predicted to exceed the available water column (approximately 7 feet). The analysis also predicts that a 1.5-gpm pumping rate should result in measurable drawdown up to 12 feet away from well T-11 after pumping for 8

hours. The predicted drawdown is sensitive to the hydraulic conductivity; therefore, this analysis will be updated based on the slug testing results conducted in Phase 1 as further described below.

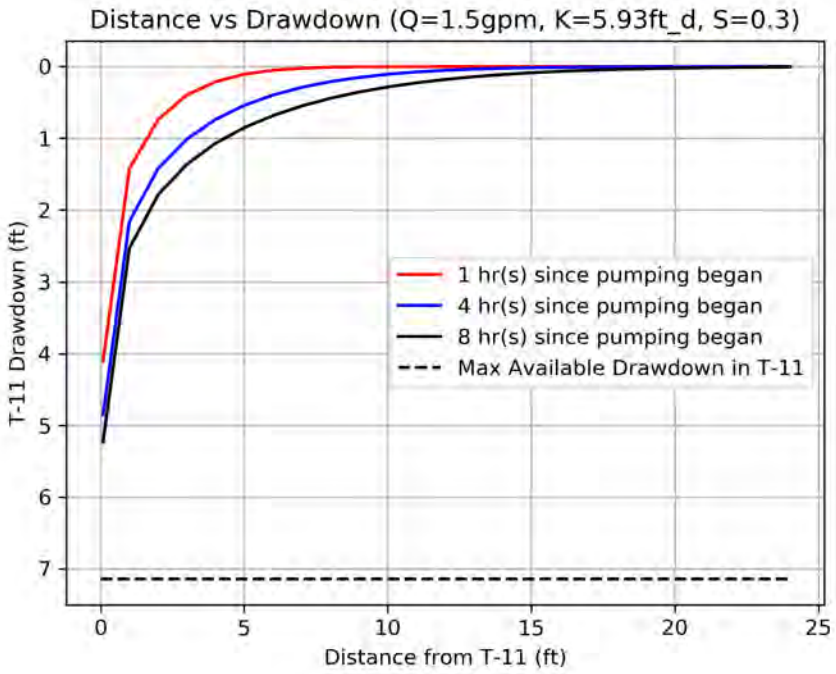


Exhibit 1. Expected drawdown using Theis equation

Based on these calculations, temporary observation wells are proposed to be placed at distances of approximately 5 feet upgradient (to the northeast), 10 feet downgradient (to the south), and 12 feet cross-gradient (to the west) from T-11 (Figure 3-6). If the hydraulic conductivity calculated from slug testing conducted in Phase 1 is significantly different (i.e., more than 1 order of magnitude [10 times] difference) than the previously reported value of 5.93 feet per day, then the proposed observation well locations will be reassessed.

The three temporary observation wells will be drilled, installed, and developed in accordance with the specified procedures in Attachment A-6 (Subsurface Drilling and Monitoring Well Installation Procedures) of the UFP-QAPP (Appendix A). Sonic drilling with temporary surface isolation casings is anticipated for installation of these wells. Based on the observed depth at which moisture occurred in well T-11, these wells will be drilled to similar depths (approximately 15 feet bgs) and have similar screened intervals (8.5 feet to 13.5 feet bgs). Since these wells will be very shallow, surface casings will only be installed to 5 feet bgs instead of the standard 10 feet specified in Attachment A-6 (Subsurface Drilling and Monitoring Well Installation Procedures) of the UFP-QAPP. Following the aquifer testing and associated data processing, and following consultation with USEPA, the three observation wells will be overdrilled, plugged, and abandoned following the procedures in Attachment A-8 (Monitoring Well/Borehole Abandonment Procedure) of the UFP-QAPP.

A geotechnical boring will be advanced to an approximate depth of 40 feet bgs to satisfy data needs for the soil and groundwater portion of the PDI in the T-11 area. This boring will also be

used as part of the groundwater program. At least one sample will be collected from the interval of 1.5–2 feet bgs, and two samples will be collected from the depth interval of the groundwater-bearing unit at 7–8 feet bgs and 9–10 feet bgs. Additional samples may be submitted for analysis to satisfy the data needs of the soil PDI (as discussed in Section 3.1.2), and all samples will be maintained under chain-of-custody protocol in accordance with Attachment A-1 (Field Sample Packing, Handling, Shipping, and Chain-of-Custody Procedures) of the UFP-QAPP (Appendix A).

Once drilling is complete, the borehole will be tremie grouted from the bottom of the borehole (approximately 40 feet bgs) to approximately 15 feet bgs before the well screen is installed to have similar construction details as the other observation wells to be installed in the T-11 area.

Phase 3: Specific-Capacity and Pump Testing

For Phase 3, aquifer pumping tests at well T-11 will be used to evaluate aquifer response and well yield for designing the groundwater extraction-and-treatment system. Aquifer testing will be conducted to evaluate groundwater response within the groundwater-bearing unit to various pumping rates. Aquifer testing will use the three temporary observation wells installed during Phase 2 and will include step-drawdown, constant-rate, and recovery tests at well T-11. Data from the test(s) will be analyzed with a commercially available software package (i.e., AQTESOLV) to derive appropriate aquifer properties and evaluate well performance. The aquifer specific-capacity test (also known as a step test) and pumping test (also known as a constant-rate test) will follow the procedures in Attachment A-11 (Specific-Capacity and Pumping Test Procedures) of the UFP-QAPP (Appendix A).

The specific-capacity test will be conducted first to determine the maximum sustainable pumping rate that can be used during the pumping test. As described in Phase 2 above, an approximate maximum pumping rate for well T-11 based on assumed parameters (including the reported hydraulic conductivity of 5.93 feet per day) was calculated to be 1.5 gpm. The hydraulic conductivity calculated from the results of the slug test conducted in Phase 1, if significantly different than 5.93 feet per day (i.e., 1 order of magnitude [10 times] difference), will be used to reevaluate the pumping rate steps described in Section 5.0 of Attachment A-11 of the UFP-QAPP.

The constant-rate pumping test will commence after groundwater levels have fully recovered to static water level conditions (or at least 24 hours) following the end of the specific-capacity test. The groundwater discharge from the aquifer pump test will be containerized, treated, and disposed of using methods like those used for purge water generated during OU-3 groundwater sampling activities. Approximately 120 gallons of the discharge water from the pump test will not be treated; instead, it will be containerized in 30-gallon drums or 5-gallon plastic totes and shipped via chain-of-custody control to SiREM in Knoxville, Tennessee, for use in the groundwater TS described in Section 4.1 and Appendix B. The actual amount of water required for the TS may be larger or smaller depending on the reported PCB concentrations of the groundwater samples collected during Phase 1.

The data from the specific-capacity test and pumping test will be analyzed using the AQTESOLV software to estimate hydraulic properties of the groundwater-bearing unit including transmissivity, hydraulic conductivity, and storativity (or storage coefficient). The results of the pumping test will be used to support the detailed design of the full-scale extraction-and-treatment system.

3.3 Snow Creek Sediment

3.3.1 Rationale and Objectives

As described in Section 2.1.3, most of the existing sediment data for the OU-1/OU-2 portion of Snow Creek were generated as part of the 1999 RFI investigation. Additional data are needed to address the data gaps listed in Section 2.2.3 and provide updated information to refine the sediment remedial extent and sediment characteristics.

The overall purpose of the sediment PDI is to further define the chemical and physical characteristics of the sediment to support the OU-1/OU-2 RD. Specific DQOs for the OU-1/OU-2 sediment PDI are as follows:

- SED-DQO 1: Provide an updated delineation of the current locations, extents, and depths of sediment deposits within the OU-1/OU-2 portion of Snow Creek as compared to the deposits depicted in the OU-1/OU-2 FS to refine removal volume estimates.
- SED-DQO 2: Refine the current understanding of sediment in the OU-1/OU-2 portion of Snow Creek that exceed the remedial criteria listed in the ROD to define the lateral and vertical limits of sediment removal.
- SED-DQO 3: Evaluate potentially efficient and cost-effective methods for dewatering and stabilizing sediment excavated from Snow Creek.
- SED-DQO 4: Evaluate treatment technologies that may be necessary to meet the requirements for discharging the supernatant that drains from sediment excavated from the creek.

Details of the proposed PDI activities to achieve SED-DQO 1 and SED-DQO 2 are provided in Section 3.3.2, and TS activities to be completed as part of the sediment PDI to achieve SED-DQO 3 and SED-DQO 4 are provided in Section 4.2.

3.3.2 Scope of Work and Investigative Methods

The sediment PDI will use a two-phase approach to evaluate the current condition of sediment deposits in the creek. Each phase will be designed to gather specific data to address the specified DQOs, as follows:

- Phase 1 will include reconnaissance and probing to identify the current locations of sediment deposits and measure their dimensions to quantify the volume of sediment deposits in the creek.
- Phase 2 will include sampling the sediment deposits identified in Phase 1 to supplement existing sediment data, support the delineation of sediment that has PCB and other

constituent concentrations above the cleanup levels defined in the ROD, and establish waste disposal requirements.

The PDI activities for each phase are described in the following subsections.

3.3.2.1 Phase 1: Sediment Probing and Reconnaissance

Phase 1 of the sediment PDI will include site reconnaissance and probing to identify the presence and extent of sediment deposits and soft sediments within the OU-1/OU-2 portion of Snow Creek. The Phase 1 probing will measure the sediment deposit dimensions, including thicknesses, to quantify the volume of sediment deposits in the creek. The physical dimensions of the creek bottom and the top elevation of the creek bed will also be measured, and the substrate types will be observed and mapped.

The Phase 1 sediment PDI approach will be like the approach used in the 1999 sediment deposit mapping and the geomorphological characterization (e.g., aggrading bar, channel deposit, terrace deposit, bank deposit, sand bar). The data generated during the PDI will be used to supplement, where appropriate, the data collected during the 1999 RFI. The Phase 1 PDI will also target the culvert beneath Quintard Mall that was constructed after the RFI was completed.

The geographical distribution of sediment in Snow Creek will be evaluated based on continuous visual inspections (access permitting) between the 11th Street Ditch and Highway 78 downstream of Quintard Mall (the termination point of OU-1/OU-2). Field reconnaissance and probing will be conducted along transects spaced approximately 100 feet apart along both sides of the creek and across the midsection of the channel to identify sediment deposits. In addition, the channel widths and bank heights will be measured at each of the 100-foot transects (Figures 3-9a through 3-9cc), and observations of bank erosion will be noted. Sediment deposits and areas of soft sediment between 100-foot transects will also be identified and mapped to determine the presence, area, and volume of each deposit. For comparison, the sediment deposits identified during the 1999 RFI and the prior sediment sampling locations are also shown on Figures 3-9a through 3-9cc. The final transect locations may be adjusted based on conditions encountered in the field. Probing will be conducted every 10 feet along each transect.

The reconnaissance and probing task will be accomplished by walking or wading along Snow Creek. To the extent possible, probing will be performed during a period of low creek flow and when significant rain is not forecasted. The probing will be performed using a small-diameter (less than 0.5 inches) steel rod advanced manually through the sediment to refusal to identify depositional areas. Areas that are penetrable with the rod by at least 4 inches and are at least 50 square feet in area will be considered sediment deposits. Each deposit will then be probed further to determine its lateral extent and the apparent average thickness. A standard survey rod (approximately 2 inches in diameter) will be used to measure water depth and identify the sediment–water interface. The field SOP included as Attachment A-3 (Sediment Probing and Sampling Procedures) of the UFP-QAPP (Appendix A) provides additional guidance and details regarding probing methods to be used.

Sediment deposits identified during probing will be characterized with respect to texture (fine vs. coarse) as well as localized geomorphological characteristics of the creek, including channel deposits, terraces, aggrading bars, and bank slopes. Each of the sediment classifications and geomorphological features will be mapped on a detailed aerial photograph of each creek section. The geographical distribution of sediment in Snow Creek will be determined based on continuous visual inspections (access permitting). Snow Creek is wadable and does not warrant the use of underwater cameras.

During the probing activities, sediment thickness, top of sediment elevation, bottom of sediment elevation, horizontal coordinates, relative sediment consistency, location, water depth, date, time, and any pertinent visual observations will be recorded. The extent of each identified sediment deposit will be sketched, and its dimensions will be marked on the sketch. The presence and location of large debris and obstructions visible in the creek will also be documented. The lateral boundaries of the sediment deposits will be recorded with a differential global positioning system (DGPS) (sub-meter accuracy) where adequate signal is available. In areas where DGPS signals are not available (e.g., in the culvert beneath Quintard Mall), sediment deposit locations will be obtained by taking measurements to known reference points.

The Highway 202 culverts (Figure 3-10) and the culvert beneath Quintard Mall (Figure 3-11) are permit-required confined spaces (Section 6.2). As such, special protocols and procedures will be followed while conducting sediment probing and sampling within the culverts. Detailed descriptions of the specific hazards and confined space entry procedures for working inside the Highway 202 culverts and the Quintard Mall culvert are presented in the *Highway 202 Confined Space Entry Plan* (Appendix D) and the *Quintard Mall Confined Space Entry Plan* (Appendix E).

The Quintard Mall culvert is accessible during periods of low or normal stream flow. At a minimum, the culvert can be accessed by walking into the culvert from the downstream end. Details for access from the upstream end of the culvert will be confirmed during PDI preparation tasks. Based on culvert foundation plan design drawings, the Quintard Mall culvert is trapezoidal with an upper width of approximately 100 feet. The height of the culvert varies and is estimated to be between 9 and 11 feet. Dimensions of the culvert will be confirmed during the PDI.

The Phase 1 sediment probing results will be provided to USEPA before the Phase 2 sediment sampling program begins (Section 3.3.2.2).

3.3.2.2 Phase 2: Sediment Sampling and Analysis

The results of the PDI Phase 1 sediment probing and mapping program will be reviewed and compared with the results from the previous field efforts to evaluate potential changes in the location and thickness of sediment deposits since the 1999 RFI. The Phase 1 measurements will be used to calculate the estimated volume of each sediment deposit similar to the process that was followed during the RFI. Phase 2 will include sampling new sediment deposits or sediment deposits in a different location or size than were identified and characterized in the RFI evaluation. For comparison, the results of the PDI sediment probing and mapping will be summarized and evaluated by segmenting Snow Creek into the seven sections (summarized in Table 3-2) like the approach used to summarize the sediment data collected as part of the 1999 RFI. The results of

this comparison will be used to identify where Phase 2 sampling is warranted. The Phase 2 sampling will occur as a separate mobilization after the sampling locations are identified. The Phase 1 sediment probing results and the Phase 2 sampling plan will be submitted to USEPA prior to sampling.

Additional sampling will not be necessary for most sediment deposits where PDI probing indicates sediment deposits in the same location and size as measured in the RFI and that were previously characterized to contain PCBs or metals above the remedial criteria identified in the ROD. For example, if the Phase 1 mapping confirms that a deposit identified for removal in the ROD is still in the same location as documented during the RFI, it will be identified for removal in the OU-1/OU-2 RD without collecting additional samples. For confirmatory purposes, a subset of sediment samples (up to five samples) will be collected from sediment deposits that were previously characterized for removal and appear to be present at the same location and size as measured in the RFI. If the PCB results for the sediment deposit are greater than or equal to 3 mg/kg, the deposit will continue to be identified for remediation. If the PCB results for a sample are less than 3 mg/kg, additional sampling may be proposed for the sediment deposit to further assess whether remediation is no longer required.

Sediment deposits identified in the Phase 1 mapping that differ in location or extent compared to those identified in the RFI mapping will be characterized under the Phase 2 sampling program. Where necessary, sampling will be conducted to provide data to supplement existing sediment data to delineate the sediment that has PCB or metals concentrations above the cleanup levels defined in the ROD.

Where additional sediment sampling is warranted, the PDI sediment sampling locations will be selected using the following approach and summarized on Figure 3-12:

- For uncharacterized sediment deposits larger than 50 cubic yards, up to five full-depth subsamples will be collected and homogenized into one composite sample.
- For uncharacterized sediment deposits larger than 5 cubic yards and smaller than 50 cubic yards, one full-depth sediment sample will be collected.
- Uncharacterized sediment deposits between 1 and 5 cubic yards will be characterized together with other nearby sediment deposits (located within five transects and are larger than 5 cubic yards when combined) by collecting and homogenizing up to five full-depth subsamples into one composite sample.
- Sediment deposits smaller than 1 cubic yard will not be sampled.
- Sediment deposited within each of the five Highway 202 box culverts (Figure 3-10) will be characterized by collecting five full-depth subsamples spaced evenly within each culvert and homogenizing the subsamples into one composite sample. Sampling within the culverts will evaluate the extent of PCBs previously detected above 50 mg/kg within this portion of the creek.
- Sediment deposited within the culvert beneath Quintard Mall will be characterized in three different sections: the upstream section between T195-00 and T200-00, the

middle section between T200-00 and T205-00, and the downstream section between T205-00 and T210-00 (Figure 3-11). Five full-depth subsamples spaced evenly within each culvert section will be collected and homogenized into one composite sample.

The additional sampling will only target sediment deposits consisting of sand, silt, or clay. Samples will not be collected from deposits that are predominantly gravel or cobbles, and samples will not be collected from underlying material that represents refusal.

Where sediment deposits exceed the remedial criteria of 3 mg/kg based on the PDI results, it is anticipated that the full depth and extent of the sediment deposit will be removed. As such, full-depth subsamples will be collected and composited across the sediment deposit to characterize the full depth and extent of the sediment deposits that will be removed as part of the remediation. Partial depth removal of sediment deposits is not anticipated during the remediation project. This current assumption is based on the relatively shallow nature of the sediment deposits that have been previously observed in Snow Creek, absent the deposits in the Highway 202 culverts and possibly the Quintard Mall culvert. The sediment deposits in the Highway 202 culverts are expected to require full removal based on the previous characterization data. Final details regarding the extent of remediation will be determined as part of the OU-1/OU-2 RD and will be based on the PDI results.

The Highway 202 culverts and the culvert beneath Quintard Mall are permit-required confined spaces (Section 6.2). Due to the potential hazards presented by these permit-required confined spaces, the Phase 2 sediment sampling will be conducted concurrently with the Phase 1 reconnaissance and probing activities within these culverts. Detailed descriptions of the specific hazards and confined space entry procedures for the work inside the culverts are presented in the *Highway 202 Confined Space Entry Plan* (Appendix D) and the *Quintard Mall Confined Space Entry Plan* (Appendix E).

The sediment core samples will be collected by walking or wading in the creek and using direct-push coring techniques in accordance with the field SOP included as Attachment A-3 (Sediment Probing and Sampling Procedures) in the UFP-QAPP (Appendix A). Sediment cores will be collected by advancing Lexan® tubing that is 2 to 4 inches in diameter through the sediment by hand or by using a driving device if necessary. The core tubing will be pushed from the top of the sediment to the full depth of the deposit to collect a continuous sediment core. Once a core is collected, the recovery date, time, location, and other pertinent information will be recorded. The recovered sediment core will be capped within the tube, labeled, and handled for processing. The sediment cores will be split longitudinally and logged using the USCS to include soil type, color, consistency, odors, and visible evidence of impacts (e.g., sheen) and then photographed. The full core depth will be collected and homogenized in a stainless steel tray/bowl or disposable equivalent in accordance with the field SOP included as Attachment A-9 (Procedures for Compositing or Homogenizing Soil or Sediment Samples) in the UFP-QAPP (Appendix A). The homogenized samples will be placed into the appropriate container provided by the analytical testing laboratory.

In areas where sediment deposits are less than 6 inches in thickness and difficult to sample using Lexan® tubing, a stainless steel bucket auger or hand trowel will be used to collect a representative sediment sample that will be processed as described previously.

When practicable, disposable equipment (e.g., core liners, bowls, spoons, aluminum pans) will be used for sampling, homogenizing, and subsampling procedures. However, when equipment is reused (e.g., stainless steel bucket auger), the equipment will be decontaminated between sampling locations and before leaving the Site in accordance with the field SOP included as Attachment A-2 (Field Cleaning and Decontamination Procedures) in the UFP-QAPP (Appendix A).

The sediment core locations will be recorded in the field with a DGPS when a sufficient signal is available. In areas, where DGPS signals are not available (e.g., in the culvert beneath Quintard Mall), the locations will be determined by collecting measurements to known reference points and recorded. Field notes will be recorded using a field tablet, in a field logbook, or on data collection forms.

Each sediment sample will be analyzed for the following parameters:

- PCBs (USEPA SW-846 8082)
- TOC (Lloyd Kahn)
- Particle size (including hydrometer for cohesive samples only; ASTM D6913/D7928)
- Atterberg Limits (cohesive samples only; ASTM D4318)
- Specific gravity (ASTM D854)
- Moisture content (ASTM D2216/D2974A)

Sediment samples collected from the downstream section of Snow Creek (downstream of T130-00) will also be analyzed for metals because prior investigations indicated the presence of metals above the remedial criteria in the downstream section of the creek and floodplain where PCB concentrations were below the remedial goals. Upstream of this location, metals concentrations were co-located with PCB detections in sediment samples. The sediment samples collected in the downstream section of Snow Creek (downstream of T130-00 [Figure 3-9r]) will be analyzed for barium, chromium (total), cobalt, lead, manganese, mercury (total), nickel, and vanadium (USEPA SW-846 6010C/7471B).

Once the samples are prepared, they will be labeled, and information will be entered onto a chain-of-custody form for transport to the laboratory in accordance with the field SOP included as Attachment A-1 (Field Sample Packing, Handling, Shipping, and Chain-of-Custody Procedures) in the UFP-QAPP (Appendix A). Sample containers will be placed in a secure area and stored on wet ice or under refrigeration at 4±2°C pending shipment to the analytical laboratory.

QA/QC samples will be collected and submitted in accordance with the UFP-QAPP (Appendix A). Sample container sizes, holding times, preservation requirements, and other information on

the analytical program, specific methods, and QA/QC procedures are provided in the UFP-QAPP (Appendix A).

Three bulk sediment samples will be collected for treatability testing from target removal areas within Snow Creek. The bulk sediment sampling locations will be selected based on the following criteria:

- To provide a range in sediment types (i.e., fine material vs. coarse material) based on historical data and data collected during Phase 1 and Phase 2 of the sediment PDI
- To focus on areas with the largest expected volume of sediment to be removed
- To be spatially representative of the sediment removal areas within Snow Creek

The bulk sediment samples will be composited from sediment samples collected in the target remedial areas. The bulk material samples collected for the treatability testing will be collected from the full-depth samples of sediment deposits at up to five locations and then composited together. It is anticipated that, at a minimum, one bulk sediment sample will be composited from samples collected upstream, within, and immediately downstream of the Highway 202 culverts, which are anticipated to be handled together as part of the remediation. Other sampling locations will be determined after the PDI survey and sampling data has been reviewed. Additional information regarding the TSs for sediment are provided in the TS work plan (Appendix C).

3.4 OU-1/OU-2-Wide Evaluations

The OU-1/OU-2-wide evaluations described in the following subsections are common elements needed to complete the OU-1/OU-2 RD for soil, groundwater, and sediment. The following tasks will be completed OU-1/OU-2-wide:

- A geophysical survey will be conducted to evaluate whether there are buried structures, pipes, utilities, or other features within the OU-1/OU-2 RD/RA areas.
- The Highway 202 and Quintard Mall culverts and other structures adjacent to OU-1/OU-2 RD/RA areas will be evaluated to assess whether special considerations will be required during the RA.
- Topographical surveys of OU-1/OU-2 will be conducted to create a survey-level base map for the RD.
- An assessment of cultural and archeological resources will be conducted to evaluate the presence of historical or archeological resources.

The rationale, objectives, and investigative methods for each of these scopes of work are presented in the subsections below.

3.4.1 Utility Surveys

Desktop and geophysical field investigations will be completed to evaluate the presence of buried structures, pipes, utilities, or other features within the OU-1/OU-2 areas as described below.

3.4.1.1 Rationale and Objectives

The presence of aboveground and underground utilities in the OU-1/OU-2 areas could pose risks during excavation to both the integrity of the utility and the safety of the workers. During the PDI, active outreach will be completed with property owners, local city building departments, area utility companies (including 811), and county and state transportation agencies regarding the presence of utilities that could impact remedy implementation, worker safety, and/or utility integrity. Utility organizations that would typically be contacted as part of the outreach process include sewer, water, gas, electric, oil, and cable/fiber optic. The data and information obtained from this work element will be integrated into the Preliminary (30%) RD submittal as existing conditions to allow the OU-1/OU-2 RD to be implemented safely in the vicinity of active utilities.

The DQO to support the utility survey as part of the OU-1/OU-2 PDI is as follows:

- US-DQO 1: Identify utilities located within or adjacent to the proposed remedial areas and gather information for those features to design the RA and potential mitigation measures to prevent damage.

A description of the work to be conducted to achieve this DQO is provided in the following subsection.

3.4.1.2 Scope of Work and Investigative Methods

Prior to starting utility survey work, P/S will complete a desktop study (e.g., review historical documents, contact Alabama One-Call) to obtain copies of the available record mapping of utilities within the OU-1/OU-2 RD/RA areas. P/S will request existing conditions surveys, permits, and topographical surveys from appropriate parties (e.g., owner, municipalities, utility agencies). This information will be used to supplement the geophysical utility survey to be completed within the OU-1/OU-2 RD/RA areas.

Geophysical surveys will be conducted over soil boring locations, ISM sampling areas, and monitoring well and piezometer locations where intrusive sampling activities will be completed as part of the PDI work. Additionally, geophysical surveys will be conducted over areas (e.g., excavation areas) where utility information is needed to support the OU-1/OU-2 RD. The following geophysical techniques will be used to locate subsurface features and utilities:

- Ground-penetrating radar (GPR) system: The GPR instrumentation emits pulses of high-frequency electromagnetic (EM) waves into the subsurface. The EM waves either dissipate or reflect back to the GPR sensor based on contact with subsurface features (e.g., a metal pipe will reflect energy back to sensor). The GPR profiles obtained during the survey will provide cross-sectional subsurface information that can be reviewed in real time. Underground storage tanks (USTs) and buried pipelines typically exhibit anomalous responses that have characteristic GPR signatures (i.e., hyperbola or inverted "U"). From the cross-sectional subsurface information, the size, orientation, and approximate depth of the identified anomalies can be estimated. GPR survey resolution is anticipated to be a minimum of 0.3 feet.

- Radio frequency (RF) instrument: An RF instrument is capable of tracing active electric lines, telecommunication lines, and other buried lines that might have an induced charge. RF instrumentation is connected directly to or through inductive coupling at a point that allows the entirety of the utility line to be traced by introducing a small current and measuring these signals at remote locations. Strong response peaks are observed directly over the lines by an RF sensor.
- EM unit: An EM instrument induces a current by applying magnetic fields into the subsurface and measuring the resulting response. EM is highly sensitive to buried metal features, such as USTs, rebar, and buried pipelines, within the upper 3.5 feet of the subsurface, which is the typical burial depth of underground utilities.

Before geophysical survey work begins in upland areas, grid markers will be established. The grid markers will be used to locate the geophysical intersecting traverse lines. GPR, RF, and EM measurements will be obtained at 10-foot intervals along parallel traverse lines that are spaced 10 feet apart. This spacing corresponds to a 10-foot by 10-foot grid pattern.

Data will be acquired in the scan mode of the various instruments throughout accessible portions of the OU-1/OU-2 RD/RA area by qualified personnel with expertise in the selected geophysical instrumentation. The instrument responses will be continuously monitored, and the locations of anomalous responses from significant buried objects will be indicated on the ground with stakes, spray paint, or other preferred marking medium. Additionally, as-built coordinates will be collected of the anomalies using a mapping-grade GPS with sub-foot accuracy. Identified anomalies will be evaluated with statistical techniques to assess if further investigation should be conducted.

3.4.2 Structural Evaluations

Desktop and survey field investigations will be completed to locate and document existing conditions of structures, slopes, and utilities along Snow Creek as described below.

3.4.2.1 Rationale and Objectives

Snow Creek passes through, under, or immediately adjacent to several structures within OU-1/OU-2 (Table 3-2, Figure 3-13). Field data and information will be collected during the PDI to support the evaluation of these structures (e.g., bridges, culverts, outfalls) along Snow Creek.

Structures adjacent to potential remedial areas in Snow Creek will be evaluated during the RD to determine whether the remedial construction will present special concerns for structural integrity. The first step in the design process will be to determine the final extent and depths of remediation to analyze whether nearby structures may be affected. The results of the Phase 2 PDI sediment sampling program (Section 3.3) will be evaluated, along with existing sediment sampling data, to refine the extent of sediment that exceeds the remedial criteria. Details regarding the locations and depths of sediment remediation will be necessary to complete a structural evaluation.

The DQO to support the structural evaluation as part of the OU-1/OU-2 PDI is as follows:

- STR-DQO 1: Identify structures, slopes, and utilities located within or adjacent to the proposed remedial areas and gather information for those features to design the RA and potential mitigative measures to prevent damaging them.

A description of the work to be conducted to achieve this DQO is provided in the following subsection.

3.4.2.2 Scope of Work and Investigative Methods

In addition to the PDI sediment investigation activities described in Section 3.3, the following investigative efforts will be undertaken to gather information about the structures along Snow Creek that may potentially be affected by remedial construction activities:

- The field crew will conduct an initial reconnaissance along Snow Creek to identify and note any visible structures not listed in Table 3-3. Each observed structure will be photographed from various angles and directions. In addition, construction material types will be noted, and field measurements will be collected from accessible portions of the structures to determine approximate dimensions. The field crew will note any debris observed at each structure and any obvious conditions related to the structures.
- P/S will review property records to identify the owners of each potentially affected structure along the OU-1/OU-2 portion of Snow Creek.
- P/S will contact the owners of the potentially affected structures to request record drawings or design drawings for the structures. In addition, P/S will request any available recent conditions survey reports for the structures.

Once the sediment remediation extents and depths are understood based on the PDI results, the engineering team will engage an experienced structural engineer to review the remediation extents, field data, and available structural records to determine whether any structures may warrant additional structural evaluations regarding the remediation work.

Structures that might be at risk from the remediation work will be evaluated further as the RD progresses. Details for the next steps in the evaluation will be determined following review of the available data and information. The structural evaluation may include outreach to owners of the structures to present the conceptual approach for remediation near the structures and to determine if they will perform a conditions survey for the structure. The results of a recent (after 2019) structural assessment conducted could also be acceptable. The assessment would need to have been performed under the supervision of a professional engineer and address maintenance-related construction activities such as the removal of sediment from within or underneath the structure. Depending on the availability of an owner-prepared structural survey that can be used to describe current baseline structural conditions, P/S may retain an Alabama-licensed structural engineer or civil engineer. The engineer would need to have a background in bridge structures to conduct a detailed existing conditions survey to document the condition of bridge-type structures and provide recommendations on best practices or mitigative measures that could be implemented prior to, during, and following the remediation work for those types of structures.

3.4.3 Base Mapping Activities

Topo-bathymetric surveys will be completed to update the Site's base map in OU-1/OU-2 areas. The surveys will use light detection and ranging (LiDAR) and be supplemented with manual survey methods to obtain structure elevations.

3.4.3.1 Rationale and Objectives

Data regarding the current topo-bathymetric surface of OU-1/OU-2, including creek bank elevations and structures (e.g., bridges, culverts, and outfalls), are needed to create a survey-level base map for the OU-1/OU-2 RD. The DQOs to support the base mapping activities as part of the OU-1/OU-2 PDI are as follows:

- BM-DQO 1: Update the project base mapping to support the development of the RD and contract drawings.
- BM-DQO 2: Gather topography and bathymetry data to support the design for the sediment remediation and to use in the hydraulic modeling of potential flooding impacts associated with potential remediation within and near the Highway 202 culverts.

3.4.3.2 Scope of Work and Investigative Methods

The survey methods discussed below have been selected for the PDI based on their ability to generate data for use in the OU-1/OU-2 RD and their applicability during the RA. LiDAR will be the primary survey method and will be supplemented with manual survey methods to obtain structure elevations.

LiDAR uses an unmanned aerial system (UAS) or survey plane equipped with a laser that emits pulses of light to the ground surface that reflect back to the UAS or survey plane to measure distance to the ground surface or structures. A UAS or survey plane equipped with a LiDAR sensor capable of measuring a minimum of 40 points per square meter with minimum relative accuracies of 1 to 4 centimeters will be flown over OU-1/OU-2 to conduct a topographic survey. Data will be collected and processed in conformance with USGS's LiDAR Base Specification Version 1.3. A point cloud will be generated, allowing for feature extraction and terrain modeling, and a bare earth terrain surface will be delivered as a digital terrain model. In addition to the LiDAR survey, additional technologies (i.e., video or photogrammetry) may be used to meet the inspection and long-term monitoring needs during construction. Ideally, the LiDAR survey will be completed during leaf-off conditions (i.e., winter) to obtain maximum coverage of OU-1/OU-2. Traditional survey work might also be required for locations where UAS or survey plane operations cannot provide adequate LiDAR coverage. The aerial-based survey will be completed in accordance with all applicable federal, state, and local (i.e., City of Oxford, City of Anniston, and Calhoun County) regulations.

Survey data for structures located in OU-1/OU-2 will be collected manually by a professional land-based surveyor. The structural survey will include locations and elevations of abutments, beams, pipe inverts, and any other details for bridges, tide gates, outfalls, and culverts located in or next to OU-1/OU-2 RD/RA areas.

The horizontal and vertical benchmarks will be established and/or verified. The horizontal datum will conform to Alabama State Plane East Zone Coordinate System, North American Datum of 1983 (NAD83) in units of feet. The vertical datum will conform to the National American Vertical Datum of 1988 (NAVD88).

A professional land surveyor will conduct surveys that conform to the requirements of the most current versions of the following guidance documents:

- United States Army Corps of Engineers (USACE) Manual No. 1110-1-1005 Control and Topographic Surveying (USACE 2007)
- USACE Manual No. 1110-1-1000 Photogrammetric Mapping (USACE 2002)
- FM 3-34.331 Topographic Surveying (USACE 2013)

Survey vertical accuracy is anticipated to be 0.1 feet.

The results of the topographic survey will be submitted to P/S by the professional land surveyor in the following formats: (1) AutoCAD files, (2) digital shapefiles and polyline coverage maps, and (3) American Standard Code for Information Interchange (ASCII) files of the XYZ coordinate data used to produce AutoCAD files. Manual elevations of structures will be presented in ASCII files of the XYZ coordinate data, and the linework of these structures will be incorporated into the AutoCAD and digital shapefile deliverables from the professional land surveyor to P/S.

3.4.4 Cultural and Archeological Resource Assessment TBD

Outreach will be completed with the Alabama State Historic Preservation Office (SHPO) to evaluate whether additional research is required regarding the potential presence of historical or archeological resources within the OU-1/OU-2 RD/RA area. If additional research or documentation is deemed necessary by the Alabama SHPO, the requested information will be provided to facilitate further review by SHPO.

3.5 Baseline Surface Water Sampling

3.5.1 Rationale and Objectives

Surface water monitoring will be conducted at the upstream and downstream ends of OU-1/OU-2 in accordance with the selected remedy described in the ROD. Most of the existing surface water data for the OU-1/OU-2 portion of Snow Creek were generated as part of the RFI and the Nonresidential Properties FSP. The PDI will include surface water sampling to establish a baseline for comparison to post-remedy monitoring.

The DQO for the OU-1/OU-2 surface water PDI is as follows:

- SW-DQO 1: Provide baseline surface water data that can be used for comparison with post-remedy surface water monitoring data.

Details of the proposed PDI activities to achieve SW-DQO 1 are provided in Section 3.5.2.

3.5.2 Scope of Work and Investigative Methods

The baseline surface water monitoring program will include collecting surface water samples from the upstream and downstream ends of the OU-1/OU-2 portion of Snow Creek during seasonal (i.e., quarterly) base flow. The samples will be collected during base flow as these conditions are present approximately 90% of the time. The sample locations are shown on Figure 3-14. The upstream sampling location is located downstream of the confluence of Snow Creek and the West 9th Street Creek on the downstream side of the West 14th Street Bridge. The downstream sampling location is at the southern end of OU-1/OU-2 at the outfall of the culvert that passes beneath the Quintard Mall. The sampling will be conducted once per quarter (i.e., seasonally, 4 times per year) during base-flow conditions and will start following USEPA approval of the PDIWP and continue for 2 years or to the start of remediation, whichever occurs first.

Baseline surface water samples will be collected using grab sampling techniques and in situ passive sampling techniques. Grab sampling techniques will be used to measure total[3] and dissolved concentrations in surface water. In situ passive samplers, specifically commercially available polyethylene-based passive samplers, will be used to measure PCB concentrations truly dissolved in surface water (i.e., not associated with suspended particles or colloids).

The general approach for sampling surface water is as follows:

- The sampling procedure will depend on the access method.
- If the location is accessed from the shoreline without entering the creek, sampling personnel will collect surface water using a stainless steel dipper.
- If the location is accessed by wading into the creek, sampling personnel will collect surface water by submerging the sample container. Sampling personnel will face upstream in areas of flow and collect the sample without disturbing the bottom sediment.
- If the location is accessed from a bridge, sampling personnel will deploy a Kemmerer sampler. Once the Kemmerer sampler has been lowered to the correct sample depth, sampling personnel will trigger the Kemmerer sampler to close by releasing the weighted messenger. Care will be taken to collect the sample without disturbing the bottom sediment.
- Filtered samples will be collected using a 0.1-micron filter to evaluate the dissolved fractions of PCBs. Filtering of surface water will be completed in the field.

Additional details regarding surface water grab sampling are in the SOP included as Attachment A-13 (Surface Water Sampling, Filtering, and Measuring Procedures) of the UFP-QAPP (Appendix A). Samples will be labeled, placed in ice coolers, and shipped to Eurofins-TestAmerica for analysis of PCB homologs by USEPA Method 680 with a detection limit less than the remedial goal of 0.014 µg/L. In addition, the filtered and unfiltered grab samples will be

[3] Total concentration in surface water includes concentrations associated with suspended particles and colloids.

analyzed for metals (lead and total chromium only) by USEPA Method 6010C and hexavalent chromium by USEPA Method 7196A.

Passive samplers will be deployed in the creek at the same upstream and downstream grab sample locations shown on Figure 3-14. Passive sampling will be conducted twice per year (for up to 2 years) at each location.[4] In situ surface water sampling will be conducted in general accordance with procedures included in Attachment A-13 of the UFP-QAPP (Appendix A). The general deployment procedures are as follows:

- Passive samplers and any support frames will be deployed such that the device is suspended mid-depth under base-flow conditions and a minimum of 3 inches above the sediment surface such that the sampler is submerged continuously over the deployment period.
- The passive samplers will be deployed for a minimum of 28 days. Sampling personnel will retrieve the passive samplers as close to the end of the 28-day deployment period as possible accounting for weekends or possible holidays. The retrieved passive sampler will be wrapped in foil in accordance with the UFP-QAPP and procedures included in Attachment A-13 of the UFP-QAPP (Appendix A).

Samples will be labeled, placed in ice coolers, and shipped to Eurofins-TestAmerica for analysis of PCB congeners by USEPA Method 1668A and PCB homologs by USEPA Method 680 using a common extract to minimize the potential for variability.

[4] In situ passive samplers will typically be deployed during the late spring to early summer and early fall to avoid extreme low-water and high-water conditions and the potential for temperatures to fall below freezing that could damage the passive sampling devices.

4. TREATABILITY STUDIES

TSs for groundwater and sediment will be conducted to support the development of the Preliminary (30%) RD. The TSs are summarized in Sections 4.1 and 4.2, respectively. Supporting details are provided for the groundwater and sediment in stand-alone TS work plans provided in Appendices B and C, respectively.

4.1 Groundwater Treatability Study

A bench-scale TS will be completed to support the design of the groundwater remedy component for the T-11 area, with specific focus on performance of the GAC component of the planned treatment. A TS to assess GAC performance for the T-11 area is recommended based on the presence of lower-chlorinated PCBs and the potential for competing demand on the carbon from naturally occurring organics that might also be present in groundwater. The overall objective and purpose of the groundwater TS is to generate data for designing and evaluating groundwater treatment using GAC filtration to satisfy the GW-DQOs 3 and 4 presented in Section 3.2.1.

Groundwater samples for the bench-scale treatability tests will be collected from monitoring well T-11 as part of the PDI sampling and aquifer testing program (Section 3.2). Bench-scale treatability tests will include an isotherm batch test to address GW-DQO 3 and a rapid small-scale column test to address GW-DQO 4. Details of these tests and the associated data analysis and reporting for the groundwater TS are provided in Appendix B.

4.2 Sediment Treatability Study

A bench-scale TS will be completed to support the design of the sediment remedy component of the OU-1/OU-2 portion of Snow Creek. The TS will generate data that will be used in designing and evaluating sediment removal, transport, processing, and disposal components of the remedy. The sediment TS will be performed specifically to satisfy SED-DQO 3 and SED-DQO 4.

The sediment to be excavated from Snow Creek might require processing, treatment, and conditioning to allow it to be loaded into trucks, to meet regulatory requirements for transportation on public roads (i.e., no free liquids permitted), and to strengthen the sediment to satisfy waste acceptance criteria for the selected off-site landfill. The results of the proposed bench-scale testing program will help identify potential methods for implementing the sediment RA.

Sediment excavated from water bodies contains excess water (referred to as supernatant) that needs to be removed through dewatering operations prior to sediment transport for disposal. The supernatant from Snow Creek sediment will likely require treatment prior to discharge. The bench-scale treatability testing program will provide data on the potential concentrations of contaminants in the supernatant for review as part of the OU-1/OU-2 RD to evaluate potential treatment requirements. During design, data from analytical testing of the supernatant will be compared to the expected discharge limits to assess treatment requirements prior to discharge. Details of these tests and the associated data analysis and reporting for the sediment TS are provided in Appendix C.

5. PROPERTY ACCESS EVALUATION

P/S are required to use best efforts, as defined under Section VIII (Property Requirements) Paragraph 21 of the CD, to secure enforceable access agreements from third parties to conduct the work identified in the CD. Properties for which access or use restriction agreements may be required have been identified as part of developing this PDIWP. These properties are listed in Table 5-1. The table includes the property identification numbers (PPINs); the owner(s); the street address; the reason(s) for access, including bridges (that conveys vehicular or rail traffic); the low-activity special use properties; locations within 100 feet of Snow Creek for sediment-related efforts; nonresidential soil and IM areas; and dredge spoil piles. There are two unique features where access will be required—a building that was built over Snow Creek and portions of the Quintard Mall parking lot that overlie Snow Creek (Table 5-1). These two features along with other structures that span Snow Creek are also listed in Table 5-2 to highlight features that might require structural evaluations (described in Section 3.4.2). While Table 5-1 includes over 120 individual PPINs, multiple parties own multiple individual properties and, as a result, the number of individual access agreements will be substantially less. In addition to the individual properties, access to right-of-way properties will be required from multiple parties, including the City of Anniston, the City of Oxford, Calhoun County, the Alabama Department of Transportation, and Norfolk Southern Railway. These parties are also identified in Table 5-1 for specific properties and, thus, access-related discussions with these entities will include both specific parcels and right-of-way areas.

The process for obtaining property access was started following submittal of the PDIWP in November 2021, recognizing the time necessary for obtaining access that may include grants allowing P/S and USEPA to access Non-Settling Owner's Affected Property (as defined by the RD/RA CD) and/or may require a Non-Settling Owner to agree to limitations on the use of Non-Settling Owner's Affected Property and to agree to refrain from interfering with or adversely affecting the implementation, integrity, or protectiveness of the RA.

In addition to requests to conduct the PDI work detailed herein, access will be required for a subset of properties to allow P/S to conduct the RA activities (including soil and sediment removal and backfill, material staging, and support activities), monitor completed RAs, and provide ingress and egress on the affected property. These future access needs will be presented in the Preliminary (30%) RD. Potential use restriction agreements may also be included in the Preliminary (30%) RD that could place restrictions on the use of groundwater for potable purposes and constraints on soil-disturbance activities.

Once the properties that will need to be accessed are finalized, the process to obtain the access and use restriction agreements required by the RD/RA CD will include the following steps:

1. The current owner of record of each Non-Settling Owner Affected Property will be identified through Calhoun County, Alabama's available on-line mapping portal (see https://gis.calhouncounty.org/Parcelviewer2/).
2. P/S will transmit a letter and access agreement or use restriction agreement or both to the current owner of record.

3. If the access agreement or use restriction agreement is not returned to P/S within 30 days from the date of delivery, a follow-up letter will be transmitted to the owner of record requesting a meeting to discuss the request for the access agreement or the use restriction agreement or both.
4. If a meeting is not scheduled within 30 days of the date of delivery of the follow-up letter or if following a meeting with P/S and exhaustion of best efforts as described in the CD, a Non-Settling Owner refuses to provide access or use restrictions, then P/S will provide a copy of all correspondence to USEPA for review and further action (i.e., assist P/S in obtaining access or use restrictions or take independent action) if deemed appropriate.

6. HEALTH AND SAFETY

Key health and safety concerns and considerations associated with implementing the PDI are discussed in the following subsections. The overarching concerns are presented in Section 6.1, and a summary of the confined space entry plans developed to support PDI activities for the Highway 202 culverts and the portion of Snow Creek that passes under the Quintard Mall parking lot are provided in Section 6.2.

6.1 Health and Safety Plan

Work performed during the PDI will be conducted in accordance with the OU-1/OU-2 RD/RA HASP. An initial version of the OU-1/OU-2 RD/RA HASP was submitted to USEPA as Appendix A of the RDWP (P/S 2021a) and an updated version (P/S 2022) was submitted as a companion document to this PDIWP. The OU-1/OU-2 RD/RA HASP is a stand-alone document that covers work conducted by P/S for the RD/RA. Job Safety Analyses (JSAs) will be developed to identify task-specific hazards and prescribe hazard control measures and emergency preparedness actions for each scope of work described in this PDIWP.

Subcontractors who are hired to carry out activities supporting the RD/RA are required to work under the OU-1/OU-2 RD/RA HASP, or they might be required to develop their own HASP. When applicable, the subcontractor's HASP will be specific to OU-1/OU-2 and the task(s) that the subcontractor will be conducting. All subcontractor-specific HASPs must also comply with the requirements of the OU-1/OU-2 RD/RA HASP and will be distributed to the project parties and kept on file electronically. P/S will approve each subcontractor-specific HASP prior to the start of work. Subcontractors are responsible for protecting their workers, and subcontractor work must be conducted in accordance with the approved HASP. Subcontractors are required to adhere to the health and safety roles, responsibilities, and commitments mutually and contractually agreed upon between themselves and P/S.

All personnel involved in implementing the PDI and TS scopes of work will be required to meet the health and safety requirements presented in the HASP. The following general health and safety requirements will be implemented during the project:

- A JSA will be developed for each PDI task or activity. The JSA will identify potential hazards for each task and describe mitigation measures and action levels to address each hazard. The JSA may incorporate procedures discussed in the OU-1/OU-2 RD/RA HASP or attachments or it may identify the need for new or updated procedures. JSAs will be developed in accordance with Section 3.2 of the OU-1/OU-2 RD/RA HASP.
- Health and safety meetings will be completed at the start of each workday, at the beginning of each new task, and when new personnel are introduced to OU-1/OU-2. As discussed in Section 3.3 of the OU-1/OU-2 RD/RA HASP, these meetings will be used to discuss specific safety topics relevant to the task and obtain personnel feedback.
- Utility avoidance and clearance activities will be performed prior to intrusive field work in accordance with Section 4.1 of the OU-1/OU-2 RD/RA HASP. Utility

clearance/avoidance activities will include contacting local public utility clearance system (e.g., contacting 811), reviewing the detailed utility plan, and visually inspecting the area for utilities. Additional utility avoidance and clearance activities (e.g., private utility-location service, hand-clearing boring location) may be performed as discussed in the OU-1/OU-2 RD/RA HASP.

- Field activities that occur within a railroad right-of-way or within 25 feet of a railroad line will be performed in accordance with Section 4.2 of the OU-1/OU-2 RD/RA HASP. Personnel will meet with a representative of the railroad company to determine what standards are required for work on that railroad property, conduct the required job briefing, and log it as an OU-1/OU-2 safety meeting.
- Soil boring and monitoring well installation activities will be performed in accordance with Section 4.4 of the OU-1/OU-2 RD/RA HASP and task-specific JSAs.
- Sediment probing and sampling will be performed in accordance with Section 4.5 of the OU-1/OU-2 RD/RA HASP and task-specific JSAs. Due to the configuration and specific hazards associated with work inside the Highway 202 culverts and the Quintard Mall culvert, these structures are considered permit-required confined spaces and will require a confined space entry permit.
- Surface water sampling will be performed in accordance with Section 4.7 of the OU-1/OU-2 RD/RA HASP and task-specific JSAs.
- Safety procedures will be included to account for applicable federal, state, local, and P/S requirements due to the COVID-19 pandemic. Individual company-specific policies and procedures can be integrated into the task-specific JSAs or subcontractor HASPs (where applicable) provided they meet the minimum requirements set by the above listed entities.

6.2 Confined Space Entry Plans

Entry into the Highway 202 culverts and the culvert beneath Quintard Mall will be necessary to complete the PDI work described in Section 3.3. Due to the configuration and specific hazards associated with work inside these structures, the Highway 202 culverts and the Quintard Mall culvert are considered permit-required confined spaces.

The Highway 202 culverts are in Anniston, Alabama, and consist of five separate concrete box culverts, each approximately 10 feet wide by 10 feet tall, that convey Snow Creek flows beneath Highway 202. The culverts are open at both ends and contain varying amounts of accumulated sediment.

The Quintard Mall culvert is in Oxford, Alabama, and conveys Snow Creek stream flow for approximately 0.3 miles beneath a portion of the parking lot and mall building. There are only two entry points to the culvert: one at the upstream end and one at the downstream end. The Quintard Mall culvert is accessible during periods of low or normal stream flow. At a minimum, the culvert can be accessed by walking into the culvert from the downstream end. Details for access from the upstream end of the culvert will be confirmed during PDI preparation tasks. Based on culvert

foundation plan design drawings, the Quintard Mall culvert is trapezoidal with an upper width of approximately 100 feet. The height of the culvert varies and is estimated to be between 9 and 11 feet. Dimensions of the culvert will be confirmed during the PDI.

These confined spaces will be entered during the PDI program to perform such tasks as surveying, visual observations, sediment probing, and sediment sampling. Due to the specific hazards presented by these structures, P/S have prepared area-specific confined space entry plans that will be followed while working within these confined spaces. Detailed descriptions of the specific hazards and confined space entry procedures for the work inside the Highway 202 culverts and the Quintard Mall culvert are presented in the *Highway 202 Confined Space Entry Plan* (Appendix D) and the *Quintard Mall Confined Space Entry Plan* (Appendix E). The work inside these culverts will be conducted in accordance with these plans and with the project-specific HASP and the associated JSAs.

7. QUALITY ASSURANCE PROJECT PLAN

The objective of the UFP-QAPP is to help generate scientifically valid analytical data. The UFP-QAPP has been developed in accordance with the requirements of the following:

- Requirements for Quality Assurance Project Plans, QA/R-5, EPA/240/B-01/003 (USEPA 2001)
- Guidance for Quality Assurance Project Plans, QA/G-5, EPA/240/R 02/009 (USEPA 2002a)
- Uniform Federal Policy for Quality Assurance Project Plans, Parts 1–3, EPA/505/B-04/900A through 900C (USEPA 2005)

The UFP-QAPP includes sampling and laboratory QA/QC programs. The sampling QA/QC program combines USEPA-approved sampling, preservation, and field monitoring techniques with the necessary documentation and chain-of-custody requirements. The sampling QA/QC program also specifies the field QA/QC samples that will be collected. The laboratory QA/QC program provides a systematic set of standards of operation and surveillance procedures. Important elements of the QA/QC program for the PDI and TS elements discussed in Sections 3 and 4 of this PDIWP are discussed in the project UFP-QAPP (Appendix A).

QC procedures will be followed to monitor the analytical process through each phase of the project from sample collection through laboratory analysis and reporting. QC sampling will be incorporated into the scope of work for the field sampling program and laboratory analysis program. Field QC samples used to assess data usability will consist of blind duplicate soil, groundwater, and sediment samples and matrix spike/matrix spike duplicate samples. The QC samples will be collected from the sample location at frequencies specified in the UFP-QAPP and will be recorded in the field book.

If additional PDIs are needed to complete the RD, the UFP-QAPP will be amended as needed to incorporate the associated means and methods and the QA/QC elements for these additional PDI scopes of work that are not currently described in the UFP-QAPP. Amended worksheets will be provided as an appendix to any subsequently developed work plans (e.g., addenda to PDIWP) and RD/RA plans as necessary.

8. PREDESIGN INVESTIGATION REPORT AND SCHEDULE

Once the PDI field investigations and TS bench-scale studies are completed, a PDI evaluation report will be prepared for submittal to USEPA. If any additional investigations are required based on the PDI findings, P/S will submit an addendum to the PDIWP and the UFP-QAPP (if necessary).

8.1 Reporting

A PDI evaluation report will be prepared in accordance with Section 3.3(b) of the SOW. The report will include the following:

- Summary of the PDI field investigations and TS bench-scale studies performed, including deviations from the PDIWP
- Summary of the results from the PDI field investigation and TS bench-scale studies
- Tables and figures presenting validated laboratory analytical results as appropriate for individual PDI field investigations and TS bench-scale studies
- Narrative interpretation of soil, groundwater, and sediment data and results
- Results of statistical and modeling analyses (if generated)
- Sampling logs, surveying reports photographs, and the laboratory analytical reports, included as appendices (or attachments)
- Conclusions and recommendations for the Preliminary (30%) RD and the identification of any additional PDIs

The data validation approach is consistent with the applicable USEPA guidelines for data verification and data validation (USEPA 2002b). Data validation will include 95% Stage 2A and 5% Stage 4 data validation per analytical test and matrix. Level IV laboratory reports will be requested for all sample delivery groups. Following validation, the data will be entered into the project database.

8.2 Schedule

To advance the schedule of PDI field work, P/S are advancing the property access process in parallel with USEPA's review of the PDIWP. If additional property access is necessary based on USEPA's review of the PDIWP, these properties will be added to Table 5-1, and the access process will be initiated for these additional properties. Field mobilization for PDI efforts following USEPA approval of the PDIWP is anticipated in the summer and fall of 2022. The field investigation and TS work efforts are expected to take 5–6 months to complete, with draft analytical chemistry results anticipated to be available in late spring 2023. The validated analytical chemistry results are anticipated to be available in late summer of 2023, and the PDI evaluation report will be submitted to USEPA within 60 calendar days of completing validation of all data.

Several interim working deliverables will also be submitted to support consultation with USEPA regarding decisions to advance the PDI program. These interim deliverables will take various forms and will generally include a brief memorandum summarizing the investigation results along with supporting tables, figures, and photographs. For those interim deliverables where analytical chemistry data are being provided, the data will be summarized in tabular form with the validated data also being provided to USEPA electronically. A list of anticipated interim working deliverables is provided below recognizing that additional deliverables may be identified as the PDI work is conducted and data are validated:

- Land use survey results for the low-activity special use properties identified in the OU-1/OU-2 ROD. These properties will be surveyed to confirm land use and could include three potential outcomes: low-activity special use, high-activity special use, or nonresidential. The surveys will be conducted in a manner such that the results could apply in part or in whole to the properties and will identify areas that are not suitable for removal. The survey results will be accompanied by a list of properties proposed for subsurface soil sampling and suitable documentation to remove properties (or portions of properties) that no longer fit the definition of low-activity special use. Following consultation with USEPA, the subsurface soil sampling will be scheduled and conducted.
- Soil and groundwater results for the Eastside Properties area will be provided to USEPA for consultation relative to the potential for additional characterization work for the Eastside Properties.
- Vertical soil PCB concentration profiling for the PB-RR-37 area will be provided to USEPA for consultation relative to the potential for installing a groundwater well at this location.
- The results of the Phase 1 sediment probing results and proposed Phase 2 sediment sampling plan will be submitted to USEPA for consultation prior to implementing the Phase 2 sediment sampling.

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TABLES

Table 1-1. SOW Requirements for the PDIWP
OU-1/OU-2 Predesign Investigation Work Plan
Anniston PCB Site, Anniston, Alabama

SOW Section 3.3(a)	Description	Location in PDIWP
(1)	An evaluation and summary of existing data and description of data gaps	Section 2
(2)	A field sampling plan (FSP) as described in SOW Section 7.7 including the media to be sampled, contaminants or parameters for which sampling will be conducted, location (aerial extent and depths), and number of samples	Sections 3 and 4
(3)	Cross references to quality assurance/quality control (QA/QC) requirements set forth in the Quality Assurance Project Plan (QAPP) as described in SOW Section 6.7(d). This may also include updates to the QAPP to reflect the work proposed in the PDIWP.	Sections 3, 4, and 7 Appendices A, B, and C

Notes:
FSP: field sampling plan
PDIWP: predesign investigation work plan
QAPP: quality assurance project plan
SOW: statement of work

Table 2-1. Summary of OU-1/OU-2 Nonresidential Surface Soil PCB Data by Exposure Unit
OU-1/OU-2 Predesign Investigation Work Plan
Anniston PCB Site, Anniston, Alabama

EU	Area (acres)	Locations	Maximum Discrete Detection (mg/kg)	Mean Surface Detection (mg/kg)	Surface UCL (mg/kg)	Nonresidential Area Of EU (%)	Weighted EU Surface Mean (mg/kg)
1	60.3	21	20	3.6	8.4	12	1.3
2	32.0	55	12	1.8	2.2	29	1.2
3	18.7	37	30	4.9	18	39	2.5
4	19.5	53	2.6	0.76	0.78	47	0.89
5	20.3	92	3,700	100	350	89	89
6	25.2	40	43	2.2	14	39	1.5
7	16.0	23	310	25	160	42	11
8	17.7	25	2.5	0.51	0.66	26	0.87
9	25.9	28	4.9	0.65	0.90	84	0.71
10	15.4	92	130	8.5	19	94	8.1
11	15.3	25	9.1	1.3	2.8	96	1.3
12	8.0	10	11	4.3	9.6	55	2.8
13	19.7	13	17	3.0	9.5	9.7	1.2
14 North	7.0	14	33	12	21	42	5.6
14 South	6.6	11	1.7	0.58	0.56	19	0.92
15/16	46.4	44	4.7	0.84	1.9	100	0.84
17	23.4	25	12	2.4	4.7	92	2.3
18	30.3	23	1.1	0.30	0.33	90	0.37
19 North	5.0	13	770	77	660	100	77
19 South	4.7	13	59	13	68	100	13
20	8.7	7	5.0	1.2	4.0	31	1.1
21	21.9	20	2.7	0.46	0.51	100	0.46
22	26.5	25	15	1.5	7.3	100	1.5
23	19.7	20	1.2	0.37	0.41	100	0.37
24	30.0	36	38	6.8	12	100	6.8
25	22.2	17	1.4	0.27	0.56	86	0.37
26	18.8	23	99	12	34	31	4.4
27	25.9	20	2.0	0.46	0.45	100	0.46
28	54.6	0	--	--	--	100	--
29	37.0	21	0.83	0.20	0.19	100	0.20
30	40.6	10	0.039	0.039	SS	1.8	0.98
ES	57.0	146	260	9.1	19	80	7.5
NS	32.7	31	160	15	72	80	12
All	**813.0**	**1,033**	**3,700**	**15**	**38**	**69**	**12**
Excluding EU5	792.8	941	770	6.2	12	68	5.0
Exlcuding EU5, NS, ES	703.1	764	770	5.3	13	66	4.3

Notes:

ProUCL 4.1 was utilized for statistical summaries and UCL calculation for EUs with sufficient sample population. Microsoft Access 2010 was utilized for base statistical summaries.

Parent and duplicate samples were combined. If both detect or both nondetect, a mean was taken. If one was detected and other was nondetect, then the detected value was utilzed to represent the pair.

Results for maximum, mean, and UCL were rounded to two significant figures.

--: not sampled

APCO: Alabama Power Company
ES: eastside
EU: exposure unit
mg/kg: milligrams per kilogram
NC: not calculated; due to low sample count and/or low number of detected concentrations.
NS: northside
UCL: upper confidence limit

Table 2-2. OU-1/OU-2 Nonresidential Subsurface Soil PCB Data by Exposure Unit
OU-1/OU-2 Predesign Investigation Work Plan
Anniston PCB Site, Anniston, Alabama

Dataset	EU	Parcel ID	Location ID	Northing	Easting	Mean PCB (mg/kg)	Maximum PCB (mg/kg)
Miscellaneous Programs	1	3224	CWM-1,2,5	1,143,889.2	649,402.3	0.72	0.72
Miscellaneous Programs	1	3224	CWM-3,4,6	1,143,893.5	649,679.4	0.15	0.15
Miscellaneous Programs	10	1790	ABS-1	1,148,535.6	653,005.0	0.18	0.28
Miscellaneous Programs	10	1790	ABS-10	1,148,677.1	653,115.1	0.57	1.0
Miscellaneous Programs	10	1790	ABS-11	1,148,654.8	653,167.9	0.40	0.46
Miscellaneous Programs	10	1790	ABS-12	1,148,701.1	653,181.3	0.20	0.24
Miscellaneous Programs	10	1790	ABS-15	1,148,721.8	653,332.6	68	130
Miscellaneous Programs	10	1790	ABS-17	1,148,729.6	653,398.8	9.1	15
Miscellaneous Programs	10	1790	ABS-18	1,148,672.8	653,408.6	21	32
Miscellaneous Programs	10	1790	ABS-19	1,148,677.6	653,358.4	8.2	16
Miscellaneous Programs	10	1790	ABS-20	1,148,663.4	653,311.2	11	19
Miscellaneous Programs	10	1790	ABS-21	1,148,637.2	653,294.1	1.9	3.2
Miscellaneous Programs	10	1790	ABS-22	1,148,606.2	653,285.3	0.79	1.5
Miscellaneous Programs	10	1790	ABS-23	1,148,595.4	653,333.5	0.090	0.096
Miscellaneous Programs	10	1790	ABS-24	1,148,626.8	653,359.1	2.9	5.8
Miscellaneous Programs	10	1790	ABS-25	1,148,625.1	653,400.5	1.5	2.9
Miscellaneous Programs	10	1790	ABS-26	1,148,539.4	653,416.7	0.51	0.93
Miscellaneous Programs	10	1790	ABS-27	1,148,773.3	653,410.0	39	78
Miscellaneous Programs	10	1790	ABS-29	1,148,762.7	653,330.1	9.9	13
Miscellaneous Programs	10	1790	ABS-30	1,148,774.0	653,329.3	17	27
Miscellaneous Programs	10	1790	ABS-34	1,148,798.4	653,404.6	16	16
Miscellaneous Programs	10	1790	ABS-36	1,148,760.6	653,277.2	11	11
Miscellaneous Programs	10	1790	ABS-5	1,148,597.8	653,170.5	0.078	0.10
Miscellaneous Programs	10	1790	ABS-6	1,148,617.3	653,073.0	0.17	0.25
Miscellaneous Programs	10	1790	ABS-7	1,148,603.8	653,008.5	0.40	0.72
Miscellaneous Programs	10	1790	ABS-8	1,148,652.9	653,005.4	0.54	1.0
Miscellaneous Programs	10	1790	ABS-9	1,148,671.4	653,061.6	0.95	1.8
Miscellaneous Programs	10	1800	CA-1	1,148,680.0	653,650.4	0.41	0.67
Miscellaneous Programs	10	1800	CA-19	1,148,828.9	653,665.6	16	26
Miscellaneous Programs	10	1800	CA-2	1,148,673.8	653,674.5	0.16	0.32
Miscellaneous Programs	10	1800	CA-9	1,148,794.7	653,662.2	8.9	22
Nonresidential OU-1/OU-2 FSP	10	1807	CA-10-1807-02	1,148,789.3	653,687.1	3.0	6.3
Miscellaneous Programs	10	1807	CA-12	1,148,777.6	653,733.7	2.6	5.7
Miscellaneous Programs	10	1807	CA-13	1,148,770.4	653,757.8	0.80	0.88
Miscellaneous Programs	10	1807	CA-24	1,148,623.0	653,757.2	0.58	1.2
Miscellaneous Programs	10	1807	CA-25	1,148,541.3	653,762.0	0.18	0.33
Miscellaneous Programs	10	1807	CA-27	1,148,758.9	653,686.1	0.77	1.8
Miscellaneous Programs	10	1807	CA-28	1,148,752.6	653,710.2	0.14	0.27
Miscellaneous Programs	10	1807	CA-29	1,148,746.0	653,734.4	0.16	0.24
Miscellaneous Programs	10	1807	CA-30	1,148,739.5	653,758.4	0.091	0.15
Nonresidential OU-1/OU-2 FSP	10	2049	CA-10-2049-11	1,148,361.8	654,359.7	64	190
Nonresidential OU-1/OU-2 FSP	10	2049	CA-10-2049-12	1,148,378.0	654,437.6	150	560
Nonresidential OU-1/OU-2 FSP	10	2049	CA-10-2049-13	1,148,183.7	654,537.6	43	72
USEPA Programs	10	2049	PB-024-60	1,148,368.6	653,945.4	0.23	0.23
Nonresidential OU-1/OU-2 FSP	12	2737	CA-12-2737-05	1,146,546.0	655,845.9	4.9	13
Nonresidential OU-1/OU-2 FSP	12	2737	CA-12-2737-06	1,146,453.0	655,880.0	12	25
Nonresidential OU-1/OU-2 FSP	12	2819	CA-12-2819-07	1,146,698.6	655,483.5	7.3	19
Nonresidential OU-1/OU-2 FSP	13	2850	CA-13-2850-02	1,146,775.7	654,975.2	2.7	5.5

Table 2-2. OU-1/OU-2 Nonresidential Subsurface Soil PCB Data by Exposure Unit
OU-1/OU-2 Predesign Investigation Work Plan
Anniston PCB Site, Anniston, Alabama

Dataset	EU	Parcel ID	Location ID	Northing	Easting	Mean PCB (mg/kg)	Maximum PCB (mg/kg)
Nonresidential OU-1/OU-2 FSP	13	2900	CA-13-2900-09	1,146,714.6	655,168.8	4.3	8.1
Nonresidential OU-1/OU-2 FSP	13	2900	CA-13-2900-10	1,146,685.5	655,212.5	12	23
Nonresidential OU-1/OU-2 FSP	14 North	3053	CA-14-3053-02	1,146,336.9	655,759.3	14	28
Nonresidential OU-1/OU-2 FSP	14 North	3053	CA-14-3053-03	1,146,362.0	655,806.1	16	46
Nonresidential OU-1/OU-2 FSP	14 North	3053	CA-14-3053-05	1,146,283.2	655,918.4	4.2	8.4
Nonresidential OU-1/OU-2 FSP	14 North	3053	CA-14-3053-06	1,146,199.9	656,047.2	5.7	11
Nonresidential OU-1/OU-2 FSP	14 North	3189	CA-14-3189-10	1,145,931.0	655,999.0	18	45
Nonresidential OU-1/OU-2 FSP	17	3536	CA-17-3536-03	1,145,418.7	657,091.8	2.7	10
Nonresidential OU-1/OU-2 FSP	17	4011	CA-17-4011-13	1,144,649.3	656,974.0	11	24
Nonresidential OU-1/OU-2 FSP	19 North	4559	CA-19-4559-01	1,143,628.9	657,326.9	4.8	8.5
Nonresidential OU-1/OU-2 FSP	19 North	4559	CA-19-4559-90	1,143,142.3	657,388.8	4.2	8.9
Nonresidential OU-1/OU-2 FSP	19 North	4559	CA-19-4559-92	1,143,564.0	657,356.6	11	31
USEPA Programs	19 North	4559	PB-010-60	1,143,066.6	657,297.1	0.26	0.26
Nonresidential OU-1/OU-2 FSP	19 North	8560	CA-19-8560-15	1,143,300.9	657,209.9	510	1500
Nonresidential OU-1/OU-2 FSP	19 North	8560	CA-19-8560-16	1,143,111.7	657,234.6	96	200
Nonresidential OU-1/OU-2 FSP	19 North	8560	CA-19-8560-91	1,143,514.6	657,167.6	9.1	27
Nonresidential OU-1/OU-2 FSP	19 South	5347	CA-19-5347-07	1,141,910.4	657,468.2	44	89
Nonresidential OU-1/OU-2 FSP	20	5368	CA-20-5368-10	1,141,955.4	657,152.7	4.7	13
USEPA Programs	22	5621	PB-021-60	1,140,587.4	657,386.5	0.034	0.034
USEPA Programs	22	5887	PB-003-60	1,139,030.4	657,293.1	0.20	0.20
Nonresidential OU-1/OU-2 FSP	24	6004	CA-24-6004-04	1,137,924.1	657,452.8	4.3	13
Nonresidential OU-1/OU-2 FSP	24	6098	CA-24-6098-14	1,137,706.3	657,477.9	16	38
Nonresidential OU-1/OU-2 FSP	24	6098	CA-24-6098-15	1,137,272.5	657,489.6	26	61
Nonresidential OU-1/OU-2 FSP	24	6098	CA-24-6098-90	1,137,318.7	657,564.2	13	28
USEPA Programs	24	6098	PB-004-60	1,137,614.4	657,680.9	0.63	0.63
Nonresidential OU-1/OU-2 FSP	24	6282	CA-24-6282-30	1,136,517.8	657,320.0	6.8	25
Nonresidential OU-1/OU-2 FSP	24	6350	CA-24-6350-35	1,135,958.5	657,420.5	10	22
Nonresidential OU-1/OU-2 FSP	26	6440	CA-26-6440-02	1,135,565.1	657,177.4	13	24
Nonresidential OU-1/OU-2 FSP	26	6629	CA-26-6629-90	1,134,987.1	657,606.2	14	31
Nonresidential OU-1/OU-2 FSP	26	6629	CA-26-6629-92	1,135,069.2	657,516.3	46	150
Nonresidential OU-1/OU-2 FSP	26	6629	CA-26-9999-91	1,135,039.7	657,588.4	36	150
Nonresidential OU-1/OU-2 FSP	26	6629	CA-26-9999-93	1,134,981.2	657,657.6	11	19
Nonresidential OU-1/OU-2 FSP	3	1806	CA-03-1806-18	1,148,796.0	651,417.9	18	45
Miscellaneous Programs	4	1353	HA-049	1,149,554.1	650,111.7	0.50	0.50
Miscellaneous Programs	4	1353	HA-050	1,149,598.6	650,112.0	0.50	0.50
Miscellaneous Programs	4	1353	HA-051	1,149,599.0	650,148.7	0.50	0.50
Miscellaneous Programs	4	1353	HA-052	1,149,643.5	650,148.6	0.50	0.50
Miscellaneous Programs	4	1353	HA-053	1,149,599.5	650,186.6	0.50	0.50
Miscellaneous Programs	5	1671	ASL-186	1,148,764.6	652,868.8	55	55
Miscellaneous Programs	5	1671	ASL-219	1,148,976.4	652,595.7	0.092	0.13
Miscellaneous Programs	5	1671	ASL-223	1,148,924.9	652,648.6	0.70	0.70
Miscellaneous Programs	5	1671	ASL-234	1,148,820.8	652,907.5	0.22	0.22
Miscellaneous Programs	5	1671	ASL-238	1,148,817.7	652,760.4	0.085	0.085
Miscellaneous Programs	5	1671	ASL-241	1,148,710.7	652,529.6	0.28	0.28
Miscellaneous Programs	5	1671	ASL-243	1,148,811.9	652,589.1	0.16	0.18
Miscellaneous Programs	5	1671	TH-1	1,148,772.1	652,956.0	52	52
Miscellaneous Programs	5	1671	TH-3	1,148,851.6	652,866.4	130	250
Nonresidential OU-1/OU-2 FSP	5	1782	CA-05-1782-03	1,148,538.9	651,412.0	20	37

Table 2-2. OU-1/OU-2 Nonresidential Subsurface Soil PCB Data by Exposure Unit
OU-1/OU-2 Predesign Investigation Work Plan
Anniston PCB Site, Anniston, Alabama

Dataset	EU	Parcel ID	Location ID	Northing	Easting	Mean PCB (mg/kg)	Maximum PCB (mg/kg)
Nonresidential OU-1/OU-2 FSP	5	1782	CA-05-1782-04	1,148,610.0	651,447.0	43	76
Nonresidential OU-1/OU-2 FSP	5	1782	CA-05-1782-05	1,148,554.9	651,562.1	170	610
Nonresidential OU-1/OU-2 FSP	5	1782	CA-05-1782-06	1,148,699.1	651,644.9	14	30
Nonresidential OU-1/OU-2 FSP	5	2006	CA-05-2006-12	1,148,136.9	650,478.1	2.7	5.4
Miscellaneous Programs	5	8583	ASL-193	1,149,021.4	652,828.4	62	65
Miscellaneous Programs	5	8583	ASL-195	1,148,955.7	652,900.4	72	110
Miscellaneous Programs	5	8583	ASL-197	1,148,894.2	652,982.7	58	88
Miscellaneous Programs	5	8583	ASL-203	1,148,942.2	653,008.7	6.6	6.6
Miscellaneous Programs	5	8583	ASL-207	1,148,945.7	653,069.4	100	110
Miscellaneous Programs	5	8583	ASL-215	1,148,924.1	653,325.4	0.21	0.32
Miscellaneous Programs	5	8583	ASL-225	1,148,952.6	652,816.1	1.3	1.3
Miscellaneous Programs	5	8583	ASL-232	1,148,909.5	652,861.4	2.5	4.8
Miscellaneous Programs	5	8583	ASL-236	1,148,827.3	652,962.3	5.6	5.6
Miscellaneous Programs	5	8583	ASL-237	1,148,787.8	653,022.1	35	62
Nonresidential OU-1/OU-2 FSP	5	8583	CA-05-8583-13	1,149,006.0	652,761.8	28	81
Nonresidential OU-1/OU-2 FSP	5	8583	CA-05-8583-14	1,148,972.0	652,940.1	150	360
Nonresidential Other	5	8583	T-11	1,148,974.6	652,936.9	1.5	3.0
Nonresidential Other	5	8583	T-11 E10	1,148,974.6	652,946.9	150	340
Nonresidential Other	5	8583	T-11 N10	1,148,984.6	652,936.9	210	500
Nonresidential Other	5	8583	T-11 S10	1,148,964.6	652,936.9	150	430
Nonresidential Other	5	8583	T-11 W10	1,148,974.6	652,926.9	560	1500
Nonresidential OU-1/OU-2 FSP	7	1520	CA-07-1520-08	1,149,169.9	652,536.0	7.2	15
Nonresidential OU-1/OU-2 FSP	9	1597	CA-09-1597-07	1,149,027.0	654,920.0	3.4	5.9
USEPA Programs	9	1720	PB-019-60	1,148,588.3	654,755.1	1.9	1.9
Eastside Properties	ES	2843	MH-03	1,146,620.4	651,048.8	0.54	0.81
Miscellaneous Programs	ES	3467	SWSL-03	1,147,567.6	650,590.0	0.40	0.99
Miscellaneous Programs	ES	3467	SWSL-10	1,147,567.8	650,467.9	0.70	1.4
Miscellaneous Programs	ES	3467	SWSL-28	1,147,666.1	650,608.3	0.83	2.3
Miscellaneous Programs	ES	3467	SWSL-35	1,147,746.6	650,607.9	0.45	1.2
Northside Properties	NS	2367	NSP-A	1,147,862.7	648,773.2	0.79	0.79
Northside Properties	NS	2367	NSP-E	1,147,087.6	648,361.0	0.55	0.55

Notes:
APCO: Alabama Power Company
ES: eastside
EU: exposure unit
FSP: field sampling program
ID: identification
mg/kg: milligrams per kilogram
ND: nondetect
NS: northside
PCB: polychlorinated biphenyl
USEPA: United States Environmental Protection Agency

Table 3-1. OU-1/OU-2 Predesign Investigation Scope of Work for Floodplain and Creek Bank Soils
OU-1/OU-2 Predesign Investigation Work Plan
Anniston PCB Site, Anniston, Alabama

Location Description		Figure Reference	Area (acres)	Sampling Method	Number of Samples Per Location[1,2]	QA/QC - Duplicate Samples (1/20)	QC/QC - MS/MSD Pairs (1/20)	Total Estimated No. of Samples	Analyses							
									PCBs Aroclors (USEPA SW-846 8082)	PCBs Homologs (USEPA SW-846 680)	Natural Moisture Content (ASTM D2216)	Particle-Size Analysis (ASTM D6913 and ASTM D7928)	Atterberg Limits (ASTM D4318)	Engineering Classification (ASTM D2487)	Unconsolidated Undrained Triaxial Compression Test (ASTM D2850)	Consolidated Undrained Triaxial Compression Test (ASTM D4767)
Dredge Spoil Pile (SC-2)		Figure 3-1	0.03	5-Point Composite	1	1	1	3	◆	-	-	-	-	-	-	-
Low-Activity Special Use Areas (PPIN/Previous Sample Identification)	16384 / 16384-BY	Figure 3-2l	0.09	5-Point Composite	3 (See Note 1)	6	6	132	◆	-	-	-	-	-	-	-
	17523 / 17523-G1	Figure 3-2g	0.12	5-Point Composite	3 (See Note 1)				◆	-	-	-	-	-	-	-
	17523 / 17523-G3		0.13	5-Point Composite	3 (See Note 1)				◆	-	-	-	-	-	-	-
	17523 / 17523-G4		0.12	5-Point Composite	3 (See Note 1)				◆	-	-	-	-	-	-	-
	17523 / 17523-G5		0.14	5-Point Composite	3 (See Note 1)				◆	-	-	-	-	-	-	-
	17523 / 17523-G6		0.08	5-Point Composite	3 (See Note 1)				◆	-	-	-	-	-	-	-
	18084 / 18084-G1	Figure 3-2g	0.08	5-Point Composite	3 (See Note 1)				◆	-	-	-	-	-	-	-
	18084 / 18084-G2		0.06	5-Point Composite	3 (See Note 1)				◆	-	-	-	-	-	-	-
	18084 / 18084-G3		0.07	5-Point Composite	3 (See Note 1)				◆	-	-	-	-	-	-	-
	18084 / 18084-G5		0.09	5-Point Composite	3 (See Note 1)				◆	-	-	-	-	-	-	-
	18311 / CA-14-3189-09, CA-14-3189-12, CA-14-SU-3368-G, CA-14-SU-3368-I	Figure 3-2j	0.39	5-Point Composite	6 (See Note 1)				◆	-	-	-	-	-	-	-
	18536 / CA-06-2205-11	Figure 3-2h	0.23	5-Point Composite	3 (See Note 1)				◆	-	-	-	-	-	-	-
	18536 / CA-6-SU-2205-L		0.15	5-Point Composite	3 (See Note 1)				◆	-	-	-	-	-	-	-
	18536 / CA-6-SU-2205-D		0.18	5-Point Composite	3 (See Note 1)				◆	-	-	-	-	-	-	-
	18536 / CA-06-2205-12		0.21	5-Point Composite	3 (See Note 1)				◆	-	-	-	-	-	-	-
	18591 / 18591-G5	Figure 3-2j	0.07	5-Point Composite	3 (See Note 1)				◆	-	-	-	-	-	-	-
	18591 / 18591-G4		0.08	5-Point Composite	3 (See Note 1)				◆	-	-	-	-	-	-	-
	18591 / 18591-G3		0.07	5-Point Composite	3 (See Note 1)				◆	-	-	-	-	-	-	-
	18671 / CA-6-SU-2498-D	Figure 3-2h	0.11	5-Point Composite	3 (See Note 1)				◆	-	-	-	-	-	-	-
	19313 /PA-118-A	Figure 3-2f	0.36	5-Point Composite	3 (See Note 1)				◆	-	-	-	-	-	-	-
	19406, 19407, 19408, Brown Ave / CA-08-1004-01	Figure 3-2c	0.08	5-Point Composite	3 (See Note 1)				◆	-	-	-	-	-	-	-
	20579 / 20579-G10	Figure 3-2d	0.16	5-Point Composite	3 (See Note 1)				◆	-	-	-	-	-	-	-
	20717 / 20717-G48	Figure 3-2d	0.1	5-Point Composite	3 (See Note 1)				◆	-	-	-	-	-	-	-
	21033 / 21033-G11	Figure 3-2b	0.15	5-Point Composite	3 (See Note 1)				◆	-	-	-	-	-	-	-
	21033 / 21033-G24		0.27	5-Point Composite	3 (See Note 1)				◆	-	-	-	-	-	-	-
	21033 / 21033-G25		0.26	5-Point Composite	3 (See Note 1)				◆	-	-	-	-	-	-	-
	21033 / 21033-G26		0.23	5-Point Composite	3 (See Note 1)				◆	-	-	-	-	-	-	-
	21033 / 21033-G27		0.26	5-Point Composite	3 (See Note 1)				◆	-	-	-	-	-	-	-
	21033 / 21033-G29		0.15	5-Point Composite	3 (See Note 1)				◆	-	-	-	-	-	-	-
	21033 / 21033-G30		0.13	5-Point Composite	3 (See Note 1)				◆	-	-	-	-	-	-	-
	30182 / 30182-G1	Figure 3-2a	0.03	5-Point Composite	3 (See Note 1)				◆	-	-	-	-	-	-	-
	30865 / FC4-0029-E	Figure 3-2e	0.10	5-Point Composite	3 (See Note 1)				◆	-	-	-	-	-	-	-
	32123 /CA-3-SU-1700-LA-B	Figure 3-2e	0.19	5-Point Composite	3 (See Note 1)				◆	-	-	-	-	-	-	-
	32375 / CA-06-2113-02 and CA-6-SU-2113-LA-A	Figure 3-2h	0.08	5-Point Composite	3 (See Note 1)				◆	-	-	-	-	-	-	-
	32487 / CA-1-3503-LA-D	Figure 3-2i	0.10	5-Point Composite	3 (See Note 1)				◆	-	-	-	-	-	-	-
	32490 / CA-1-SU-3402-B	Figure 3-2i	0.09	5-Point Composite	3 (See Note 1)				◆	-	-	-	-	-	-	-
	32612 / 4005-LA-A	Figure 3-2k	0.16	5-Point Composite	3 (See Note 1)				◆	-	-	-	-	-	-	-
T-11 Area		Figure 3-6	--	Geotechnical	TBD (See Note 2)	Not Applicable	Not Applicable	TBD (See Note 2)	-	-	◆	◆	◆	◆	◆	◆
PB-RR-37		Figure 3-5	--	Discrete	TBD (See Note 3)	TBD (See Note 3)	TBD (See Note 3)	TBD (See Note 3)	◆	◆	-	-	-	-	-	-
			0.05	Composite	4 (See Note 1)	1	1	7	◆	◆	-	-	-	-	-	-
Drainage Ditch downstream of PB-RR-37		Figure 3-5	--	Discrete	2 (from 0 to 1 foot bgs)	1	1	5	◆	-	-	-	-	-	-	-
			0.19	Composite	4 (See Note 1)	1	1	7	◆	-	-	-	-	-	-	-
Drainage Ditch upstream of PB-RR-37			0.11	Composite	4 (See Note 1)	1	1	7	◆	-	-	-	-	-	-	-
IM Areas	PB-ASP-08	Figure 3-3a	0.002	Composite	3 (See Note 1)	1	1	24	◆	-	-	-	-	-	-	-
	Brown Auto Parts (ESL093)	Figure 3-3a	0.006	Composite	3 (See Note 1)				◆	-	-	-	-	-	-	-
	WEL042	Figure 3-3b	0.035	Composite	3 (See Note 1)				◆	-	-	-	-	-	-	-
	GASCOCL07	Figure 3-3b	0.009	Composite	3 (See Note 1)				◆	-	-	-	-	-	-	-
	SED1430R225	Figure 3-3c	0.03	Composite	3 (See Note 1)				◆	-	-	-	-	-	-	-
	SED0010R25 and SED0010R50	Figure 3-3d	0.01	Composite	3 (See Note 1)				◆	-	-	-	-	-	-	-
	Drainage Ditch near Miller Property	Figure 3-3e	0.1	Composite	3 (See Note 1)				◆	-	-	-	-	-	-	-
Nonresidential Areas	Exposure Unit 5 (area sourth of Snow Creek)	Figure 3-4a	0.17	Composite	3 (See Note 1)	2	2	36	◆	-	-	-	-	-	-	-
	Exposure Unit 14 North	Figure 3-4b	0.33	Composite	6 (See Note 1)				◆	-	-	-	-	-	-	-
	Exposure Unit 19 North	Figure 3-4c	0.15	Composite	3 (See Note 1)				◆	-	-	-	-	-	-	-
	Exposure Unit 24	Figure 3-4e	0.26	Composite	6 (See Note 1)				◆	-	-	-	-	-	-	-
	Exposure Unit 26	Figure 3-4f	0.19	Composite	3 (See Note 1)				◆	-	-	-	-	-	-	-
	Highway 202 Area (south)	Figure 3-4g	0.49	Composite	6 (See Note 1)				◆	-	-	-	-	-	-	-
	North of APCO	Figure 3-4h	0.05	Composite	3 (See Note 1)				◆	-	-	-	-	-	-	-

Table 3-1. OU-1/OU-2 Predesign Investigation Scope of Work for Floodplain and Creek Bank Soils
OU-1/OU-2 Predesign Investigation Work Plan
Anniston PCB Site, Anniston, Alabama

Location Description		Figure Reference	Area (acres)	Sampling Method	Number of Decision Units Per Location	Number of Samples per Decision Unit	ISM Field Replicates per Location[5]	Total Estimated No. of Samples and Field Repliates	PCBs Aroclors (USEPA SW-846 8082)	PCBs Homologs (USEPA SW-846 680)	Natural Moisture Content (ASTM D2216)	Particle-Size Analysis (ASTM D6913 and ASTM D7928)	Atterberg Limits (ASTM D4318)	Engineering Classification (ASTM D2487)	Unconsolidated Undrained Triaxial Compression Test (ASTM D2850)	Consolidated Undrained Triaxial Compression Test (ASTM D4767)
Nonresidential Areas[4]	Exposure Unit 5 (area west of T-11 Area)	Figure 3-4a	0.79	ISM	3 (See Note 4)	1	Y (All DUs; See Note 5)	9	◆	-	-	-	-	-	-	-
	Exposure Unit 19 South	Figure 3-4d	0.61	ISM	3 (See Note 4)	1	--	3	◆	-	-	-	-	-	-	-
	Highway 202 Area (north)	Figure 3-4g	1.32	ISM	3 (See Note 4)	1	--	3	◆	-	-	-	-	-	-	-

Notes:
1. Low-activity special use areas, IM areas, nonresidential areas, and the drainage ditch near PB-RR-37 that are less then 0.5 acres in size will be sampled as one 5-point composite per 0.25 acre area. Each 5-point composites will be collected at three sample depths from low-activity special use areas, IM areas, and nonresidential areas: 1 to 2 feet bgs, 2 to 3 feet bgs, and 3 to 4 feet bgs. Samples from 2 to 3 feet bgs and 3 to 4 feet bgs will be archived for analysis pending results of the shallower depth horizon sample. 5-point composites will be collected from four sample depths for the PB-RR-37 area and the drainage ditch downstream of PB-RR-37: 0 to 1 foot bgs, 1 to 2 feet bgs, 2 to 3 feet bgs, and 3 to 4 feet bgs. Samples from 1 to 2 feet bgs, 2 to 3 feet bgs, and 3 to 4 feets bgs will be archived pending analysis of the shallower depth horizon.

2. Four geotechnical borings will be advanced in the T-11 area. Split-spoon samples will be collected continuously to boring termination at approximately 40 feet bgs or refusal whichever is encountered first. Split-spoon samples from each boring will be collected from the geotechnical borings for the following geotechnical laboratory analyses: Natural Moisture Content (ASTM D2216), Particle-Size Analysis (ASTM D6913 and ASTM D7928), Atterberg Limits (ASTM D4318), and Engineering Classification (ASTM D2487).
One thin-walled Shelby-tube sample from each of the borings will be collected from cohesive soil horizons encountered in the borings. Representative Shelby-tube samples will be submitted for the following geotechnical laboratory analyses: Unconsolidated Undrained Triaxial Compression Test (ASTM D2850) and Consolidated Undrained Triaxial Compression Test (ASTM D4767).
3. The PB-RR-37 area will be sampled continuously until soil-groundwater interface is achieved based on observations made during boring advancement.
4. Nonresidential areas greater than 0.5 acres will be sampled using ISM as three DUs based on sample depth: 1 to 2 feet bgs, 2 to 3 feet bgs, and 3 to 4 feet bgs. The incremental sample for each DU will consist of 30 increments collected and composited. Samples from 2 to 3 feet bgs and 3 to 4 feet bgs will be archived for analysis pending results of the shallower depth horizon sample.
5. ISM field triplicates will be collected at a rate of 10% of number of DUs.
APCO: Alabama Power Company
ASTM: ASTM International
bgs: below ground surface
DU: decision unit
IM: interim measure
ISM: incremental sampling methodology
MS/MSD: matrix spike/matrix spike duplicate
PCB: polychlorinated biphenyl
PPIN: property identification numbers
QA/QC: quality assurance/quality control
TBD: to be determined
USEPA: United States Environmental Protection Agency

Table 3-2. Snow Creek Sediment Characterization Areas
OU-1/OU-2 Predesign Work Plan
Anniston PCB Site, Anniston, Alabama

Area	General Description
1	From the 11th Street Ditch to West 9th Street Bridge
2	From the West 9th Street Bridge to the Railroad Bridge at the southwest corner of Calhoun County Jail
3	From the southwest corner of Calhoun County Jail to the end of the concrete spillway
4	From the end of the concrete spillway to downstream of the Sandy Lumber Company property
5A[1]	From downstream of the Sandy Lumber Company property to upstream of Quintard Mall culvert
5B[1]	Quintard Mall culvert
5C[1]	From downstream of the Quintard Mall culvert to Highway 78

1. Area 5 was segmented into three separate subreaches to avoid confusion with Area 6, which was characterized as part of the 1999 Resource Conservation and Recovery Act Facility Investigation and is located in OU-4 downstream of Highway 78.

Table 3-3. Summary of Known Structures along Snow Creek
OU-1/OU-2 Predesign Investigation Work Plan
Anniston PCB Site, Anniston, Alabama

Transect	Location (nearest street)	Structure Type
T0-00	West 11th Street	Bridge
T0-50	West 11th Street	Railroad Bridge
T19-50	West 10th Street	Bridge
T25-00	West 9th Street	Bridge (limited use)
T31-00	Highway 202	Box Culvert
T35-50	West 7th Street	Bridge
T42-50	Glenaddie Avenue	Bridge
T51-00	Calhoun County Jail	Railroad Bridge
T56-50	Calhoun County Jail	Culvert (private)
T61-50	West 4th Street	Bridge
T66-00	West 3rd Street	Structure Over Creek (private)
T69-50	West 2nd Street	Railroad Bridge
T93-50	East F Street	Bridge
T136-00	South Noble Street	Railroad Bridge
T137-50	South Noble Street	Bridge
T161-00	South Quintard Avenue	Railroad Bridge
T174-50	South Quintard Avenue	Bridge
T194-50	Snow Street	Bridge
T195-50	Quintard Mall	Culvert beneath Mall (0.27 mile)
T210-50	Highway 78	Bridge

Table 5-1. Summary of Access Needs for Parcels
OU-1/OU-2 Predesign Investigation Work Plan
Anniston PCB Site, Anniston, Alabama

PPIN	Owner (2021)	Location (nearest street)	Predesign Investigation Category
2091	Private Party (Clevenger)	901 W 10th St	100FT Floodplain
2092	Private Party (Clevenger)	905 W 10th St	100FT Floodplain
16299	Oxford Mall LLC	Quintard Ave	100FT Floodplain
16384	City of Oxford	E Side of Hale St	SU
16470	Oxford Mall LLC	700 Quintard Dr	Creek, 100FT Floodplain
16471	Oxford Mall LLC	Quintard Ave	100FT Floodplain
16795	Oxford Mall LLC	Snow St	100FT Floodplain
17521	Regional Medical Center Board	9th St	SU
17523	Health Care Authority	351 E 8th St	SU
17930	Health Care Authority	E 9th St	SU
18084	Episcopal Protestant Church	10th & Quintard Ave	SU
18125	Private Party (Pettus)	500 Chestnut Ave	NRS, 100FT Floodplain
18303	Calhoun County	West 4th St	100FT Floodplain
18311	Alabama Power Co	W 4th St	NRS, SU (to be confirmed)
18533	Private Party (Wilborn)	830 W 10th St	100FT Floodplain
18536	Anniston Department of Parks and Recreation	Zinn Dr	SU
18565	Private Party (Munro)	815 W 10th St	100FT Floodplain
18566	Private Party (Owens)	Glen Addie Ave	100FT Floodplain
18591	City of Anniston	Palmetto Ave	SU
18612	Private Party (Brewster)	616 Glenaddie Ave	100FT Floodplain
18613	Private Party (Barclay)	Pine St	100FT Floodplain
18622	Housing Development Corporation	613 Mulberry Ave	100FT Floodplain
18623	Private Party (Summers)	629 Mulberry Ave	100FT Floodplain
18624	Sam's Properties LLC	709 Mulberry Ave	100FT Floodplain
18671	City of Anniston	Moses St	SU
18702	Cherokee Controls Inc	909 W 10th St	100FT Floodplain
18703	Cherokee Controls Inc	911 W 10th St	100FT Floodplain
18704	Private Party (Starr)	917 W 10th St	100FT Floodplain
18705	Private Party (McDonald)	929 W 10th St	NRS, 100FT Floodplain
18719	Private Party (Collins)	Glen Addie Ave	100FT Floodplain
18720	Private Party (Beason)	529 Glenaddie Ave	100FT Floodplain
18757	Private Party (Ray)	Pine St	100FT Floodplain
18758	Private Party (Woodrow)	600 Pine Ave	100FT Floodplain
18759	Private Party (Ray)	W 6th St	100FT Floodplain
18772	Private Party (Oliver)	701 Mulberry Ave	100FT Floodplain

Table 5-1. Summary of Access Needs for Parcels
OU-1/OU-2 Predesign Investigation Work Plan
Anniston PCB Site, Anniston, Alabama

PPIN	Owner (2021)	Location (nearest street)	Predesign Investigation Category
18773	Private Party (Barclay)	626 Pine Ave	100FT Floodplain
18812	Private Party (Wilborn)	9th & Mulberry Ave	100FT Floodplain
19256	Friendship Baptist Church of Anniston	1130 W 14th Street	SU
19313	Anniston City Board of Education	12th St West	SU
19406	Friendship Baptist Church of Anniston	1316 White Ave	SU
19407	Friendship Baptist Church of Anniston	1324 White Ave	SU
19408	Friendship Baptist Church of Anniston	1328 White Ave	SU
19438	Friendship Baptist Church of Anniston	1311 Brown Ave	SU
20579	Anniston City Board of Education	1109 Woodstock Ave	SU
20717	Anniston City Board of Education	1301 Woodstock Ave	SU
21033	Anniston City Board of Education	500 E 18th St	SU
30182	Anniston City Board of Education	2200 W 17th St	SU
30865	Private Party (Bass)	1716 W 12th St	SU
32053	Solutia Inc	HWY 202	IM
32122	Private Party (Howard)	1107 Clydesdale Ave	SU
32123	Private Party (Hampton)	1105 Clydesdale Ave	SU
32133	Solutia Inc	Ferron & W 7th St	IM, NRS
32175	Solutia Inc	820 Boynton Ave	IM
32365	Alabama Power Co	2309 Ada,s St	NRS
32375	American Legion	W 10th St	SU
32487	International Church	1St Ave	SU
32490	First Wesleyan Church of Anniston	2500 W Jefferson St	SU
32491	First Wesleyan Church of Anniston	2502 W Jefferson St	SU
32593	Solutia Inc	Bancroft Ave	NRS Non-PCB
32612	New Covenant Church of God	2511 Old Birmingham Hwy	SU
32670	International Church	AL HWY 202	SU
32746	Solutia Inc	AL HWY 202	NRS
32763	Worsham Mildred K Trust & Roberts	AL HWY 202	NRS
62844	First Missionary Baptist Church	1100 Pine Grove Rd	NRS, 100FT Floodplain
62849	Private Party (Mitchell)	1231 Pine Grove Rd	IM
63046	Private Party (Smallwood)	Mulberry Ave	100FT Floodplain
63047	Private Party (Smallwood)	Pine St	Dredge Spoils, 100FT Floodplain
63048	Private Party (Phillips)	710 Pine Ave	100FT Floodplain
63049	Private Party (Swink)	706 Pine Ave	100FT Floodplain
63050	Private Property (Barclay)	700 Pine Ave	100FT Floodplain

Table 5-1. Summary of Access Needs for Parcels
OU-1/OU-2 Predesign Investigation Work Plan
Anniston PCB Site, Anniston, Alabama

PPIN	Owner (2021)	Location (nearest street)	Predesign Investigation Category
63216	Danny Isbell Transportation LLC	J Street	100FT Floodplain
63259	Private Party (Smith)	West 3rd St	Creek, 100FT Floodplain
63443	Estes Railcar Services LLC	Noble St	100FT Floodplain
63444	Private Party (Hall)	510 Chestnut Ave	NRS Non-PCB, 100FT Floodplain
64445	King of Anniston Inc	S Quintard Ave	100FT Floodplain
64446	Williams Scrap Metal Inc	1704 S Noble St	NRS, NRS Non-PCB, 100FT Floodplain
64447	Dukes Properties LLC	AL St HWY 21	100FT Floodplain
64448	MAFS Property Holdings LLC	ALA HWY 21	100FT Floodplain
64449	Private Party (Wingard)	1700 S Quintard Ave	100FT Floodplain
64534	Mueller Property Holdings LLC	Front St	NRS Non-PCB
64630	Retail Development of Oxford LLC	675 Creekside Dr	100FT Floodplain
64632	Mike's Romac Cleaners Inc	635 Creekside Dr	100FT Floodplain
65286	City of Oxford	AL HWY 21	SU
66392	Sandy M L Lumber Sales Co Inc	1000 Front St	NRS, 100FT Floodplain
66429	Estes Railcar Services LLC	330 S Noble St	100FT Floodplain
66607	Estes Railcar Services LLC	402 S Noble St	100FT Floodplain
66614	Private Party (Barker)	502 S Noble St	100FT Floodplain
66676	Estes Railcar Services LLC	Noble St	NRS, 100FT Floodplain
66812	Sandy M L Lumber Sales Co Inc	J Street	100FT Floodplain
66813	Private Party (Traina 2005 Trust)	Poplar Street	100FT Floodplain
66814	Private Party (Stringham)	E of 1205 Front St	100FT Floodplain
66815	Private Party (Miles)	Front St	100FT Floodplain
67093	McCormick Properties LLC	Front St	100FT Floodplain
67094	Private Party (Miles)	Front Street	100FT Floodplain
67096	Alacote Inc	E of Front St	100FT Floodplain
67539	PAC Enterprises LLC	641 Snow St	100FT Floodplain
67580	Spirit Master Funding IX LLC	515 Quintard Dr	100FT Floodplain
67593	Oxford City Water and Sewer Board	114 Hall Ave	NRS, 100FT Floodplain
67594	Oxford City Water and Sewer Board	111 Hall Ave	100FT Floodplain
67633	MAFS Property Holdings LLC	1704 S Quintard Ave	100FT Floodplain
67642	Anniston I LLC	1510 S Quintard Ave	100FT Floodplain
67746	Private Party (Young)	425 Quintard Dr	100FT Floodplain
67750	Private Party (Lee)	503 Quintard Dr	100FT Floodplain
67788	Tull Chemical Co	322 Burton St	NRS, 100FT Floodplain
67803	D G & W Properties LLC	1724 S Quintard Ave	Dredge Spoils, 100FT Floodplain

Table 5-1. Summary of Access Needs for Parcels
OU-1/OU-2 Predesign Investigation Work Plan
Anniston PCB Site, Anniston, Alabama

PPIN	Owner (2021)	Location (nearest street)	Predesign Investigation Category
67805	Williams Scrap Metal Inc	1800 S Noble St	100FT Floodplain
67806	Williams Scrap Metal Inc	S Noble	100FT Floodplain
67807	Southern Railway System	S Noble St	100FT Floodplain
67810	Private Party (Wigley)	Burton St	NRS
67811	Private Party (Wigley)	Hines St	100FT Floodplain
67814	Private Party (Gay)	1830 S Quintard Ave	Dredge Spoils, 100FT Floodplain
67820	Dukes Properties LLC	1710 S Quintard Ave	100FT Floodplain
67821	Private Party (Chen)	1800 S Quintard Ave	Dredge Spoils, 100FT Floodplain
67822	Private Party (Jennings)	1720 S Quintard Ave	Dredge Spoils, 100FT Floodplain
67823	Private Party (Sills)	1726 S Quintard Ave	Dredge Spoils, 100FT Floodplain
68000	Mueller Property Holdings LLC	W of HWY 21	Dredge Spoils, 100FT Floodplain
68004	Williams Scrap Metal Inc	S Noble St	100FT Floodplain
68005	Mueller Property Holdings LLC	S Noble St	NRS, 100FT Floodplain
68194	Hudson Properties LLC	1909 Gwin St	100FT Floodplain
68396	Private Party (Cardenas)	44 Gwin St	100FT Floodplain
68404	Oxford City Water and Sewer Board	114 Hall Ave	100FT Floodplain
68720	Mueller Property Holdings LLC	S Noble St	NRS Non-PCB, 100FT Floodplain
68920	Private Party (Garfrerick)	655 Creekside Dr	100FT Floodplain
69009	SOLUTIA INC	West 11th (Pine Grove Rd)	NRS, T11, 100FT Floodplain
75901	Private Party (Durden)	621 Pine Ave	100FT Floodplain
88497	Northeast Alabama Aluminum LLC	1517 Front St	100FT Floodplain
88498	Northeast Alabama Aluminum LLC	1517 Front St	100FT Floodplain

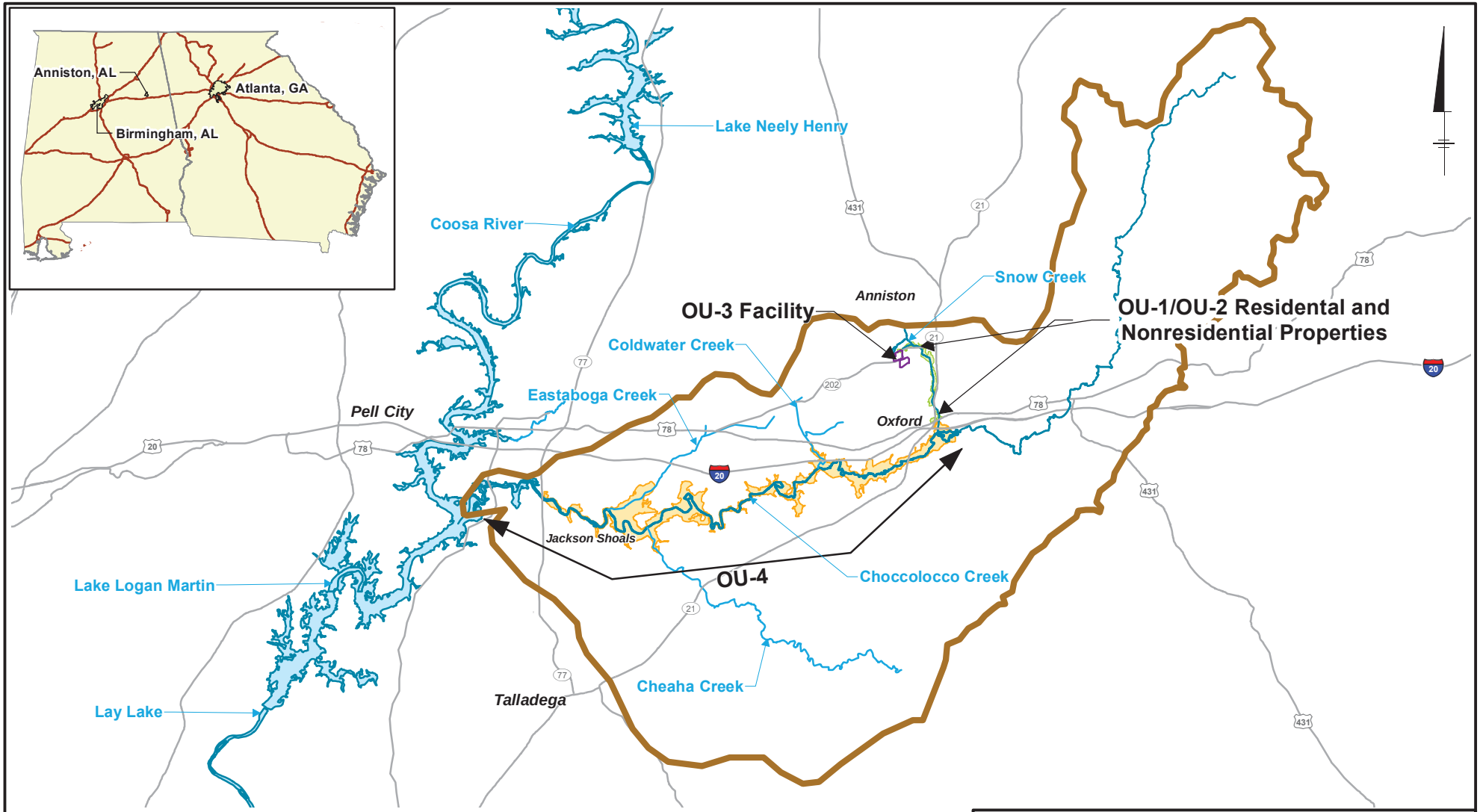
Notes:
IM: interim measure
NRS: nonresidential soils
PCB: polychlorinated biphenyl
PPIN: property identification numbers
SU: special use

Table 5-2. Summary of Access Needs for Structures along Snow Creek
OU-1/OU-2 Predesign Investigation Work Plan
Anniston PCB Site, Anniston, Alabama

Transect	Owner (2021)	Location (nearest street)	Structure Type
T0-00	City of Anniston	West 11th Street	Bridge
T0-50	Norfolk Southern	West 11th Street	Railroad Bridge
T19-50	City of Anniston	West 10th Street	Bridge
T25-00	City of Anniston	West 9th Street	Bridge (limited use)
T31-00	Alabama Department of Transportation (ALDOT)	Highway 202	Box Culvert
T35-50	City of Anniston	West 7th Street	Bridge
T42-50	City of Anniston	Glenaddie Avenue	Bridge
T51-00	Norfolk Southern	Calhoun County Jail	Railroad Bridge
T56-50	Calhoun County	Calhoun County Jail	Culvert (private)
T61-50	City of Anniston	West 4th Street	Bridge
T66-00	Private Party (Smith)	West 3rd Street	Structure Over Creek (private)
T69-50	Norfolk Southern	West 2nd Street	Railroad Bridge
T93-50	City of Anniston	East F Street	Bridge
T136-00	City of Anniston	South Noble Street	Railroad Bridge
T137-50	City of Anniston	South Noble Street	Bridge
T161-00	Norfolk Southern	South Quintard Avenue	Railroad Bridge
T174-50	ALDOT	South Quintard Avenue	Bridge
T194-50	City of Oxford	Snow Street	Bridge
T195-50	Oxford Mall LLC	Quintard Mall	Culvert beneath Mall (0.27 mile)
T210-50	ALDOT	Highway 78	Bridge

FIGURES

City: SYR Div/Group: SWG Created By: K.IVES Last Saved By: ldrum
Anniston (B0010291.2010.00001)
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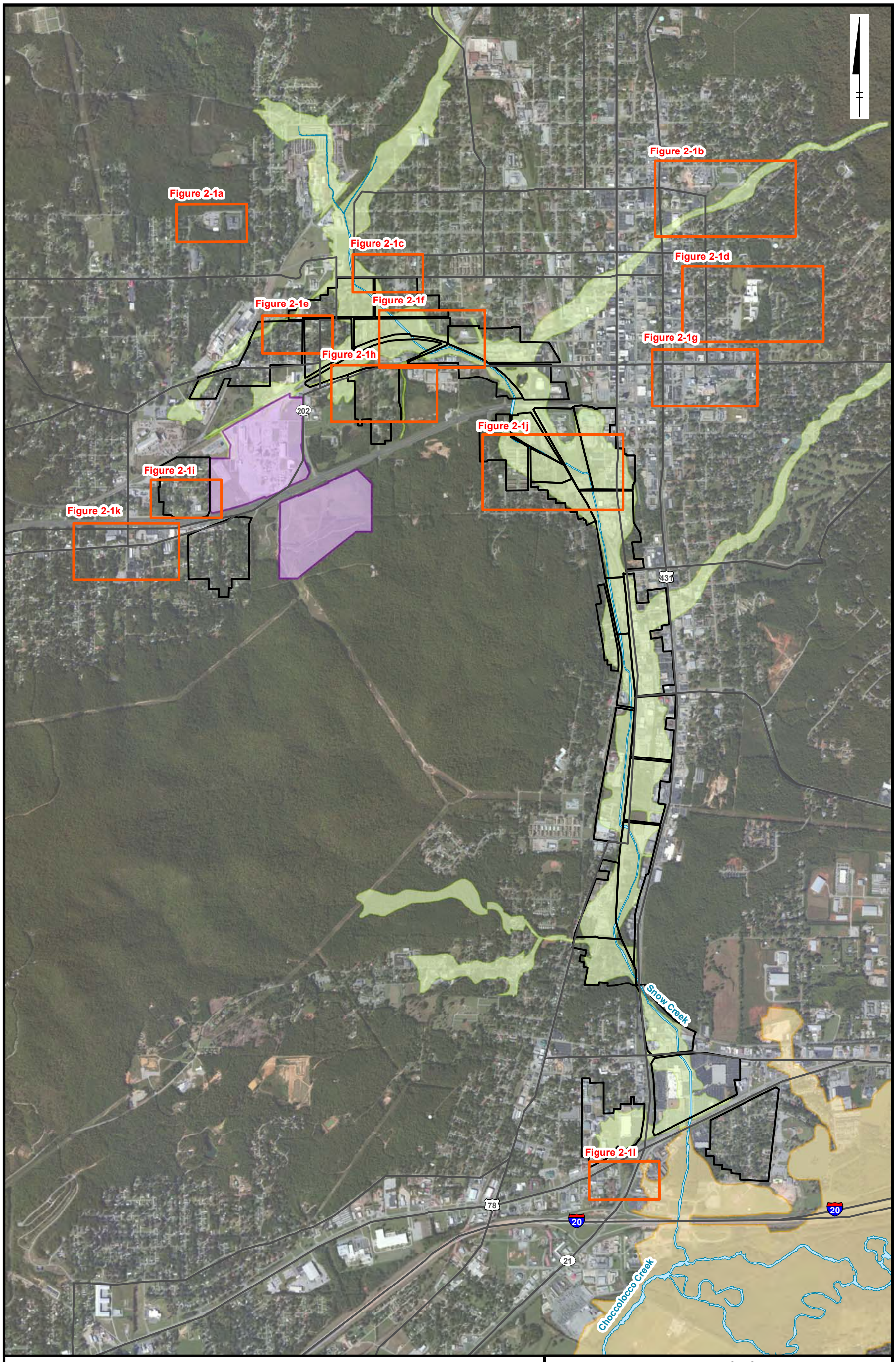
Anniston PCB Site
Anniston, Alabama

OU-1/OU-2 Predesign Investigation Work Plan

Site Location Map

Solutia

Figure
1-1



Legend

- Major Roads
- Figure Extent
- Exposure Unit
- Creek
- Operable Unit 3
- Snow Creek 100-Year Floodplain and Drainage
- OU-4 100-Year Floodplain

Notes:
1. Aerial imagery used is Clarity from ArcGIS.
2. OU: operable unit
   PCB: polychlorinated biphenyl

0 0.5 1 Miles

Graphic Scale

Anniston PCB Site
Anniston, Alabama
OU-1/OU-2 Predesign Investigation Work Plan

Low-Activity Special Use - Site Map

Solutia

Figure
2-1

City: SYR Div/Group: SWG Created By: K. Ives Last Saved By: cnelson
Anniston
N:\AAnniston\GIS\MXD\20220202_OU1_OU2_PDIWP\Rev2\Fig2-1_PDIWP_OU1_OU2_LA_Special_Use_Site Map_rev2.mxd 2/23/2022 10:41:53 AM



Legend

Historical Surface Soil
PCB Results (mg/kg)
- ND or < 1
- ≥ 1 to <10
- All Parcels
- Special Use Parcel Boundary
- Special Use: Low-Activity Area ≥1 mg/kg
- Low-Activity Subarea

0 100 200 Feet
Graphic Scale

Notes:
1. Aerial imagery provided by Calhoun and Talladega Counties.
2. Surface concentrations depicted represent the average of data from the 0- to 1-foot interval.
3. mg/kg: milligrams per kilogram
   ND: nondetect
   OU: operable unit
   PCB: polychlorinated biphenyl

Anniston PCB Site
Anniston, Alabama
OU-1/OU-2 Predesign Investigation Work Plan

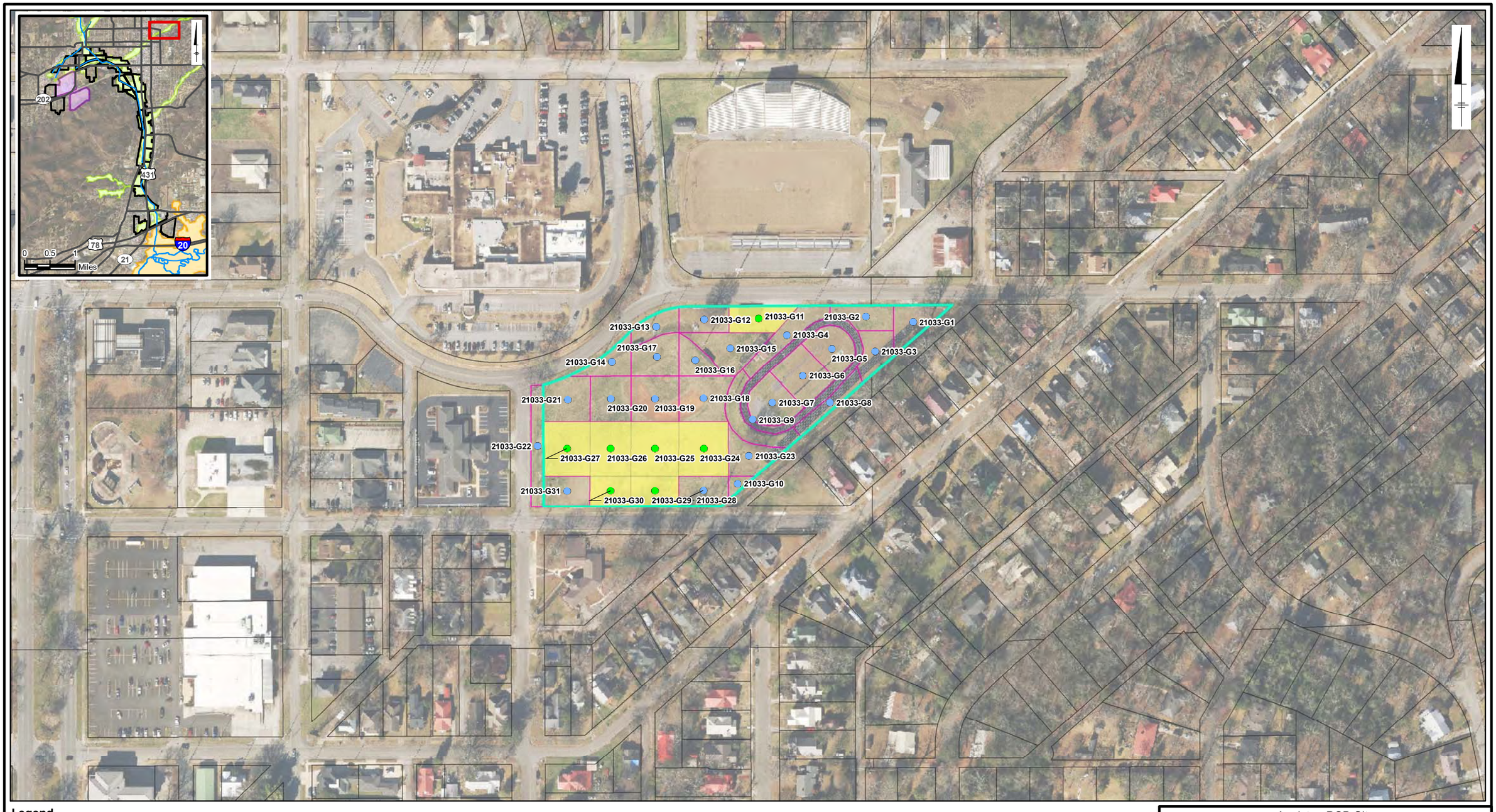
Low-Activity Special Use Soil PCB Results

PPIN: 30182

Solutia

Figure 2-1a

City: SYR Div/Group: SWG Created By: K. Ives Last Saved By: cnelson
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Legend

Historical Surface Soil
PCB Results (mg/kg)

- ND or < 1
- ≥ 1 to <10
- All Parcels
- Special Use Parcel Boundary
- Special Use: Low-Activity Area ≥1 mg/kg
- Low-Activity Subarea

0 200 400
Feet
Graphic Scale

Notes:
1. Aerial imagery provided by Calhoun and Talladega Counties.
2. Surface concentrations depicted represent the average of data from the 0- to 1-foot interval.
3. mg/kg: milligrams per kilogram
   ND: nondetect
   OU: operable unit
   PCB: polychlorinated biphenyl

Anniston PCB Site
Anniston, Alabama
OU-1/OU-2 Predesign Investigation Work Plan

Low-Activity Special Use Soil PCB Results

PPIN: 21033

Solutia

Figure 2-1b

City: SYR Div/Group: SWG Created By: K. Ives Last Saved By: cnelson
Anniston
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Legend

- Historical Analytes other than PCB and Lead Analyzed
- Historical Surface Soil PCB Results (mg/kg)
  - ND or < 1
  - ≥ 1 to <10
- Snow Creek
- All Parcels
- Special Use Parcel Boundary
- Exposure Unit
- Special Use: Low-Activity Area ≥1 mg/kg
- Low-Activity Subarea

0 100 200 Feet
Graphic Scale

Notes:
1. Aerial imagery provided by Calhoun and Talladega Counties.
2. Surface concentrations depicted represent the average of data from the 0- to 1-foot interval.
3. EU: exposure unit
   mg/kg: milligrams per kilogram
   ND: nondetect
   OU: operable unit
   PCB: polychlorinated biphenyl

Anniston PCB Site
Anniston, Alabama
OU-1/OU-2 Predesign Investigation Work Plan

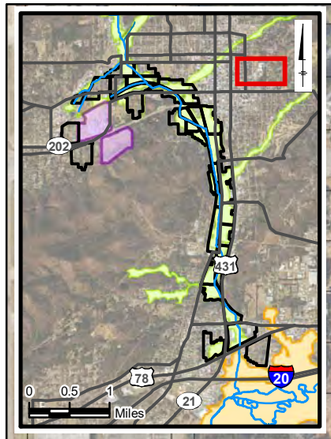
Low-Activity Special Use Soil PCB Results

PPIN: 19256, 19463, 30711 & 80736

Solutia

Figure **2-1c**

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20579-G22
20579-G23
20579-G20
20579-G21
20717-G24
20717-G43
20717-G44
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20579-G19
20579-G18
20717-G20
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20579-G13
20717-G13
20717-G53
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20717-G52
20717-G55
20579-G11
20579-G12
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20717-G50
20717-G54
20717-G12
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20579-G4
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20579-G1
20579-G2

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Anniston
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Legend

Historical Surface Soil PCB Results (mg/kg)
- ND or < 1
- ≥ 1 to <10

All Parcels
Special Use Parcel Boundary
Special Use: Low-Activity Area ≥1 mg/kg
Low-Activity Subarea

Inner and outer circles reflect surface and subsurface PCB concentrations, respectively.

0 200 400 Feet
Graphic Scale

Notes:
1. Aerial imagery provided by Calhoun and Talladega Counties.
2. Surface concentrations depicted represent the average of data from the 0- to 1-foot interval.
3. Subsurface concentrations depicted represent the average of data from the 0- to 4-foot interval.
4. mg/kg: milligrams per kilogram
   ND: nondetect
   OU: operable unit
   PCB: polychlorinated biphenyl

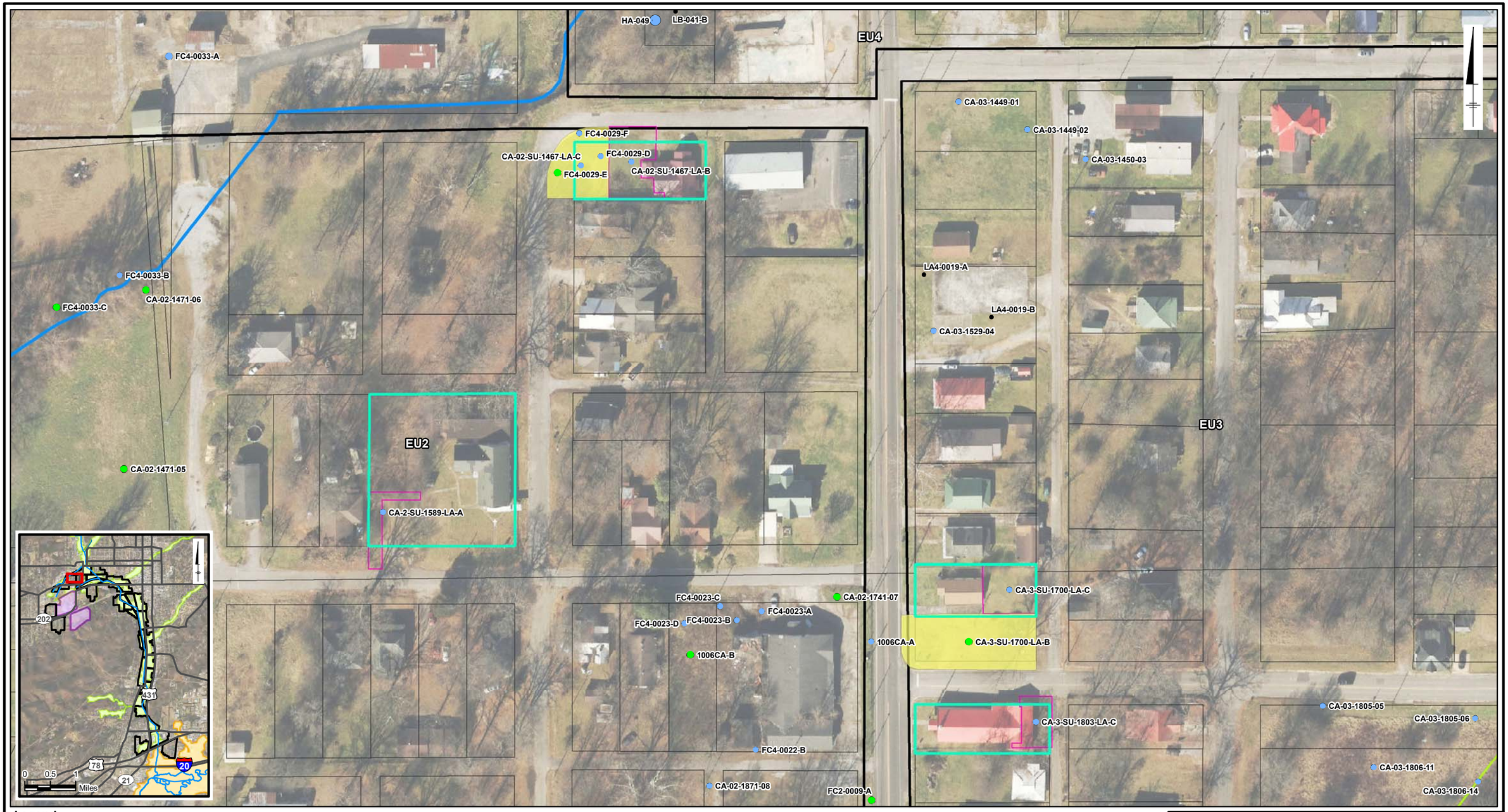
Anniston PCB Site
Anniston, Alabama
OU-1/OU-2 Predesign Investigation Work Plan

Low-Activity Special Use Soil PCB Results

PPIN: 20579, 20717

Solutia

Figure
2-1d



Legend

- Historical Analytes other than PCB and Lead Analyzed

Historical Surface Soil PCB Results (mg/kg)

- ND or < 1
- ≥ 1 to <10
- West 9th Street Creek
- All Parcels
- Exposure Unit
- Special Use Parcel Boundary
- Special Use: Low-Activity Area ≥1 mg/kg
- Low-Activity Subarea

Inner and outer circles reflect surface and subsurface PCB concentrations, respectively.

0 100 200 Feet

Graphic Scale

Notes:
1. Aerial imagery provided by Calhoun and Talladega Counties.
2. Surface concentrations depicted represent the average of data from the 0- to 1-foot interval.
3. Subsurface concentrations depicted represent the average of data from the 0- to 4-foot interval.
4. EU: exposure unit
   mg/kg: milligrams per kilogram
   ND: nondetect
   OU: operable unit
   PCB: polychlorinated biphenyl

Anniston PCB Site
Anniston, Alabama
OU-1/OU-2 Predesign Investigation Work Plan

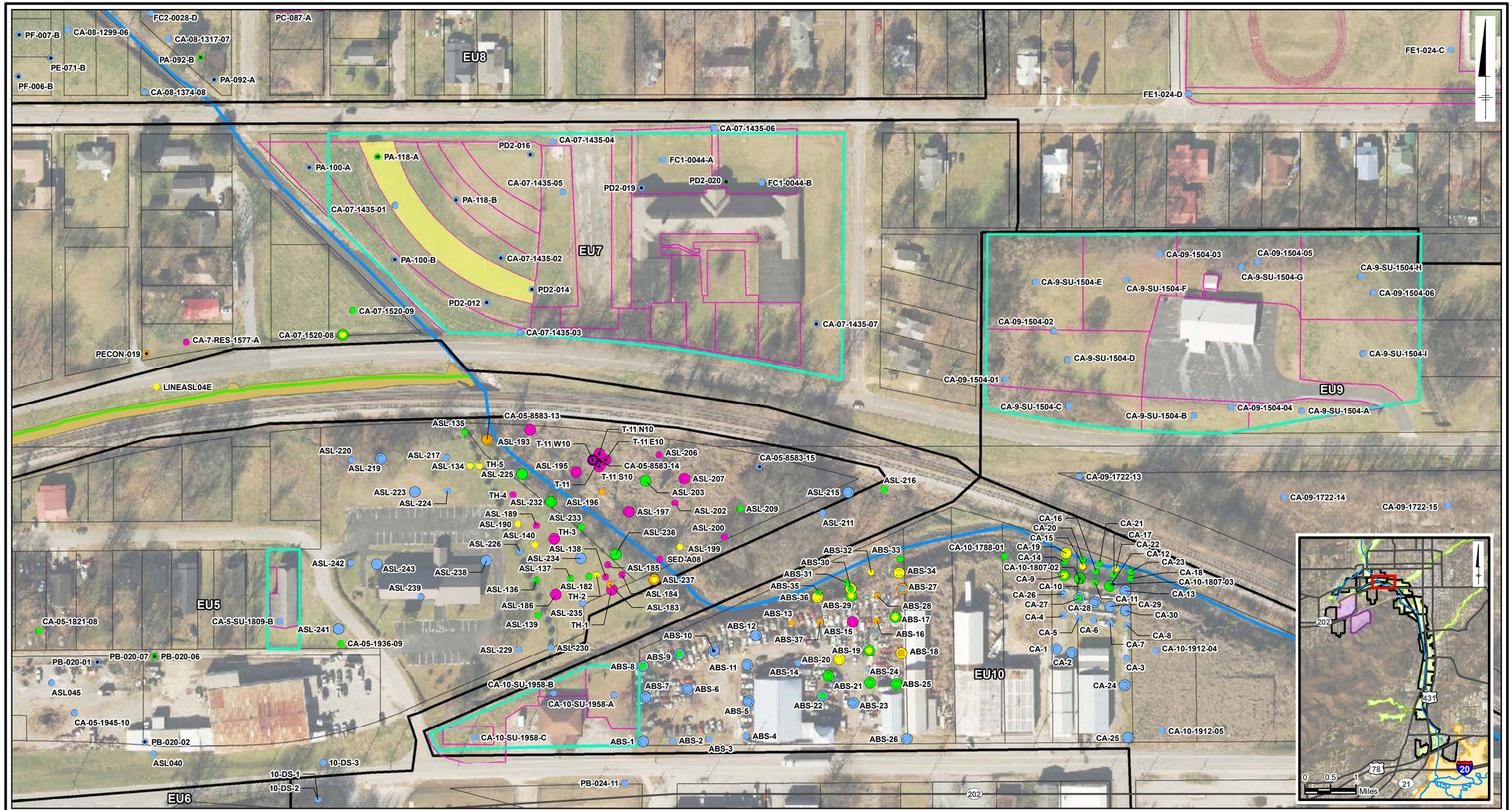
Low-Activity Special Use Soil PCB Results

PPIN: 30861, 30865, 32122 & 32277

Solutia

Figure **2-1e**

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Anniston
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Legend

- Historical Analytes other than PCB and Lead Analyzed

Historical Surface Soil PCB Results (mg/kg)

- ND or < 1
- ≥ 1 to <10
- ≥ 10 to < 25
- ≥ 25 to < 50
- ≥ 50 to < 500
- ≥ 500

- 11th Street Ditch
- Snow Creek
- All Parcels
- Exposure Unit
- Special Use Parcel Boundary
- Special Use: Low-Activity Area ≥1 mg/kg
- Low-Activity Subarea

Inner and outer circles reflect surface and subsurface PCB concentrations, respectively.

0 150 300 Feet

Graphic Scale

Notes:

1. Aerial imagery provided by Calhoun and Talladega Counties.
2. Surface concentrations depicted represent the average of data from the 0- to 1-foot interval.
3. Subsurface concentrations depicted represent the average of data from the 0- to 4-foot interval.
4. EU: exposure unit
   mg/kg: milligrams per kilogram
   ND: nondetect
   OU: operable unit
   PCB: polychlorinated biphenyl

Anniston PCB Site
Anniston, Alabama
OU-1/OU-2 Predesign Investigation Work Plan

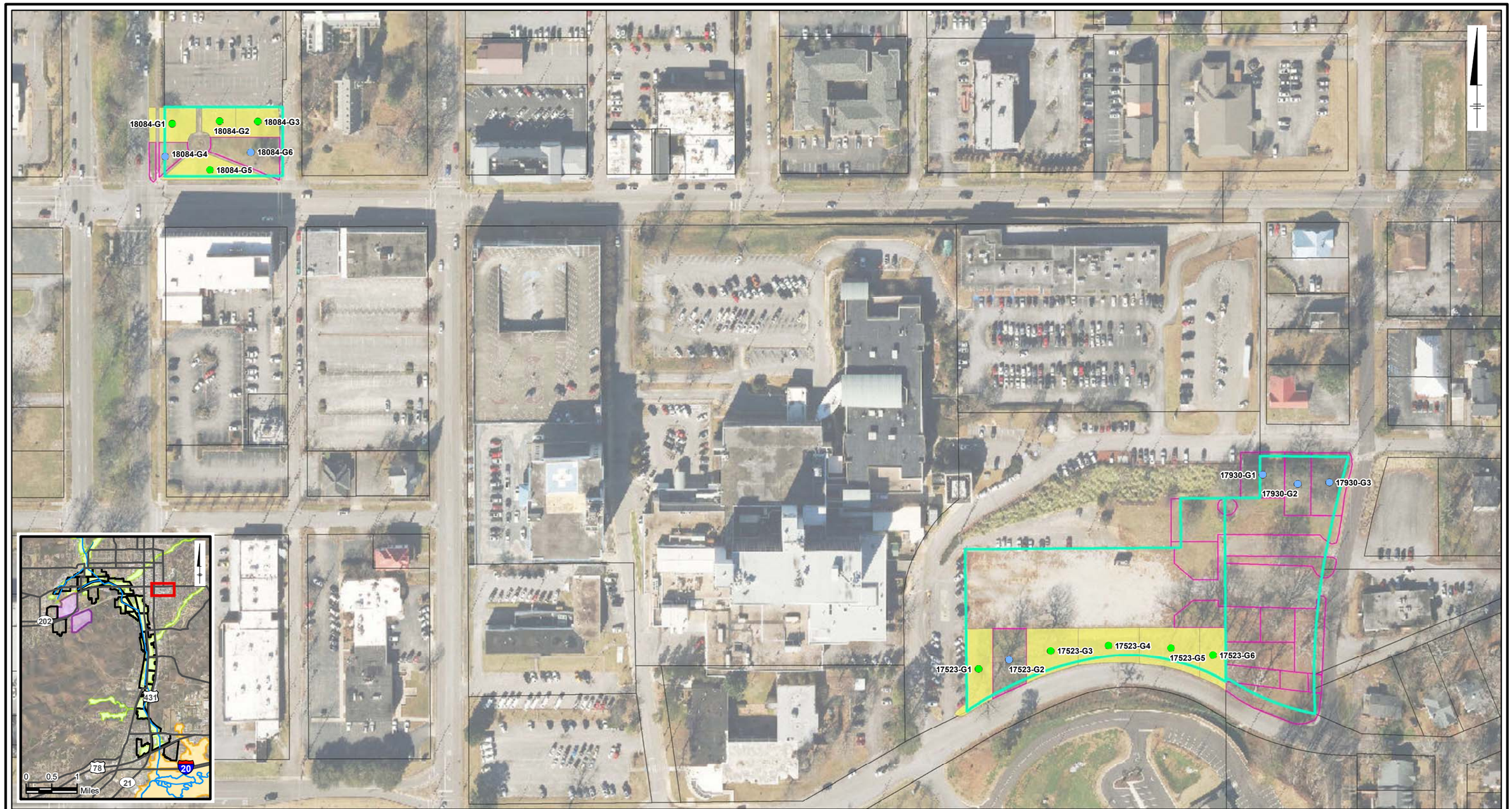
Low-Activity Special Use Soil PCB Results

PPIN: 18700, 18713, 19122 & 19313

Solutia

Figure 2-1f

City: SYR Div/Group: SWG Created By: K. Ives Last Saved By: cnelson
Anniston
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Legend

Historical Surface Soil
PCB Results (mg/kg)

- ND or < 1
- ≥ 1 to <10
- All Parcels
- Special Use Parcel Boundary
- Special Use: Low-Activity Area ≥1 mg/kg
- Low-Activity Subarea

0 150 300
Feet
Graphic Scale

Notes:
1. Aerial imagery provided by Calhoun and Talladega Counties.
2. Surface concentrations depicted represent the average of data from the 0- to 1-foot interval.
3. mg/kg: milligrams per kilogram
   ND: nondetect
   OU: operable unit
   PCB: polychlorinated biphenyl

Anniston PCB Site
Anniston, Alabama
OU-1/OU-2 Predesign Investigation Work Plan

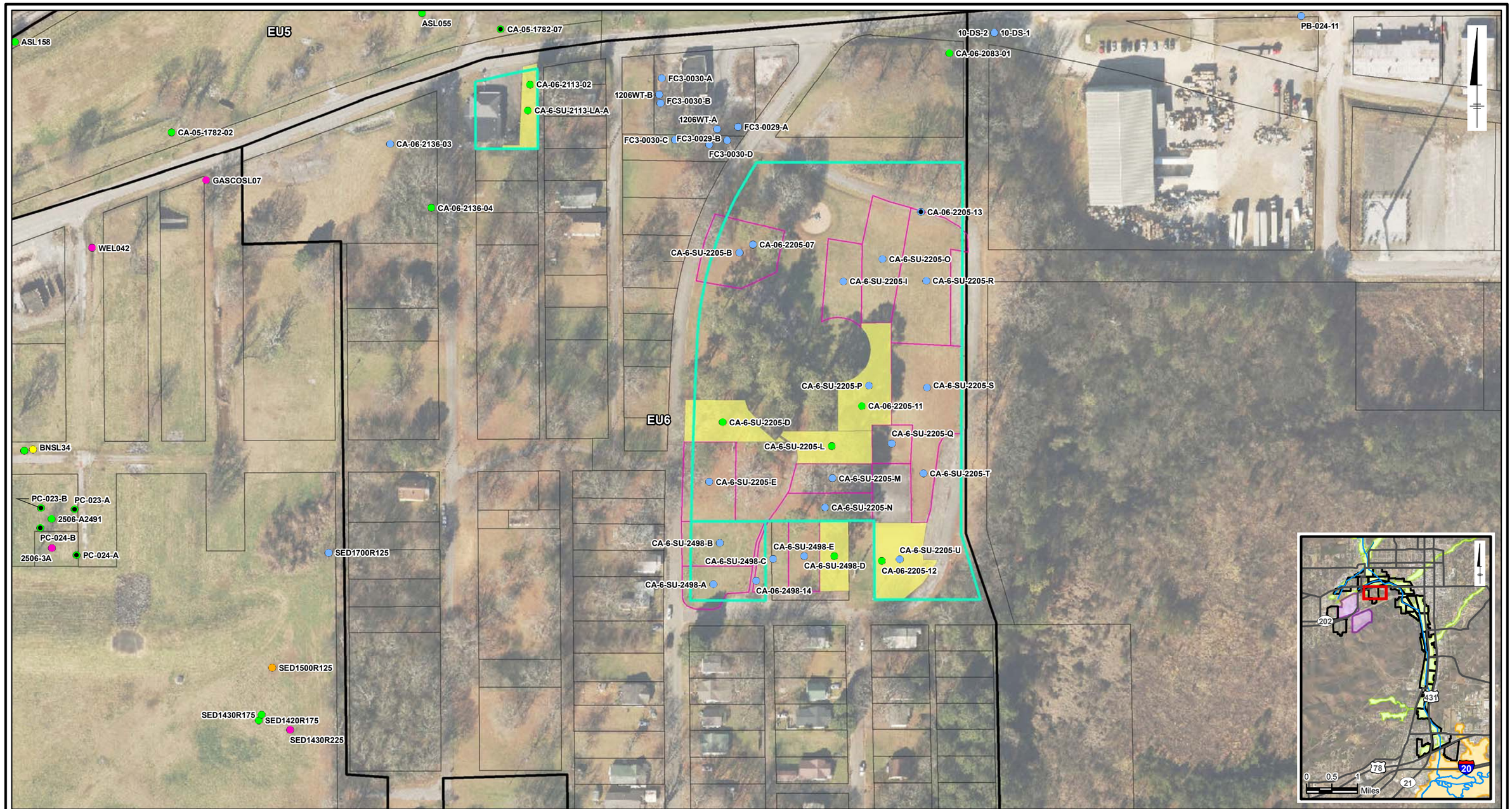
Low-Activity Special Use Soil PCB Results

PPIN: 17523,17930 & 18084

Solutia

Figure
2-1g

City: SYR Div/Group: SWG Created By: K. Ives Last Saved By: cnelson
Anniston
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Legend

- Historical Analytes other than PCB and Lead Analyzed

Historical Surface Soil PCB Results (mg/kg)

- ND or < 1
- ≥ 1 to <10
- ≥ 10 to < 25
- ≥ 25 to < 50
- ≥ 50 to < 500

- All Parcels
- Exposure Unit
- Special Use Parcel Boundary
- Special Use: Low-Activity Area ≥1 mg/kg
- Low-Activity Subarea

0 150 300 Feet

Graphic Scale

Notes:
1. Aerial imagery provided by Calhoun and Talladega Counties.
2. Surface concentrations depicted represent the average of data from the 0- to 1-foot interval.
3. EU: exposure unit
   mg/kg: milligrams per kilogram
   ND: nondetect
   OU: operable unit
   PCB: polychlorinated biphenyl

Anniston PCB Site
Anniston, Alabama
OU-1/OU-2 Predesign Investigation Work Plan

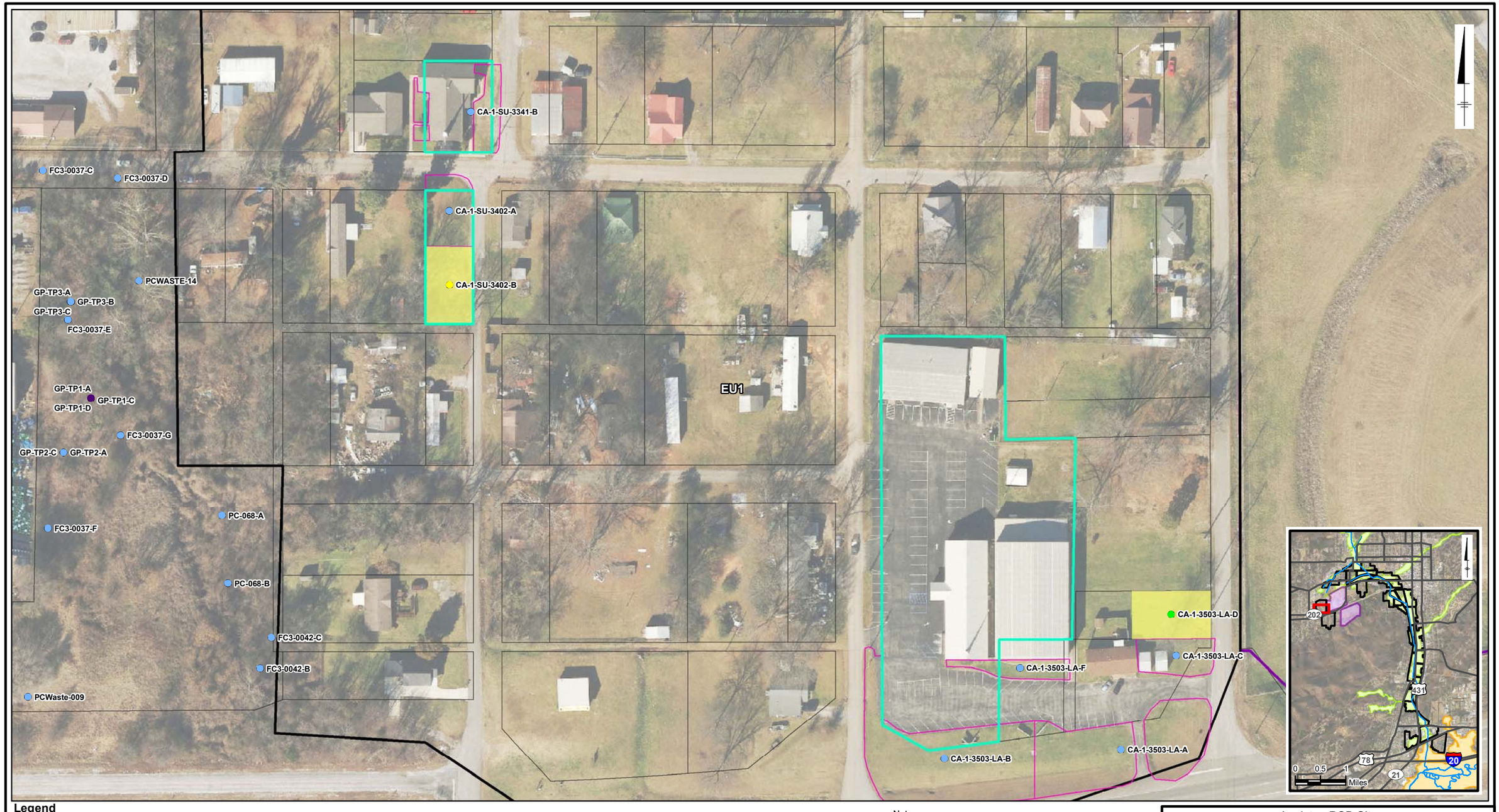
Low-Activity Special Use Soil PCB Results

PPIN: 18536, 32375 & 62824

Solutia

Figure 2-1h

City: SYR Div/Group: SWG Created By: K. Ives Last Saved By: cnelson
Anniston
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Legend

Historical Surface Soil PCB Results (mg/kg)

- ND or < 1
- ≥ 1 to <10
- ≥ 10 to < 25
- ≥ 500
- All Parcels
- Exposure Unit
- Special Use Parcel Boundary
- Special Use: Low-Activity Area ≥1 mg/kg
- Low-Activity Subarea

0 100 200 Feet

Graphic Scale

Notes:

1. Aerial imagery provided by Calhoun and Talladega Counties.
2. Surface concentrations depicted represent the average of data from the 0- to 1-foot interval.
3. EU: exposure unit
   mg/kg: milligrams per kilogram
   ND: nondetect
   OU: operable unit
   PCB: polychlorinated biphenyl

Anniston PCB Site
Anniston, Alabama
OU-1/OU-2 Predesign Investigation Work Plan

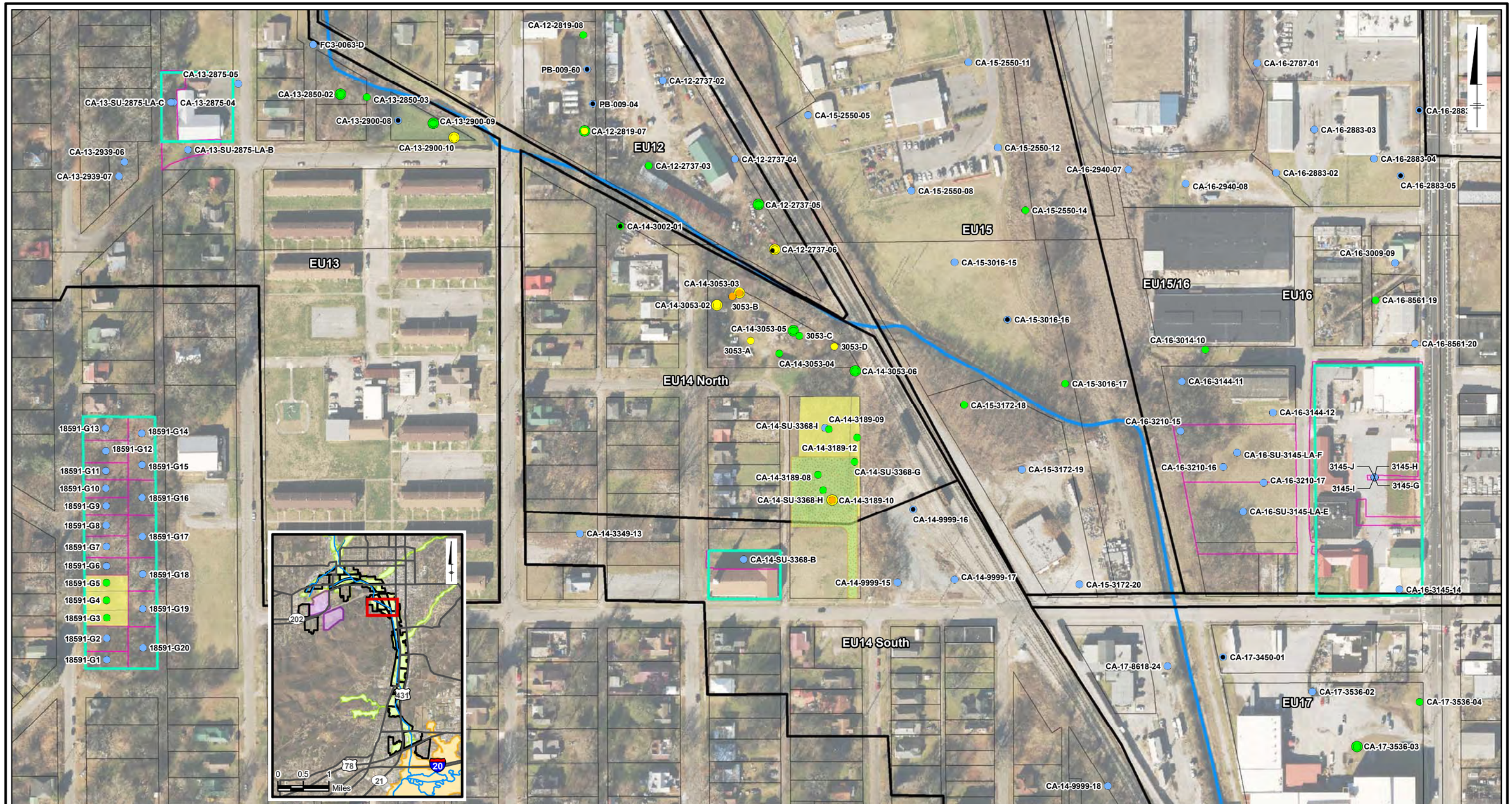
Low-Activity Special Use Soil PCB Results

PPIN: 32322, 32490 & 32664

Solutia

Figure **2-1i**

City: SYR Div/Group: SWG Created By: K. Ives Last Saved By: cnelson
Anniston
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Legend

- Historical Analytes other than PCB and Lead Analyzed

Historical Surface Soil PCB Results (mg/kg)

- ND or < 1
- ≥ 1 to <10
- ≥ 10 to < 25
- ≥ 25 to < 50
- Snow Creek
- All Parcels
- Exposure Unit
- Special Use Parcel Boundary
- Special Use: Low-Activity Area ≥1 mg/kg
- Low-Activity Subarea
- Alabama Power West 4th Street Customer Substation Development Excavation Area

Inner and outer circles reflect surface and subsurface PCB concentrations, respectively.

0 200 400 Feet

Graphic Scale

Notes:

1. Aerial imagery provided by Calhoun and Talladega Counties.
2. Surface concentrations depicted represent the average of data from the 0- to 1-foot interval.
3. Subsurface concentrations depicted represent the average of data from the 0- to 4-foot interval.
4. The Alabama Power West 4th Street customer substation construction was completed in December 2021. Limits of excavation are show as documented in the Alabama Power West 4th Street Customer Substation Construction Project Completion Report.
5. EU: exposure unit
   mg/kg: milligrams per kilogram
   ND: nondetect
   OU: operable unit
   PCB: polychlorinated biphenyl

Anniston PCB Site
Anniston, Alabama
OU-1/OU-2 Predesign Investigation Work Plan

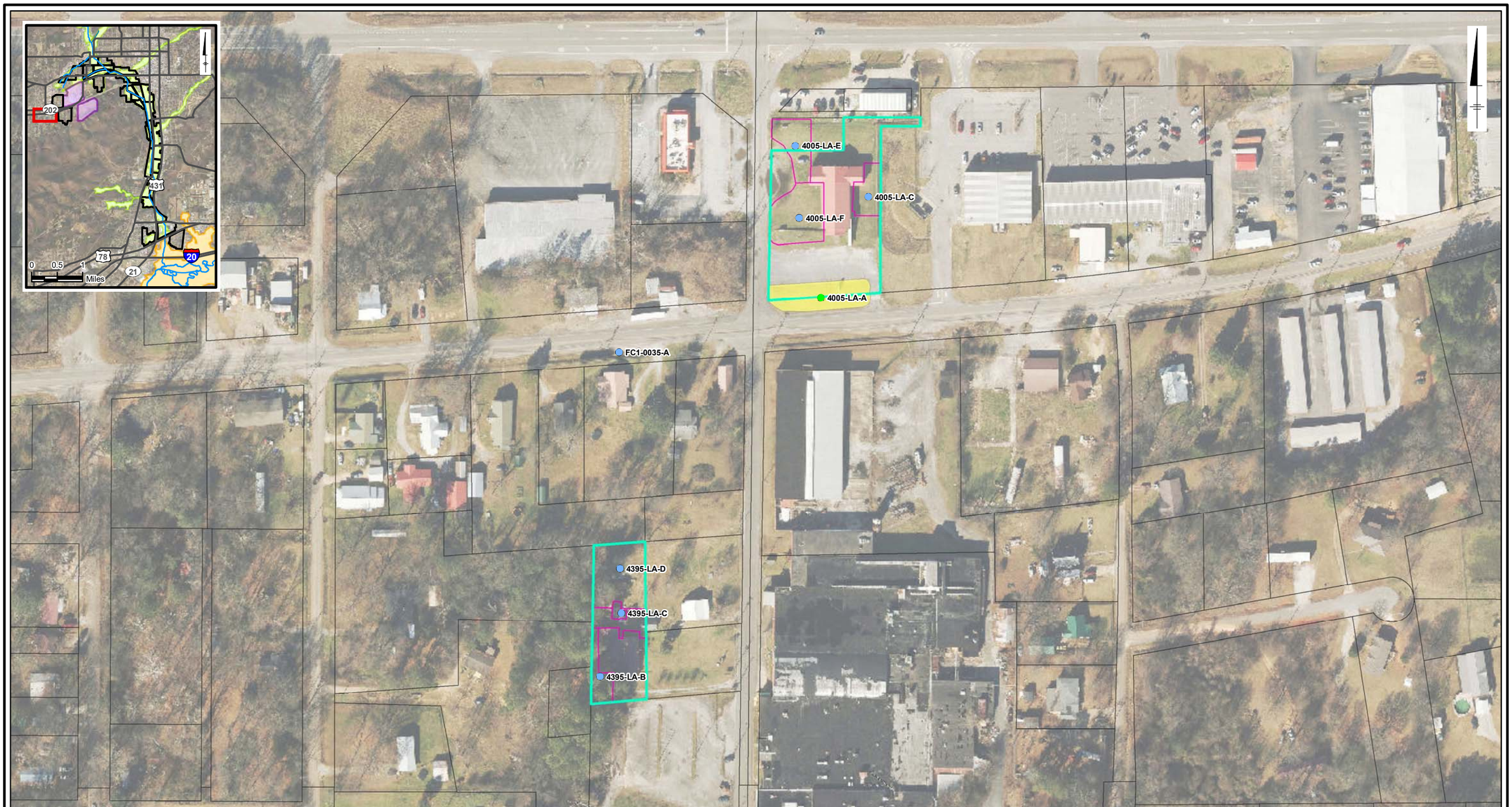
Low-Activity Special Use Soil PCB Results

PPIN: 18122, 18591, 18628 & 63275

Solutia

Figure
2-1j

City: SYR Div/Group: SWG Created By: K. Ives Last Saved By: cnelson
Anniston N:\AAnniston\GIS\MXD\20220202_OU1_OU2_PDIWP\Rev2\Fig2-1j_PDIWP_OU1_OU2_LA_Special_Use_PCB_Results_rev2.mxd 2/23/2022 11:28:27 AM



Legend

Historical Surface Soil
PCB Results (mg/kg)

- ND or < 1
- ≥ 1 to <10
- All Parcels
- Special Use Parcel Boundary
- Special Use: Low-Activity Area ≥1 mg/kg
- Low-Activity Subarea

0 150 300
Feet
Graphic Scale

Notes:
1. Aerial imagery provided by Calhoun and Talladega Counties.
2. Surface concentrations depicted represent the average of data from the 0- to 1-foot interval.
3. mg/kg: milligrams per kilogram
   ND: nondetect
   OU: operable unit
   PCB: polychlorinated biphenyl

Anniston PCB Site
Anniston, Alabama
OU-1/OU-2 Predesign Investigation Work Plan

Low-Activity Special Use Soil PCB Results

PPIN: 31947 & 32612

Solutia

Figure 2-1k

City: SYR Div/Group: SWG Created By: K. Ives Last Saved By: cnelson
Anniston
N:\AA\Anniston\GIS\MXD\20220202_OU1_OU2_PDIWP\Rev2\Fig2-1k_PDIWP_OU1_OU2_LA_Special_Use_PCB_Results_rev2.mxd 2/23/2022 11:31:43 AM



Legend

- Historical Analytes other than PCB and Lead Analyzed

Historical Surface Soil PCB Results (mg/kg)

- ND or < 1
- ≥ 1 to <10
- All Parcels
- Exposure Unit
- Special Use Parcel Boundary
- Special Use: Low-Activity Area ≥1 mg/kg
- Low-Activity Subarea

0 100 200
Feet
Graphic Scale

Notes:
1. Aerial imagery provided by Calhoun and Talladega Counties.
2. Surface concentrations depicted represent the average of data from the 0- to 1-foot interval.
3. EU: exposure unit
   mg/kg: milligrams per kilogram
   ND: nondetect
   OU: operable unit
   PCB: polychlorinated biphenyl

Anniston PCB Site
Anniston, Alabama
OU-1/OU-2 Predesign Investigation Work Plan

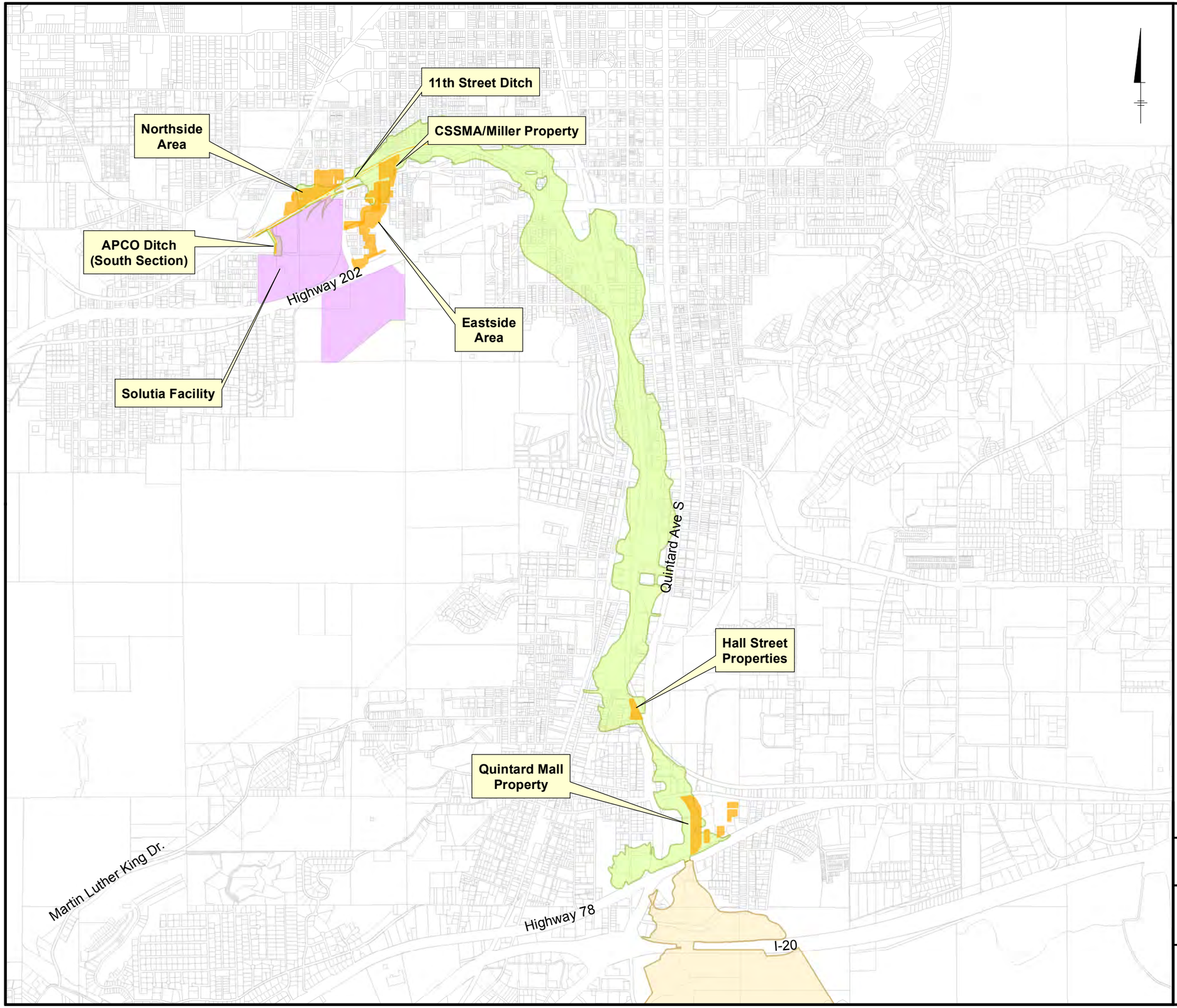
Low-Activity Special Use Soil PCB Results

PPIN: 16384

Solutia

Figure 2-1l

City: SYR Div/Group: SWG Created By: K. Ives Last Saved By: cnelson
Anniston N:\AAnniston\GIS\MXD\20220202_OU1_OU2_PDIWP\Rev2\Fig2-1l_PDIWP_OU1_OU2_LA_Special_Use_PCB_Results_rev2.mxd 2/23/2022 11:36:36 AM



Legend

- OU-3 Boundary
- Interim Measure Locations
- OU-4 100 Year Floodplain
- OU-1/OU-2 Downgradient Floodplain
- Parcels

Notes

1. The OU-3 boundary shown has been modified since issuance of the OU-3 IROD. P/S are currently working with USEPA to formally revise the boundary.

APCO: Alabama Power Company
CSSMA: central staging and soil management area
IROD: Interim Record of Decision
OU: operable unit
P/S: Pharmacia LLC and Solutia Inc.
USEPA: United States Environmental Protection Agency

References

Calhoun County Tax Parcels, 2011.

0 1,250 2,500 5,000 Feet

Anniston PCB Site
Anniston, Alabama
OU-1/OU-2 Predesign Investigation Work Plan

OU-1/OU-2 Interim Measures

Solutia

Figure 2-2

Figure 2-3e
Figure 2-3a
Figure 2-3b
202
Figure 2-3c
Figure 2-3d
431
Figure 2-3f
78
20
21
20

Legend

- Major Roads
- Figure Extent
- Exposure Unit
- Creek
- Operable Unit 3
- Snow Creek 100-Year Floodplain and Drainage
- OU-4 100-Year Floodplain

Notes:
1. Aerial imagery used is Clarity from ArcGIS.
2. OU: operable unit
   PCB: polychlorinated biphenyl

0 0.5 1 Miles

Graphic Scale

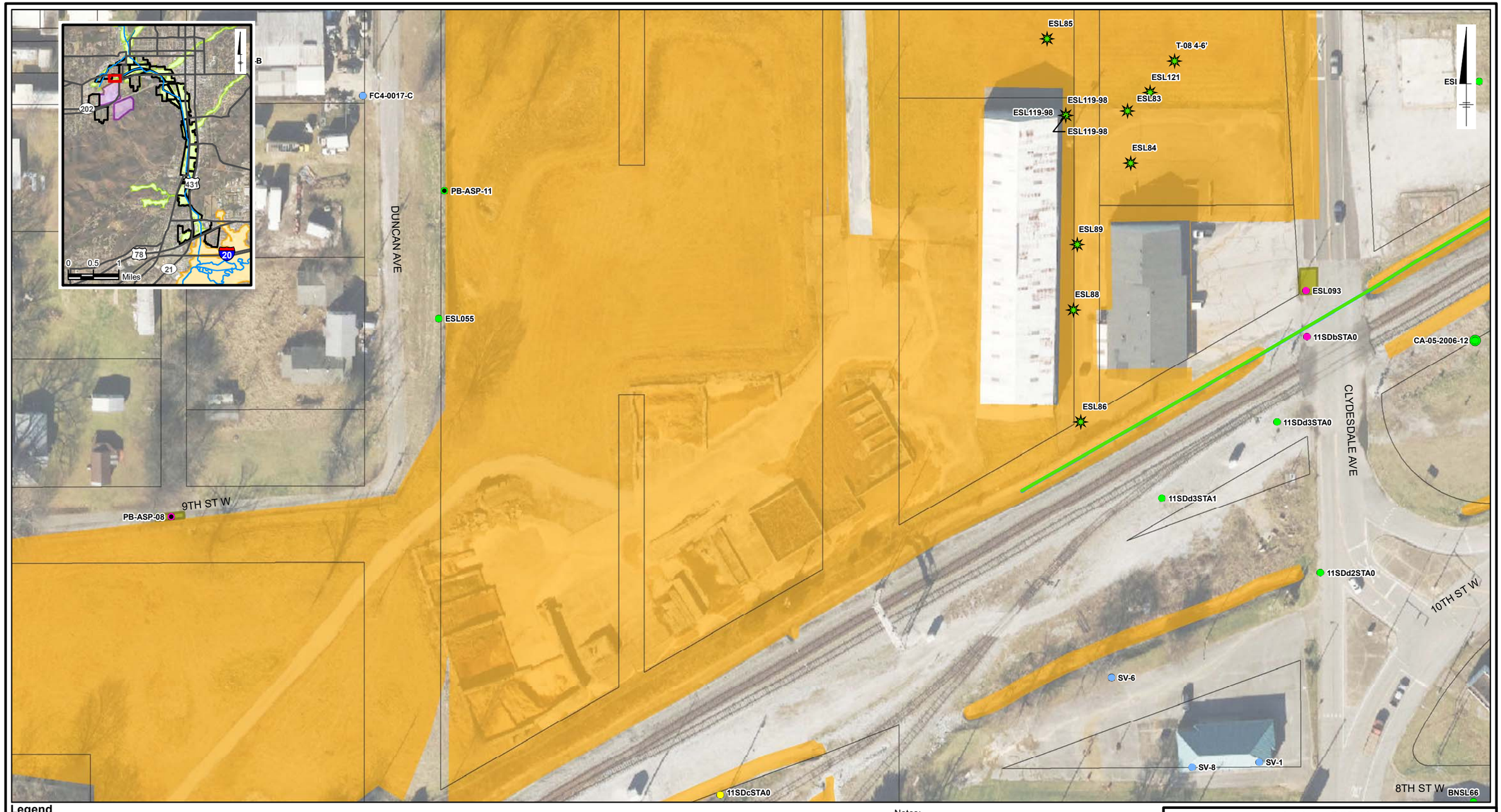
Anniston PCB Site
Anniston, Alabama
OU-1/OU-2 Predesign Investigation Work Plan

Remediation Footprints for Interim Measure Areas - Site Map

Solutia

Figure
2-3

City: SYR Div/Group: SWG Created By: K. Ives Last Saved By: cnelson
Anniston
N:\A\Anniston\GIS\MXD\20220202_OU1_OU2_PDIWP\Rev2\Fig2-3_PDIWP_OU1_OU2_Remediation_FP_IMA_Site_Map_rev2.mxd 2/23/2022 12:13:19 PM



Legend

- Historical Analytes other than PCB and Lead Analyzed
- Historical Surface Soil PCB Results (mg/kg)
  - ND or < 1
  - ≥ 1 to <10
  - ≥ 10 to < 25
  - ≥ 25 to < 50
  - ≥ 50 to < 500
- Historical Locations Beneath Cover System with Maximum PCB Results ≥500 mg/kg
- 11th Street Ditch
- Interim Measure Area Remediation Footprint
- Interim Measure and Other Early Actions
- All Parcels

Inner and outer circles reflect surface and subsurface PCB concentrations, respectively.

0 75 150 Feet

Graphic Scale

Notes:
1. Aerial imagery provided by Calhoun and Talladega Counties.
2. Surface concentrations depicted represent the average of data from the 0- to 1-foot interval.
3. Subsurface concentrations depicted represent the average of data from the 0- to 4-foot interval.
4. mg/kg: milligrams per kilogram
   ND: nondetect
   OU: operable unit
   PCB: polychlorinated biphenyl

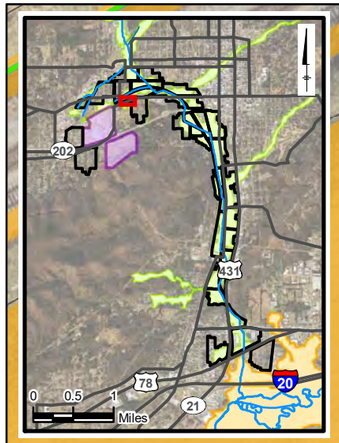
Anniston PCB Site
Anniston, Alabama
OU-1/OU-2 Predesign Investigation Work Plan

Remediation Footprints for Interim Measure Areas: Northside Area

Solutia

Figure
2-3a

City: SYR Div/Group: SWG Created By: K. Ives Last Saved By: cnelson
Anniston
N:\Anniston\GIS\MXD\20220202_OU1_OU2_PDIWP\Rev2\Fig2-3a_PDIWP_OU1_OU2_Remediation_FP_IMA_rev2.mxd 2/23/2022 11:49:15 AM



ASL157
ASL158
CA-05-1782-02
CA-06-2136-03
10TH ST W
MP-08
GASCOSL07
CA-05-1782-01
CA-06-2136-04
MONTROSE AVE
BOYNTON AVE
WEL042
HP-1
BNSL73
HP-2
WEL043
BNSL74
BNSL75
HP-3
BNSL72

Legend

Historical Surface Soil
PCB Results (mg/kg)
- ND or < 1
- ≥ 1 to <10
- ≥ 10 to < 25
- Interim Measure Area Remediation Footprint
- Interim Measure Area Remediation Footprint (0-to-1 foot bgs only)
- Interim Measure and Other Early Actions
- All Parcels

0 60 120
Feet
Graphic Scale

Notes:
1. Aerial imagery provided by Calhoun and Talladega Counties.
2. Surface concentrations depicted represent the average of data from the 0- to 1-foot interval.
3. bgs: below ground surface
   mg/kg: milligrams per kilogram
   ND: nondetect
   OU: operable unit
   PCB: polychlorinated biphenyl

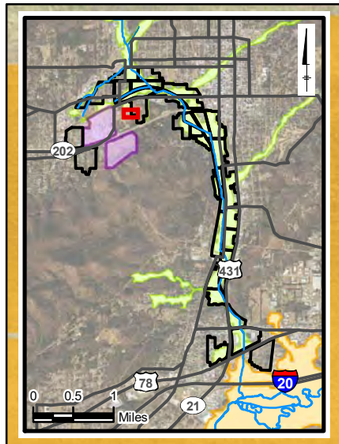
Anniston PCB Site
Anniston, Alabama
OU-1/OU-2 Predesign Investigation Work Plan

Remediation Footprints for Interim Measure Areas: Eastside Area

Solutia

Figure
2-3b

City: SYR Div/Group: SWG Created By: K. Ives Last Saved By: cnelson
Anniston
N:\AV\Anniston\GIS\MXD\20220202_OU1_OU2_PDIWP\Rev2\Fig2-3b_PDIWP_OU1_OU2_Remediation_FP_IMA_rev2.mxd 5/17/2022 10:25:22 AM



SED1500R125

SED1430R175

SED1420R175

SED1430R225

SED1300

MONTROSE AVE

SED-5

OFSL18

WEL006

WEL005

OFSL19

Legend

Historical Surface Soil PCB Results (mg/kg)
- ND or < 1
- ≥ 1 to <10
- ≥ 25 to < 50
- ≥ 50 to < 500

Historical Locations Beneath Cover System with Maximum PCB Results ≥500 mg/kg

Interim Measure Area Remediation Footprint

Interim Measure and Other Early Actions

All Parcels

0 60 120 Feet

Graphic Scale

Notes:
1. Aerial imagery provided by Calhoun and Talladega Counties.
2. Surface concentrations depicted represent the average of data from the 0- to 1-foot interval.
3. mg/kg: milligrams per kilogram
   ND: nondetect
   OU: operable unit
   PCB: polychlorinated biphenyl

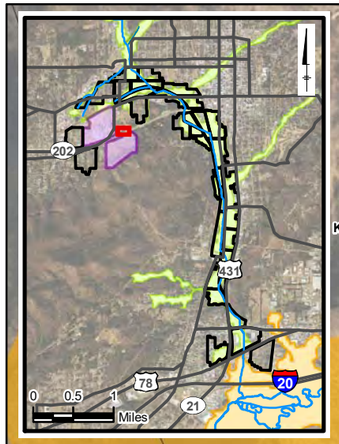
Anniston PCB Site
Anniston, Alabama
OU-1/OU-2 Predesign Investigation Work Plan

Remediation Footprints for Interim Measure Areas: Eastside Area

Solutia

Figure
2-3c

City: SYR Div/Group: SWG Created By: K. Ives Last Saved By: cnelson
Anniston
N:\Anniston\GIS\MXD\20220202_OU1_OU2_PDIWP\Rev2\Fig2-3c_PDIWP_OU1_OU2_Remediation_FP_IMA_rev2.mxd 2/23/2022 11:56:41 AM



KNSL07

SED0200R50

SED0010R50

SED0010R25

SED0010

SED0010L25

SED0010L50

AL HWY 202

CLYDESDALE AVE

MDNSL05

Legend

Historical Surface Soil PCB Results (mg/kg)
- ND or < 1
- ≥ 10 to < 25
- ≥ 50 to < 500

Historical Locations Beneath Cover System with Maximum PCB Results ≥500 mg/kg

Interim Measure Area Remediation Footprint

Interim Measure and Other Early Actions

All Parcels

Nonresidential Soil Remediation Area

0 50 100
Feet
Graphic Scale

Notes:
1. Aerial imagery provided by Calhoun and Talladega Counties.
2. Surface concentrations depicted represent the average of data from the 0- to 1-foot interval.
3. mg/kg: milligrams per kilogram
   ND: nondetect
   OU: operable unit
   PCB: polychlorinated biphenyl

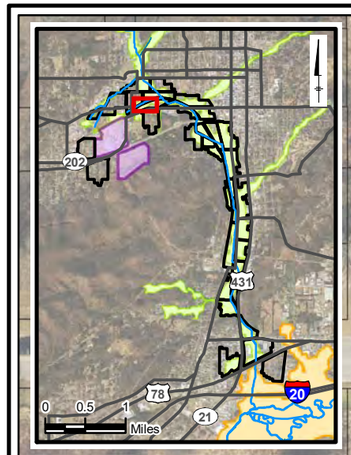
Anniston PCB Site
Anniston, Alabama
OU-1/OU-2 Predesign Investigation Work Plan

Remediation Footprints for Interim Measure Areas: Eastside Area

Solutia

Figure
2-3d

City: SYR Div/Group: SWG Created By: K. Ives Last Saved By: cnelson
Anniston
N:\A\Anniston\GIS\MXD\20220202_OU1_OU2_PDIWP\Rev2\Fig2-3d_PDIWP_OU1_OU2_Remediation_FP_IMA_rev2.mxd 2/23/2022 12:01:50 PM



CRAWFORD AVE
BOYNTON AVE
PROJECT DR
MCDANIEL AVE
11TH ST W
PINE GROVE RD
10TH ST W
MONTROSE AVE
MCDANIEL AVE
ZINN PARKWAY DR
MOSES ST

LINEASL03D
PB-RR-40
PB-RR-37
PB-RR-42
ASED550
ASED500
ASED450
ASED450R25
ASED400
LINEASL02F
LINEASL02E
LINEASL02D
ASED350
SED-C1
ASED300
ASED250
ASED100R50
CA-05-1782-06
CA-05-1782-04
CA-05-1782-05
CA-05-1782-03
CA-05-1782-07
CA-05-1782-02
CA-06-2136-03
CA-06-2113-02
CA-6-SU-2113-LA-A
ASL056
ASL055
ASL158
ASL157
CA-03-1806-17
CA-03-1806-18
CA-03-1806-11
CA-03-1806-14
CA-03-1806-12
CA-03-1806-16
CA-03-1806-13
CA-03-1806-15
CA-03-1806-10
LINEASL01F
LINEASL01E
LINEASL01D
CA-05-1821-08
PB-020-07
PB-020-06
PB-020-01
ASL045
CA-05-1945-10
CA-05-1996-11
PB-020-02
ASL040
FC3-0030-A
1206WT-B
FC3-0030-B
FC3-0029-A
1206WT-A
FC3-0029-B
FC3-0030-C
FC3-0030-D

Legend

- Historical Analytes other than PCB and Lead Analyzed

Historical Surface Soil PCB Results (mg/kg)
- ND or < 1
- ≥ 1 to <10
- ≥ 10 to < 25
- ≥ 25 to < 50
- ≥ 50 to < 500
- ≥ 500

- Historical Locations Beneath Cover System with Maximum PCB Results ≥500 mg/kg
- Historical Locations with Maximum PCB Results ≥500 mg/kg
- 11th Street Ditch
- Interim Measure Area Remediation Footprint
- Interim Measure Area Remediation Footprint (0-to-1 foot bgs only)
- Interim Measure and Other Early Actions
- All Parcels

Inner and outer circles reflect surface and subsurface PCB concentrations, respectively.

0 100 200 Feet
Graphic Scale

Notes:
1. Aerial imagery provided by Calhoun and Talladega Counties.
2. Surface concentrations depicted represent the average of data from the 0- to 1-foot interval.
3. Subsurface concentrations depicted represent the average of data from the 0- to 4-foot interval.
4. bgs: below ground surface
   mg/kg: milligrams per kilogram
   ND: nondetect
   OU: operable unit
   PCB: polychlorinated biphenyl

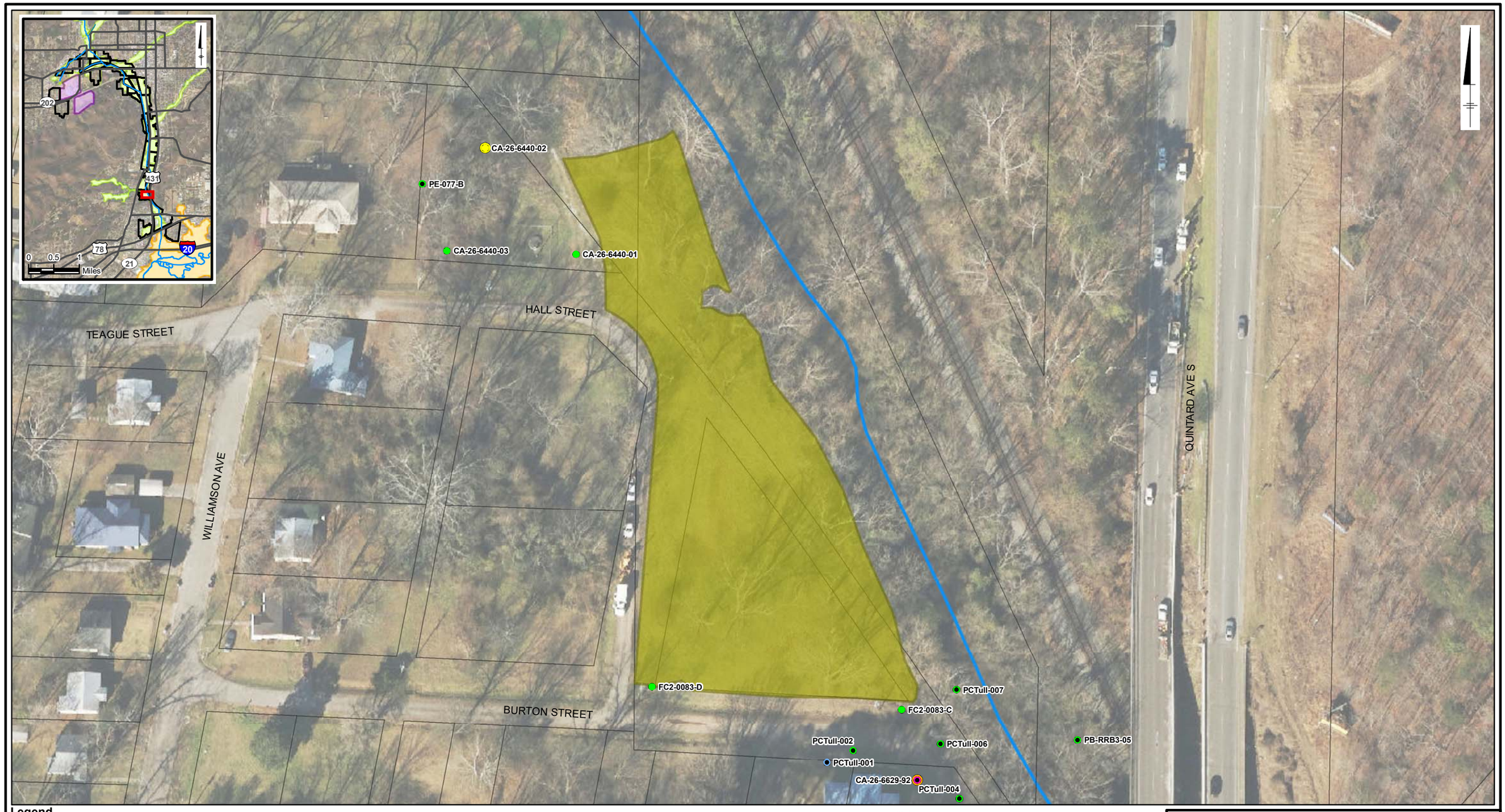
Anniston PCB Site
Anniston, Alabama
OU-1/OU-2 Predesign Investigation Work Plan

Remediation Footprints for Interim Measure Areas: Eastside Drainage Way

Solutia

Figure
2-3e

City: SYR Div/Group: SWG Created By: K. Ives Last Saved By: cnelson
Anniston
N:\AA\Anniston\GIS\MXD\20220202_OU1_OU2_PDIWP\Rev2\Fig2-3e_PDIWP_OU1_OU2_Remediation_FP_IMA_rev2.mxd 5/17/2022 11:23:37 AM



Legend

- Historical Analytes other than PCB and Lead Analyzed

Historical Surface Soil PCB Results (mg/kg)

- ND or < 1
- ≥ 1 to <10
- ≥ 10 to < 25
- ≥ 25 to < 50
- ≥ 50 to < 500

Snow Creek

Interim Measure Area Remediation Footprint

All Parcels

Inner and outer circles reflect surface and subsurface PCB concentrations, respectively.

0 75 150 Feet

Graphic Scale

Notes:

1. Aerial imagery provided by Calhoun and Talladega Counties.
2. Surface concentrations depicted represent the average of data from the 0- to 1-foot interval.
3. Subsurface concentrations depicted represent the average of data from the 0- to 4-foot interval.
4. mg/kg: milligrams per kilogram
   ND: nondetect
   OU: operable unit
   PCB: polychlorinated biphenyl

Anniston PCB Site
Anniston, Alabama
OU-1/OU-2 Predesign Investigation Work Plan

Remediation Footprints for Interim Measure Areas: Hall Street

Solutia

Figure
2-3f

City: SYR Div/Group: SWG Created By: K. Ives Last Saved By: cnelson
Anniston
N:\AAnniston\GIS\MXD\20220202_OU1_OU2_PDIWP\Rev2\Fig2-3f_PDIWP_OU1_OU2_Remediation_FP_IMA_rev2.mxd 2/23/2022 12:10:05 PM

EU10
EU11
8th
SC-2
SC-1
202
202
SC-3
Glenaddie
EU15
Mulberry
7th
8th
Pine
EU12
EU13
SC-4
SC-5
SC-6
EU25
SC-7
SC-8
EU24
EU26

Legend

- Snow Creek
- 100-Year Floodplain and Drainage Areas
- Exposure Unit
- Dredge Spoil Pile in Place
- Dredge Spoil Pile Removed

Note:
1. Dredge spoil areas SC-3, SC-4, SC-5, and SC-6 were removed in 2009.

0 1,600 3,200 Feet

Graphic Scale

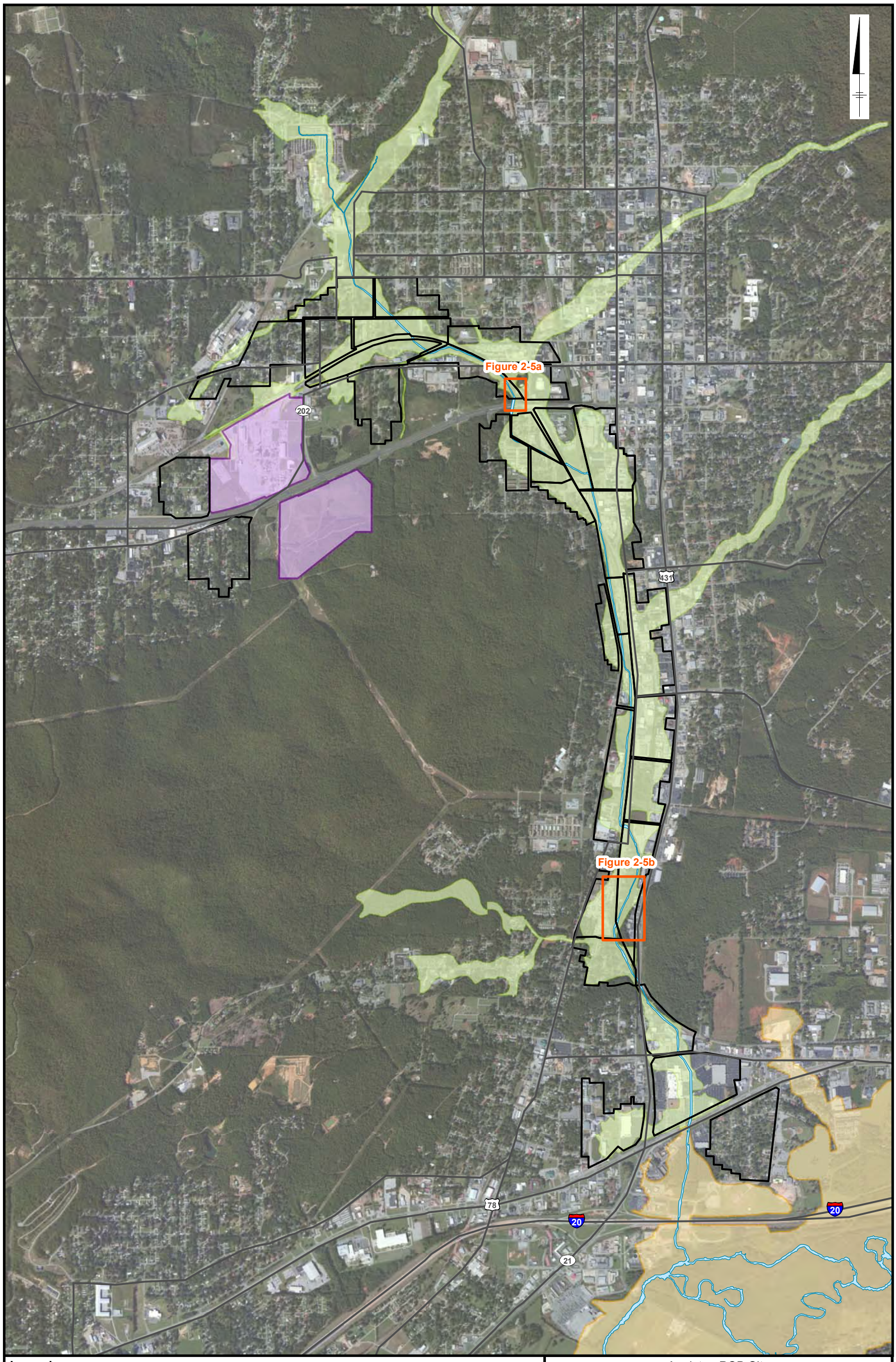
Anniston PCB Site
Anniston, Alabama

OU-1/OU-2 Predesign Investigation Work Plan

Dredge Spoils - Snow Creek

Solutia

Figure
2-4



Legend

- Major Roads
- Figure Extent
- Exposure Unit
- Creek
- Operable Unit 3
- Snow Creek 100-Year Floodplain and Drainage
- OU-4 100-Year Floodplain

Notes:
1. Aerial imagery used is Clarity from ArcGIS.
2. OU: operable unit
   PCB: polychlorinated biphenyl

0 0.5 1 Miles

Graphic Scale

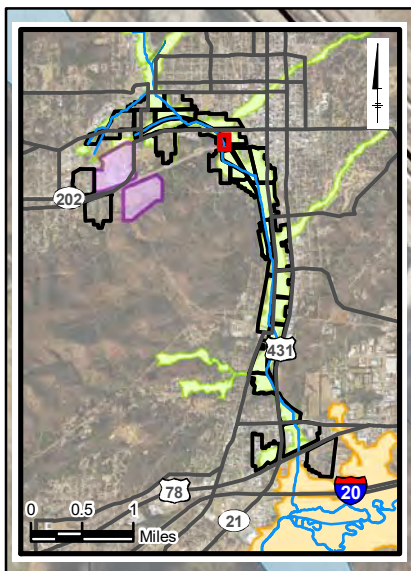
Anniston PCB Site
Anniston, Alabama
OU-1/OU-2 Predesign Investigation Work Plan

Dredge Spoil Piles - Site Map

Solutia

Figure **2-5**

City: SYR Div/Group: SWG Created By: K. Ives Last Saved By: cnelson
Anniston
N:\A\Anniston\GIS\MXD\20220202_OU1_OU2_PDIWP\Rev2\Fig2-5_PDIWP_OU1_OU2_Dredge_Spoil_Piles_SiteMap_rev2.mxd 2/23/2022 12:17:30 PM



EU11

EU10

CP-C-6

CP-C-8

CP-C-7

CA-11-9999-02

Dredge Spoil Pile SC-2

Dredge Spoil Pile SC-1

Location	Top Depth (ft)	Bottom Depth (ft)	Analyte	Result (mg/kg)
SC-1	0.83	3.00	Total PCBs	20
SC-1	0.83	3.00	Mercury	0.25

AL HWY 202

Legend

- Historical Analytes other than PCB and Lead Analyzed

Historical Surface Soil PCB Results (mg/kg)

- ND or < 1
- ≥ 1 to <10
- ≥ 10 to < 25

- Dredge Spoil Pile In Place
- All Parcels
- 100-Year Floodplain Drainage Area
- Exposure Unit
- Snow Creek

0 50 100 Feet

Graphic Scale

Notes:
1. Aerial imagery provided by Calhoun and Talladega Counties.
2. No sampling was conducted in pile SC-2.
3. Surface concentrations depicted represent the average of data from the 0- to 1-foot interval.
4. Subsurface concentrations depicted represent the average of data from the 0- to 4-foot interval.
5. EU: exposure unit
   ft: feet
   mg/kg: milligrams per kilogram
   ND: nondetect
   OU: operable unit
   PCB: polychlorinated biphenyl

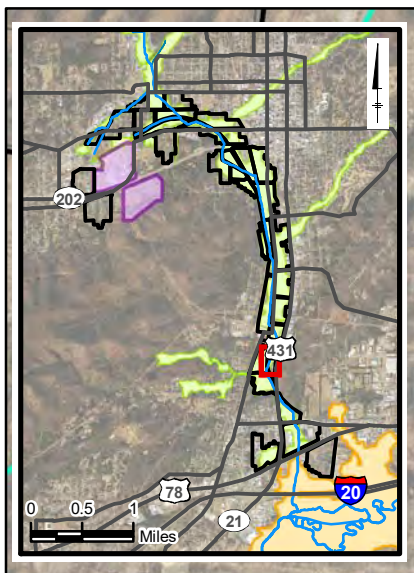
Anniston PCB Site
Anniston, Alabama
OU-1/OU-2 Predesign Investigation Work Plan

Dredge Spoil Piles SC-1 and SC-2

Solutia

Figure 2-5a

City: SYR Div/Group: SWG Created By: K. Ives Last Saved By: cnelson
Anniston
N:\A\Anniston\GIS\MXD\20220202_OU1_OU2_PDIWP\Rev2\Fig2-5a_PDIWP_OU1_OU2_Dredge_Spoil_Piles_rev2.mxd 2/23/2022 12:43:09 PM



CA-24-6164-23
CA-24-6164-24
CA-24-6196-25
CA-24-6196-26
SC-8-11
CA-25-6156-14
SC-8-10
CA-24-6246-27
SC-8-9
CA-24-6267-29
SC-8-8
SC-7-9
CA-25-6156-12
SC-7-8
SC-8-7
CA-24-6267-28
SC-7-7
EU24
SC-8-6
CA-24-6282-30
SC-7-6
EU25
Dredge Spoil Pile SC-8
SC-8-5
Dredge Spoil Pile SC-7
SC-7-5
SC-8-4
CA-24-6282-31
PB-005-04
CA-25-6156-10
SC-8-3
SC-7-4
SC-7-3
SC-8-2
SC-7-2
SC-8-20
SC-7-1
SC-8-1
CA-24-6282-32
CA-25-6156-09
CA-25-6156-13
CA-24-6321-33
CA-24-6321-34
PB-005-02
CA-25-6156-15
CA-24-6350-35
CA-24-6388-36
EU26

Location	Top Depth (ft)	Bottom Depth (ft)	Analyte	Result (mg/kg)
SC-7-1	1.67	1.92	Total PCBs	4.6
SC-7-2	1.50	1.75	Total PCBs	1.3
SC-7-3	2.50	2.75	Total PCBs	1.5
SC-7-4	1.67	1.92	Total PCBs	1.1
SC-7-4	1.67	1.92	Mercury	0.19
SC-7-5	2.00	2.25	Total PCBs	0.52
SC-7-6	1.25	1.50	Total PCBs	5.3
SC-7-7	2.25	2.50	Total PCBs	1.7
SC-7-8	1.50	1.75	Total PCBs	1.5
SC-7-9	2.00	2.25	Total PCBs	19
SC-8-1	1.00	1.25	Total PCBs	1.2
SC-8-1	1.00	1.25	Mercury	0.27
SC-8-2	2.00	2.25	Total PCBs	29
SC-8-2	2.00	2.25	Total PCBs	27
SC-8-3	1.67	2.25	Total PCBs	2.2
SC-8-4	0.75	1.00	Total PCBs	0.75
SC-8-5	0.83	1.08	Total PCBs	2.63
SC-8-6	0.67	0.92	Total PCBs	6.8
SC-8-7	2.50	2.75	Total PCBs	14
SC-8-8	0.83	1.08	Total PCBs	5.4
SC-8-9	2.50	2.75	Total PCBs	9.9
SC-8-10	1.75	2.00	Total PCBs	34
SC-8-11	2.33	2.58	Total PCBs	46

Legend

- Historical Analytes other than PCB and Lead Analyzed
- Historical Surface Soil PCB Results (mg/kg)
  - ND or < 1
  - ≥ 1 to <10
  - ≥ 10 to < 25
  - ≥ 25 to < 50
- Dredge Spoil Pile In Place
- All Parcels
- 100-Year Floodplain Drainage Area
- Exposure Unit
- Snow Creek
- Inner and outer circles reflect surface and subsurface PCB concentrations, respectively.

Notes:
1. Aerial imagery provided by Calhoun and Talladega Counties.
2. Surface concentrations depicted represent the average of data from the 0- to 1-foot interval.
3. Subsurface concentrations depicted represent the average of data from the 0- to 4-foot interval.
4. EU: exposure unit
   ft: feet
   mg/kg: milligrams per kilogram
   ND: nondetect
   OU: operable unit
   PCB: polychlorinated biphenyl

0 100 200 Feet
Graphic Scale

Anniston PCB Site
Anniston, Alabama
OU-1/OU-2 Predesign Investigation Work Plan

Dredge Spoil Piles SC-7 and SC-8

Solutia

Figure **2-5b**

City: SYR Div/Group: SWG Created By: K. Ives Last Saved By: cnelson
Anniston
N:\A\Anniston\GIS\MXD\20220202_OU1_OU2_PDIWP\Rev2\Fig2-5b_PDIWP_OU1_OU2_Dredge_Spoil_Piles_rev2.mxd 2/23/2022 12:55:18 PM

Notes:
1. EU: exposure unit
   mg/kg: milligrams per kilogram
   ND: nondetect
   OU: operable unit
   PCB: polychlorinated biphenyls

2. Aerial imagery provided by Calhoun and Talladega Counties.

3. Surface concentrations depicted represent the average of data from the 0- to 1-foot interval.

4. OU-1/OU-2 EU Area includes EU1-30, northside properties, and eastside properties.

0 4,400 8,800 Feet

Graphic Scale

Legend

Soil Sample Location
Surface PCB Inside
OU-1/OU-2 EU Area (mg/kg)
- ND or < 1
- ≥ 1 or <10
- ≥ 10 to < 25
- ≥ 25 to < 50
- ≥ 50 to < 500
- ≥ 500

Soil Sample Location
Surface PCB Outside
OU-1/OU-2 EU Area (mg/kg)
- ND or < 1
- ≥ 1 or <10
- ≥ 10 to < 25
- ≥ 25 to < 50
- ≥ 50 to < 500
- ≥ 500

- Analytes other than PCB and Lead Analyzed
- Snow Creek
- 11th Street Ditch
- West 9th Street Creek
- Operable Unit 3
- Northside and Eastside Areas
- 100-Year Floodplain and Drainage Areas
- Exposure Unit
- Zone B Boundary
- Choccoloco Creek

Anniston PCB Site
Anniston, Alabama

OU-1/OU-2 Predesign Investigation Work Plan

Nonresidential Surface Soil PCB Results

Solutia

Figure
2-6

Notes:
1. EU: exposure unit
   mg/kg: milligrams per kilogram
   ND: nondetect
   OU: operable unit
   PCB: polychlorinated biphenyls

2. Aerial imagery provided by Calhoun and Talladega Counties.

3. Subsurface concentrations depicted represent the average of data from the 0- to 4-feet interval

4. OU-1/OU-2 EU Area includes EU1-30, northside properties, and eastside properties.

0 4,400 8,800 Feet

Graphic Scale

Legend

Soil Sample Location
Subsurface PCB Inside
OU-1/OU-2 EU Area (mg/kg)
- ND or < 1
- ≥ 1 or <10
- ≥ 10 to < 25
- ≥ 25 to < 50
- ≥ 50 to < 500
- ≥ 500

Soil Sample Location
Subsurface PCB Outside
OU-1/OU-2 EU Area (mg/kg)
- ND or < 1
- ≥ 1 or <10
- ≥ 10 to < 25
- ≥ 25 to < 50
- ≥ 50 to < 500
- ≥ 500
- Snow Creek

- 11th Street Ditch
- West 9th Street Creek
- Operable Unit 3
- 100-Year Floodplain and Drainage Areas
- Exposure Unit
- Zone B Boundary
- Choccoloco Creek
- Northside and Eastside Areas

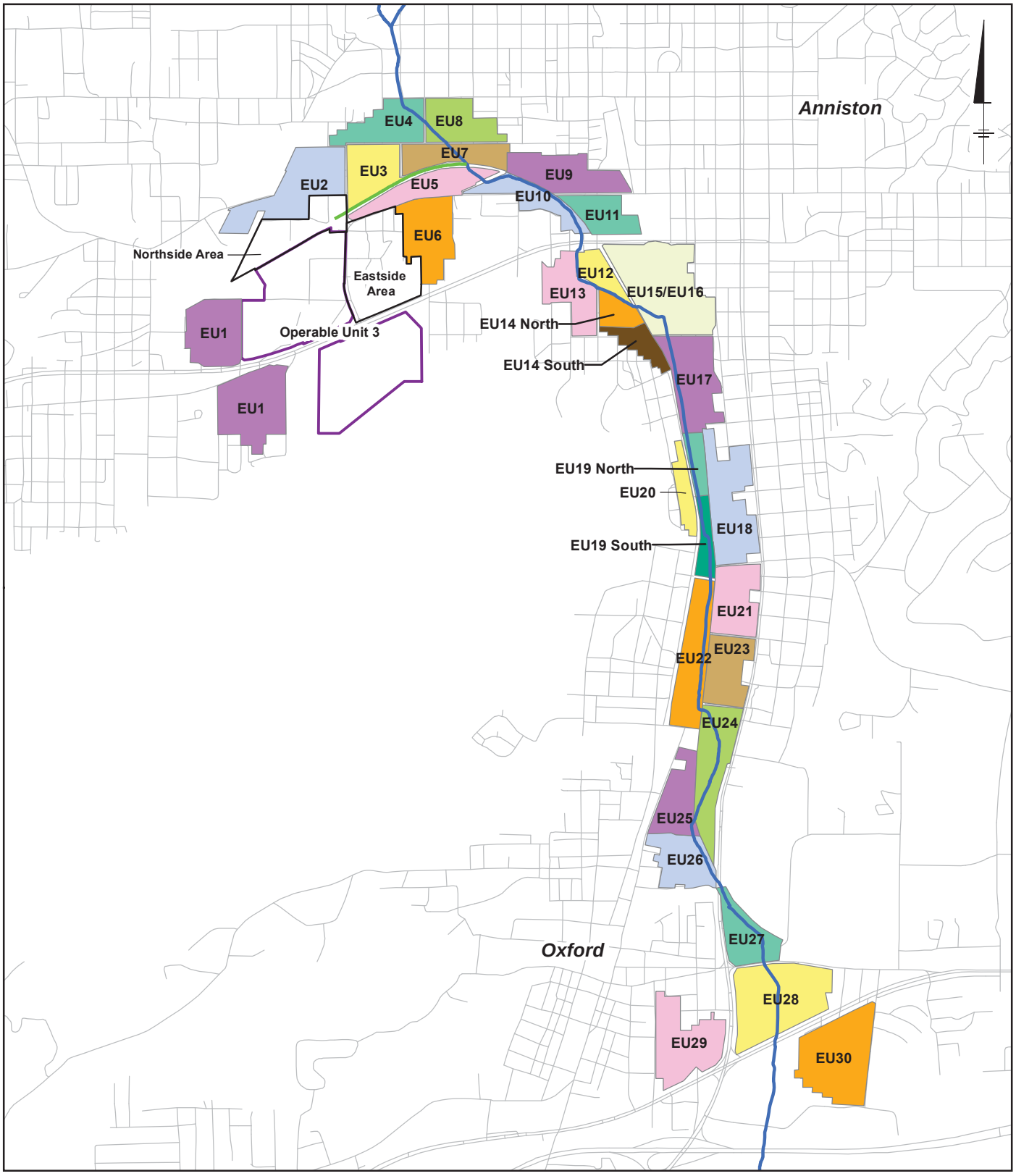
Anniston PCB Site
Anniston, Alabama

OU-1/OU-2 Predesign Investigation Work Plan

Nonresidential Subsurface Soil PCB Results

Solutia

Figure
2-7



0 2,750 5,500 Feet

GRAPHIC SCALE

Notes:
EU: exposure unit
OU: operable unit
PCB: polychlorinated biphenyl

Anniston PCB Site
Anniston, Alabama

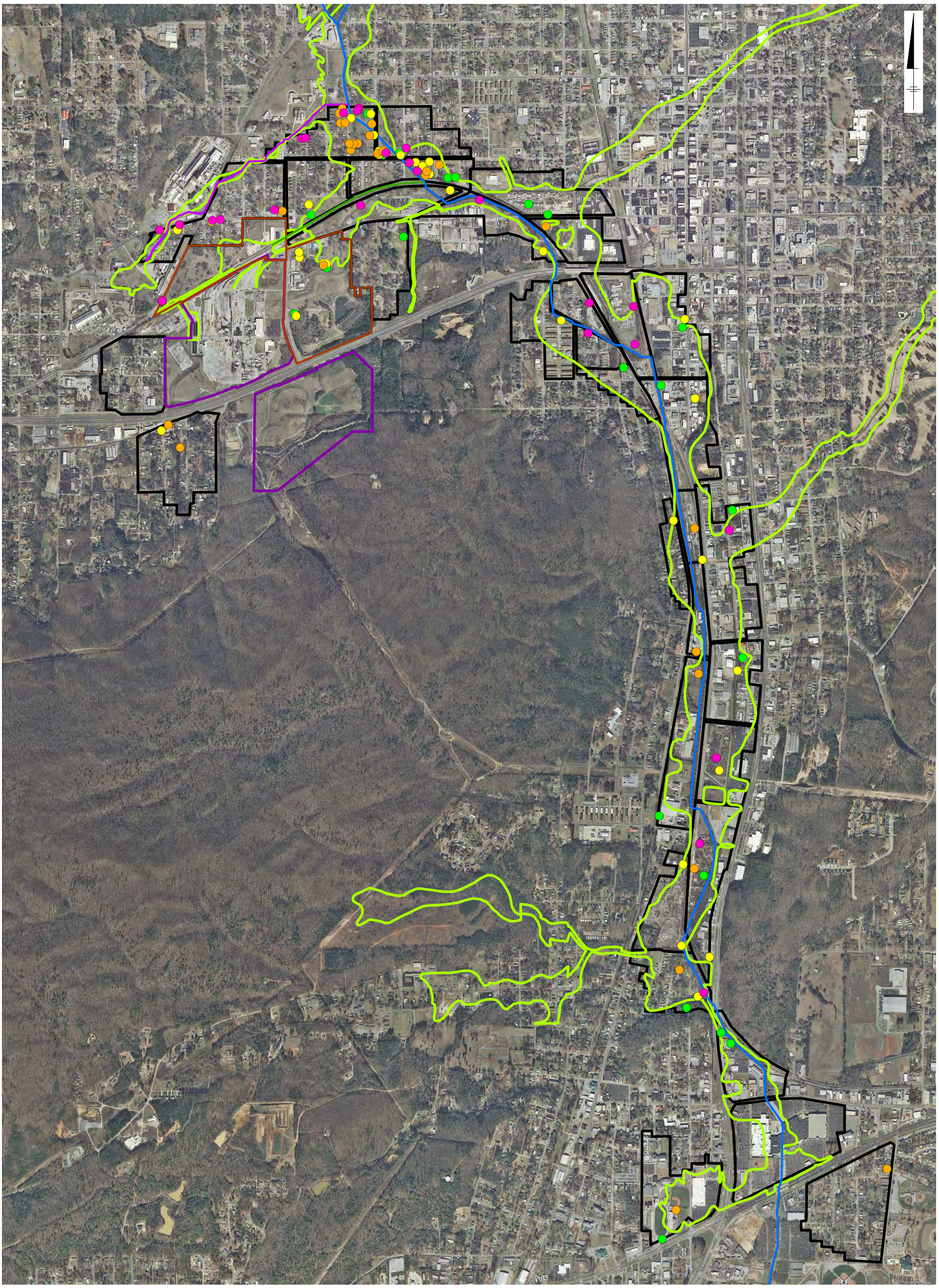
OU-1/OU-2 Predesign Invesigation Work Plan

Exposure Units

Solutia

Figure
2-8

City: SYR Div/Group: SWG Created By: Kives Last Saved By: ldrum
Anniston
Q:\Anniston_PCB_site\AnnistonALLMXDs_Printfiles\Reports\OU1_2_FeasibilityStudy\mxd\Figure2-3_ExposureUnits.mxd 3/5/2016 8:51:48 AM



Legend

Soil Sample Location

Benzo(a)pyrene Equivalents (mg/kg)

- ND - 0.090
- 0.090 - 0.360
- 0.360 - 0.720
- 0.720 - 2.50
- > 2.50

- Snow Creek
- 11th Street Ditch
- West 9th Street Creek
- Northside and Eastside Areas
- Operable Unit 3
- 100-Year Floodplain and Drainage Areas
- Exposure Unit

Notes:

1. mg/kg: milligrams per kilogram
   ND: nondetect
   OU: operable unit
   PAH: polycyclic aromatic hydrocarbon
2. Aerial imagery provided by Calhoun County.

0 1,600 3,200 Feet

Graphic Scale

Anniston PCB Site
Anniston, Alabama

OU-1/OU-2 Predesign Investigation Work Plan

PAH Concentrations in Surface Soil

Solutia

Figure
2-9

Legend

Soil Sample Location
Chromium Concentration (mg/kg)
- 0-20
- 20-38
- 38-50
- 50-100
- 100-500
- >500

- Snow Creek
- 11th Street Ditch
- West 9th Street Creek
- Northside and Eastside Areas
- Operable Unit 3
- 100-Year Floodplain and Drainage Areas
- Exposure Unit

Notes:
1. mg/kg: milligrams per kilogram
   OU: operable unit

2. Aerial imagery provided by Calhoun County.

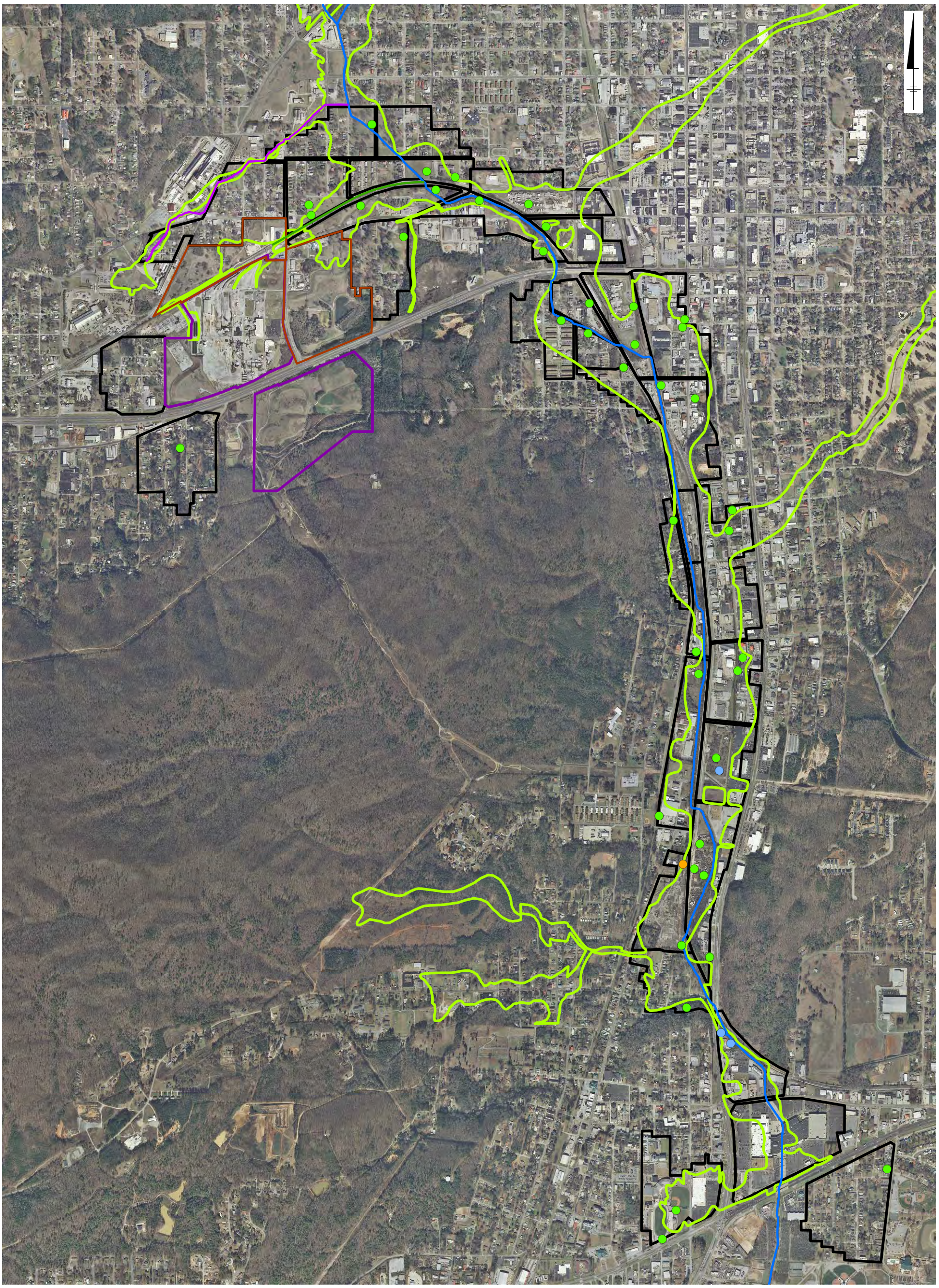
0 1,600 3,200 Feet
Graphic Scale

Anniston PCB Site
Anniston, Alabama
OU-1/OU-2 Predesign Investigation Work Plan

Chromium Concentrations in Surface Soil

Solutia

Figure
2-10



Legend

Soil Sample Location

PCDD/DF TEQ Concentration (µg/kg)

- < 0.001
- 0.001 - 0.25
- 0.25 - 2
- > 2

- Snow Creek
- 11th Street Ditch
- West 9th Street Creek
- Northside and Eastside Areas
- 100-Year Floodplain and Drainage Areas
- Operable Unit 3
- Exposure Unit

Notes:

1. OU: operable unit
   PCDD/DF: polychlorinated dibenzodioxins/dibenzofurans
   TEQ: toxic equivalency
   µg/kg: micrograms per kilogram
2. Aerial imagery provided by Calhoun County.

0 1,600 3,200 Feet

Graphic Scale

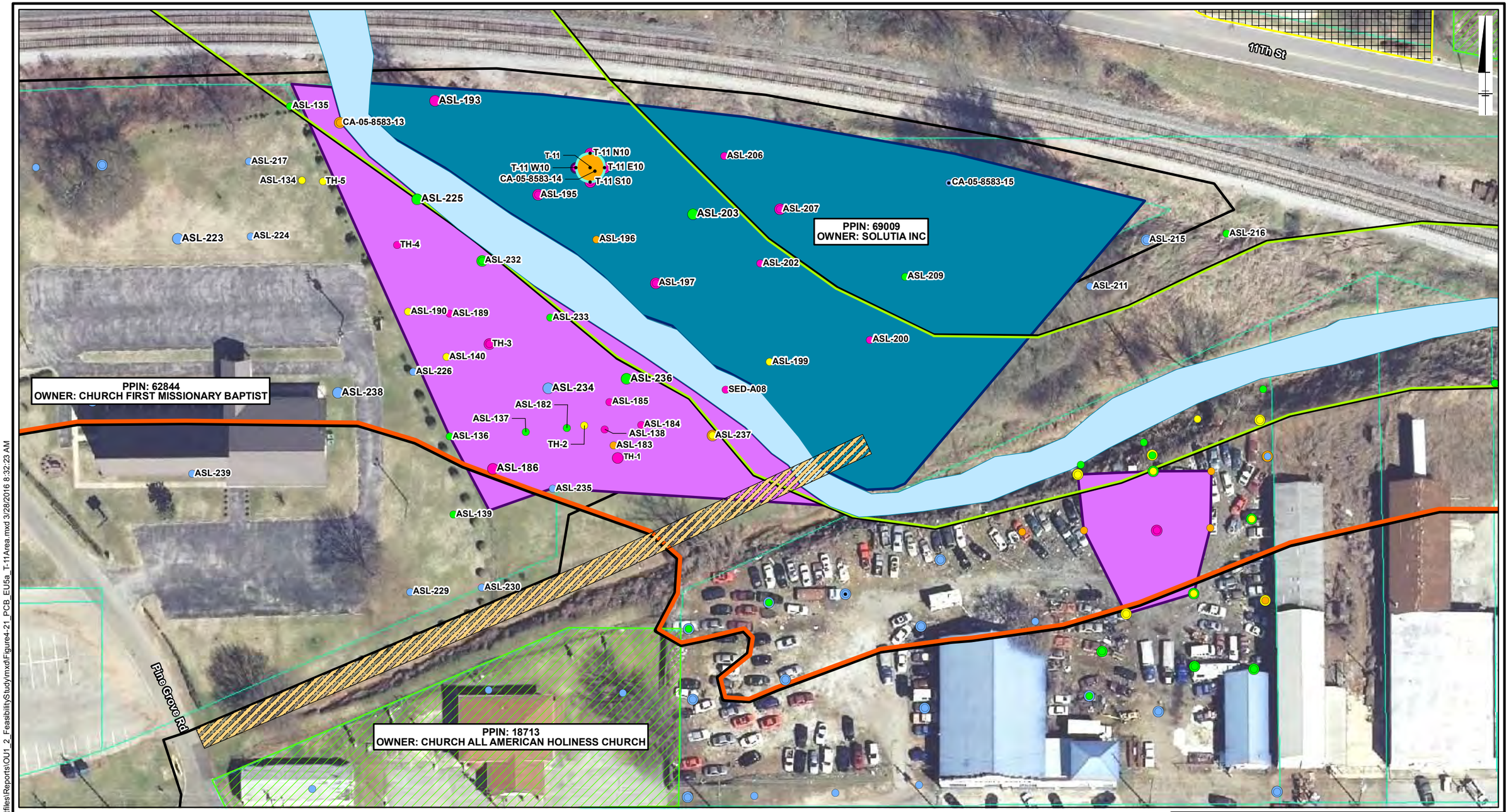
Anniston PCB Site
Anniston, Alabama

OU-1/OU-2 Predesign Investigation Work Plan

PCDD/DF TEQ Concentrations in Surface Soil

Solutia

Figure
2-11



City: SYR Div/Group: SWG Created By: K.IVES Last Saved By: jbistrovich
Anniston
Q:\Anniston_PCB_site\AnnistonALL\MXDs_Printfiles\Reports\OU1_2_FeasibilityStudy\mxd\Figure4-21_PCB_EU5a_T-11Area.mxd 3/28/2016 8:32:23 AM

Legend

Soil Sample Location
Surface PCB (mg/kg)
- ND or < 1
- ≥ 1 or <10
- ≥ 10 to < 25
- ≥ 25 to < 50
- ≥ 50 to < 500

- Snow Creek
- Analytes other than PCB and Lead Analyzed
- Potential Access Road
- Exposure Unit
- FEMA Floodway
- OU-1/OU-2 Downgradient Floodplain
- Parcel Boundaries
- Completed Removal Area
- Residential < 1 (mg/kg)
- Principal Threat Waste Area
- Nonresidential Soil Remediation Footprint
- T-11 Area Remediation Footprint

0 75 150 Feet
Graphic Scale

Inner and outer circles reflect surface and subsurface PCB concentrations, respectively.

Notes:
1. 2013 Aerial imagery provided by Calhoun County.
2. Surface concentrations depicted represent the average of data from the 0- to 1-foot interval.
3. Subsurface concentrations depicted represent the average of data from the 0- to 4-foot interval.
4. Bridge location is not finalized.
5. FEMA: Federal Emergency Management Agency
mg/kg: milligrams per kilogram
ND: nondetect
OU: operable unit
PCB: polychlorinated biphenyl
PPIN: Property Parcel Identification Number

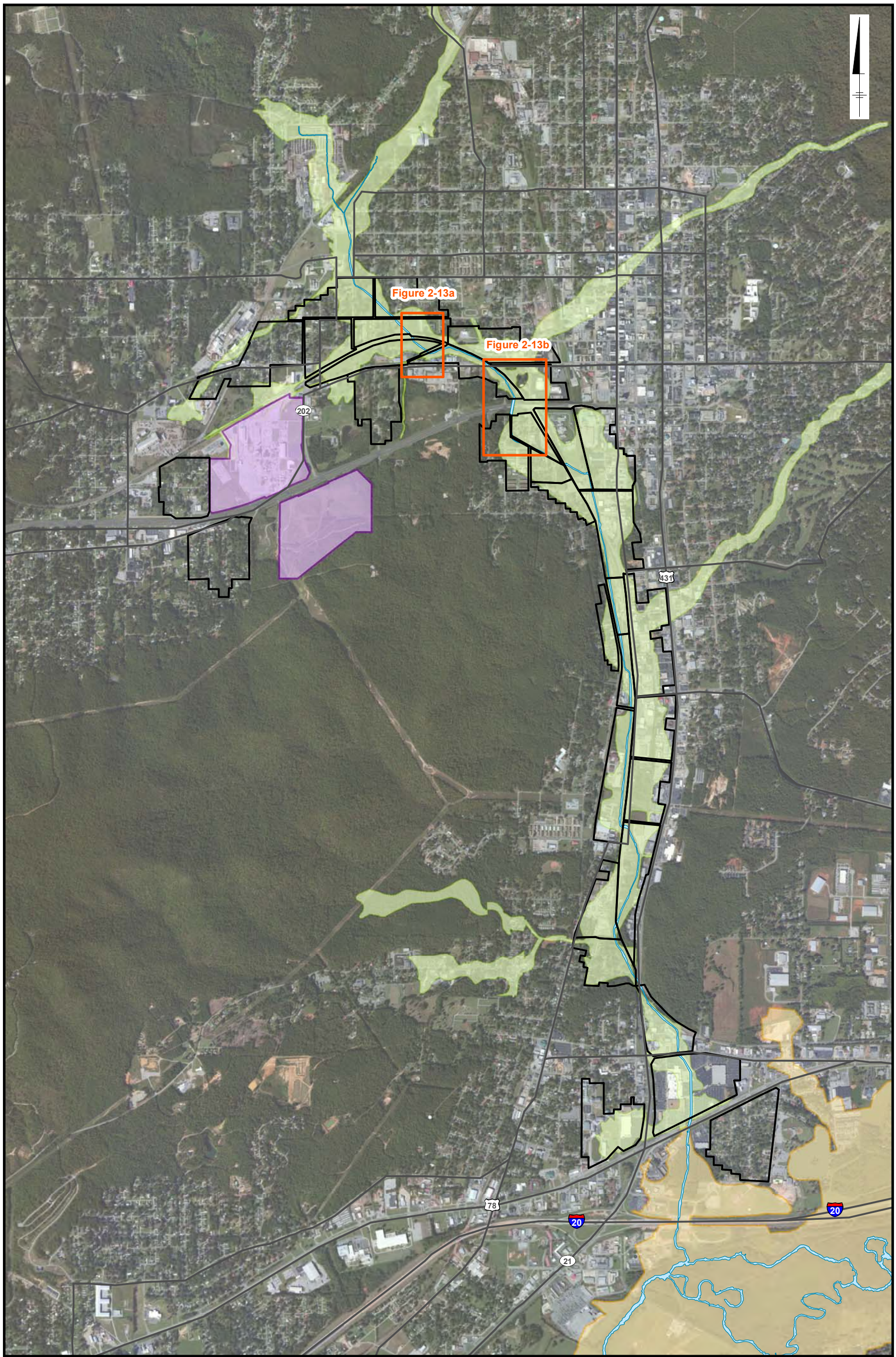
Anniston PCB Site: OU-1/OU-2
Anniston, Alabama

OU-1/OU-2 Predesign Investigation Work Plan

Target Remedial Area in Exposure Unit 5 - T-11 Area

Solutia

Figure
2-12



Legend

- Major Roads
- Figure Extent
- Exposure Unit
- Creek
- Operable Unit 3
- Snow Creek 100-Year Floodplain and Drainage
- OU-4 100-Year Floodplain

Notes:
1. Aerial imagery used is Clarity from ArcGIS.
2. OU: operable unit
   PCB: polychlorinated biphenyl

0 0.5 1 Miles

Graphic Scale

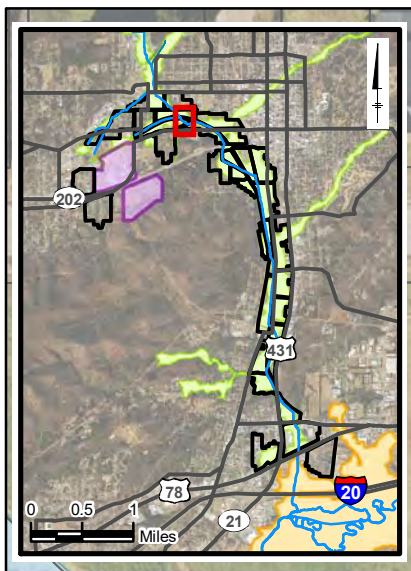
Anniston PCB Site
Anniston, Alabama
OU-1/OU-2 Predesign Investigation Work Plan

Snow Creek Banks with Stability Concerns - Site Map

Solutia

Figure **2-13**

City: SYR Div/Group: SWG Created By: K. Ives Last Saved By: cnelson
Anniston
N:\A\Anniston\GIS\MXD\20220202_OU1_OU2_PDIWP\Rev2\Fig2-13_PDIWP_OU1_OU2_BankStability_SiteMap_rev2.mxd 2/23/2022 2:18:01 PM



S-1-01		
Sample Name	Depth Interval (in)	Total PCB (mg/kg)
S10021	0 to 2	3.76 J
S10022	2 to 8	30.5 J

S-1-02		
Sample Name	Depth Interval (in)	Total PCB (mg/kg)
S10023	0 to 2	8

S-1-05		
Sample Name	Depth Interval (in)	Total PCB (mg/kg)
S10026	0 to 2	11.2 J

S-1-04		
Sample Name	Depth Interval (in)	Total PCB (mg/kg)
S10024	0 to 2	14
S10025	2 to 5	16.5

S-1-07		
Sample Name	Depth Interval (in)	Total PCB (mg/kg)
S10027	0 to 2	16.2 J
S10028	2 to 12	1.21 J
S10029	12 to 23	0.17 U

S-1-08		
Sample Name	Depth Interval (in)	Total PCB (mg/kg)
S10030	0 to 2	31.5 J
S10031	2 to 12	12.3
S10032 (duplicate)	2 to 12	4.3
S10033	12 to 14.5	37 J

S-1-10		
Sample Name	Depth Interval (in)	Total PCB (mg/kg)
S10034	0 to 2	11.6 J
S10035	2 to 12	28.5 J
S10036	12 to 16.5	18.3

S-1-12		
Sample Name	Depth Interval (in)	Total PCB (mg/kg)
S10041	0 to 2	0.67
S10042	2 to 5	2.08

S-1-11a		
Sample Name	Depth Interval (in)	Total PCB (mg/kg)
S10037	0 to 2	2.23
S10038	2 to 12	0.2 U
S10039	12 to 24	0.39 J

S-1-11b		
Sample Name	Depth Interval (in)	Total PCB (mg/kg)
S10040	0 to 2.5	11.8

Notes:
1. Aerial imagery provided by Calhoun and Talladega Counties.
2. Sediment sample locations are approximate.
3. Snow Creek deposits are based on field probing conducted in 1999.
4. Estimated PCB concentrations for sediment deposits with no sampling data were interpreted based on PCB data for nearby sediment samples.
5. in: inches
   mg/kg: milligrams per kilogram
   OU: operable unit
   PCB: polychlorinated biphenyl
   J: The compound was positively identified; however, the associated numerical value is an estimated concentration only.
   U: The compound was analyzed for but not detected. The associated value is the compound quantitation limit.

Legend

- Sediment Sample Location
- Engineered Bank Stability Measures
- Natural Bank Stability Measures
- 11th Street Ditch
- Snow Creek
- OU-1/OU-2 Downgradient Floodplain
- All Parcels

Sediment Deposit Average PCB Concentration (mg/kg)
- < 3
- ≥ 3

Sediment Deposit Estimated PCB Concentration (mg/kg) (see Note 4)
- ≥ 3

0 100 200 Feet
Graphic Scale

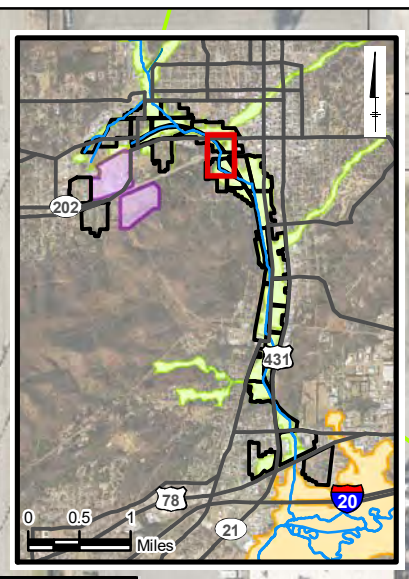
Anniston PCB Site
Anniston, Alabama
OU-1/OU-2 Predesign Investigation Work Plan

Snow Creek Banks with Stability Concerns

Solutia

Figure
2-13a

City: SYR Div/Group: SWG Created By: K. Ives Last Saved By: cnelson
Anniston
N:\A\Anniston\GIS\MXD\20220202_OU1_OU2_PDIWP\Rev2\Fig2-13a_PDIWP_OU1_OU2_BankStability_rev2.mxd 2/23/2022 2:21:06 PM



S-1-17

CP-C-9		
Sample Name	Depth Interval (in)	Total PCB (mg/kg)
CP-C-9-0-4"	0 to 4	2.04

S-2-02		
Sample Name	Depth Interval (in)	Total PCB (mg/kg)
S10045	0 to 3.5	18.9

SED-A10		
Sample Name	Depth Interval (in)	Total PCB (mg/kg)
SED-A10	0 to 3	6.9

CP-C-10		
Sample Name	Depth Interval (in)	Total PCB (mg/kg)
CP-C-10-0-2"	0 to 2	2.89
CP-C-10-2-6"	2 to 6	3.55
CP-C-10-6-12"	6 to 12	12.2 J

S-2-03a		
Sample Name	Depth Interval (in)	Total PCB (mg/kg)
S10046	0 to 3	3.82

CP-C-11		
Sample Name	Depth Interval (in)	Total PCB (mg/kg)
CP-C-11-0-2"	0 to 2	1.55
CP-C-11-2-6"	2 to 6	1.18
CP-C-11-6-12"	6 to 12	7.51

S-2-05		
Sample Name	Depth Interval (in)	Total PCB (mg/kg)
S10048	0 to 2	5.35
S10049	2 to 5	6.4

S-MED-1		
Sample Name	Depth Interval (in)	Total PCB (mg/kg)
S10134	0 to 8	60

S-2-06A		
Sample Name	Depth Interval (in)	Total PCB (mg/kg)
S10060	0 to 2	21.7
S10061	2 to 5	8.9

S-2-06B		
Sample Name	Depth Interval (in)	Total PCB (mg/kg)
S10053	0 to 2	12.8
S10054	2 to 12	11.4
S10055	12 to 20.5	34.3
S10131 (duplicate)	12 to 20.5	60

S-HIGH-1		
Sample Name	Depth Interval (in)	Total PCB (mg/kg)
S10135	0 to 18	4.22 J
S10137 (duplicate)	0 to 18	2.32 J

S-2-06C		
Sample Name	Depth Interval (in)	Total PCB (mg/kg)
S10056	0 to 2	30.1
S10057	2 to 12	13.9
S10058	12 to 24	23.2
S10059	24 to 27	15.3

S-2-08		
Sample Name	Depth Interval (in)	Total PCB (mg/kg)
S10050	0 to 2	20.3
S10051	2 to 12	19.9
S10052	12 to 16	4

SED-A11		
Sample Name	Depth Interval (in)	Total PCB (mg/kg)
SED-A11	0 to 3	15.6

PECON-011		
Sample Name	Depth Interval (in)	Total PCB (mg/kg)
PECON-011	0 to 3	4.29

PECON-012		
Sample Name	Depth Interval (in)	Total PCB (mg/kg)
PECON-012	0 to 3	8.46

S-2-01
S-2-02
S-2-03B
S-2-03A
S-2-04A
S-2-04B
S-2-05
S-2-06
S-2-08
S-2-07
S-2-09
S-2-10
S-2-11
S-2-12
S-2-13
S-2-14

Notes:
1. Aerial imagery provided by Calhoun and Talladega Counties.
2. Sediment sample locations are approximate.
3. Snow Creek deposits are based on field probing conducted in 1999.
4. Estimated PCB concentrations for sediment deposits with no sampling data were interpreted based on PCB data for nearby sediment samples.
5. in: inches
   mg/kg: milligrams per kilogram
   OU: operable unit
   PCB: polychlorinated biphenyl
   PRG: preliminary remediation goal
   J: The compound was positively identified; however, the associated numerical value is an estimated concentration only.
6. Metals results from deposit S-2-03a exceeded PRG but the corresponding PCB concentration was greater than 3 mg/kg and less than 10 mg/kg.

Legend

- Sediment Sample Location
- Natural Bank Stability Measures
- Snow Creek
- OU-1/OU-2 Downgradient Floodplain
- All Parcels

Sediment Deposit Average PCB Concentration (mg/kg)
- ≥ 3

Sediment Deposit Estimated PCB Concentration (mg/kg) (see Note 4)
- ≥ 3

0 150 300 Feet
Graphic Scale

Anniston PCB Site
Anniston, Alabama
OU-1/OU-2 Predesign Investigation Work Plan

Snow Creek Banks with Stability Concerns

Solutia

Figure
2-13b

City: SYR Div/Group: SWG Created By: K. Ives Last Saved By: cnelson
Anniston
N:\A\Anniston\GIS\MXD\20220202_OU1_OU2_PDIWP\Rev2\Fig2-13b_PDIWP_OU1_OU2_BankStability_rev2.mxd 2/24/2022 8:10:36 AM

Legend

Total PCB Aroclor Concentrations in Soil

(mg/kg) (See Note 2)

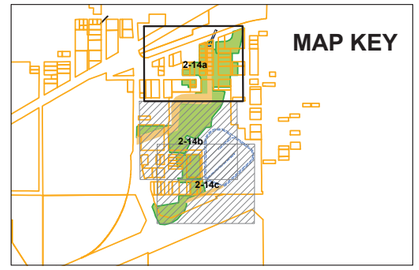
- <1 (Including nondetects)
- ≥1 to <10
- ≥10 to <25
- ≥25 to <50
- ≥50 to <500
- ≥500

Total PCB Screening Result (Immunoassay)

(ppm) (See Note 2)

- <1; <2; <5; <10
- <50
- >10; >25
- >1; >5
- >50

- OU-3 Boundary
- Solutia-Owned Parcels
- Eastside Area Boundary
- OU-1/OU-2 Residential Property
- OU-1/OU-2 Exposure Units
- Active Groundwater Monitoring Wells
- Fence
- Box Culverts
- Ditch
- Drainage Basin
- Soil-Geotextile Cover
- Soil-HDPE Geomembrane Liner (See Note 1)
- Emergency Spillway
- Concrete Inlet
- Clean-Out
- Manhole
- Pipe Inlet Area
- Outfall
- Berm
- Stormwater Piping



Notes

1. This includes areas where the HDPE liner is interpreted to be present based on field observations and discussions with parties involved in the IM construction.

2. Figure presents analytical and screening PCB data in the Eastside Area. Maximum PCB concentration at any depth in analysis profile shown for each location. Screening data is presented where no analytical data is available.

3. Where IM cover systems were constructed, the data shown represent conditions below the cover, except for samples: PC-023-A, PC-023-B, PC-024-B, 2506-3A, CA-06-2136-03, PA-126-A and PA-126-B.

AL: Alabama
AVE: avenue
DR: drive
HDPE: high density polyethylene
HWY: highway
IM: interim measure
LN: lane
mg/kg: milligrams per kilogram
OU: operable unit
PCB: polychlorinated biphenyl
ppm: parts per million
ST: street

0 25 50 100 Feet

Anniston PCB Site
Anniston, Alabama
OU-1/OU-2 Predesign Investigation Work Plan

Eastside Area
Interim Measures and
Total PCB Concentrations in Soil

Solutia
Ingenuity Productivity Performance

Figure 2-14a

Legend

Total PCB Aroclor Concentrations in Soil

(mg/kg) (See Note 2)

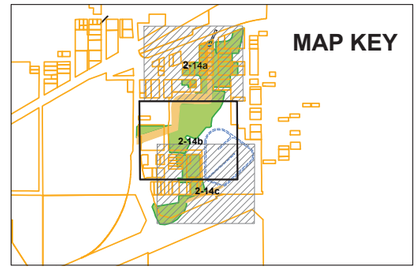
- <1 (Including nondetects)
- ≥1 to <10
- ≥10 to <25
- ≥25 to <50
- ≥50 to <500
- ≥500

Total PCB Screening Result (Immunoassay)

(ppm) (See Note 2)

- <1; <2; <5; <10
- <50
- >10; >25
- >1; >5
- >50

- OU-3 Boundary
- Solutia-Owned Parcels
- Eastside Area Boundary
- OU-1/OU-2 Residential Property
- OU-1/OU-2 Exposure Units
- Active Groundwater Monitoring Wells
- Fence
- Box Culverts
- Ditch
- Drainage Basin
- Soil-Geotextile Cover
- Soil-HDPE Geomembrane Liner (See Note 1)
- Emergency Spillway
- Concrete Inlet
- Clean-Out
- Manhole
- Pipe Inlet Area
- Outfall
- Berm
- Stormwater Piping



Notes

1. This includes areas where the HDPE liner is interpreted to be present based on field observations and discussions with parties involved in the IM construction.

2. Figure presents analytical and screening PCB data in the Eastside Area. Maximum PCB concentration at any depth in analysis profile shown for each location. Screening data is presented where no analytical data is available.

3. Where IM cover systems were constructed, the data shown represent conditions below the cover, except for samples: PC-023-A, PC-023-B, PC-024-B, 2506-3A, CA-06-2136-03, PA-126-A and PA-126-B.

AL: Alabama
AVE: avenue
DR: drive
HDPE: high density polyethylene
HWY: highway
IM: interim measure
LN: lane
mg/kg: milligrams per kilogram
OU: operable unit
PCB: polychlorinated biphenyl
ppm: parts per million
ST: street

0 25 50 100 Feet

Anniston PCB Site
Anniston, Alabama
OU-1/OU-2 Predesign Investigation Work Plan

Eastside Area
Interim Measures and
Total PCB Concentrations in Soil

Solutia
Ingenuity Productivity Performance

Figure 2-14b



Legend

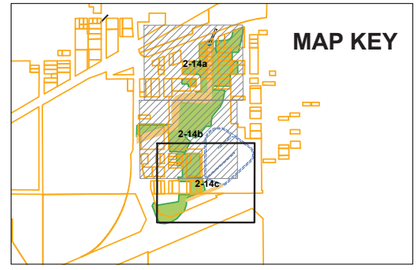
Total PCB Aroclor Concentrations in Soil (mg/kg) (See Note 2)

- <1 (Including nondetects)
- ≥1 to <10
- ≥10 to <25
- ≥25 to <50
- ≥50 to <500
- ≥500

Total PCB Screening Result (Immunoassay) (ppm) (See Note 2)

- <1; <2; <5; <10
- <50
- >10; >25
- >1; >5
- >50

- OU-3 Boundary
- Solutia-Owned Parcels
- Eastside Area Boundary
- OU-1/OU-2 Residential Property
- OU-1/OU-2 Exposure Units
- Active Groundwater Monitoring Wells
- Fence
- Box Culverts
- Ditch
- Drainage Basin
- Soil-Geotextile Cover
- Soil-HDPE Geomembrane Liner (See Note 1)
- Emergency Spillway
- Concrete Inlet
- Clean-Out
- Manhole
- Pipe Inlet Area
- Outfall
- Berm
- Stormwater Piping



Notes

1. This includes areas where the HDPE liner is interpreted to be present based on field observations and discussions with parties involved in the IM construction.

2. Figure presents analytical and screening PCB data in the Eastside Area. Maximum PCB concentration at any depth in analysis profile shown for each location. Screening data is presented where no analytical data is available.

3. Where IM cover systems were constructed, the data shown represent conditions below the cover, except for samples: PC-023-A, PC-023-B, PC-024-B, 2506-3A, CA-06-2136-03, PA-126-A and PA-126-B.

AL: Alabama
AVE: avenue
DR: drive
HDPE: high density polyethylene
HWY: highway
IM: interim measure
LN: lane
mg/kg: milligrams per kilogram
OU: operable unit
PCB: polychlorinated biphenyl
ppm: parts per million
ST: street

0 25 50 100 Feet

Anniston PCB Site
Anniston, Alabama
OU-1/OU-2 Predesign Investigation Work Plan

Eastside Area
Interim Measures and
Total PCB Concentrations in Soil

Solutia
Ingenuity Productivity Performance

Figure 2-14c

Legend

Sediment Sample Location Program
- 9th Street Ditch
- Anniston Lead Site
- EPA Programs
- Miscellaneous Programs
- Nonresidential OU-1/OU-2 FSP
- RFI Sediment

- Snow Creek
- 11th Street Ditch
- West 9th Street Creek
- Operable Unit 3
- 100-Year Floodplain and Drainage Areas
- Exposure Unit

Notes:
1. FSP: field sampling plan
   OU: operable unit
   RCRA: Resource Conservation and Recovery Act
   RFI: RCRA Facility Investigation
   USEPA: United States Environmental Protection Agency
2. Aerial imagery provided by Calhoun County.

0 1,600 3,200 Feet

Graphic Scale

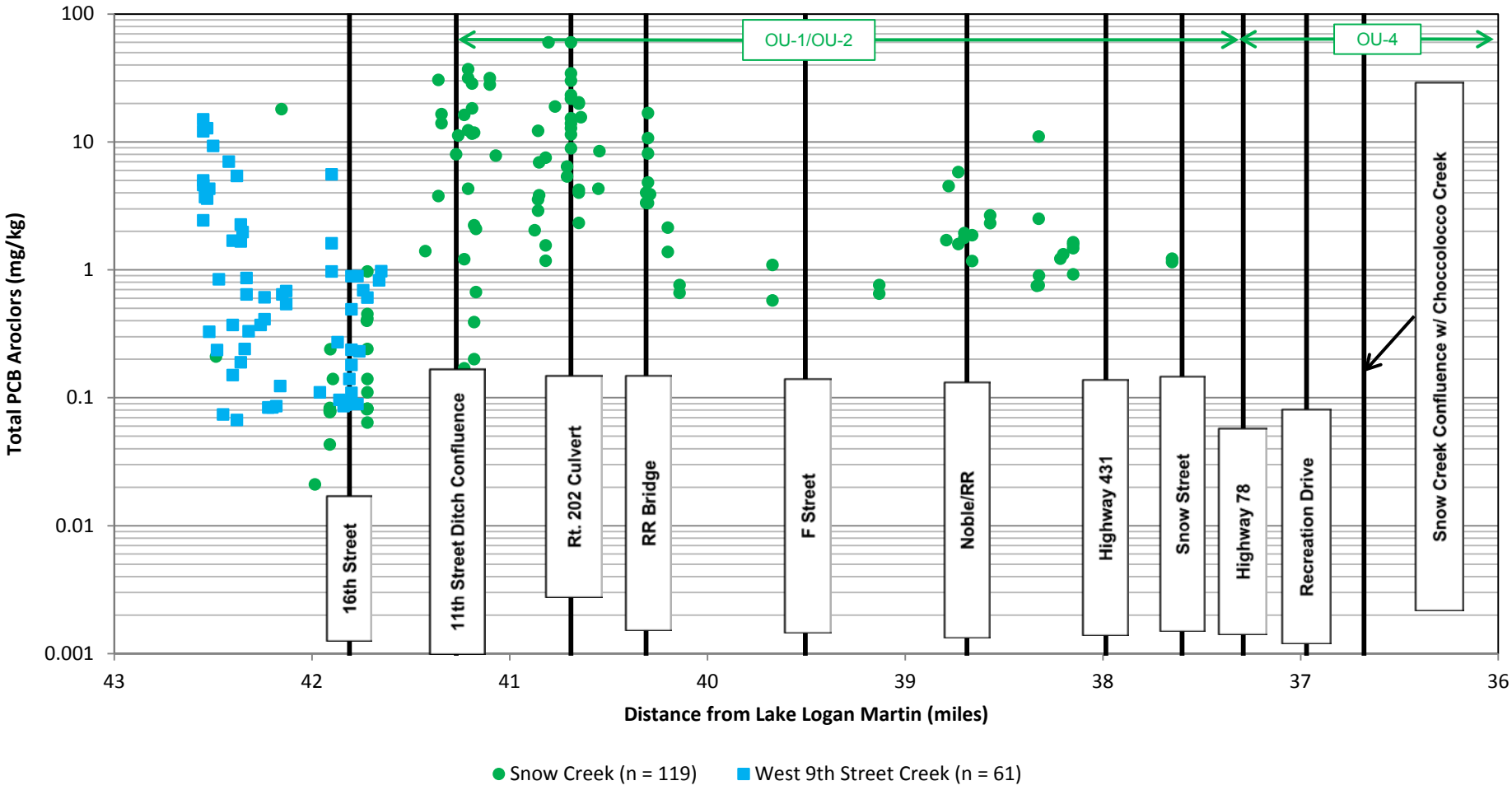
Anniston PCB Site
Anniston, Alabama

OU-1/OU-2 Predesign Investigation Work Plan

Sediment Sample Locations

Solutia

Figure
2-15



Notes:

1. Data include results from Snow Creek OU-1/OU-2 (n = 96) and Snow Creek upstream of 11th Street (n = 23).
2. Data include West 9th Street Creek Sediment Data (n = 61).
3. For total PCB Aroclor calculation, nondetects were given a value of zero; if all PCB Aroclors were nondetect, then the maximum individual reporting limit was utilized.

mg/kg: milligrams per kilogram
n: sample count
OU: operable unit
PCB: polychlorinated biphenyl
RR: railroad

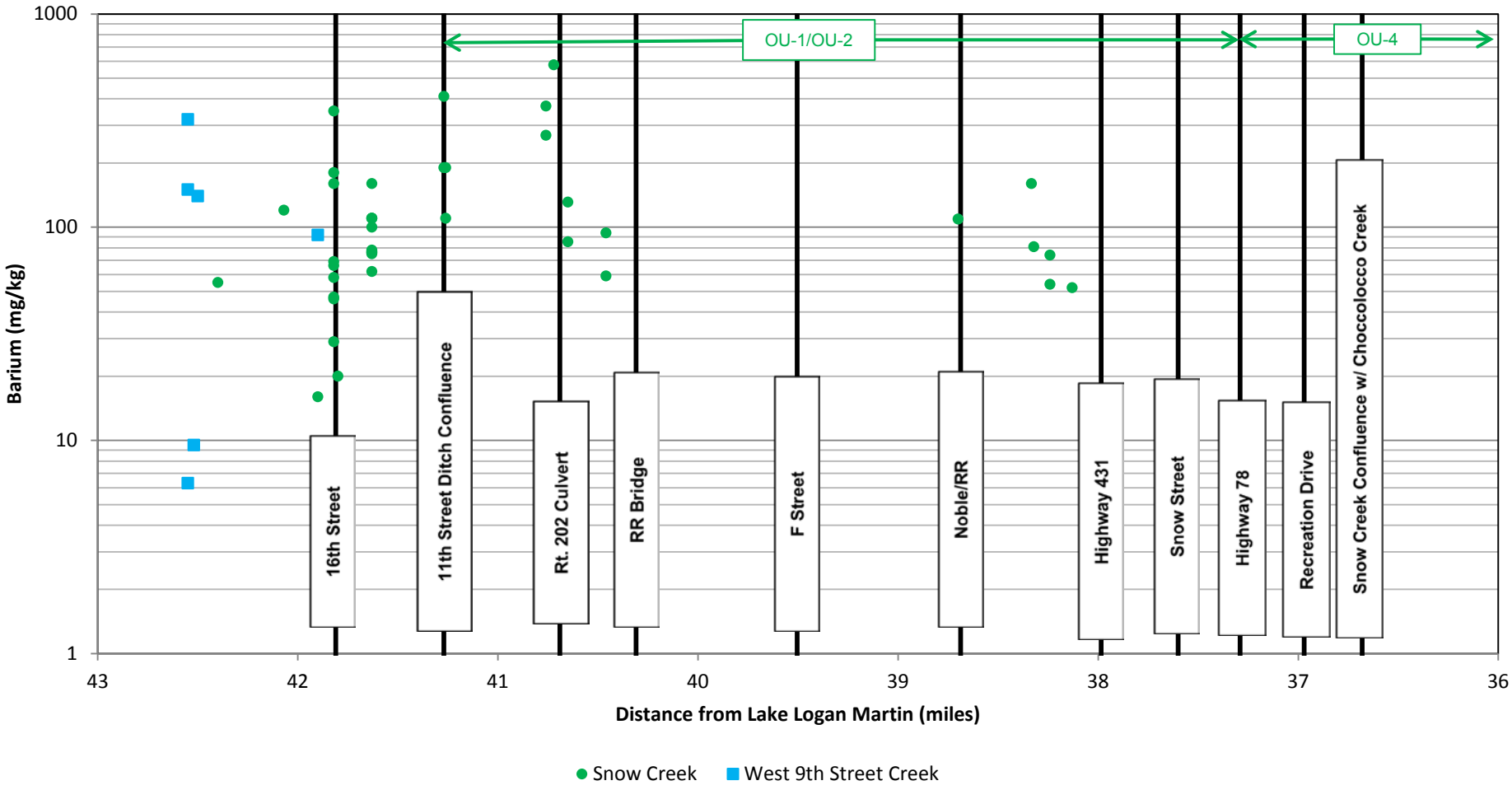
Anniston PCB Site
Anniston, Alabama

OU-1/OU-2 Predesign Investigation Work Plan

Total PCB Concentrations in Snow Creek Sediment with Distance from Lake Logan Martin

Solutia
Ingenuity Productivity Performance

FIGURE
2-16



Notes:
mg/kg: milligrams per kilogram
OU: operable unit
RR: railroad
Rt.: route

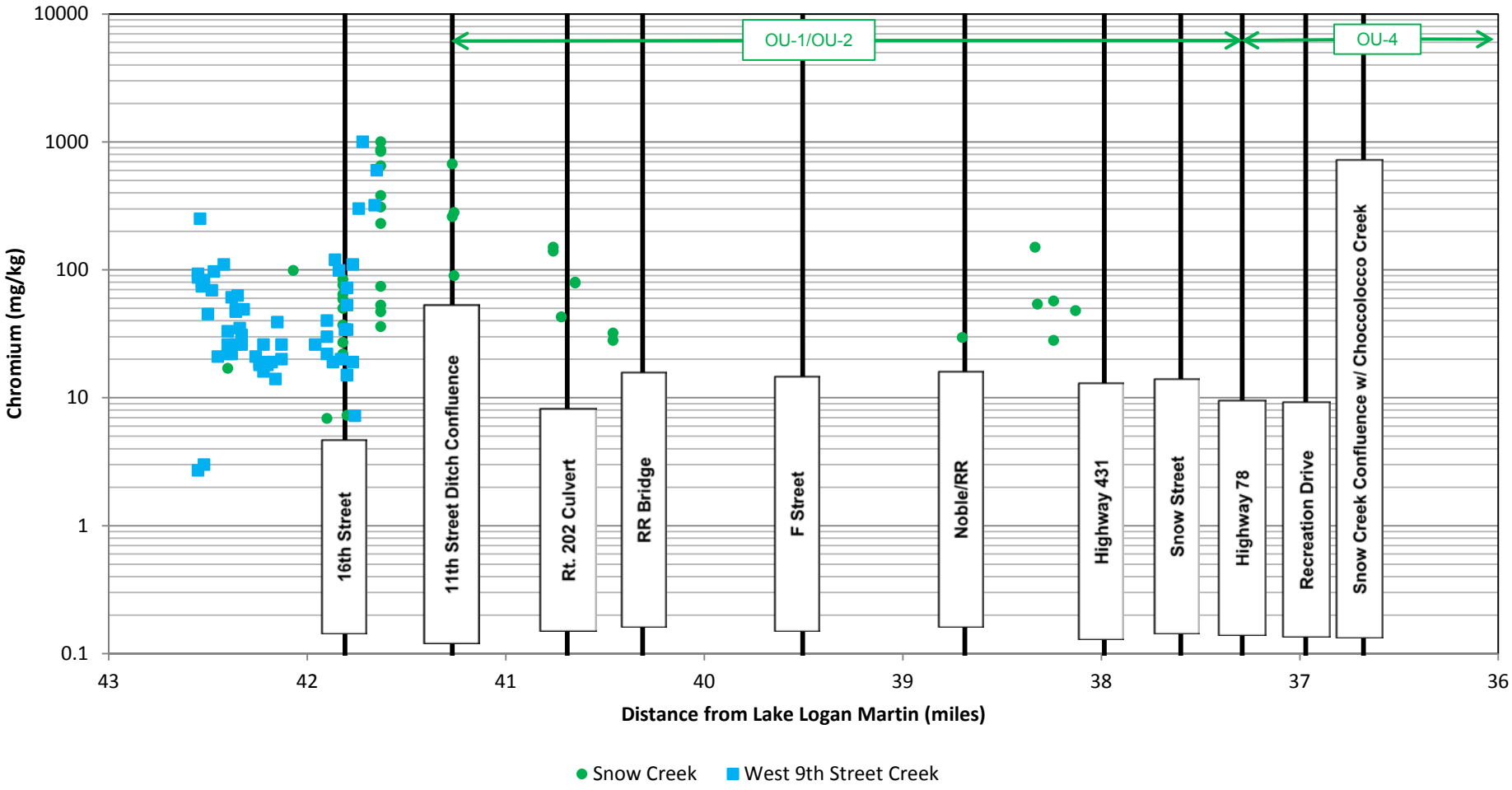
Anniston PCB Site
Anniston, Alabama

OU-1/OU-2 Predesign Investigation Work Plan

Barium Concentrations in Snow Creek Sediment with Distance from Lake Logan Martin

Solutia
Ingenuity Productivity Performance

FIGURE
2-17



Notes:
mg/kg: milligrams per kilogram
OU: operable unit
RR: railroad
Rt.: route

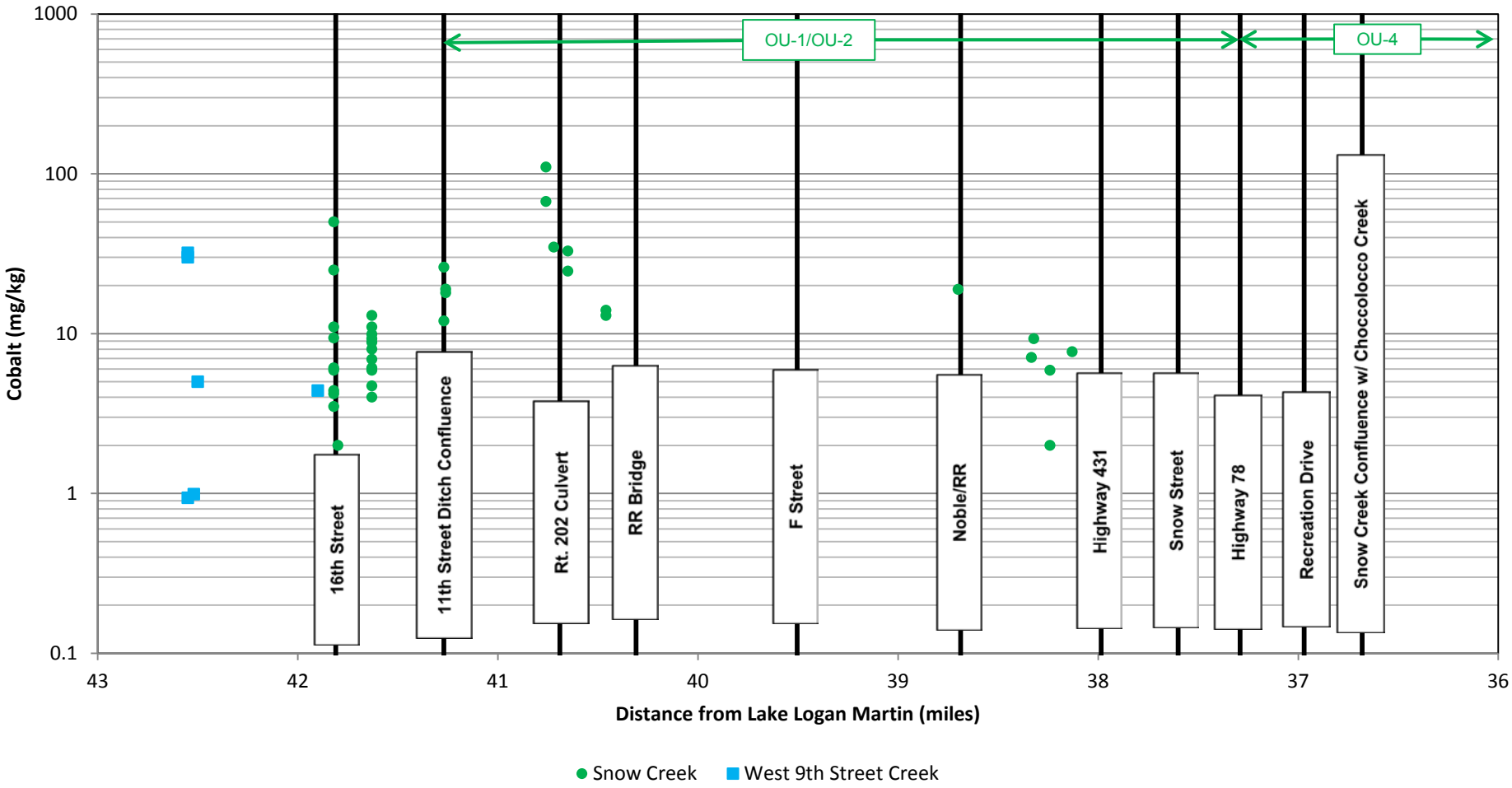
Anniston PCB Site
Anniston, Alabama

OU-1/OU-2 Predesign Investigation Work Plan

Chromium Concentrations in Snow Creek Sediment with Distance from Lake Logan Martin

Solutia
Ingenuity Productivity Performance

FIGURE
2-18



Notes:
mg/kg: milligrams per kilogram
OU: operable unit
RR: railroad
Rt.: route

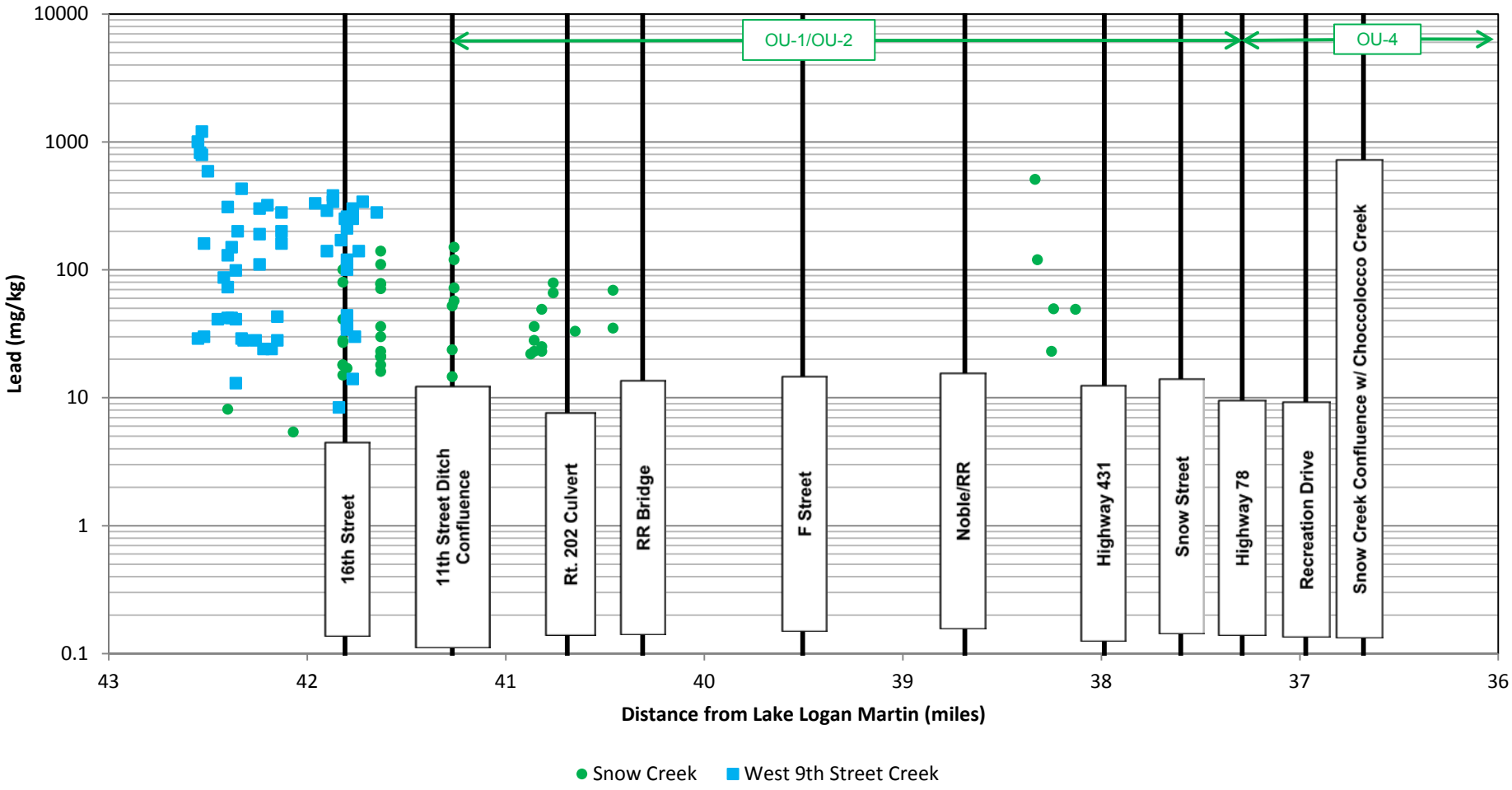
Anniston PCB Site
Anniston, Alabama

OU-1/OU-2 Predesign Investigation Work Plan

Cobalt Concentrations in Snow Creek Sediment with Distance from Lake Logan Martin

Solutia
Ingenuity Productivity Performance

FIGURE
2-19



Notes:
mg/kg: milligrams per kilogram
OU: operable unit
RR: railroad
Rt.: route

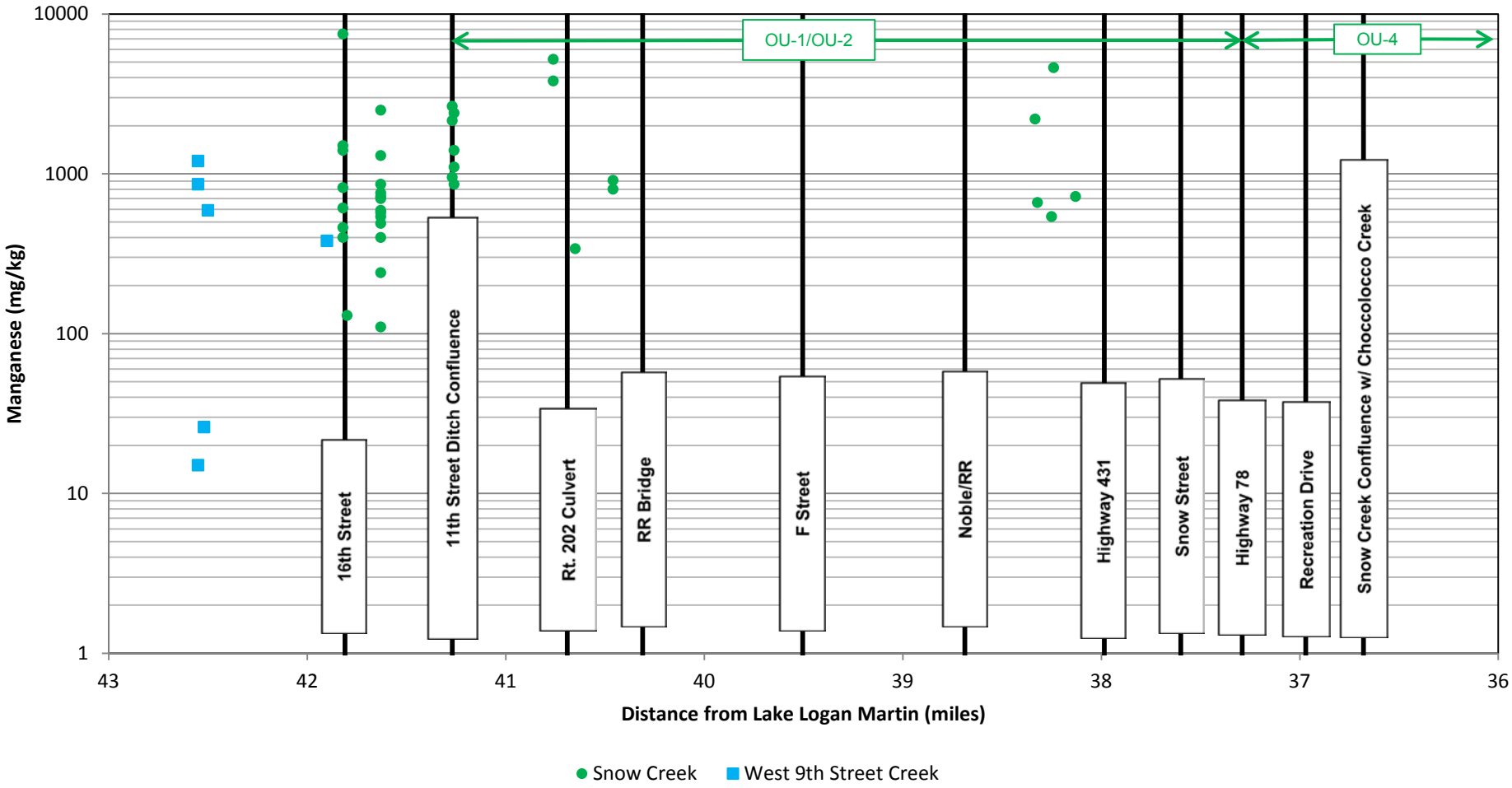
Anniston PCB Site
Anniston, Alabama

OU-1/OU-2 Predesign Investigation Work Plan

Lead Concentrations in Snow Creek Sediment with Distance from Lake Logan Martin

Solutia
Ingenuity Productivity Performance

FIGURE
2-20



Notes:
mg/kg: milligrams per kilogram
OU: operable unit
RR: railroad
Rt.: route

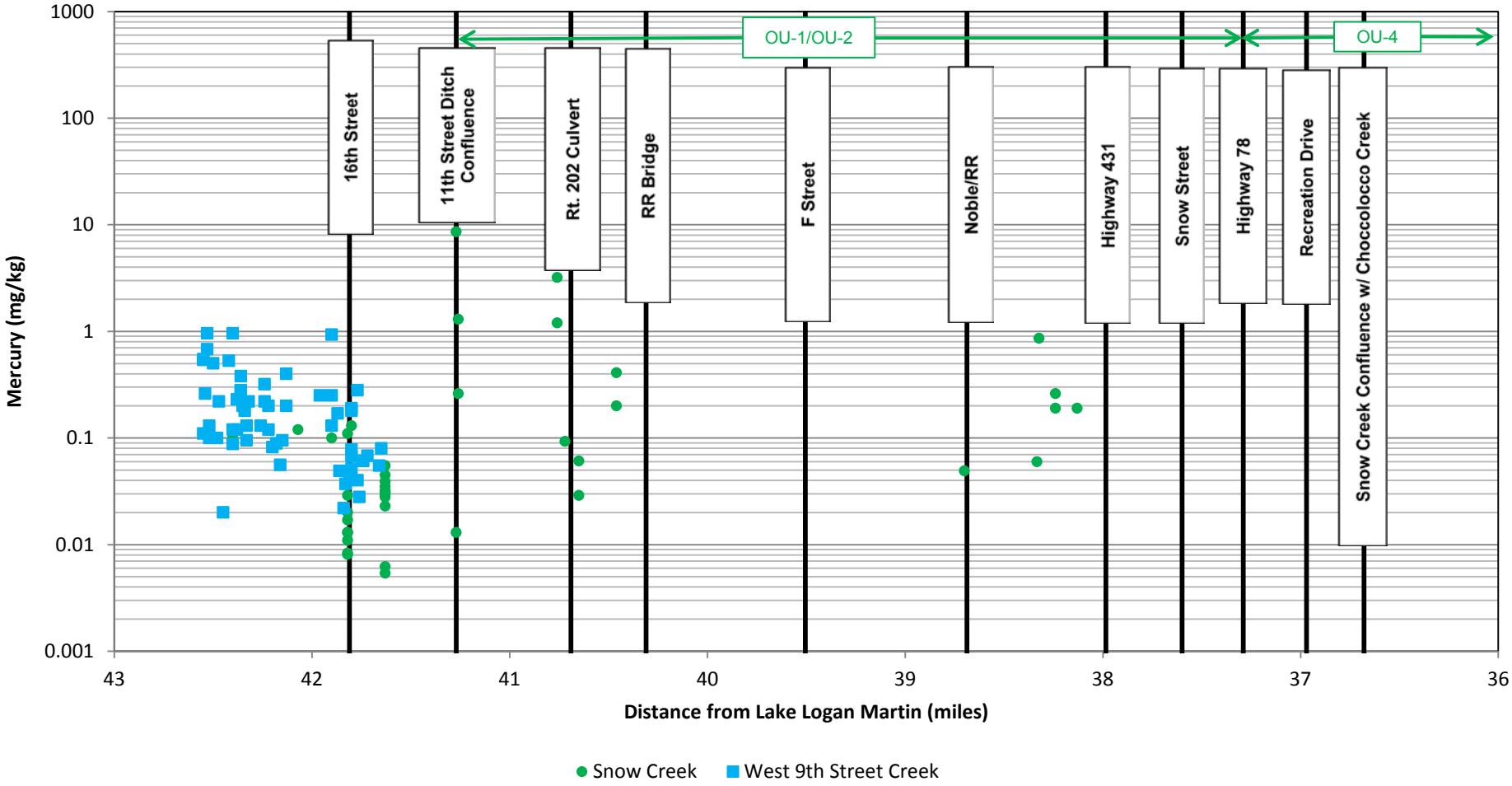
Anniston PCB Site
Anniston, Alabama

OU-1/OU-2 Predesign Investigation Work Plan

Manganese Concentrations in Snow Creek Sediment with Distance from Lake Logan Martin

Solutia
Ingenuity Productivity Performance

FIGURE
2-21



Notes:
mg/kg: milligrams per kilogram
OU: operable unit
RR: railroad
Rt.: route

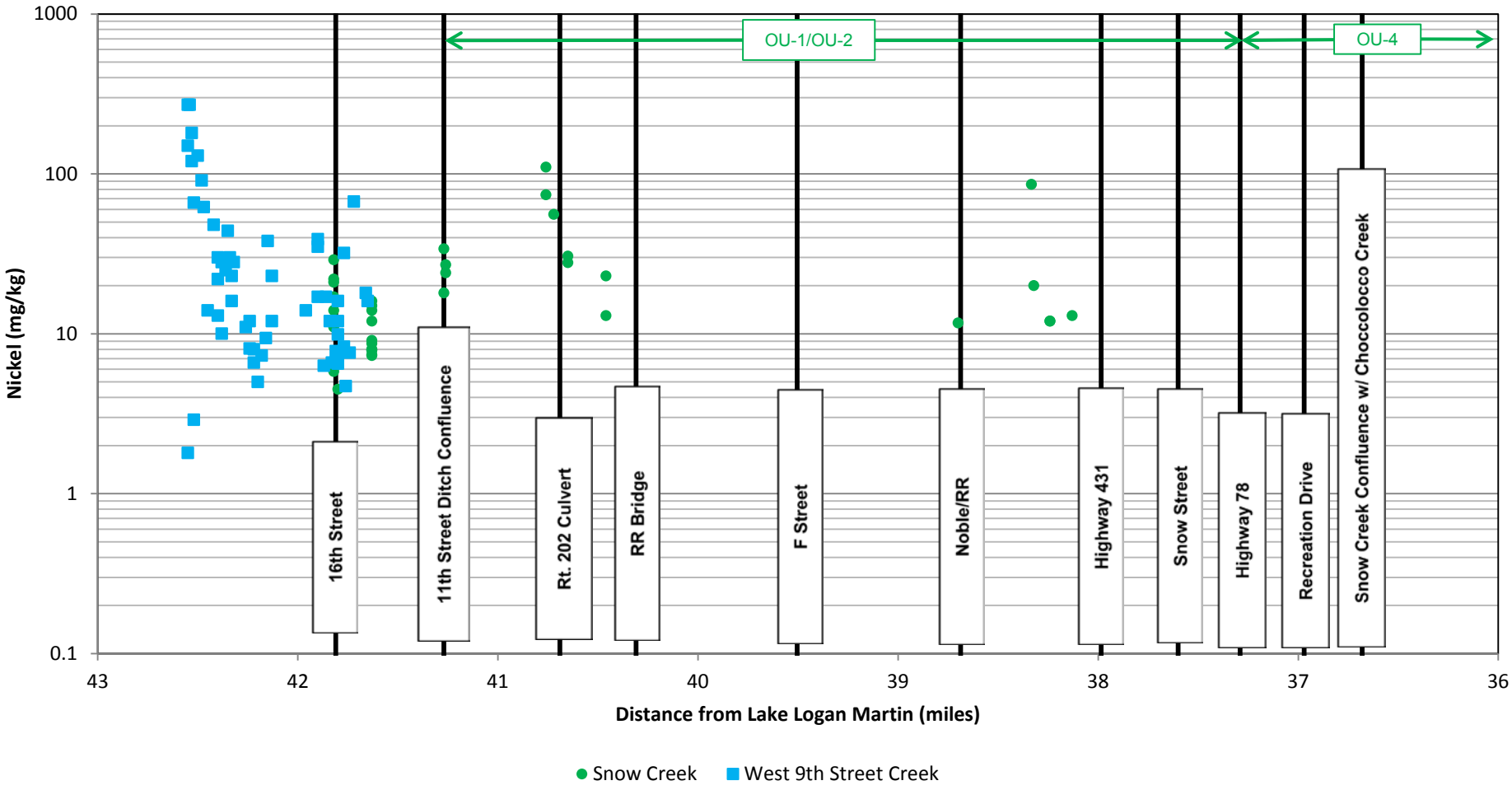
Anniston PCB Site
Anniston, Alabama

OU-1/OU-2 Predesign Investigation Work Plan

Mercury Concentrations in Snow Creek Sediment with Distance from Lake Logan Martin

Solutia
Ingenuity Productivity Performance

FIGURE
2-22



Notes:
mg/kg: milligrams per kilogram
OU: operable unit
RR: railroad
Rt.: route

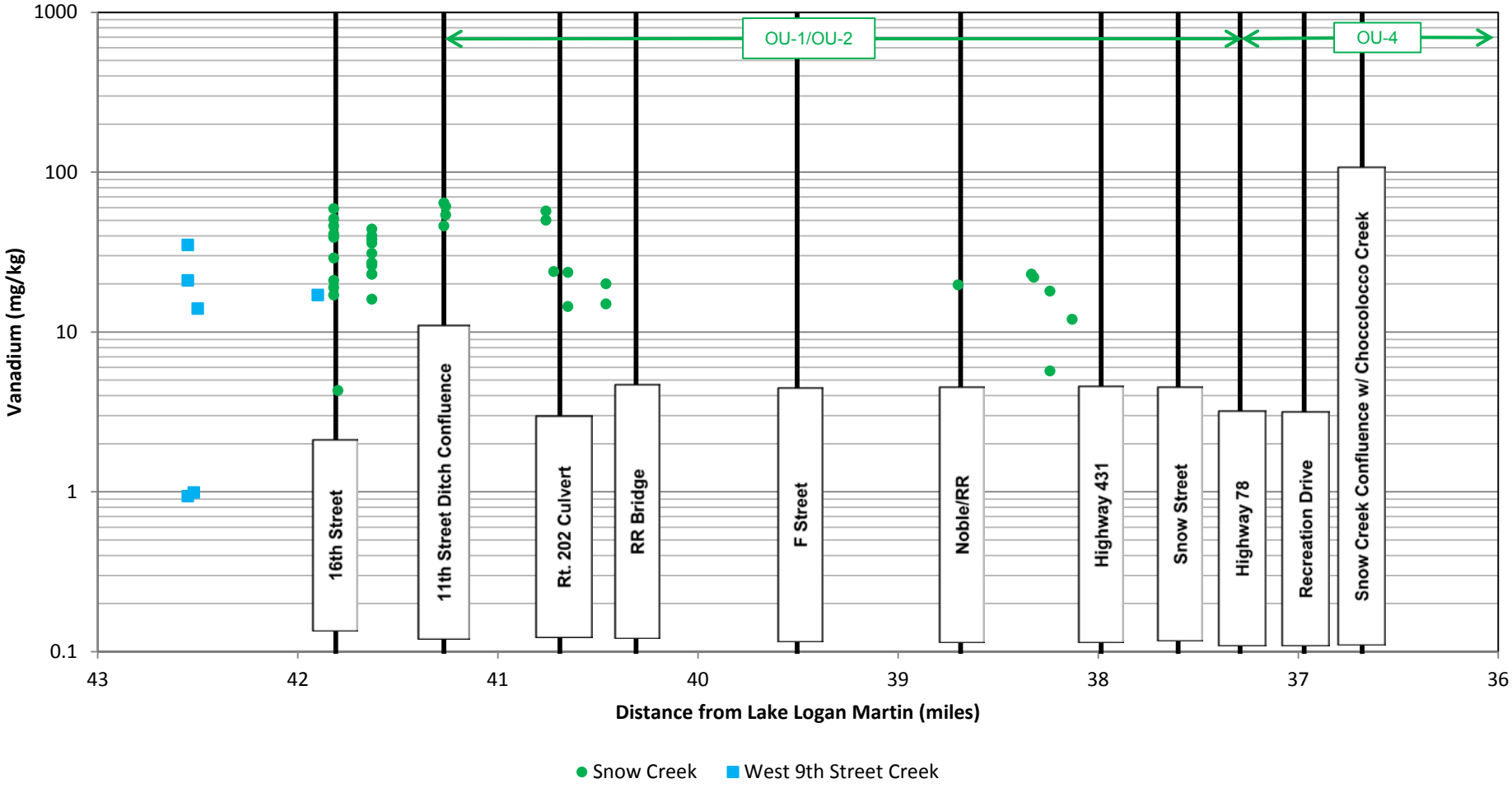
Anniston PCB Site
Anniston, Alabama

OU-1/OU-2 Predesign Investigation Work Plan

Nickel Concentrations in Snow Creek Sediment with Distance from Lake Logan Martin

Solutia
Ingenuity Productivity Performance

FIGURE
2-23



Notes:
mg/kg: milligrams per kilogram
OU: operable unit
RR: railroad
Rt.: route

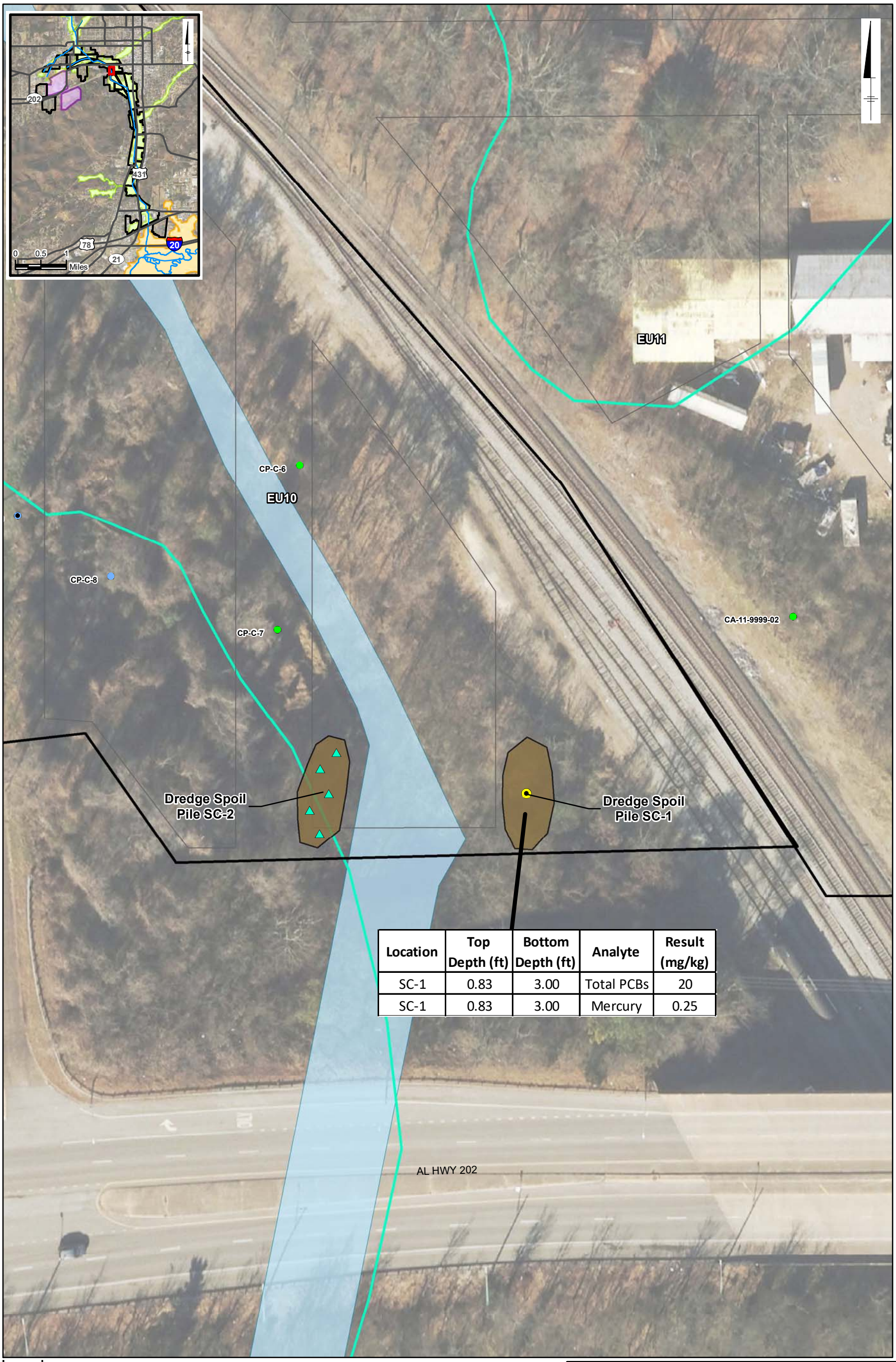
Anniston PCB Site
Anniston, Alabama

OU-1/OU-2 Predesign Investigation Work Plan

Vanadium Concentrations in Snow Creek Sediment with Distance from Lake Logan Martin

Solutia
Ingenuity Productivity Performance

FIGURE
2-24



Location	Top Depth (ft)	Bottom Depth (ft)	Analyte	Result (mg/kg)
SC-1	0.83	3.00	Total PCBs	20
SC-1	0.83	3.00	Mercury	0.25

Legend

- Proposed Dredge Spoil Pile Soil Sample Location for 5-Point Composite
- Historical Analytes other than PCB and Lead Analyzed

Historical Surface Soil PCB Results (mg/kg)

- ND or < 1
- ≥ 1 to <10
- ≥ 10 to < 25

- Dredge Spoil Pile In Place
- All Parcels
- 100-Year Floodplain Drainage Area
- Exposure Unit
- Snow Creek

0 50 100 Feet
Graphic Scale

Notes:
1. Aerial imagery provided by Calhoun and Talladega Counties.
2. No sampling was conducted in pile SC-2.
3. Surface concentrations depicted represent the average of data from the 0- to 1-foot interval.
4. Subsurface concentrations depicted represent the average of data from the 0- to 4-foot interval.
5. EU: exposure unit
   ft: feet
   mg/kg: milligrams per kilogram
   ND: nondetect
   OU: operable unit
   PCB: polychlorinated biphenyl
6. Proposed dredge spoil pile samples will be composited from five sample locations within each low-activity special use area. Sample locations are for illustration purposes and may be revised in the field based on site conditions.

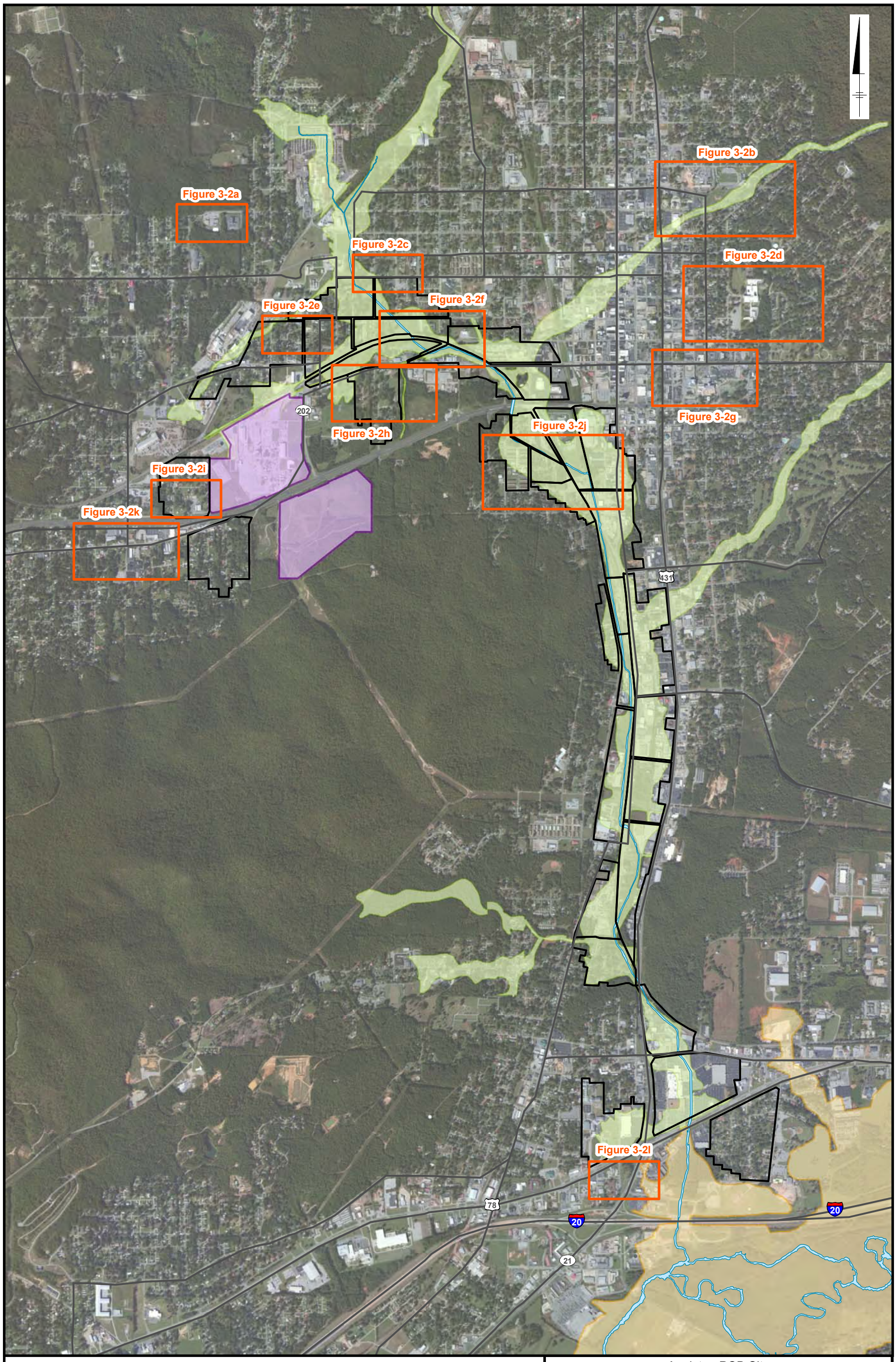
Anniston PCB Site
Anniston, Alabama
OU-1/OU-2 Predesign Investigation Work Plan

Proposed Soil Sampling: Dredge Spoil Pile SC-2

Solutia

Figure 3-1

City: SYR Div/Group: SWG Created By: K. Ives Last Saved By: cnelson
Anniston N:\A\Anniston\GIS\MXD\20220202_OU1_OU2_PDIWP\Rev2\Fig3-1_PDIWP_OU1_OU2_Proposed_Dredge_Spoil_Piles_Samples_rev2.mxd 2/23/2022 12:33:05 PM



Notes:
1. Aerial imagery used is Clarity from ArcGIS.
2. OU: operable unit
   PCB: polychlorinated biphenyl

Anniston PCB Site
Anniston, Alabama
OU-1/OU-2 Predesign Investigation Work Plan

Proposed Soil Sampling: Low-Activity Special Use Areas Site Map

Solutia

Figure
3-2

City: SYR Div/Group: SWG Created By: K. Ives Last Saved By: cnelson
Anniston
N:\A\Anniston\GIS\MXD\20220202_OU1_OU2_PDIWP\Rev2\Fig3-2_PDIWP_OU1_OU2_LowUse_Proposed_Soil_Sampling_rev2.mxd 2/23/2022 2:29:34 PM



Legend

- Proposed Low-Activity Special Use Soil Sample Location for 5-Point Composite

Historical Surface Soil
PCB Results (mg/kg)

- ND or < 1
- ≥ 1 to <10
- All Parcels
- Special Use Parcel Boundary
- Special Use: Low-Activity Area ≥1 mg/kg
- Low-Activity Subarea

0 100 200 Feet
Graphic Scale

Notes:
1. Aerial imagery provided by Calhoun and Talladega Counties.
2. Surface concentrations depicted represent the average of data from the 0- to 1-foot interval.
3. mg/kg: milligrams per kilogram
   ND: nondetect
   OU: operable unit
   PCB: polychlorinated biphenyl
4. Samples from the proposed low-activity special use areas will be composited from five sample locations within each low-activity special use area. Sample locations are for illustration purposes and may be revised in the field based on site conditions.

Anniston PCB Site
Anniston, Alabama
OU-1/OU-2 Predesign Investigation Work Plan

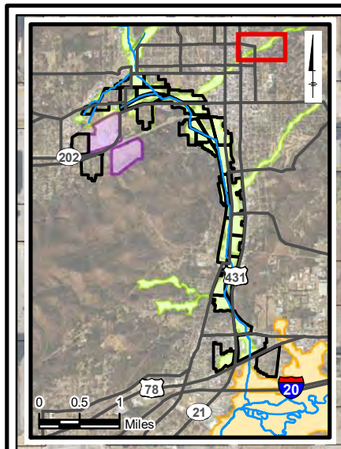
Proposed Soil Sampling: Low-Activity Special Use Areas

PPIN: 30182

Solutia

Figure 3-2a

City: SYR Div/Group: SWG Created By: K. Ives Last Saved By: cnelson
Anniston
N:\AA\Anniston\GIS\MXD\20220202_OU1_OU2_PDIWP\Rev2\Fig3-2a_PDIWP_OU1_OU2_LowUse_Proposed_Soil_Sampling_rev2.mxd 2/23/2022 2:38:33 PM



21033-G11
21033-G12
21033-G2
21033-G1
21033-G13
21033-G4
21033-G17
21033-G15
21033-G3
21033-G14
21033-G5
21033-G16
21033-G6
21033-G21
21033-G18
21033-G7
21033-G8
21033-G20
21033-G19
21033-G26
21033-G9
21033-G22
21033-G23
21033-G27
21033-G25
21033-G24
21033-G31
21033-G10
21033-G30
21033-G28
21033-G29

Legend

- Proposed Low-Activity Special Use Soil Sample Location for 5-Point Composite

Historical Surface Soil PCB Results (mg/kg)

- ND or < 1
- ≥ 1 to <10
- All Parcels
- Special Use Parcel Boundary
- Special Use: Low-Activity Area ≥1 mg/kg
- Low-Activity Subarea

0 200 400 Feet
Graphic Scale

Notes:
1. Aerial imagery provided by Calhoun and Talladega Counties.
2. Surface concentrations depicted represent the average of data from the 0- to 1-foot interval.
3. mg/kg: milligrams per kilogram
   ND: nondetect
   OU: operable unit
   PCB: polychlorinated biphenyl
4. Samples from the proposed low-activity special use areas will be composited from five sample locations within each low-activity special use area. Sample locations are for illustration purposes and may be revised in the field based on site conditions.

Anniston PCB Site
Anniston, Alabama
OU-1/OU-2 Predesign Investigation Work Plan

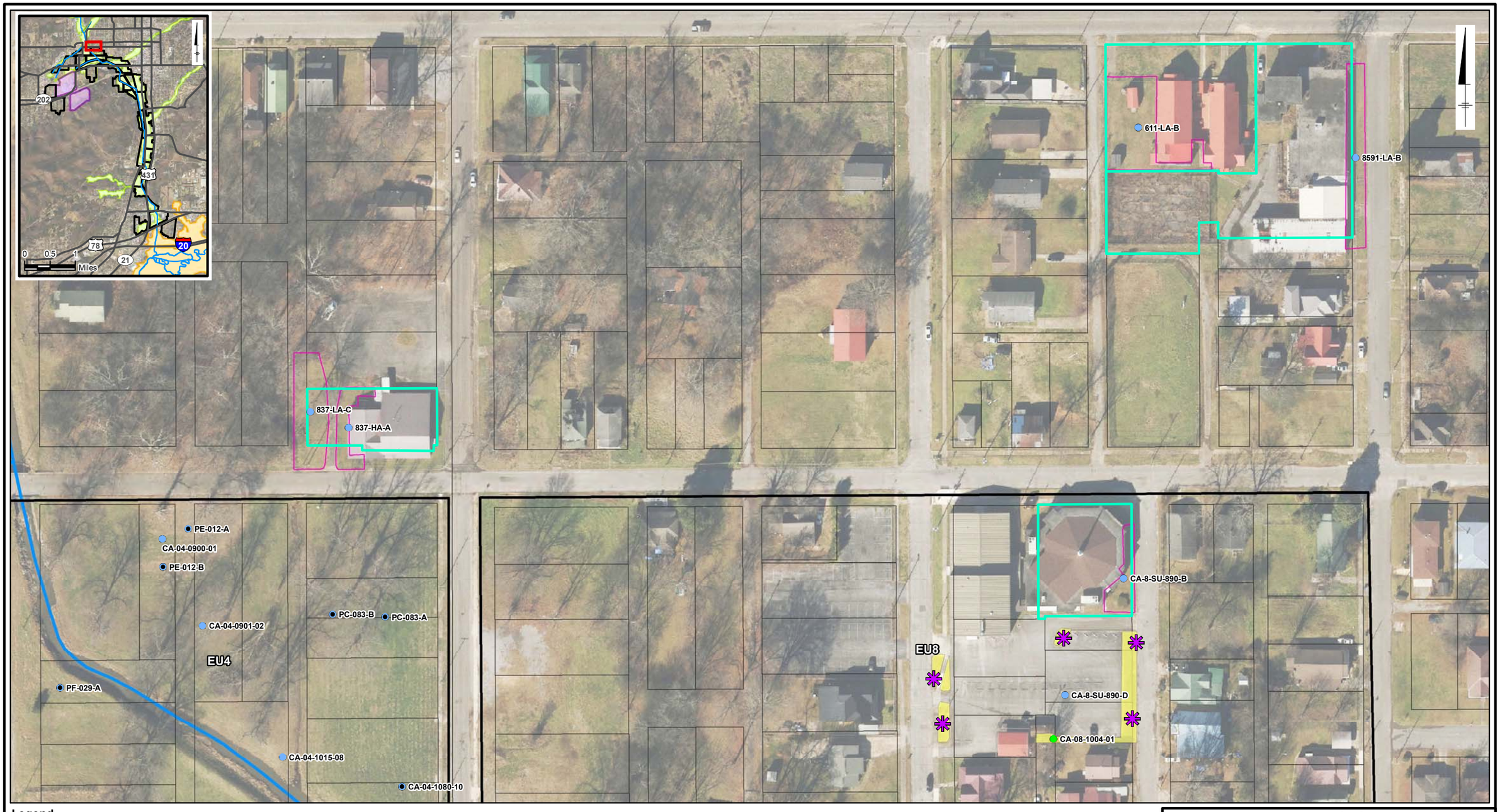
Proposed Soil Sampling: Low-Activity Special Use Areas

PPIN: 21033

Solutia

Figure 3-2b

City: SYR Div/Group: SWG Created By: K. Ives Last Saved By: cnelson
Anniston N:\AAnniston\GIS\MXD\20220202_OU1_OU2_PDIWP\Rev2\Fig3-2b_PDIWP_OU1_OU2_LowUse_Proposed_Soil_Sampling_rev2.mxd 2/23/2022 2:42:05 PM



Legend

- Proposed Low-Activity Special Use Soil Sample Location for 5-Point Composite
- Historical Analytes other than PCB and Lead Analyzed

Historical Surface Soil PCB Results (mg/kg)

- ND or < 1
- ≥ 1 to <10
- Snow Creek
- All Parcels
- Special Use Parcel Boundary
- Exposure Unit
- Special Use: Low-Activity Area ≥1 mg/kg
- Low-Activity Subarea

0 100 200 Feet

Graphic Scale

Notes:

1. Aerial imagery provided by Calhoun and Talladega Counties.
2. Surface concentrations depicted represent the average of data from the 0- to 1-foot interval.
3. EU: exposure unit
   mg/kg: milligrams per kilogram
   ND: nondetect
   OU: operable unit
   PCB: polychlorinated biphenyl
4. Samples from the proposed low-activity special use areas will be composited from five sample locations within each low-activity special use area. Sample locations are for illustration purposes and may be revised in the field based on site conditions.

Anniston PCB Site
Anniston, Alabama
OU-1/OU-2 Predesign Investigation Work Plan

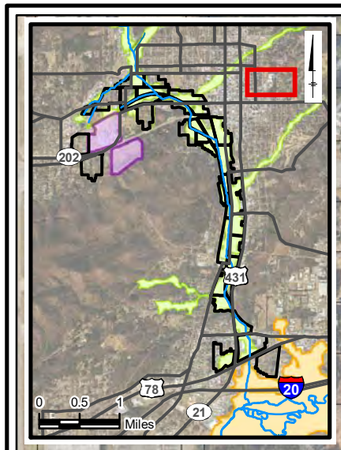
Proposed Soil Sampling: Low-Activity Special Use Areas

PPIN: 19256, 19463, 30711 & 80736

Solutia

Figure
3-2c

City: SYR Div/Group: SWG Created By: K. Ives Last Saved By: cnelson
Anniston
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20579-G22 20579-G23 20579-G20 20579-G21 20717-G24 20717-G43 20717-G44 20717-G19 20579-G19 20579-G18 20717-G20 20717-G21 20717-G47 20717-G48 20717-G17 20717-G18 20579-G16 20717-G46 20579-G17 20717-G15 20717-G16 20717-G45 20579-G14 20579-G15 20717-G49 20717-G14 20579-G13 20717-G53 20717-G13 20717-G51 20717-G52 20717-G55 20579-G11 20579-G12 206605-G75-LA-B 20717-G7 20717-G50 20717-G54 20579-G10 20717-G8 20717-G12 20579-G8 20579-G9 20717-G11 20717-G9 20717-G10 20579-G7 20717-G3 20717-G6 20717-G5 20717-G70 20717-G4 20579-G6 20717-G71 20717-G72 20579-G5 20717-G73 20717-G74 20579-G3 20579-G4 20717-G1 20579-G1 20579-G2

Legend

- Proposed Low-Activity Special Use Soil Sample Location for 5-Point Composite
- Historical Surface Soil PCB Results (mg/kg)
  - ND or < 1
  - ≥ 1 to <10
- All Parcels
- Special Use Parcel Boundary
- Special Use: Low-Activity Area ≥1 mg/kg
- Low-Activity Subarea

Inner and outer circles reflect surface and subsurface PCB concentrations, respectively.

0 200 400 Feet

Graphic Scale

Notes:

1. Aerial imagery provided by Calhoun and Talladega Counties.
2. Surface concentrations depicted represent the average of data from the 0- to 1-foot interval.
3. Subsurface concentrations depicted represent the average of data from the 0- to 4-foot interval.
4. mg/kg: milligrams per kilogram
   ND: nondetect
   OU: operable unit
   PCB: polychlorinated biphenyl
5. Samples from the proposed low-activity special use areas will be composited from five sample locations within each low-activity special use area. Sample locations are for illustration purposes and may be revised in the field based on site conditions.

Anniston PCB Site
Anniston, Alabama
OU-1/OU-2 Predesign Investigation Work Plan

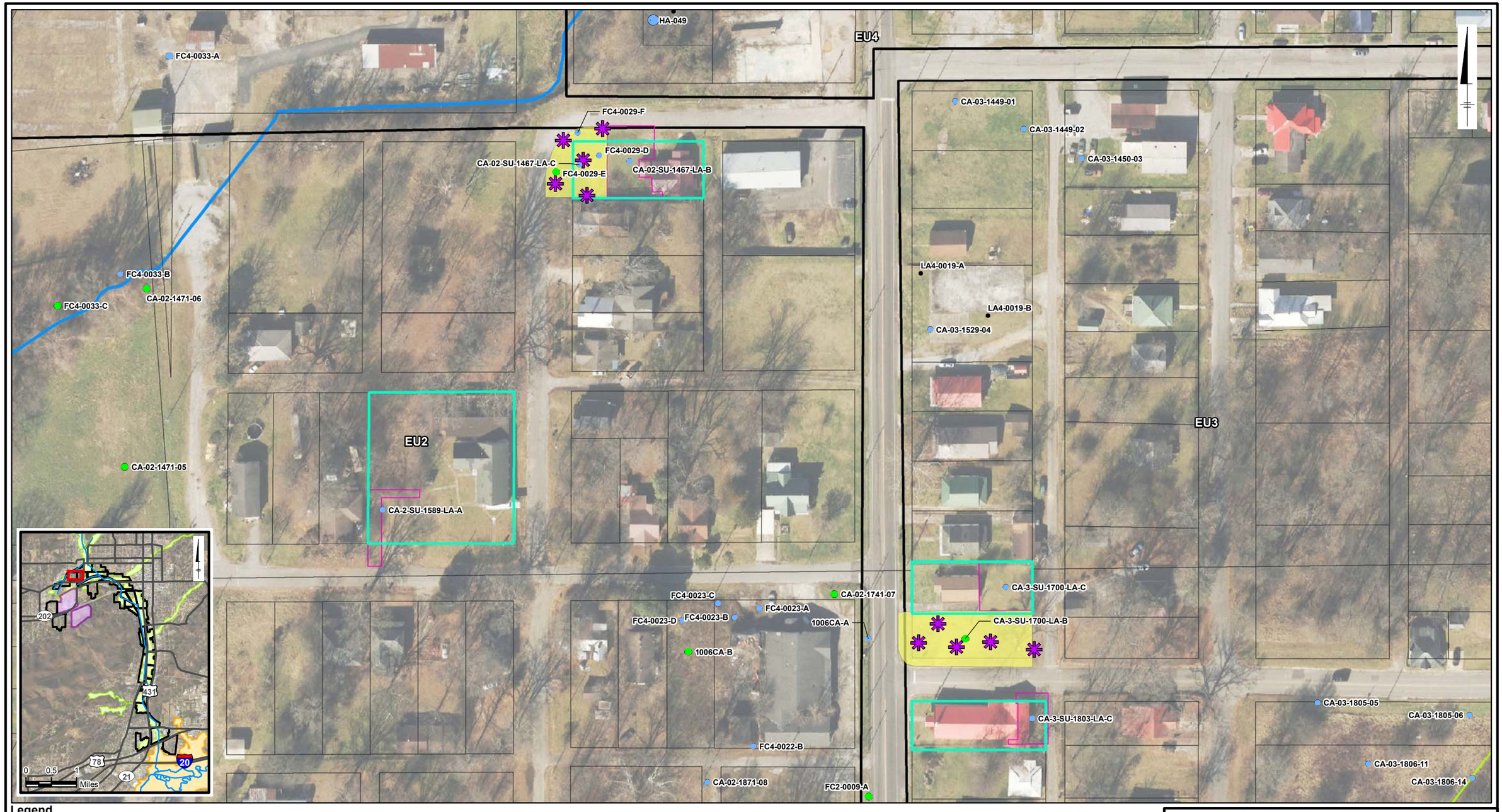
Proposed Soil Sampling: Low-Activity Special Use Areas

PPIN: 20579, 20717

Solutia

Figure 3-2d

City: SYR Div/Group: SWG Created By: K. Ives Last Saved By: cnelson
Anniston
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Legend

- Proposed Low-Activity Special Use Soil Sample Location for 5-Point Composite
- Historical Analytes other than PCB and Lead Analyzed

Historical Surface Soil PCB Results (mg/kg)

- ND or < 1
- ≥ 1 to <10
- West 9th Street Creek
- All Parcels
- Exposure Unit
- Special Use Parcel Boundary
- Special Use: Low-Activity Area ≥1 mg/kg
- Low-Activity Subarea

Inner and outer circles reflect surface and subsurface PCB concentrations, respectively.

0 100 200 Feet
Graphic Scale

Notes:
1. Aerial imagery provided by Calhoun and Talladega Counties.
2. Surface concentrations depicted represent the average of data from the 0- to 1-foot interval.
3. Subsurface concentrations depicted represent the average of data from the 0- to 4-foot interval.
4. EU: exposure unit
   mg/kg: milligrams per kilogram
   ND: nondetect
   OU: operable unit
   PCB: polychlorinated biphenyl
5. Samples from the proposed low-activity special use areas will be composited from five sample locations within each low-activity special use area. Sample locations are for illustration purposes and may be revised in the field based on site conditions.

Anniston PCB Site
Anniston, Alabama
OU-1/OU-2 Predesign Investigation Work Plan

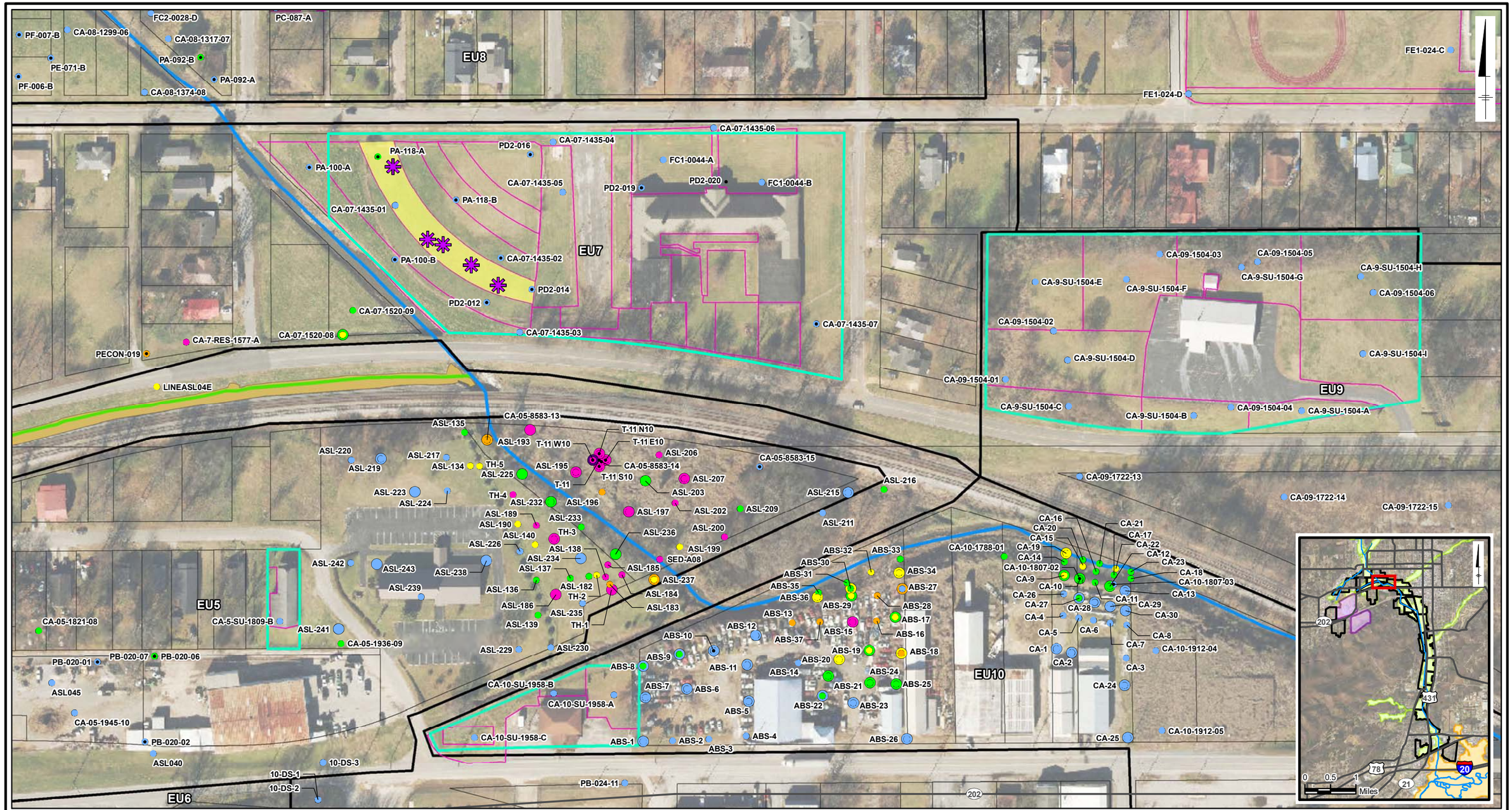
Proposed Soil Sampling: Low-Activity Special Use Areas

PPIN: 30861, 30865, 32122 & 32277

Solutia

Figure
3-2e

City: SYR Div/Group: SWG Created By: K. Ives Last Saved By: cnelson
Anniston
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Legend

- Proposed Low-Activity Special Use Soil Sample Location for 5-Point Composite
- Historical Surface Soil PCB Results (mg/kg)
  - ND or < 1
  - ≥ 1 to <10
  - ≥ 10 to < 25
  - ≥ 25 to < 50
  - ≥ 50 to < 500
  - ≥ 500
- Historical Analytes other than PCB and Lead Analyzed
- 11th Street Ditch
- Snow Creek
- All Parcels
- Exposure Unit
- Special Use Parcel Boundary
- Special Use: Low-Activity Area ≥1 mg/kg
- Low-Activity Subarea
- Inner and outer circles reflect surface and subsurface PCB concentrations, respectively.

0 150 300 Feet

Graphic Scale

Notes:
1. Aerial imagery provided by Calhoun and Talladega Counties.
2. Surface concentrations depicted represent the average of data from the 0- to 1-foot interval.
3. Subsurface concentrations depicted represent the average of data from the 0- to 4-foot interval.
4. EU: exposure unit
   mg/kg: milligrams per kilogram
   ND: nondetect
   OU: operable unit
   PCB: polychlorinated biphenyl
5. Samples from the proposed low-activity special use areas will be composited from five sample locations within each low-activity special use area. Sample locations are for illustration purposes and may be revised in the field based on site conditions.

Anniston PCB Site
Anniston, Alabama
OU-1/OU-2 Predesign Investigation Work Plan

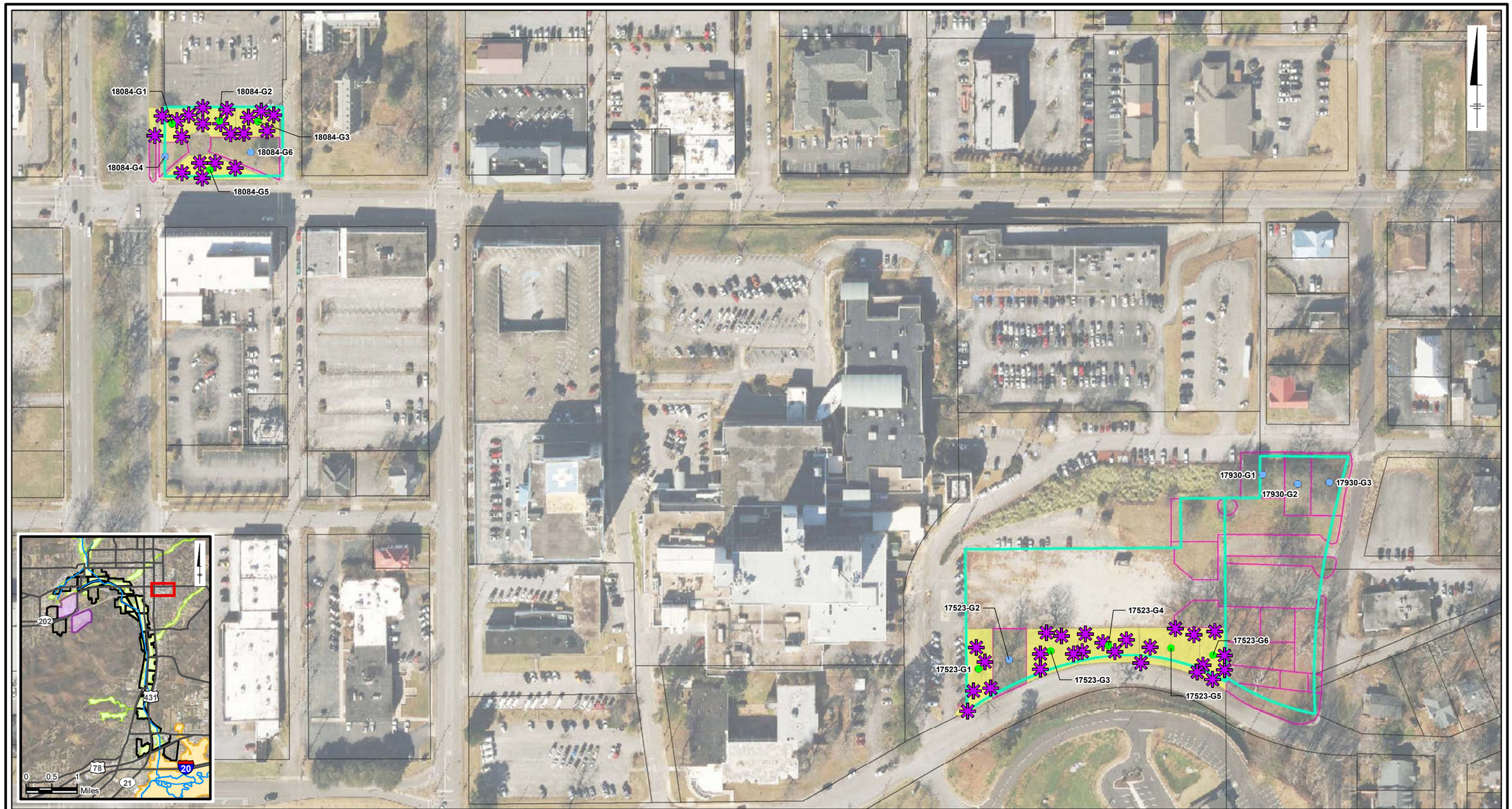
Proposed Soil Sampling: Low-Activity Special Use Areas

PPIN: 18700, 18713, 19122 & 19313

Solutia

Figure 3-2f

City: SYR Div/Group: SWG Created By: K. Ives Last Saved By: cnelson
Anniston
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Legend

- Proposed Low-Activity Special Use Soil Sample Location for 5-Point Composite

Historical Surface Soil PCB Results (mg/kg)

- ND or < 1
- ≥ 1 to <10
- All Parcels
- Special Use Parcel Boundary
- Special Use: Low-Activity Area ≥1 mg/kg
- Low-Activity Subarea

0 150 300 Feet

Graphic Scale

Notes:

1. Aerial imagery provided by Calhoun and Talladega Counties.
2. Surface concentrations depicted represent the average of data from the 0- to 1-foot interval.
3. mg/kg: milligrams per kilogram
   ND: nondetect
   OU: operable unit
   PCB: polychlorinated biphenyl
4. Samples from the proposed low-activity special use areas will be composited from five sample locations within each low-activity special use area. Sample locations are for illustration purposes and may be revised in the field based on site conditions.

Anniston PCB Site
Anniston, Alabama
OU-1/OU-2 Predesign Investigation Work Plan

Proposed Soil Sampling: Low-Activity Special Use Areas

PPIN: 17523,17930 & 18084

Solutia

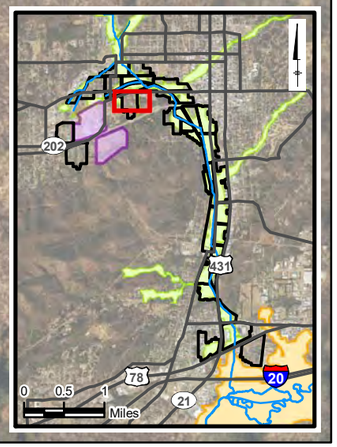
Figure 3-2g

City: SYR Div/Group: SWG Created By: K. Ives Last Saved By: cnelson
Anniston
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EU5

EU6

ASL158
ASL055
CA-05-1782-07
CA-05-1782-02
CA-06-2136-03
GASCOSL07
CA-06-2136-04
WEL042
BNSL34
PC-023-B
PC-023-A
2506-A2491
PC-024-B
2506-3A
PC-024-A
SED1700R125
SED1500R125
SED1430R175
SED1420R175
SED1430R225
CA-06-2113-02
CA-6-SU-2113-LA-A
1206WT-B
FC3-0030-A
FC3-0030-B
1206WT-A
FC3-0029-A
FC3-0030-C
FC3-0029-B
FC3-0030-D
10-DS-2
10-DS-1
CA-06-2083-01
PB-024-11
CA-06-2205-13
CA-06-2205-07
CA-6-SU-2205-B
CA-6-SU-2205-O
CA-6-SU-2205-I
CA-6-SU-2205-R
CA-6-SU-2205-P
CA-6-SU-2205-S
CA-06-2205-11
CA-6-SU-2205-D
CA-6-SU-2205-Q
CA-6-SU-2205-L
CA-6-SU-2205-E
CA-6-SU-2205-M
CA-6-SU-2205-T
CA-6-SU-2205-N
CA-6-SU-2498-B
CA-6-SU-2498-E
CA-6-SU-2205-U
CA-6-SU-2498-C
CA-6-SU-2498-D
CA-06-2205-12
CA-6-SU-2498-A
CA-06-2498-14



Legend

- Proposed Low-Activity Special Use Soil Sample Location for 5-Point Composite
- Historical Analytes other than PCB and Lead Analyzed

Historical Surface Soil PCB Results (mg/kg)

- ND or < 1
- ≥ 1 to <10
- ≥ 10 to < 25
- ≥ 25 to < 50
- ≥ 50 to < 500

- All Parcels
- Exposure Unit
- Special Use Parcel Boundary
- Special Use: Low-Activity Area ≥1 mg/kg
- Low-Activity Subarea

0 150 300 Feet

Graphic Scale

Notes:

1. Aerial imagery provided by Calhoun and Talladega Counties.
2. Surface concentrations depicted represent the average of data from the 0- to 1-foot interval.
3. EU: exposure unit
   mg/kg: milligrams per kilogram
   ND: nondetect
   OU: operable unit
   PCB: polychlorinated biphenyl
4. Samples from the proposed low-activity special use areas will be composited from five sample locations within each low-activity special use area. Sample locations are for illustration purposes and may be revised in the field based on site conditions.

Anniston PCB Site
Anniston, Alabama
OU-1/OU-2 Predesign Investigation Work Plan

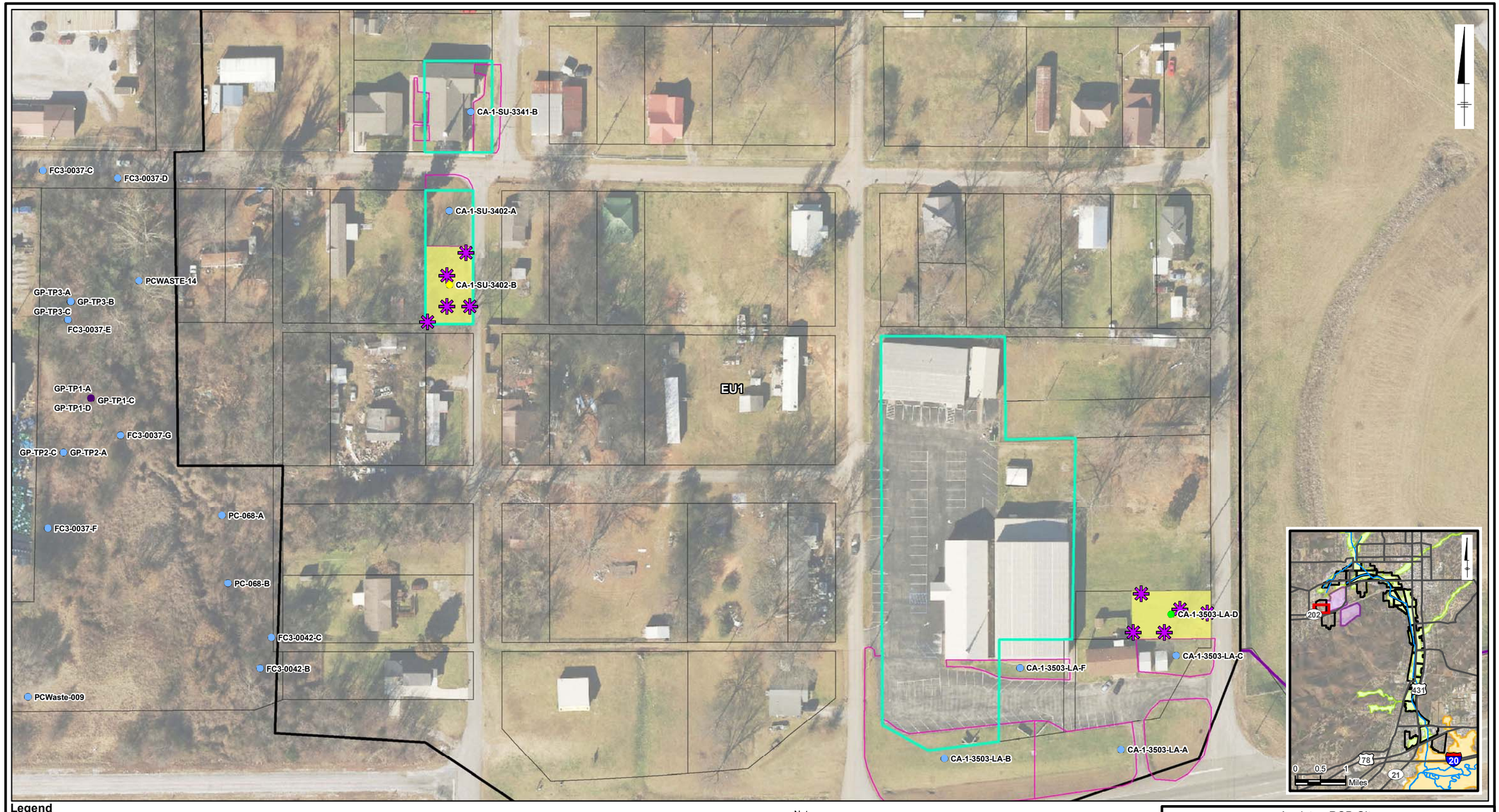
Proposed Soil Sampling: Low-Activity Special Use Areas

PPIN: 18536, 32375 & 62824

Solutia

Figure 3-2h

City: SYR Div/Group: SWG Created By: K. Ives Last Saved By: cnelson
Anniston N:\AAnniston\GIS\MXD\20220202_OU1_OU2_PDIWP\Rev2\Fig3-2h_PDIWP_OU1_OU2_LowUse_Proposed_Soil_Sampling_rev2.mxd 2/23/2022 3:04:50 PM



Legend

- Proposed Low-Activity Special Use Soil Sample Location for 5-Point Composite

Historical Surface Soil PCB Results (mg/kg)

- ND or < 1
- ≥ 1 to <10
- ≥ 10 to < 25
- ≥ 500
- All Parcels

- Exposure Unit
- Special Use Parcel Boundary
- Special Use: Low-Activity Area ≥1 mg/kg
- Low-Activity Subarea

0 100 200 Feet

Graphic Scale

Notes:

1. Aerial imagery provided by Calhoun and Talladega Counties.
2. Surface concentrations depicted represent the average of data from the 0- to 1-foot interval.
3. EU: exposure unit
   mg/kg: milligrams per kilogram
   ND: nondetect
   OU: operable unit
   PCB: polychlorinated biphenyl
4. Samples from the proposed low-activity special use areas will be composited from five sample locations within each low-activity special use area. Sample locations are for illustration purposes and may be revised in the field based on site conditions.

Anniston PCB Site
Anniston, Alabama
OU-1/OU-2 Predesign Investigation Work Plan

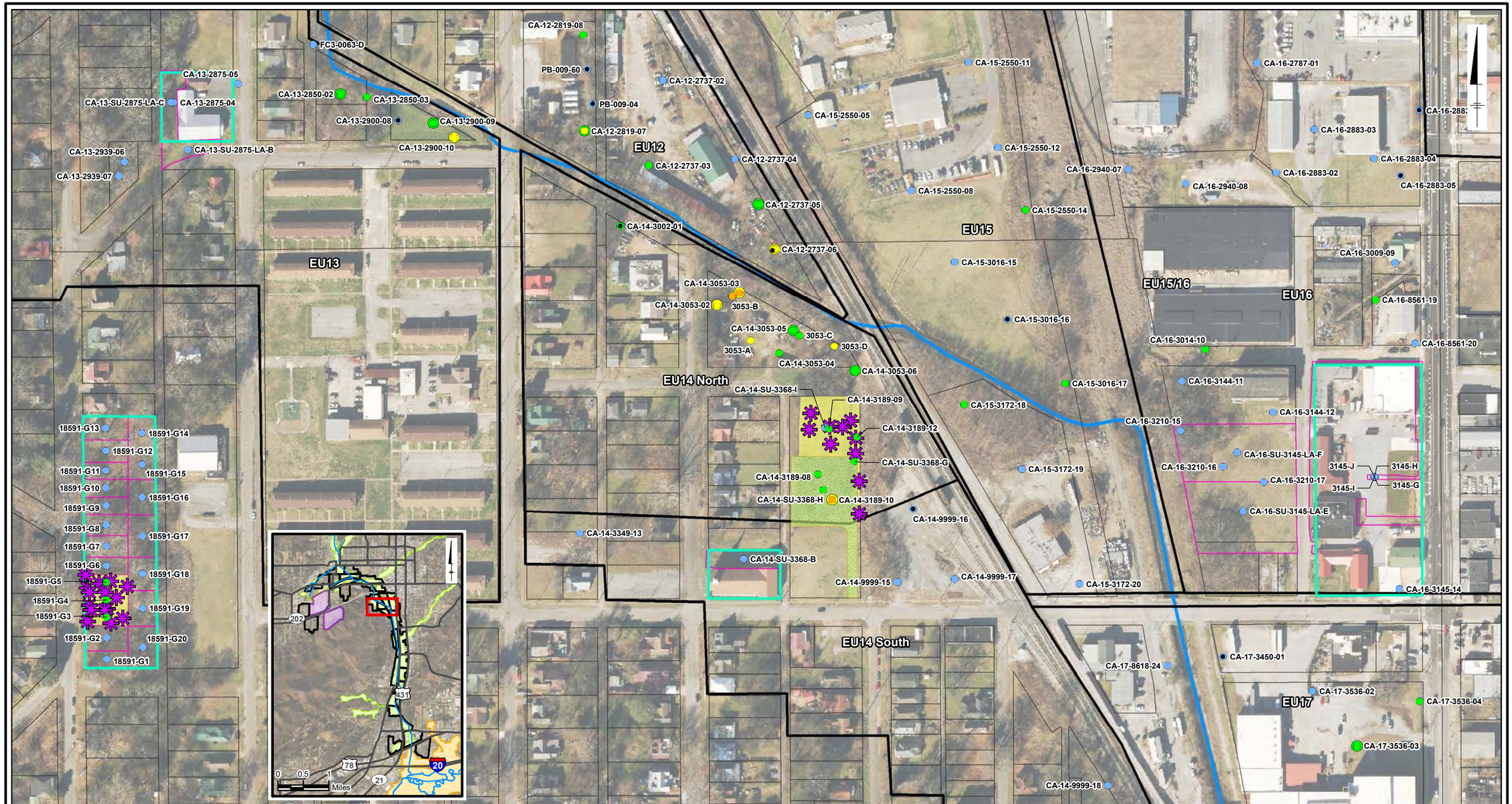
Proposed Soil Sampling: Low-Activity Special Use Areas

PPIN: 32322, 32490 & 32664

Solutia

Figure 3-2i

City: SYR Div/Group: SWG Created By: K. Ives Last Saved By: cnelson
Anniston
N:\AAnniston\GIS\MXD\20220202_OU1_OU2_PDIWP\Rev2\Fig3-2i_PDIWP_OU1_OU2_LowUse_Proposed_Soil_Sampling_rev2.mxd 2/23/2022 3:07:57 PM



Legend

- Proposed Low-Activity Special Use Soil Sample Location for 5-Point Composite
- Historical Analytes other than PCB and Lead Analyzed

Historical Surface Soil PCB Results (mg/kg)

- ND or < 1
- ≥ 1 to <10
- ≥ 10 to < 25
- ≥ 25 to < 50

- Snow Creek
- All Parcels
- Exposure Unit
- Special Use Parcel Boundary
- Special Use: Low-Activity Area ≥1 mg/kg
- Low-Activity Subarea
- Alabama Power West 4th Street Customer Substation Development Excavation Area

Inner and outer circles reflect surface and subsurface PCB concentrations, respectively.

0 200 400 Feet

Graphic Scale

Notes:
1. Aerial imagery provided by Calhoun and Talladega Counties.
2. Surface concentrations depicted represent the average of data from the 0- to 1-foot interval.
3. Subsurface concentrations depicted represent the average of data from the 0- to 4-foot interval.
4. The Alabama Power West 4th Street customer substation construction was completed in December 2021. Limits of excavation are show as documented in the Alabama Power West 4th Street Customer Substation Construction Project Completion Report.
5. EU: exposure unit
   mg/kg: milligrams per kilogram
   ND: nondetect
   OU: operable unit
   PCB: polychlorinated biphenyl
6. Samples from the proposed low-activity special use areas will be composited from five sample locations within each low-activity special use area. Sample locations are for illustration purposes and may be revised in the field based on site conditions.

Anniston PCB Site
Anniston, Alabama
OU-1/OU-2 Predesign Investigation Work Plan

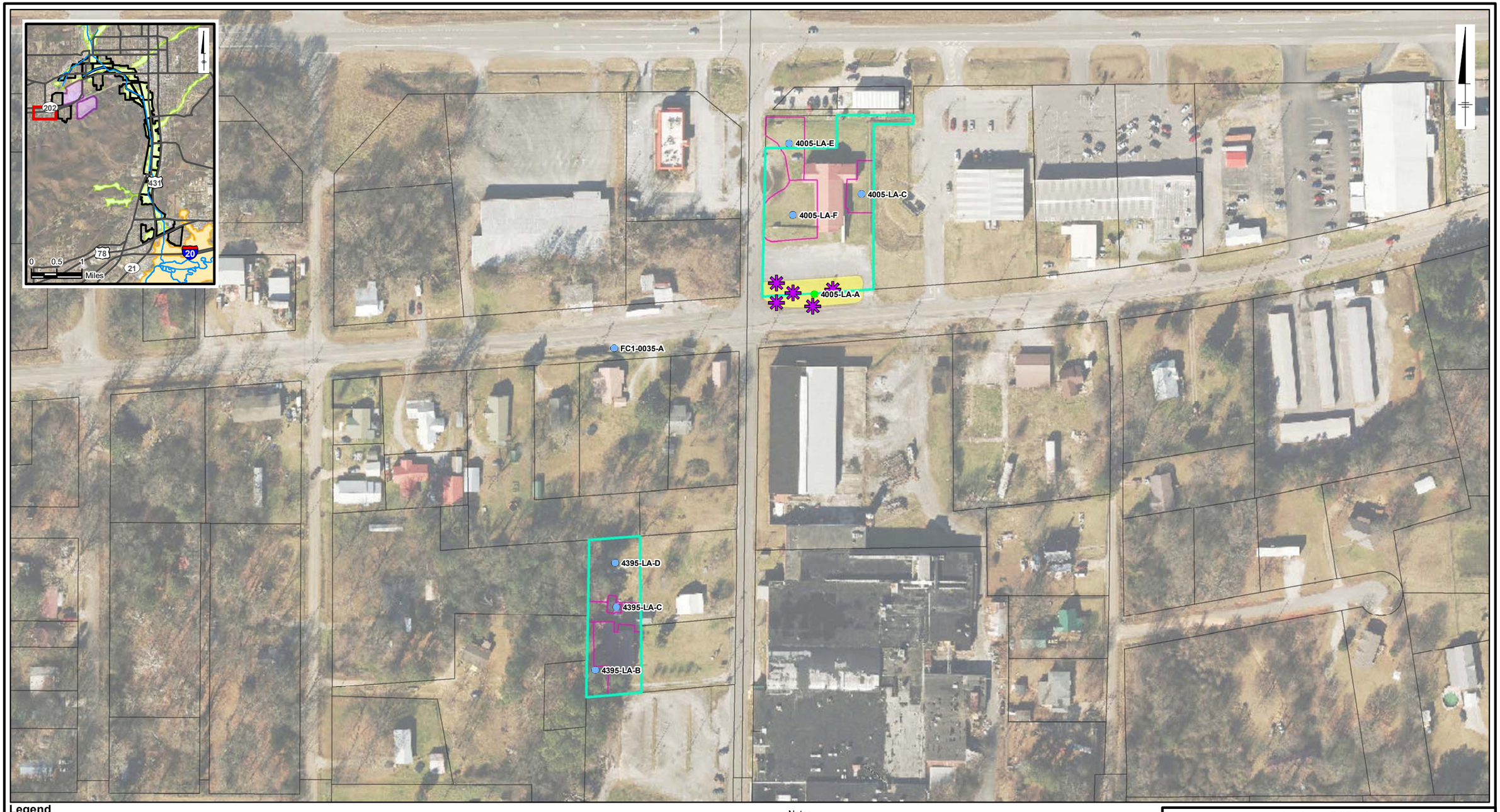
Proposed Soil Sampling: Low-Activity Special Use Areas

PPIN: 18122, 18591, 18628 & 63275

Solutia

Figure
3-2j

City: SYR Div/Group: SWG Created By: K. Ives Last Saved By: cnelson
Anniston
N:\Anniston\GIS\MXD\20220202_OU1_OU2_PDIWP\Rev2\Fig3-2j_PDIWP_OU1_OU2_LowUse_Proposed_Soil_Sampling_rev2.mxd 2/23/2022 3:10:59 PM



Legend

- Proposed Low-Activity Special Use Soil Sample Location for 5-Point Composite

Historical Surface Soil PCB Results (mg/kg)

- ND or < 1
- ≥ 1 to <10
- All Parcels
- Special Use Parcel Boundary
- Special Use: Low-Activity Area ≥1 mg/kg
- Low-Activity Subarea

0 150 300 Feet

Graphic Scale

Notes:

1. Aerial imagery provided by Calhoun and Talladega Counties.
2. Surface concentrations depicted represent the average of data from the 0- to 1-foot interval.
3. mg/kg: milligrams per kilogram
   ND: nondetect
   OU: operable unit
   PCB: polychlorinated biphenyl
4. Samples from the proposed low-activity special use areas will be composited from five sample locations within each low-activity special use area. Sample locations are for illustration purposes and may be revised in the field based on site conditions.

Anniston PCB Site
Anniston, Alabama
OU-1/OU-2 Predesign Investigation Work Plan

Proposed Soil Sampling: Low-Activity Special Use Areas

PPIN: 31947 & 32612

Solutia

Figure 3-2k

City: SYR Div/Group: SWG Created By: K. Ives Last Saved By: cnelson
Anniston
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Legend

- Proposed Low-Activity Special Use Soil Sample Location for 5-Point Composite
- Historical Analytes other than PCB and Lead Analyzed

Historical Surface Soil PCB Results (mg/kg)

- ND or < 1
- ≥ 1 to <10
- All Parcels
- Exposure Unit
- Special Use Parcel Boundary
- Special Use: Low-Activity Area ≥1 mg/kg
- Low-Activity Subarea

0 100 200 Feet

Graphic Scale

Notes:

1. Aerial imagery provided by Calhoun and Talladega Counties.
2. Surface concentrations depicted represent the average of data from the 0- to 1-foot interval.
3. EU: exposure unit
   mg/kg: milligrams per kilogram
   ND: nondetect
   OU: operable unit
   PCB: polychlorinated biphenyl
4. Samples from the proposed low-activity special use areas will be composited from five sample locations within each low-activity special use area. Sample locations are for illustration purposes and may be revised in the field based on site conditions.

Anniston PCB Site
Anniston, Alabama
OU-1/OU-2 Predesign Investigation Work Plan

Proposed Soil Sampling: Low-Activity Special Use Areas

PPIN: 16384

Solutia

Figure 3-2I

City: SYR Div/Group: SWG Created By: K. Ives Last Saved By: cnelson
Anniston
N:\AAnniston\GIS\MXD\20220202_OU1_OU2_PDIWP\Rev2\Fig3-2I_PDIWP_OU1_OU2_LowUse_Proposed_Soil_Sampling_rev2.mxd 2/23/2022 3:18:38 PM

Figure 3-3e
Figure 3-3a
Figure 3-3b
202
Figure 3-3c
Figure 3-3d
431
Figure 3-3f
78
20
20
21

Legend

- Major Roads
- Figure Extent
- Exposure Unit
- Creek
- Operable Unit 3
- Snow Creek 100-Year Floodplain and Drainage
- OU-4 100-Year Floodplain

Notes:
1. Aerial imagery used is Clarity from ArcGIS.
2. OU: operable unit
   PCB: polychlorinated biphenyl

0 0.5 1 Miles

Graphic Scale

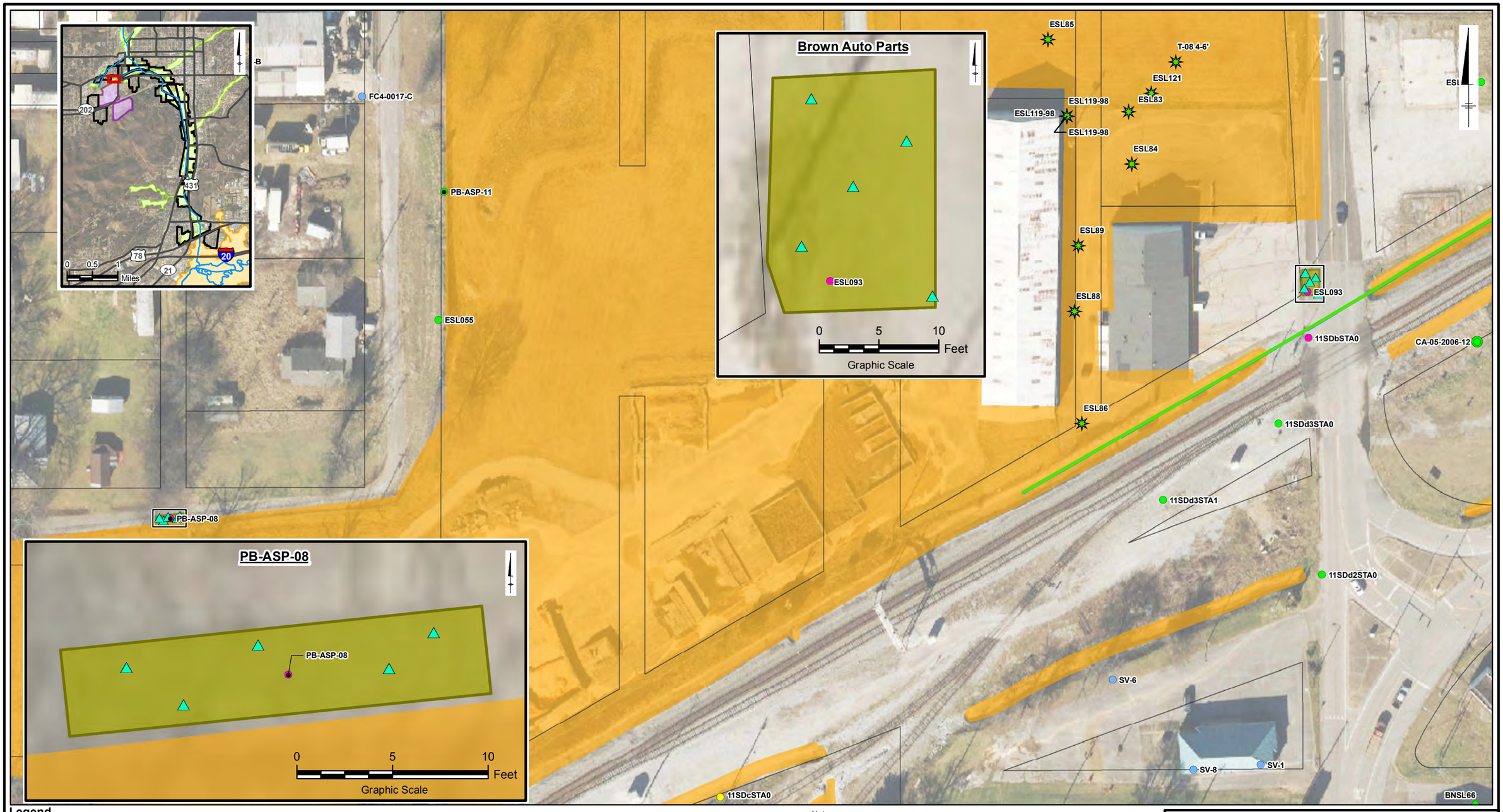
Anniston PCB Site
Anniston, Alabama
OU-1/OU-2 Predesign Investigation Work Plan

Proposed Soil Sampling: Interim Measure Areas

Solutia

Figure 3-3

City: SYR Div/Group: SWG Created By: K. Ives Last Saved By: NSekhon
Anniston
N:\Anniston\GIS\MXD\20220202_OU1_OU2_PDIWP\Rev2\Fig3-3_PDIWP_OU1_OU2_IMA_Proposed_Soil_Sampling_rev2.mxd 6/7/2022 10:38:09 AM



Legend

- Historical Analytes other than PCB and Lead Analyzed

Historical Surface Soil PCB Results (mg/kg)

- ND or < 1
- ≥ 1 to <10
- ≥ 10 to < 25
- ≥ 25 to < 50
- ≥ 50 to < 500

- Historical Locations Beneath Cover System with Maximum PCB Results ≥500 mg/kg
- Proposed IM Soil Sample Location for 5-Point Composite
- 11th Street Ditch
- Interim Measure Area Remediation Footprint: 0-to-1 foot Removal and Subsurface Soil Sampling During PDI
- Interim Measure and Other Early Actions
- All Parcels

Inner and outer circles reflect surface and subsurface PCB concentrations, respectively.

0 75 150
Feet
Graphic Scale

Notes:

1. Aerial imagery provided by Calhoun and Talladega Counties.
2. Surface concentrations depicted represent the average of data from the 0- to 1-foot interval.
3. Subsurface concentrations depicted represent the average of data from the 0- to 4-foot interval.
4. IM: interim measure
   mg/kg: milligrams per kilogram
   ND: nondetect
   OU: operable unit
   PCB: polychlorinated biphenyl
   PDI: pre-design investigation
5. Proposed IM samples will be composited from five sample locations within each IM area. Sample locations are for illustration purposes and may be revised in the field based on site conditions.

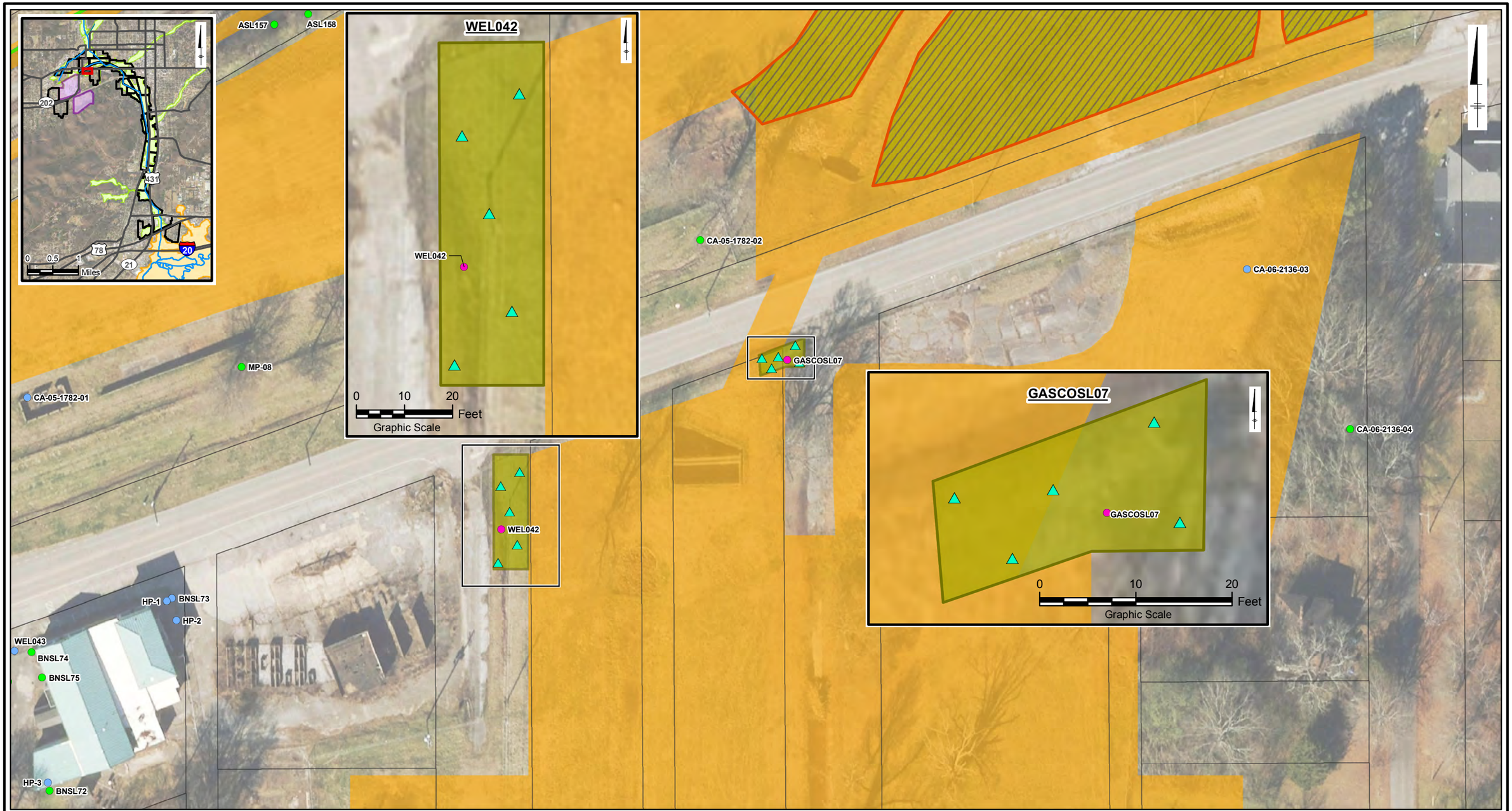
Anniston PCB Site
Anniston, Alabama
OU-1/OU-2 Predesign Investigation Work Plan

Proposed Soil Sampling: Interim Measure Areas: Northside Area

Solutia
Ingenuity Productivity Performance

Figure
3-3a

City: SYR Div/Group: SWG Created By: K. Ives Last Saved By: NSekhon
Anniston
N:\Anniston\GIS\MXD\20220202_OU1_OU2_PDIWP\Rev2\Fig3-3a_PDIWP_OU1_OU2_IMA_Proposed_Soil_Sampling_rev2.mxd 6/7/2022 9:55:25 AM



Legend

Historical Surface Soil
PCB Results (mg/kg)
- ND or < 1
- ≥ 1 to <10
- ≥ 10 to < 25
- Proposed IM Soil Sample Location for 5-Point Composite
- Interim Measure Area Remediation Footprint: 0-to-1 foot Removal and Subsurface Soil Sampling During PDI
- Interim Measure Area Remediation Footprint: 0-to-1 foot Removal and No Subsurface Soil Sampling During PDI
- Interim Measure and Other Early Actions
- All Parcels

0 60 120
Feet
Graphic Scale

Notes:
1. Aerial imagery provided by Calhoun and Talladega Counties.
2. Surface concentrations depicted represent the average of data from the 0- to 1-foot interval.
3. bgs: below ground surface
   IM: interim measure
   mg/kg: milligrams per kilogram
   ND: nondetect
   OU: operable unit
   PCB: polychlorinated biphenyl
   PDI: pre-design investigation
4. Proposed IM samples will be composited from five sample locations within each IM area. Sample locations are for illustration purposes and may be revised in the field based on site conditions.

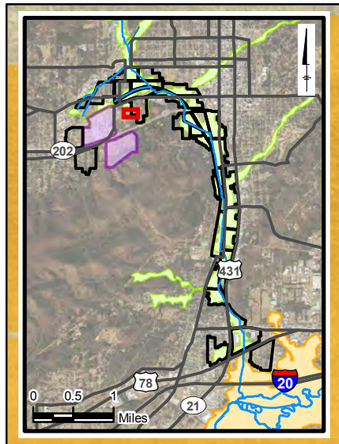
Anniston PCB Site
Anniston, Alabama
OU-1/OU-2 Predesign Investigation Work Plan

Proposed Soil Sampling: Interim Measure Areas: Eastside Area

Solutia

Figure
3-3b

City: SYR Div/Group: SWG Created By: K. Ives Last Saved By: NSekhon
Anniston
N:\Anniston\GIS\MXD\20220202_OU1_OU2_PDIWP\Rev2\Fig3-3b_PDIWP_OU1_OU2_IMA_Proposed_Soil_Sampling_rev2.mxd 6/7/2022 10:02:38 AM



SED1500R125

SED1430R175

SED1420R175

SED1430R225

SED1300

SED-5

OFSL18

OFSL19

WEL006

WEL005

SED1430R225

SED1430R225

0 20 40 Feet

Graphic Scale

Legend

Historical Surface Soil PCB Results (mg/kg)

- ND or < 1
- ≥ 1 to <10
- ≥ 25 to < 50
- ≥ 50 to < 500

Historical Locations Beneath Cover System with Maximum PCB Results ≥500 mg/kg

Proposed IM Soil Sample Location for 5-Point Composite

Interim Measure Area Remediation Footprint: 0-to-1 foot Removal and Subsurface Soil Sampling During PDI

Interim Measure and Other Early Actions

All Parcels

0 60 120 Feet

Graphic Scale

Notes:

1. Aerial imagery provided by Calhoun and Talladega Counties.
2. Surface concentrations depicted represent the average of data from the 0- to 1-foot interval.
3. IM: interim measure
   mg/kg: milligrams per kilogram
   ND: nondetect
   OU: operable unit
   PCB: polychlorinated biphenyl
   PDI: pre-design investigation
4. Proposed IM samples will be composited from five sample locations within each IM area. Sample locations are for illustration purposes and may be revised in the field based on site conditions.

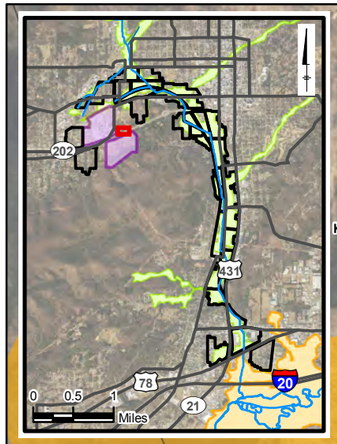
Anniston PCB Site
Anniston, Alabama
OU-1/OU-2 Predesign Investigation Work Plan

Proposed Soil Sampling: Interim Measure Areas: Eastside Area

Solutia

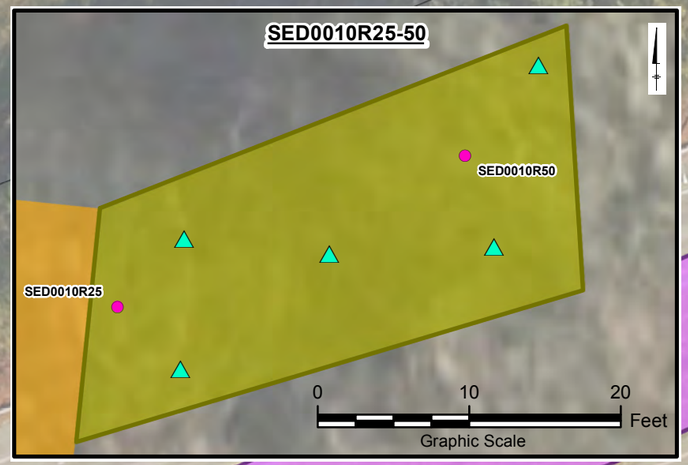
Figure
3-3c

City: SYR Div/Group: SWG Created By: K. Ives Last Saved By: NSekhon
Anniston
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KNSL07

SED0200R50



SED0010R50
SED0010
SED0010R25
SED0010L25
SED0010L50
MDNSL05

Legend

Historical Surface Soil PCB Results (mg/kg)
- ND or < 1
- ≥ 10 to < 500
- ≥ 50 to < 500

Historical Locations Beneath Cover System with Maximum PCB Results ≥500 mg/kg

Proposed IM Soil Sample Location for 5-Point Composite

Interim Measure Area Remediation Footprint: 0-to-1 foot Removal and Subsurface Soil Sampling During PDI

Interim Measure and Other Early Actions

All Parcels

Nonresidential Soil Remediation Area

0 50 100 Feet
Graphic Scale

Notes:
1. Aerial imagery provided by Calhoun and Talladega Counties.
2. Surface concentrations depicted represent the average of data from the 0- to 1-foot interval.
3. IM: interim measure
   mg/kg: milligrams per kilogram
   ND: nondetect
   OU: operable unit
   PCB: polychlorinated biphenyl
   PDI: pre-design investigation
4. Proposed IM samples will be composited from five sample locations within each IM area. Sample locations are for illustration purposes and may be revised in the field based on site conditions.

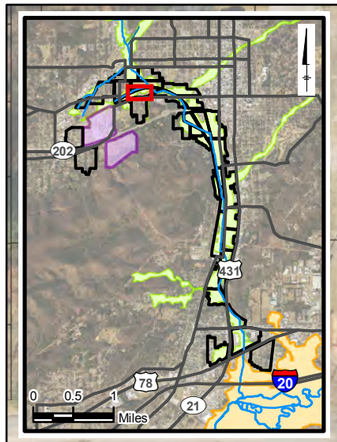
Anniston PCB Site
Anniston, Alabama
OU-1/OU-2 Predesign Investigation Work Plan

Proposed Soil Sampling: Interim Measure Areas: Eastside Area

Solutia

Figure **3-3d**

City: SYR Div/Group: SWG Created By: K. Ives Last Saved By: NSekhon
Anniston
N:\AVAnniston\GIS\MXD\20220202_OU1_OU2_PDIWP\Rev2\Fig3-3d_PDIWP_OU1_OU2_IMA_Proposed_Soil_Sampling_rev2.mxd 6/7/2022 10:04:57 AM



PECON-019
LINEASL04E
LINEASL03D
PB-RR-37
PB-RR-40
PB-RR-42
ASED550
ASED500
ASED450
ASED400
ASED350
ASED450R25
ASED300
SED-C1
ASED250
ASED100R50
CA-03-1806-18
LINEASL02E
LINEASL02F
LINEASL02D
CA-05-1782-06
CA-05-1782-04
CA-05-1782-05
CA-05-1782-03
ASL056
ASL055
CA-05-1782-07
CA-06-2113-02
CA-6-SU-2113-LA-A
FC3-0030-A
1206WT-B
FC3-0030-B
ASL135
CA-05-8583-13
ASL220
ASL219
ASL217
ASL134
TH-5
ASL224
ASL242
ASL239
CA-5-SU-1809-B
CA-05-1821-08
CA-05-1936-09
PB-020-07
PB-020-06
PB-020-01
ASL045
CA-05-1945-10
CA-05-1996-11
PB-020-02
ASL040
CA-10-SU-1958-C
10-DS-3
10-DS-2
10-DS-1
CA-06-2083-01

Legend

- Historical Analytes other than PCB and Lead Analyzed
- Historical Surface Soil PCB Results (mg/kg)
  - ND or < 1
  - ≥ 1 to <10
  - ≥ 10 to < 25
  - ≥ 25 to < 50
  - ≥ 50 to < 500
  - ≥ 500
- Historical Locations Beneath Cover System with Maximum PCB Results ≥500 mg/kg
- Historical Locations with Maximum PCB Results ≥500 mg/kg
- Proposed IM Soil Sample Location for 5-Point Composite
- Proposed Drainage Ditch Near PB-RR-37 Discrete Soil Sample Location
- 11th Street Ditch
- Snow Creek
- Interim Measure Area Remediation Footprint: 0-to-1 foot Removal and Subsurface Soil Sampling During PDI
- Interim Measure Area Remediation Footprint: 0-to-1 foot Removal and No Subsurface Soil Sampling During PDI
- Interim Measure Area Remediation Footprint: Surface and Subsurface Soil Sampling During PDI
- Interim Measure and Other Early Actions
- Drainage Ditch Near PB-RR-37 Area
- All Parcels
- Inner and outer circles reflect surface and subsurface PCB concentrations, respectively.

0 100 200 Feet
Graphic Scale

Notes:
1. Aerial imagery provided by Calhoun and Talladega Counties.
2. Surface concentrations depicted represent the average of data from the 0- to 1-foot interval.
3. Subsurface concentrations depicted represent the average of data from the 0- to 4-foot interval.
4. bgs: below ground surface
   IM: interim measure
   mg/kg: milligrams per kilogram
   ND: nondetect
   OU: operable unit
   PCB: polychlorinated biphenyl
   PDI: pre-design investigation
5. Proposed IM samples will be composited from five sample locations within each area. Sample locations are for illustration purposes and may be revised in the field based on site conditions.
6. Five-point composite soil samples will be collected from the drainage ditches located upstream and downstream of PB-RR-37. Composite samples will be collected from four sample depths: 0 to 1 foot bgs, 1 to 2 feet bgs, 2 to 3 feet bgs, and 3 to 4 feet bgs. Additionally, during collection of the 5-point composite sample, discrete samples for the two locations closest to Snow Creek will be collected from the same depth intervals. Samples collected from the soil depths of 1 to 2 feet bgs and below will be archived at the analytical laboratory pending analysis of samples collected from 0 to 1 foot bgs.
7. Five-point composite soil samples will be collected from the interim measure area remediation footprint surrounding PB-RR-37. Composite samples will be collected from four sample depths: 0 to 1 foot bgs, 1 to 2 feet bgs, 2 to 3 feet bgs, and 3 to 4 feet bgs. One of the composite locations will be co-located with the discrete boring at PB-RR-37 that is separately implemented to profile PCB concentrations in soil from ground surface to the water table. Split samples will be collected at this location to be submitted as part of the composite sample and as a discrete sample.

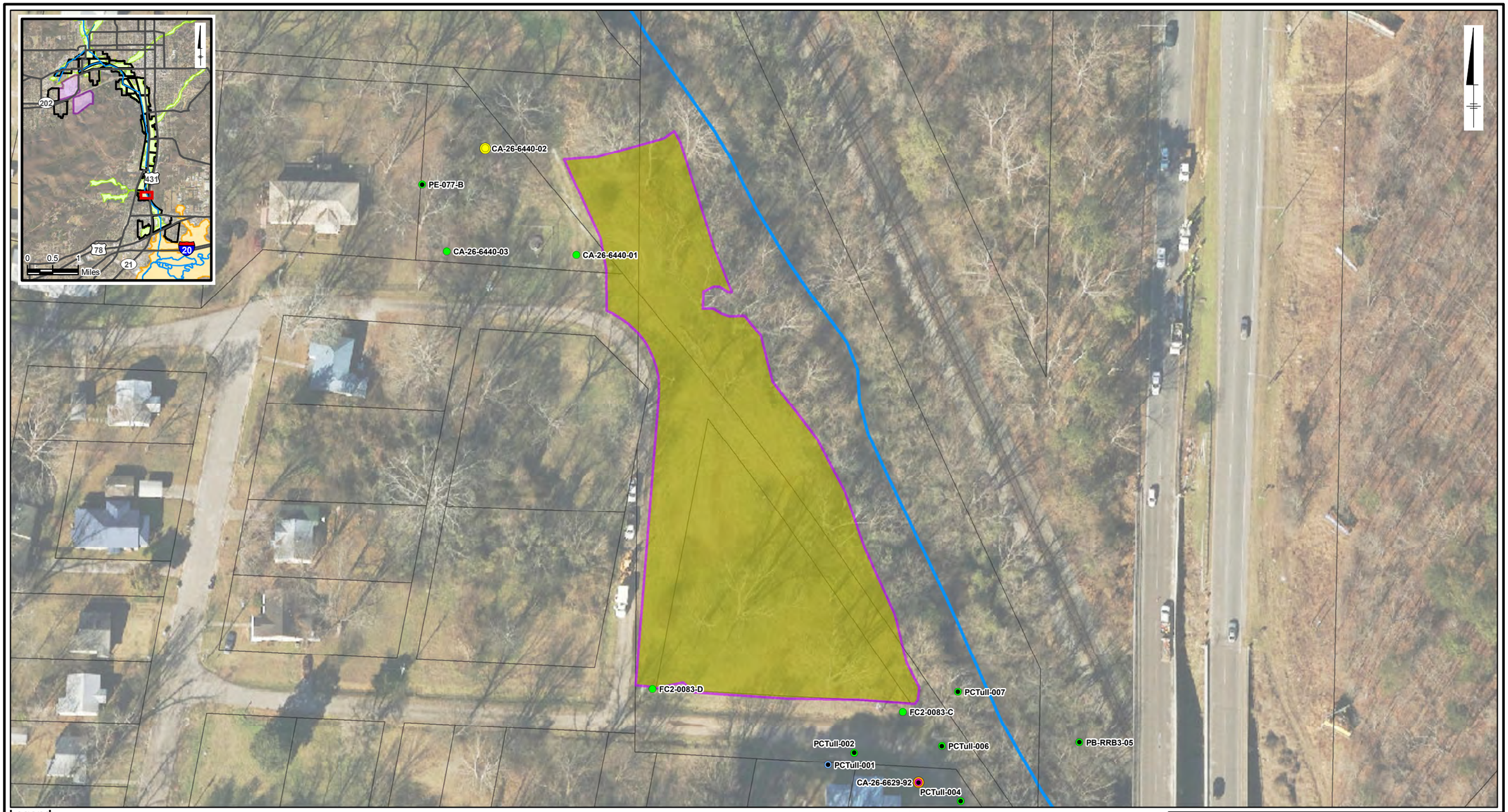
Anniston PCB Site
Anniston, Alabama
OU-1/OU-2 Predesign Investigation Work Plan

Proposed Soil Sampling: Interim Measure Areas: Eastside Drainage Way

Solutia
Ingenuity Productivity Performance

Figure
3-3e

City: SYR Div/Group: SWG Created By: K. Ives Last Saved By: NSekhon
Anniston
N:\A\Anniston\GIS\MXD\20220202_OU1_OU2_PDIWP\Rev2\Fig3-3e_PDIWP_OU1_OU2_IMA_Proposed_Soil_Sampling_rev2.mxd 6/7/2022 10:07:52 AM



Legend

- Historical Analytes other than PCB and Lead Analyzed

Historical Surface Soil PCB Results (mg/kg)

- ND or < 1
- ≥ 1 to <10
- ≥ 10 to < 25
- ≥ 25 to < 50
- ≥ 50 to < 500
- Snow Creek

- Interim Measure Area Remediation Footprint: Visual Inspection During PDI
- All Parcels

Inner and outer circles reflect surface and subsurface PCB concentrations, respectively.

0 75 150 Feet

Graphic Scale

Notes:

1. Aerial imagery provided by Calhoun and Talladega Counties.
2. Surface concentrations depicted represent the average of data from the 0- to 1-foot interval.
3. Subsurface concentrations depicted represent the average of data from the 0- to 4-foot interval.
4. mg/kg: milligrams per kilogram
   ND: nondetect
   OU: operable unit
   PCB: polychlorinated biphenyl
   PDI: pre-design investigation

Anniston PCB Site
Anniston, Alabama
OU-1/OU-2 Predesign Investigation Work Plan

Proposed Soil Sampling: Interim Measure Areas: Hall Street

Solutia

Figure
3-3f

City: SYR Div/Group: SWG Created By: K. Ives Last Saved By: NSekhon
Anniston
N:\Anniston\GIS\MXD\20220202_OU1_OU2_PDIWP\Rev2\Fig3-3f_PDIWP_OU1_OU2_Remediation_FP_IMA_rev2.mxd 6/7/2022 10:09:43 AM

Figure 3-4a
Figure 3-4h
Figure 3-4g
Figure 3-4b
Figure 3-4c
Figure 3-4d
Figure 3-4e
Figure 3-4f
202
431
78
20
21

Legend

- Major Roads
- Figure Extent
- Exposure Unit
- Creek
- Operable Unit 3
- Snow Creek 100-Year Floodplain and Drainage
- OU-4 100-Year Floodplain

Notes:
1. Aerial imagery used is Clarity from ArcGIS.
2. OU: operable unit
   PCB: polychlorinated biphenyl

0 0.5 1 Miles

Graphic Scale

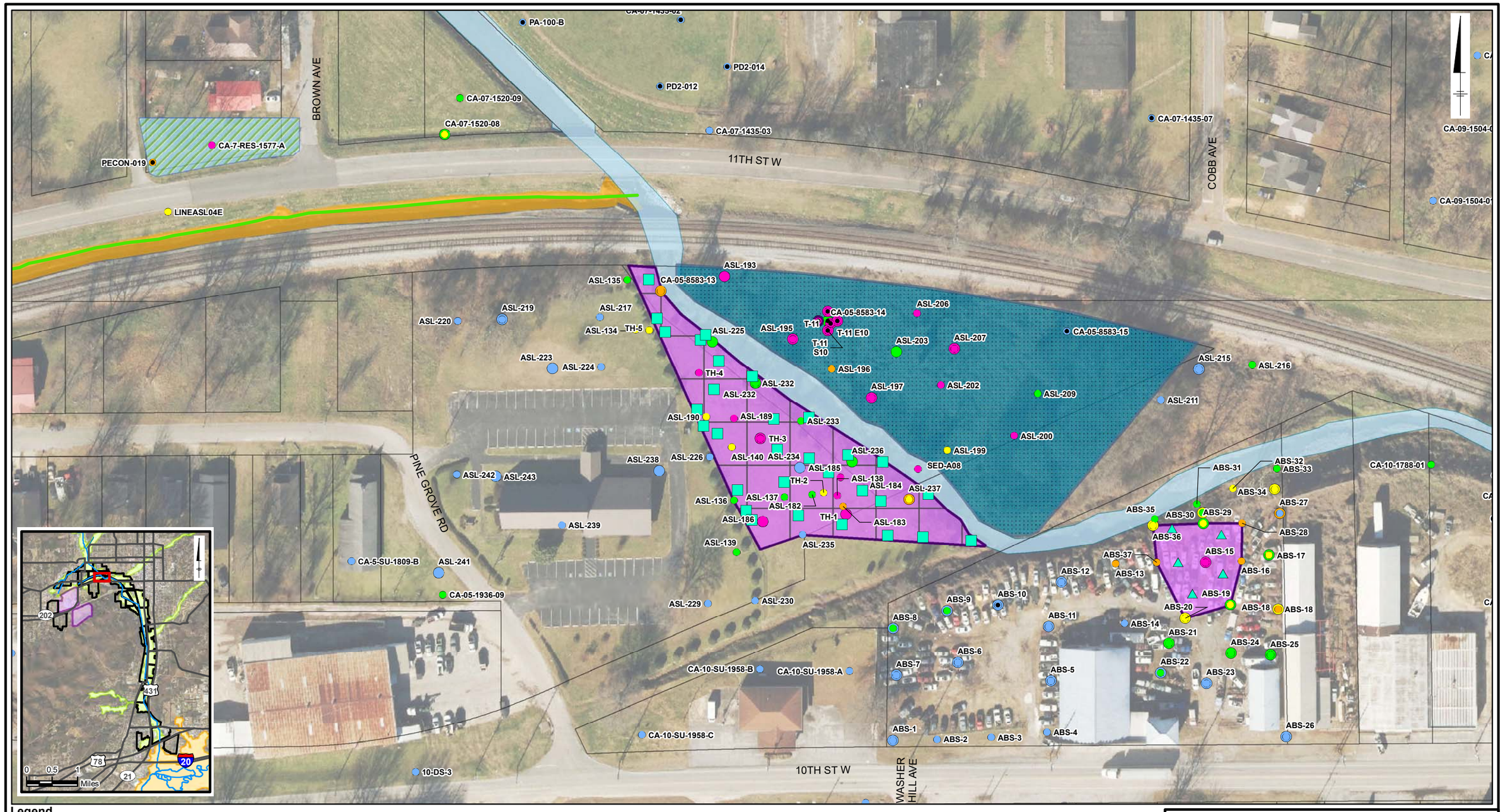
Anniston PCB Site
Anniston, Alabama
OU-1/OU-2 Predesign Investigation Work Plan

Proposed Soil Sampling: Nonresidential Areas Site Map

Solutia

Figure 3-4

City: SYR Div/Group: SWG Created By: K. Ives Last Saved By: cnelson
Anniston
N:\A\Anniston\GIS\MXD\20220202_OU1_OU2_PDIWP\Rev2\Fig3-4_PDIWP_OU1_OU2_Nonresidential_Proposed_Soil_Sampling_rev2.mxd 2/23/2022 4:13:52 PM



Legend

- Historical Analytes other than PCB and Lead Analyzed
- Historical Surface Soil PCB Results (mg/kg)
  - ND or < 1
  - ≥ 1 to <10
  - ≥ 10 to < 25
  - ≥ 25 to < 50
  - ≥ 50 to < 500
  - ≥ 500
- Proposed Nonresidential Soil Sample Location for 5-Point Composite
- Proposed Nonresidential ISM Soil Sampling Grid Location
- 11th Street Ditch
- All Parcels
- Snow Creek
- Interim Measure and Other Early Actions
- Previously Remediated Area
- T-11 Area Remediation Footprint
- Remediation Footprint for Nonresidential Soils (PCBs)
- Proposed Nonresidential ISM Sampling Grid
- Inner and outer circles reflect surface and subsurface PCB concentrations, respectively.

0 100 200 Feet
Graphic Scale

Notes:
1. Aerial imagery provided by Calhoun and Talladega Counties.
2. Surface concentrations depicted represent the average of data from the 0- to 1-foot interval.
3. Subsurface concentrations depicted represent the average of data from the 0- to 4-foot interval.
4. ISM: incremental sampling methodology
   mg/kg: milligrams per kilogram
   ND: nondetect
   OU: operable unit
   PCB: polychlorinated biphenyl
5. Sample locations from the proposed ISM grid areas are for illustration purposes and may be revised in the field based on site conditions. Thirty increment samples will make up an ISM increment for each nonresidential area.
6. Proposed nonresidential samples will be composited from five sample locations within each nonresidential soils (PCBs) remediation footprint. Sample locations are for illustration purposes and may be revised in the field based on site conditions.

Anniston PCB Site
Anniston, Alabama
OU-1/OU-2 Predesign Investigation Work Plan

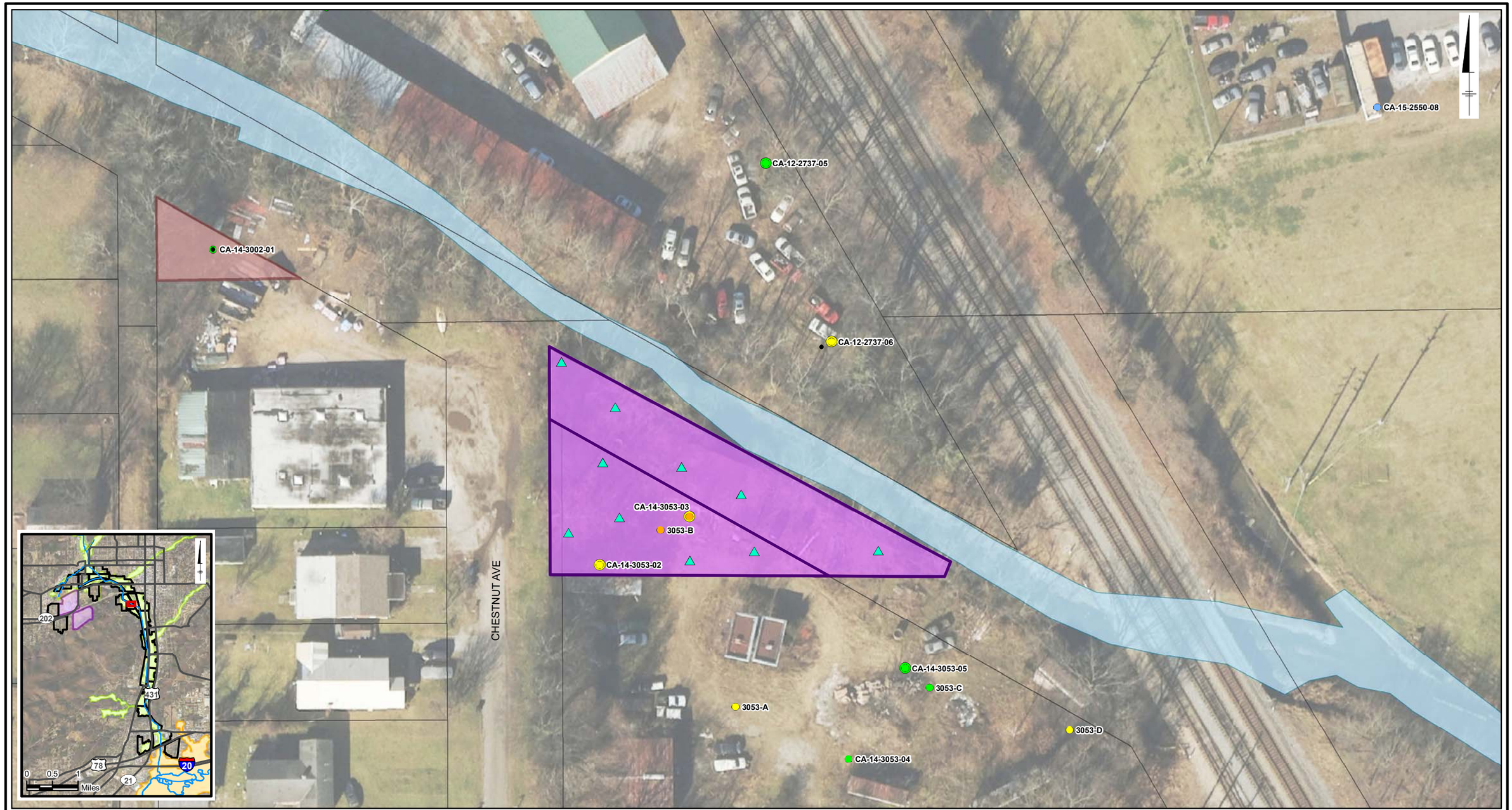
Proposed Soil Sampling Nonresidential Areas: Exposure Unit 5

PPIN: 18705, 69009 & 62844

Solutia

Figure **3-4a**

City: SYR Div/Group: SWG Created By: K. Ives Last Saved By: cnelson
Anniston
N:\AAnniston\GIS\MXD\20220202_OU1_OU2_PDIWP\Rev2\Fig3-4a_PDIWP_OU1_OU2_Nonresidential_Proposed_Soil_Sampling_rev2.mxd 2/23/2022 4:43:06 PM



Legend

- Historical Analytes other than PCB and Lead Analyzed
- Historical Surface Soil PCB Results (mg/kg)
  - ND or < 1
  - ≥ 1 to <10
  - ≥ 10 to < 25
  - ≥ 25 to < 50
- Proposed Nonresidential Soil Sample Location for 5-Point Composite
- All Parcels
- Snow Creek
- Remediation Footprints for Nonresidential Soils (non PCB constituents)
- Remediation Footprint for Nonresidential Soils (PCBs)

Inner and outer circles reflect surface and subsurface PCB concentrations, respectively.

0 50 100 Feet

Graphic Scale

Notes:
1. Aerial imagery provided by Calhoun and Talladega Counties.
2. Surface concentrations depicted represent the average of data from the 0- to 1-foot interval.
3. Subsurface concentrations depicted represent the average of data from the 0- to 4-foot interval.
4. mg/kg: milligrams per kilogram
   ND: nondetect
   OU: operable unit
   PCB: polychlorinated biphenyl
5. Proposed nonresidential samples will be composited from five sample locations within each nonresidential soils (PCBs) remediation footprint. Sample locations are for illustration purposes and may be revised in the field based on site conditions.

Anniston PCB Site
Anniston, Alabama
OU-1/OU-2 Predesign Investigation Work Plan

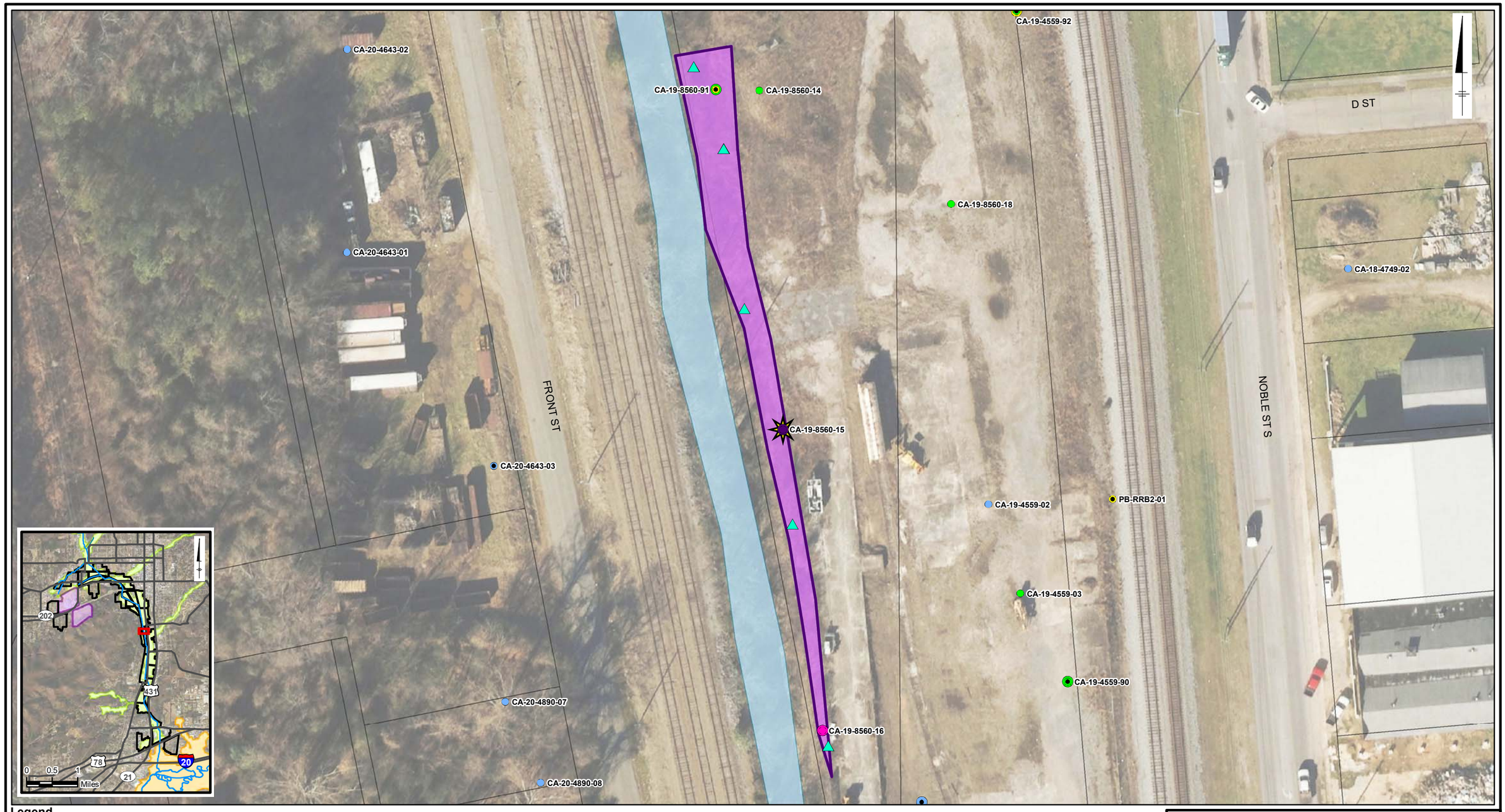
Proposed Soil Sampling
Nonresidential Areas: Exposure Unit 14 North

PPIN: 18125

Solutia

Figure 3-4b

City: SYR Div/Group: SWG Created By: K. Ives Last Saved By: cnelson
Anniston
N:\Al\Anniston\GIS\MXD\20220202_OU1_OU2_PDIWP\Rev2\Fig3-4b_PDIWP_OU1_OU2_Nonresidential_Proposed_Soil_Sampling_rev2.mxd 2/24/2022 8:09:06 AM



Legend

- Historical Analytes other than PCB and Lead Analyzed

Historical Surface Soil PCB Results (mg/kg)

- ND or < 1
- ≥ 1 to <10
- ≥ 10 to < 25
- ≥ 25 to < 50
- ≥ 50 to < 500
- ≥ 500

- Historical Locations with Maximum PCB Results ≥500 mg/kg
- Proposed Nonresidential Soil Sample Location for 5-Point Composite
- All Parcels
- Snow Creek
- Remediation Footprint for Nonresidential Soils (PCBs)

Inner and outer circles reflect surface and subsurface PCB concentrations, respectively.

0 60 120 Feet

Graphic Scale

Notes:
1. Aerial imagery provided by Calhoun and Talladega Counties.
2. Surface concentrations depicted represent the average of data from the 0- to 1-foot interval.
3. Subsurface concentrations depicted represent the average of data from the 0- to 4-foot interval.
4. mg/kg: milligrams per kilogram
   ND: nondetect
   OU: operable unit
   PCB: polychlorinated biphenyl
5. Proposed nonresidential samples will be composited from five sample locations within each nonresidential soils (PCBs) remediation footprint. Sample locations are for illustration purposes and may be revised in the field based on site conditions.

Anniston PCB Site
Anniston, Alabama
OU-1/OU-2 Predesign Investigation Work Plan

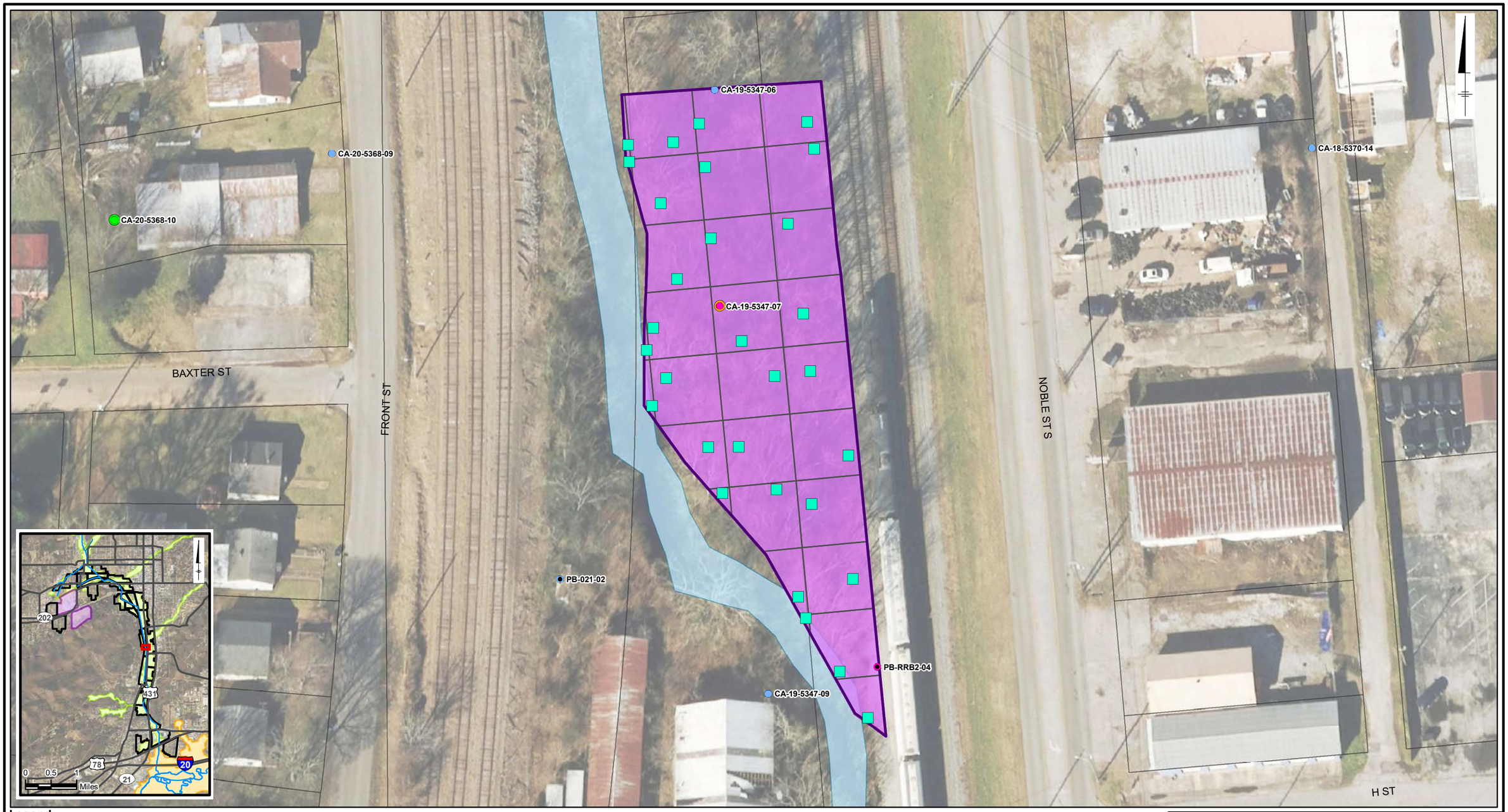
Proposed Soil Sampling Nonresidential Areas: Exposure Unit 19 North

PPIN: 66676

Solutia

Figure 3-4c

City: SYR Div/Group: SWG Created By: K. Ives Last Saved By: cnelson
Anniston
N:\AA\Anniston\GIS\MXD\20220202_OU1_OU2_PDIWP\Rev2\Fig3-4c_PDIWP_OU1_OU2_Nonresidential_Proposed_Soil_Sampling_rev2.mxd 2/23/2022 5:39:52 PM



Legend

- Historical Analytes other than PCB and Lead Analyzed

Historical Surface Soil PCB Results (mg/kg)
- ND or < 1
- ≥ 1 to <10
- ≥ 25 to < 50
- ≥ 50 to < 500

- Proposed Nonresidential ISM Soil Sampling Grid Location
- All Parcels
- Snow Creek
- Remediation Footprint for Nonresidential Soils (PCBs)
- Proposed Nonresidential ISM Sampling Grid

Inner and outer circles reflect surface and subsurface PCB concentrations, respectively.

0 50 100 Feet

Graphic Scale

Notes:
1. Aerial imagery provided by Calhoun and Talladega Counties.
2. Surface concentrations depicted represent the average of data from the 0- to 1-foot interval.
3. Subsurface concentrations depicted represent the average of data from the 0- to 4-foot interval.
4. ISM: incremental sampling methodology
   mg/kg: milligrams per kilogram
   ND: nondetect
   OU: operable unit
   PCB: polychlorinated biphenyl
5. Sample locations from the proposed ISM grid areas are for illustration purposes and may be revised in the field based on site conditions. Thirty increment samples will make up an ISM increment for each nonresidential area.

Anniston PCB Site
Anniston, Alabama
OU-1/OU-2 Predesign Investigation Work Plan

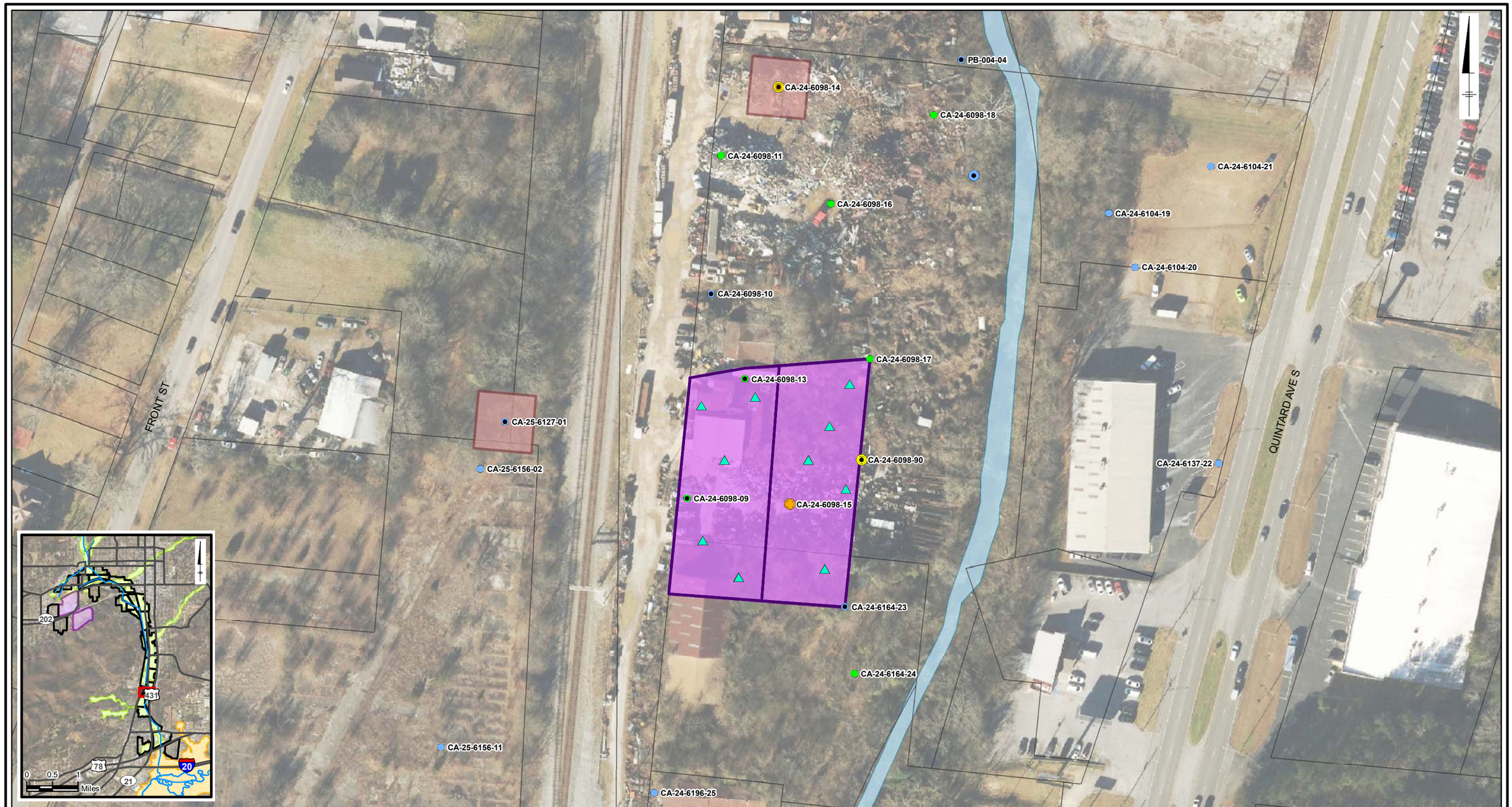
Proposed Soil Sampling Nonresidential Areas: Exposure Unit 19 South

PPIN: 66392

Solutia

Figure 3-4d

City: SYR Div/Group: SWG Created By: K. Ives Last Saved By: cnelson
Anniston
N:\Anniston\GIS\MXD\20220202_OU1_OU2_PDIWP\Rev2\Fig3-4d_PDIWP_OU1_OU2_Nonresidential_Proposed_Soil_Sampling_rev2.mxd 2/23/2022 5:49:55 PM



Legend

- Historical Analytes other than PCB and Lead Analyzed

Historical Surface Soil PCB Results (mg/kg)
- ND or < 1
- ≥ 1 to <10
- ≥ 10 to < 25
- ≥ 25 to < 50

- Proposed Nonresidential Soil Sample Location for 5-Point Composite
- All Parcels
- Snow Creek
- Remediation Footprints for Nonresidential Soils (non PCB constituents)
- Remediation Footprint for Nonresidential Soils (PCBs)

Inner and outer circles reflect surface and subsurface PCB concentrations, respectively.

0 100 200 Feet

Graphic Scale

Notes:
1. Aerial imagery provided by Calhoun and Talladega Counties.
2. Surface concentrations depicted represent the average of data from the 0- to 1-foot interval.
3. Subsurface concentrations depicted represent the average of data from the 0- to 4-foot interval.
4. mg/kg: milligrams per kilogram
   ND: nondetect
   OU: operable unit
   PCB: polychlorinated biphenyl
5. Proposed nonresidential samples will be composited from five sample locations within each nonresidential soils (PCBs) remediation footprint. Sample locations are for illustration purposes and may be revised in the field based on site conditions.

Anniston PCB Site
Anniston, Alabama
OU-1/OU-2 Predesign Investigation Work Plan

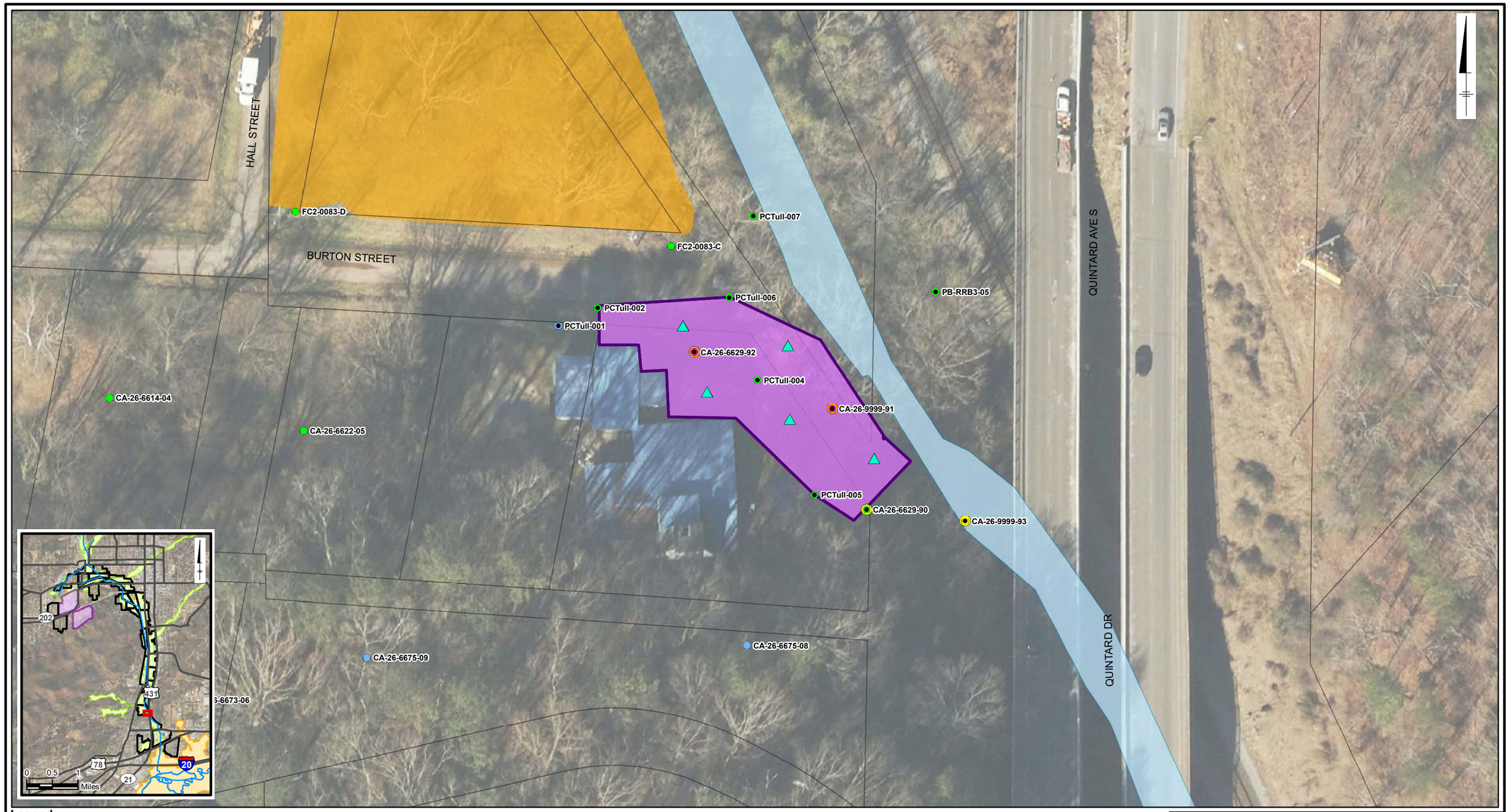
Proposed Soil Sampling Nonresidential Areas: Exposure Unit 24

PPIN: 64446 & 68005

Solutia

Figure **3-4e**

City: SYR Div/Group: SWG Created By: K. Ives Last Saved By: cnelson
Anniston
N:\AAnniston\GIS\MXD\20220202_OU1_OU2_PDIWP\Rev2\Fig3-4e_PDIWP_OU1_OU2_Nonresidential_Proposed_Soil_Sampling_rev2.mxd 2/24/2022 8:03:09 AM



Legend

- Historical Analytes other than PCB and Lead Analyzed

Historical Surface Soil PCB Results (mg/kg)

- ND or < 1
- ≥ 1 to <10
- ≥ 10 to < 25
- ≥ 25 to < 50
- ≥ 50 to < 500

- Proposed Nonresidential Soil Sample Location for 5-Point Composite
- All Parcels
- Snow Creek
- Interim Measure and Other Early Actions
- Remediation Footprint for Nonresidential Soils (PCBs)

Inner and outer circles reflect surface and subsurface PCB concentrations, respectively.

0 50 100 Feet

Graphic Scale

Notes:

1. Aerial imagery provided by Calhoun and Talladega Counties.
2. Surface concentrations depicted represent the average of data from the 0- to 1-foot interval.
3. Subsurface concentrations depicted represent the average of data from the 0- to 4-foot interval.
4. mg/kg: milligrams per kilogram
   ND: nondetect
   OU: operable unit
   PCB: polychlorinated biphenyl
5. Proposed nonresidential samples will be composited from five sample locations within each nonresidential soils (PCBs) remediation footprint. Sample locations are for illustration purposes and may be revised in the field based on site conditions.

Anniston PCB Site
Anniston, Alabama
OU-1/OU-2 Predesign Investigation Work Plan

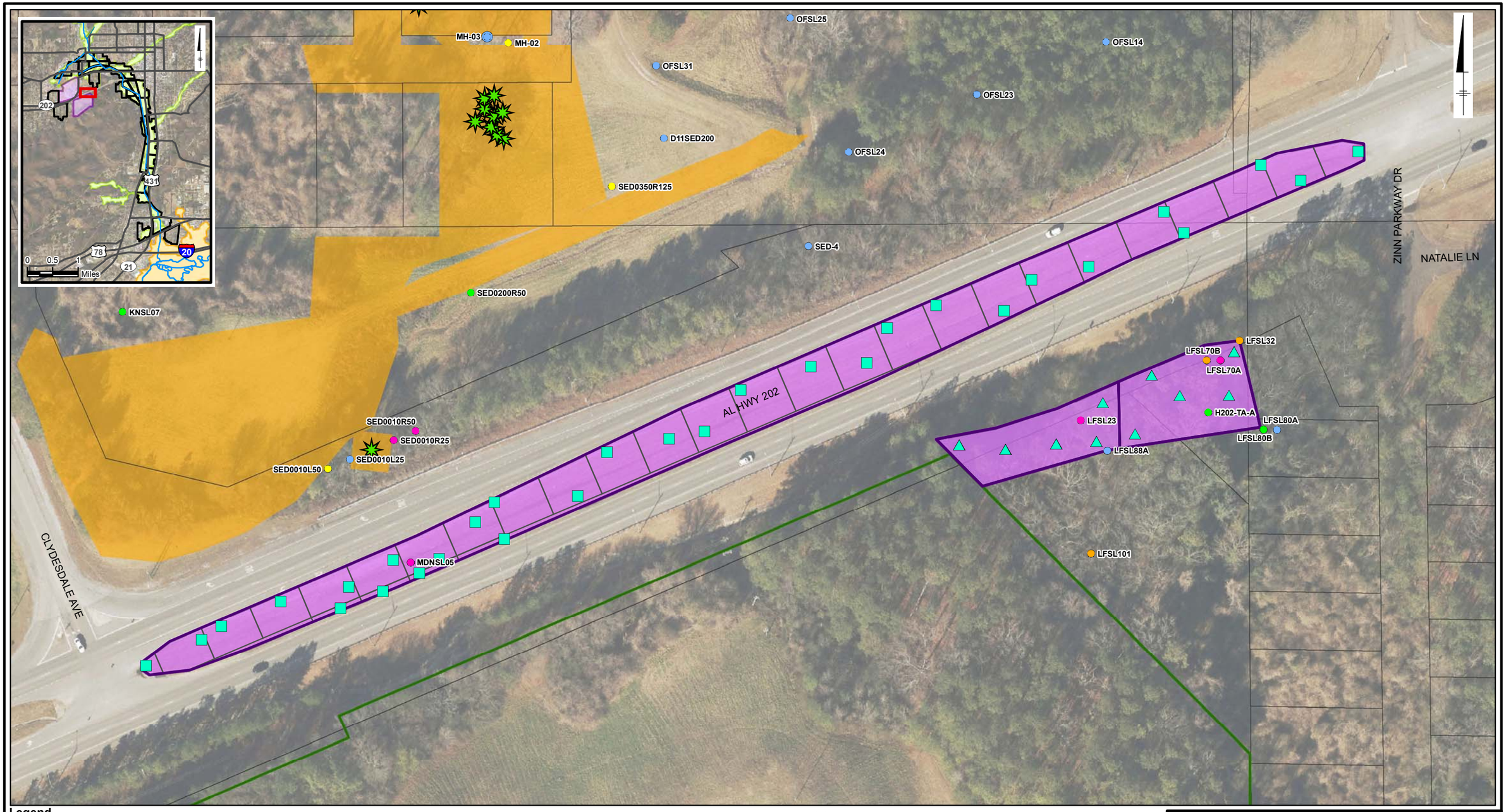
Proposed Soil Sampling
Nonresidential Areas: Exposure Unit 26

PPIN: 67593 & 67788

Solutia

Figure 3-4f

City: SYR Div/Group: SWG Created By: K. Ives Last Saved By: cnelson
Anniston
N:\AAnniston\GIS\MXD\20220202_OU1_OU2_PDIWP\Rev2\Fig3-4f_PDIWP_OU1_OU2_Nonresidential_Proposed_Soil_Sampling_rev2.mxd 2/24/2022 1:10:45 AM



Legend

Historical Surface Soil PCB Results (mg/kg)
- ND or < 1
- ≥ 1 to <10
- ≥ 10 to < 25
- ≥ 25 to < 50
- ≥ 50 to < 500

Historical Locations Beneath Cover System with Maximum PCB Results ≥500 mg/kg

Proposed Nonresidential Soil Sample Location for 5-Point Composite

Proposed Nonresidential ISM Soil Sampling Grid Location

All Parcels

Operable Unit 3

Interim Measure and Other Early Actions

Remediation Footprint for Nonresidential Soils (PCBs)

Proposed Nonresidential ISM Sampling Grid

Inner and outer circles reflect surface and subsurface PCB concentrations, respectively.

0 100 200 Feet

Graphic Scale

Notes:
1. Aerial imagery provided by Calhoun and Talladega Counties.
2. Surface concentrations depicted represent the average of data from the 0- to 1-foot interval.
3. Subsurface concentrations depicted represent the average of data from the 0- to 4-foot interval.
4. ISM: incremental sampling methodology
   mg/kg: milligrams per kilogram
   ND: nondetect
   OU: operable unit
   PCB: polychlorinated biphenyl
5. Sample locations from the proposed ISM grid areas are for illustration purposes and may be revised in the field based on site conditions. Thirty increment samples will make up an ISM increment for each nonresidential area.
6. Proposed nonresidential samples will be composited from five sample locations within each nonresidential soils (PCBs) remediation footprint. Sample locations are for illustration purposes and may be revised in the field based on site conditions.

Anniston PCB Site
Anniston, Alabama
OU-1/OU-2 Predesign Investigation Work Plan

Proposed Soil Sampling Nonresidential Areas: Highway 202

PPIN: 32133, 32746 & 32763

Solutia

Figure **3-4g**

City: SYR Div/Group: SWG Created By: K. Ives Last Saved By: cnelson
Anniston
N:\AAnniston\GIS\MXD\20220202_OU1_OU2_PDIWP\Rev2\Fig3-4g_PDIWP_OU1_OU2_Nonresidential_Proposed_Soil_Sampling_rev2.mxd 2/24/2022 1:42:48 AM



Legend

- Historical Analytes other than PCB and Lead Analyzed

Historical Surface Soil PCB Results (mg/kg)

- ND or < 1
- ≥ 50 to < 500
- Proposed Nonresidential Soil Sample Location for 5-Point Composite
- All Parcels
- Operable Unit 3
- Interim Measure and Other Early Actions
- Remediation Footprints for Nonresidential Soils (non PCB constituents)
- Remediation Footprint for Nonresidential Soils (PCBs)

Inner and outer circles reflect surface and subsurface PCB concentrations, respectively.

0 50 100 Feet

Graphic Scale

Notes:

1. Aerial imagery provided by Calhoun and Talladega Counties.
2. Surface concentrations depicted represent the average of data from the 0- to 1-foot interval.
3. Subsurface concentrations depicted represent the average of data from the 0- to 4-foot interval.
4. APCO: Alabama Power Corporation
   mg/kg: milligrams per kilogram
   ND: nondetect
   OU: operable unit
   PCB: polychlorinated biphenyl
5. Proposed nonresidential samples will be composited from five sample locations within each nonresidential soils (PCBs) remediation footprint. Sample locations are for illustration purposes and may be revised in the field based on site conditions.

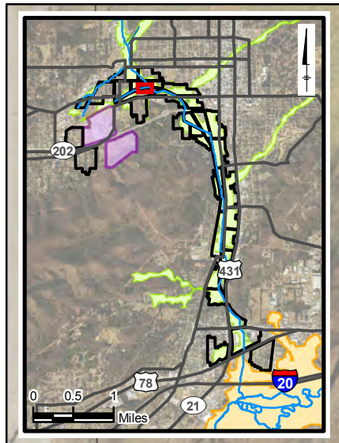
Anniston PCB Site
Anniston, Alabama
OU-1/OU-2 Predesign Investigation Work Plan

Proposed Soil Sampling Nonresidential Areas: North of APCO

Solutia

Figure 3-4h

City: SYR Div/Group: SWG Created By: K. Ives Last Saved By: cnelson
Anniston
N:\AlAnniston\GIS\MXD\20220202_OU1_OU2_PDIWP\Rev2\Fig3-4h_PDIWP_OU1_OU2_Nonresidential_Proposed_Soil_Sampling_rev2.mxd 2/24/2022 2:02:04 AM



PD2-012
CA-07-1520-09
CA-07-1520-08
CA-07-1435-03
CA-7-RES-1577-A
PECON-019
LINEASL04E
ASL-135
CA-05-8583-13
PB-RR-40
PB-RR-37
ASL-220
ASL-219
ASL-217
ASL-134
TH-5
ASL-224
TH-4
ASL-190
ASL-226
ASL-242

Legend

- Historical Analytes other than PCB and Lead Analyzed
- Historical Surface Soil PCB Results (mg/kg)
  - ND or < 1
  - ≥ 1 to <10
  - ≥ 10 to < 25
  - ≥ 25 to < 50
  - ≥ 50 to < 500
  - ≥ 500
- Historical Locations with Maximum PCB Results ≥500 mg/kg
- Proposed Soil Boring in PB-RR-37 Area
- 5-Point Composite Soil Sample Location
- Discrete Soil Sample Location Downstream of PB-RR-37
- 11th Street Ditch
- Snow Creek
- Interim Measure Area Remediation Footprint: Surface and Subsurface Soil Sampling During PDI
- Interim Measure and Other Early Actions
- Drainage Ditch Downstream PB-RR-37 Area
- All Parcels
- Inner and outer circles reflect surface and subsurface PCB concentrations, respectively.

0 60 120 Feet
Graphic Scale

Notes:
1. Aerial imagery provided by Calhoun and Talladega Counties.
2. Surface concentrations depicted represent the average of data from the 0- to 1-foot interval.
3. Subsurface concentrations depicted represent the average of data from the 0- to 4-foot interval.
4. bgs: below ground surface
   mg/kg: milligrams per kilogram
   ND: nondetect
   OU: operable unit
   PCB: polychlorinated biphenyl
   PDI: pre-design investigation
5. Five-point composite soil samples will be collected from the drainage ditch adjacent to and downstream of PB-RR-37. Composite samples will be collected from four sample depths: 0 to 1 foot bgs, 1 to 2 feet bgs, 2 to 3 feet bgs, and 3 to 4 feet bgs. Additionally, during collection of the 5-point composite sample, discrete samples for the two locations closest to Snow Creek will be collected from the same depth intervals. Samples collected from the soil depths of 1 to 2 feet bgs and below will be archived at the analytical laboratory pending analysis of samples collected from 0 to 1 foot bgs.
6. Five-point composite soil samples will be collected from the interim measure area remediation footprint surrounding PB-RR-37. Composite samples will be collected from four sample depths: 0 to 1 foot bgs, 1 to 2 feet bgs, 2 to 3 feet bgs, and 3 to 4 feet bgs. One of the composite locations will be co-located with the discrete boring at PB-RR-37 that is separately implemented to profile PCB concentrations in soil from ground surface to the water table. Split samples will be collected at this location to be submitted as part of the composite sample and as a discrete sample.

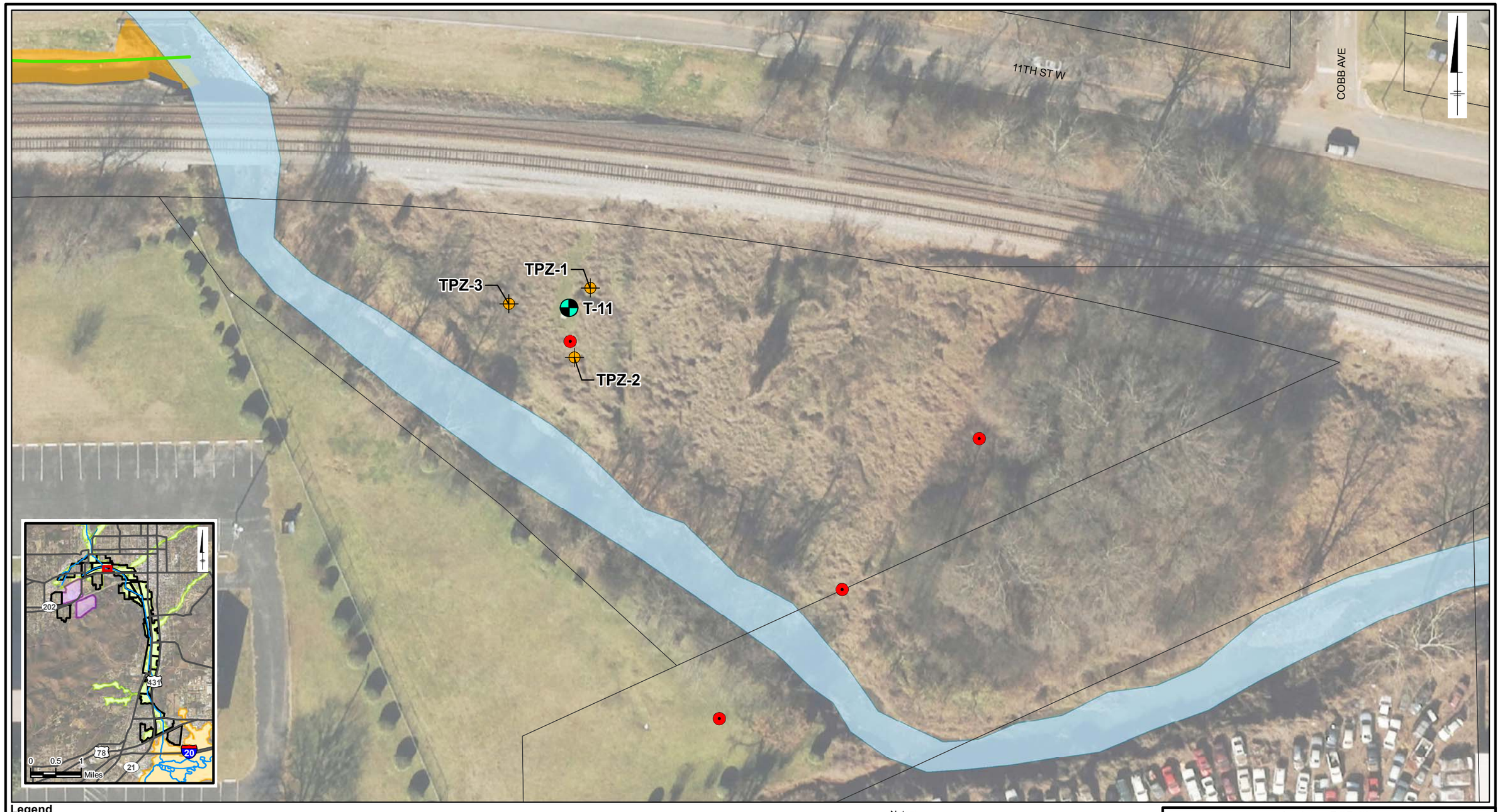
Anniston PCB Site
Anniston, Alabama
OU-1/OU-2 Predesign Investigation Work Plan

Proposed Soil Sampling in the PB-RR-37 Area

Solutia

Figure 3-5

City: SYR Div/Group: SWG Created By: K. Ives Last Saved By: NSekhon
Anniston
N:\Anniston\GIS\MXD\20220202_OU1_OU2_PDIWP\Rev2\Fig3-5_PDIWP_OU1_OU2_PB-RR-37_Area_ProposedSamples_rev2.mxd 6/7/2022 10:14:49 AM



Legend

- Existing Monitoring Well
- Proposed Temporary Observation Well Location
- Proposed Geotechnical Boring Location
- 11th Street Ditch
- Interim Measure and Other Early Actions
- All Parcels
- Snow Creek

Notes:
1. Aerial imagery provided by Calhoun and Talladega Counties.
2. OU: operable unit
   PCB: polychlorinated biphenyl

0 50 100 Feet
Graphic Scale

Anniston PCB Site
Anniston, Alabama
OU-1/OU-2 Predesign Investigation Work Plan

Proposed Geotechnical Borings and Piezometer Locations at the T-11 Area

Solutia

Figure 3-6

City: SYR Div/Group: SWG Created By: K. Ives Last Saved By: cnelson
Anniston
N:\AlAnniston\GIS\MXD\20220202_OU1_OU2_PDIWP\Rev2\Fig3-6_PDIWP_OU1_OU2_Proposed_GeotechBorings_PZ Locations_T-11_Area_rev2.mxd 2/24/2022 2:30:32 AM

Footprint of
Former Church

SED0600
3,300

MH-9 (0-6")
1,800

MH-8 (0-4")
2,200

MH-7 (0-6")
6,200

SED0420L5
24,000

SED0420R5
25,000

SED0410L10
49,000

SED0400
200,000

SED0400L5
22,000

SED0400R5
60,000

SED0400L25
1,200

SED0390L10
3,300

SED0370R10
11,000

SED0380L5
79,000

740

750

Northside Property Boundary

Eastside Property Boundary

LEGEND

Total PCB Concentration in Soil (mg/kg) (See Note 3)
- ND to < 1
- ≥ 1 to < 10
- ≥ 10 to < 25
- ≥ 25 to < 50
- ≥ 50 to < 500
- ≥ 500

Total PCB Screening Result (Immunoassay) (ppm) (See Note 3)
- < 1; < 5; < 10
- > 10; > 25
- Soil-Geotextile Cover
- OU-3 Boundary

- Berm
- HDPE Geomembrane Liner (See Note 2)
- Exposure Unit
- Solutia-Owned Parcels
- Footprint of Former
- Shallow Residuum Potentiometric Surface Contours from May 2020 (in feet above mean sea level)

- Monitoring well for PCB sampling and water level monitoring
- Piezometer for water level monitoring only (to be plugged after gauging)
- Soil boring with PCB depth profile sampling during well installation

Notes:

1. Aerial imagery taken from https://gis.calhouncounty.org/arcgis6/rest/services/ParcelOrthos/MapServer.
2. This includes areas where the HDPE liner is interpreted to be present based on field observations in the Interim Measure construction.
3. Maximum PCB concentration at any depth in analysis profile shown for each location. Screening data are presented where no analytical data are available. Analytical and screening data are present in the OU-1/OU-2 Remedial Investigation Report.
4. ND: nondetect
   HDPE: high-density polyethylene
   mg/kg: milligrams per kilogram
   OU: operable unit
   PCB: polychlorinated biphenyl
   ppm: parts per million

0 50 100 Feet

Graphic Scale

Anniston PCB Site
Anniston, Alabama
OU-1/OU-2 Predesign Investigation Work Plan

Proposed Monitoring Well and Piezometer Locations at the Eastside Properties

Solutia

Figure 3-7

Div/Group: GSI; Drawn by: CDM; Checked by: WBS
Path: J:\6101-6150\6124 Solutia Anniston OU-1OU-2 PDIWP Groundwater 2022\Figures\GIS\MXDs\6124_002_01_Fig3-7_EastsideProperties_ProposedMW.mxd



LEGEND

- Existing Monitoring Well
- Decommissioned Monitoring Well
- Monitoring well for PCB sampling and water level monitoring
- Piezometer for water level monitoring only (to be plugged after gauging)
- Soil boring with PCB depth profile sampling during well installation
- 756.78 — Measured Potentiometric Surface Elevation (in feet above mean sea level)
- Shallow Residuum Potentiometric Surface Contours from May 2020 (in feet above mean sea level)

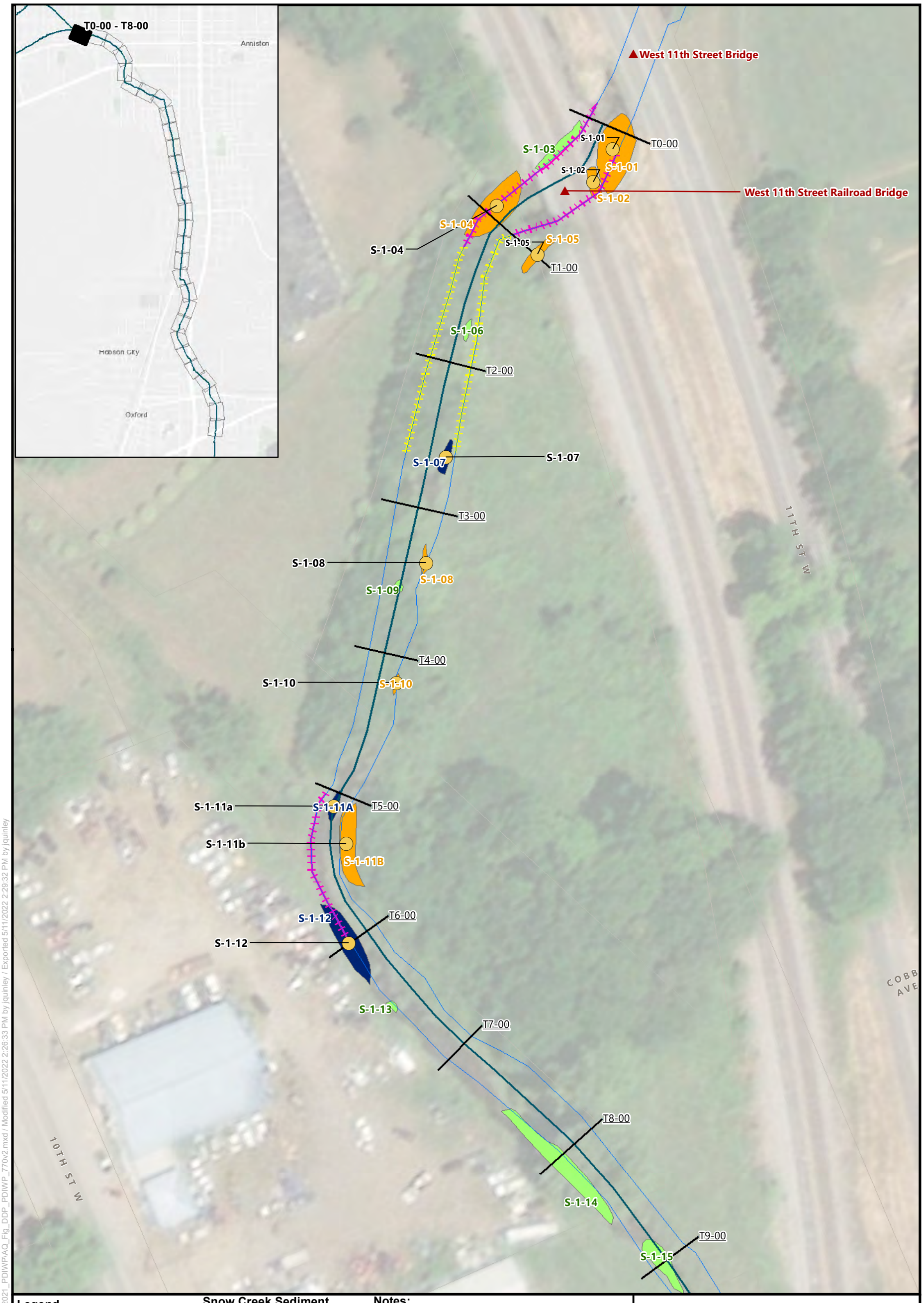
Anniston PCB Site
Anniston, Alabama
OU-1/OU-2 Predesign Investigation Work Plan

Proposed and Existing Monitoring Wells and Proposed Piezometers in the Eastside Properties Area

Solutia

Figure 3-8

Div/Group: GSI; Drawn by: CDM; Checked by: STR
Path: J:\6101-6150\6124 Solutia Anniston OU-1OU-2 PDIWP Groundwater 2022\Figures\GIS\MXDs\6124_002_02_Fig3-8_EP_Proposed-Existing_MW.mxd



Legend

- Known Structures along Snow Creek
- Previous Sediment Sample Location
- PDI Stations/Transects (100-foot spacing)
- Planned Engineered Bank Measures
- Planned Natural Bank Stability Measures
- Snow Creek Centerline
- Snow Creek Shoreline
- Calhoun County Parcels

Snow Creek Sediment Deposits

- ≥ 3 mg/kg PCB (estimated concentration)
- < 3 mg/kg PCB (average concentration)
- ≥ 3 mg/kg PCB (average concentration)

Notes:

1. Aerial imagery is from Esri basemaps service (accessed: 5/11/2022).

2. Transect station lines are spaced every 100 feet perpendicular to the Snow Creek centerline.

3. Snow Creek sediment deposits are based on the 1999 Resource Conservation Recovery Act Facility Investigation (RFI) geomorphological mapping of the creek.

mg/kg: milligrams per kilogram
PCB: polychlorinated biphenyl
PDI: predesign investigation

0 50 100 Feet

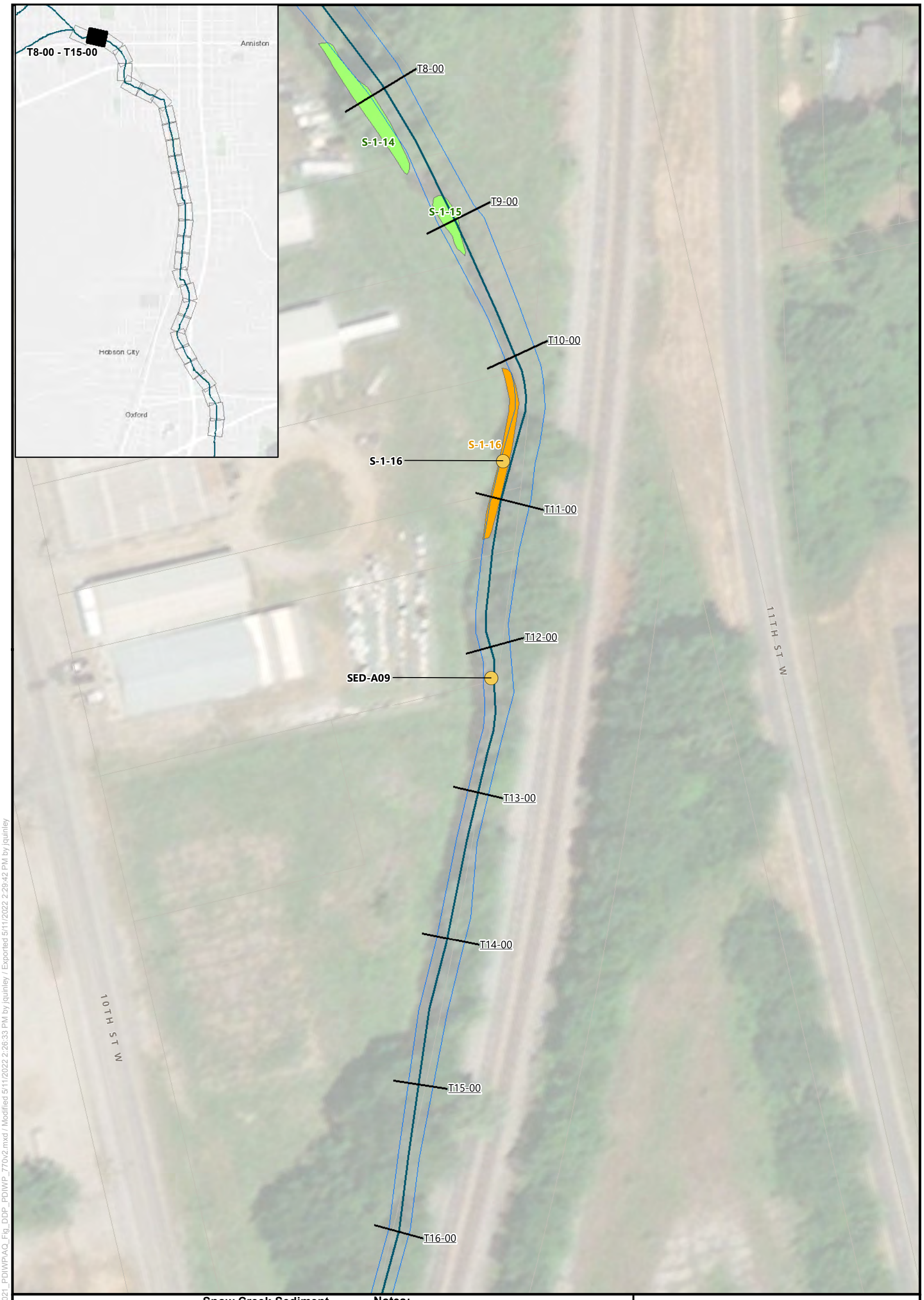
Anniston PCB Site
Anniston, Alabama
OU-1/OU-2 Predesign Investigation Work Plan

Snow Creek Predesign Investigation Transects T0-00 through T8-00

Solutia

Figure 3-9a

File Path: \\orcas\GIS\Jobs\Solutia\Inc_1718\Anniston_PCB_SiteMaps\2021_PDIWP\AQ_Fig_DDP_PDIWP_770v2.mxd / Modified 5/11/2022 2:26:33 PM by jquinley / Exported 5/11/2022 2:29:32 PM by jquinley



Legend

- Previous Sediment Sample Location
- PDI Stations/Transects (100-foot spacing)
- Snow Creek Centerline
- Snow Creek Shoreline
- Calhoun County Parcels

Snow Creek Sediment Deposits

- ≥ 3 mg/kg PCB (estimated concentration)
- ≥ 3 mg/kg PCB (average concentration)

Notes:

1. Aerial imagery is from Esri basemaps service (accessed: 5/11/2022).

2. Transect station lines are spaced every 100 feet perpendicular to the Snow Creek centerline.

3. Snow Creek sediment deposits are based on the 1999 Resource Conservation Recovery Act Facility Investigation (RFI) geomorphological mapping of the creek.

mg/kg: milligrams per kilogram
PCB: polychlorinated biphenyl
PDI: predesign investigation

0 50 100 Feet

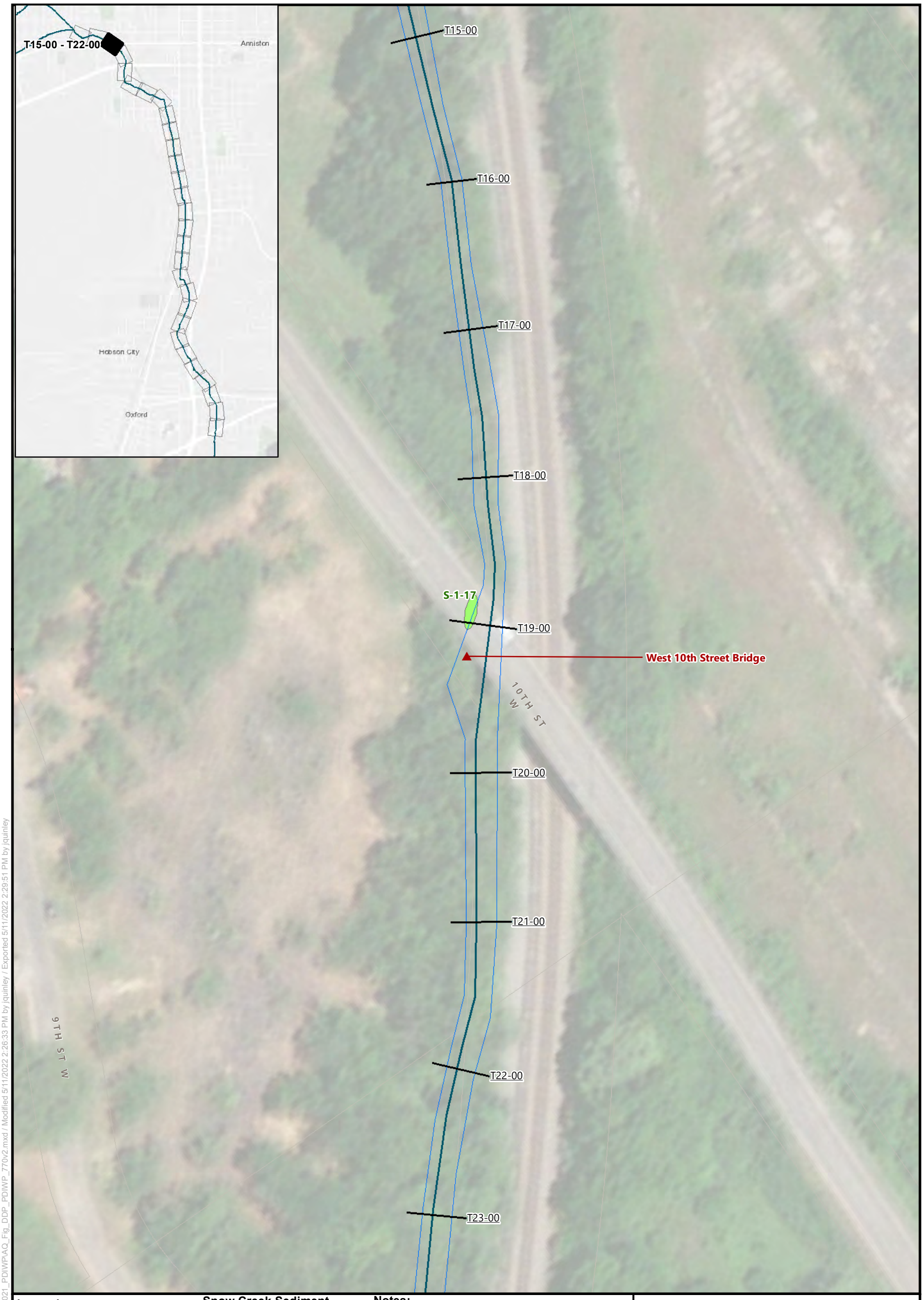
Anniston PCB Site
Anniston, Alabama
OU-1/OU-2 Predesign Investigation Work Plan

Snow Creek
Predesign Investigation Transects
T8-00 through T15-00

Solutia
Ingenuity Productivity Performance

Figure 3-9b

File Path: \\orcas\GIS\Jobs\Solutia\nc_1718\Anniston_PCB_SiteMaps\2021_PDIWPAAQ_Fig_DDP_PDIWP_770v2.mxd / Modified 5/11/2022 2:26:33 PM by jquinley / Exported 5/11/2022 2:29:42 PM by jquinley



Legend

- Known Structures along Snow Creek
- PDI Stations/Transects (100-foot spacing)
- Snow Creek Centerline
- Snow Creek Shoreline
- Calhoun County Parcels

Snow Creek Sediment Deposits

- ≥ 3 mg/kg PCB (estimated concentration)

Notes:

1. Aerial imagery is from Esri basemaps service (accessed: 5/11/2022).

2. Transect station lines are spaced every 100 feet perpendicular to the Snow Creek centerline.

3. Snow Creek sediment deposits are based on the 1999 Resource Conservation Recovery Act Facility Investigation (RFI) geomorphological mapping of the creek.

mg/kg: milligrams per kilogram
PCB: polychlorinated biphenyl
PDI: predesign investigation

0 50 100 Feet

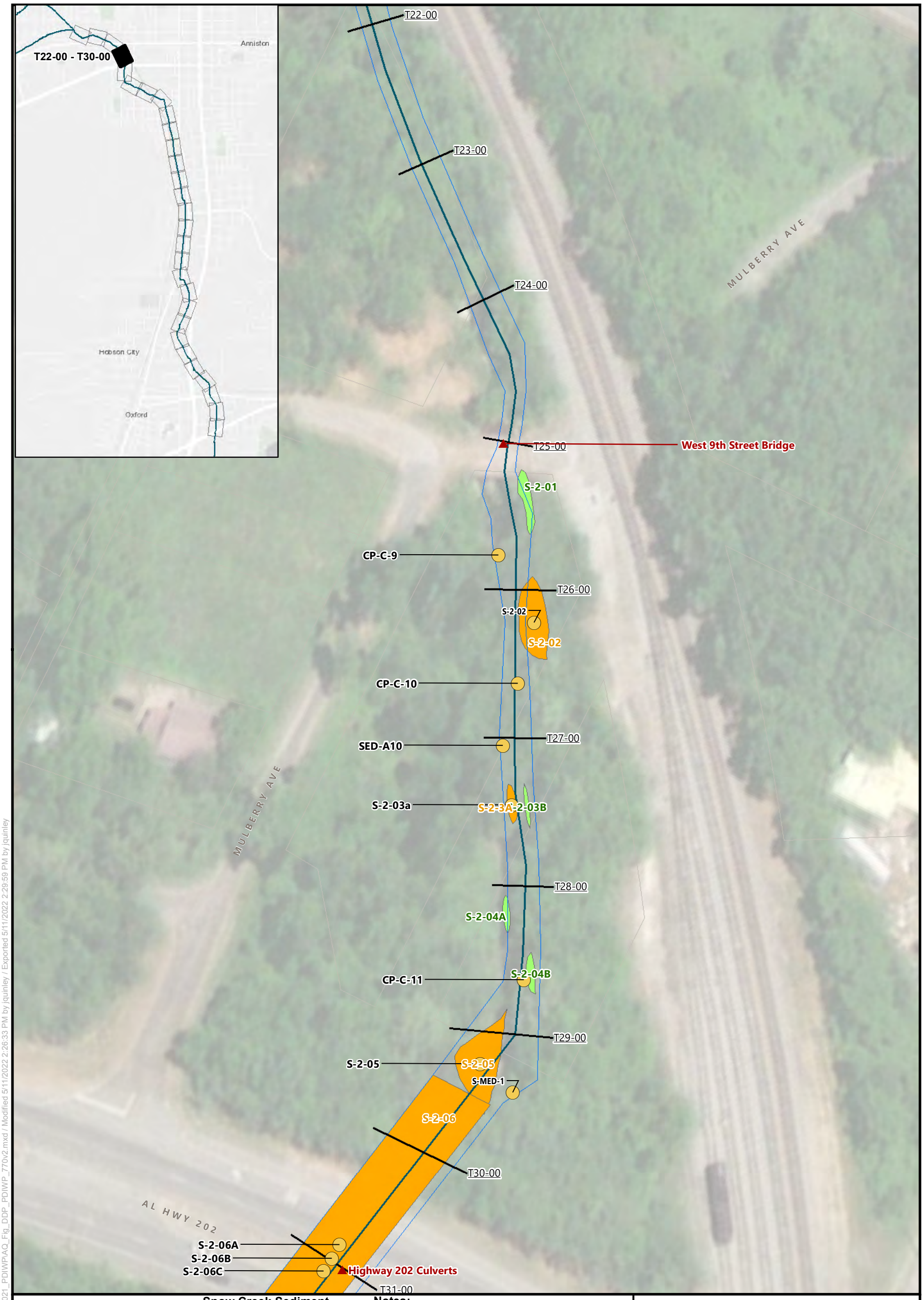
Anniston PCB Site
Anniston, Alabama
OU-1/OU-2 Predesign Investigation Work Plan

Snow Creek
Predesign Investigation Transects
T15-00 through T22-00

Solutia Ingenuity Productivity Performance

Figure 3-9c

File Path: \\orcas\GISJobs\Solutia\Inc_1718\Anniston_PCB_SiteMaps\2021_PDIWPAQ_Fig_DDP_PDIWP_770v2.mxd / Modified 5/11/2022 2:26:33 PM by jquinley / Exported 5/11/2022 2:29:51 PM by jquinley



Legend

- Known Structures along Snow Creek
- Previous Sediment Sample Location
- PDI Stations/Transects (100-foot spacing)
- Snow Creek Centerline
- Snow Creek Shoreline
- Calhoun County Parcels

Snow Creek Sediment Deposits

- ≥ 3 mg/kg PCB (estimated concentration)
- ≥ 3 mg/kg PCB (average concentration)

Notes:

1. Aerial imagery is from Esri basemaps service (accessed: 5/11/2022).

2. Transect station lines are spaced every 100 feet perpendicular to the Snow Creek centerline.

3. Snow Creek sediment deposits are based on the 1999 Resource Conservation Recovery Act Facility Investigation (RFI) geomorphological mapping of the creek.

mg/kg: milligrams per kilogram
PCB: polychlorinated biphenyl
PDI: predesign investigation

0 50 100 Feet

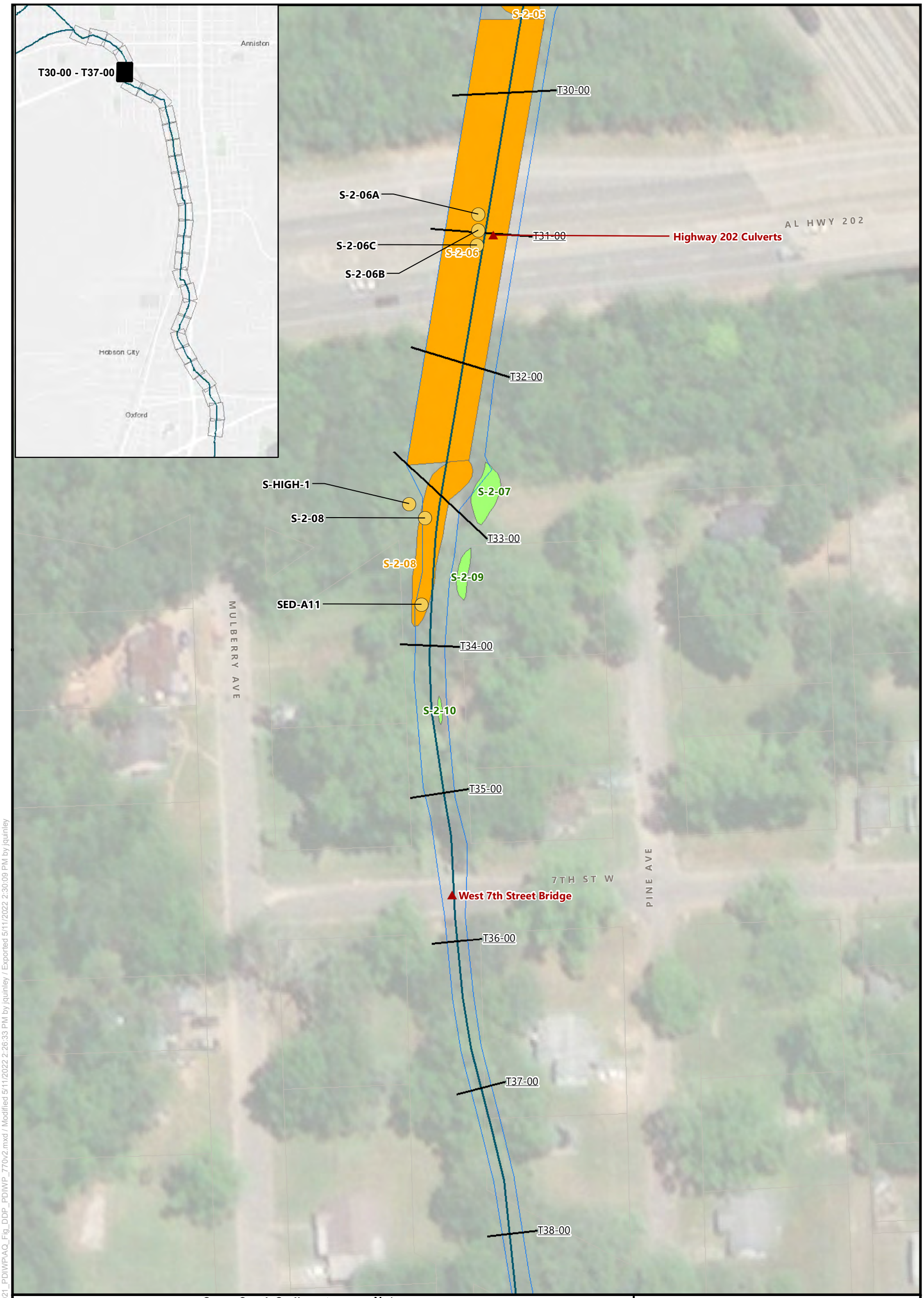
Anniston PCB Site
Anniston, Alabama
OU-1/OU-2 Predesign Investigation Work Plan

Snow Creek
Predesign Investigation Transects
T22-00 through T30-00

Solutia
Ingenuity Productivity Performance

Figure 3-9d

File Path: \\orcas\GISJobs\Solutia\nc_1718\Anniston_PCB_SiteMaps\2021_PDIWPAQ_Fig_DDP_PDIWP_770v2.mxd / Modified 5/11/2022 2:26:33 PM by jquinley / Exported 5/11/2022 2:29:59 PM by jquinley



Legend

- Known Structures along Snow Creek
- Previous Sediment Sample Location
- PDI Stations/Transects (100-foot spacing)
- Snow Creek Centerline
- Snow Creek Shoreline
- Calhoun County Parcels

Snow Creek Sediment Deposits

- ≥ 3 mg/kg PCB (estimated concentration)
- ≥ 3 mg/kg PCB (average concentration)

Notes:

1. Aerial imagery is from Esri basemaps service (accessed: 5/11/2022).

2. Transect station lines are spaced every 100 feet perpendicular to the Snow Creek centerline.

3. Snow Creek sediment deposits are based on the 1999 Resource Conservation Recovery Act Facility Investigation (RFI) geomorphological mapping of the creek.

mg/kg: milligrams per kilogram
PCB: polychlorinated biphenyl
PDI: predesign investigation

0 50 100 Feet

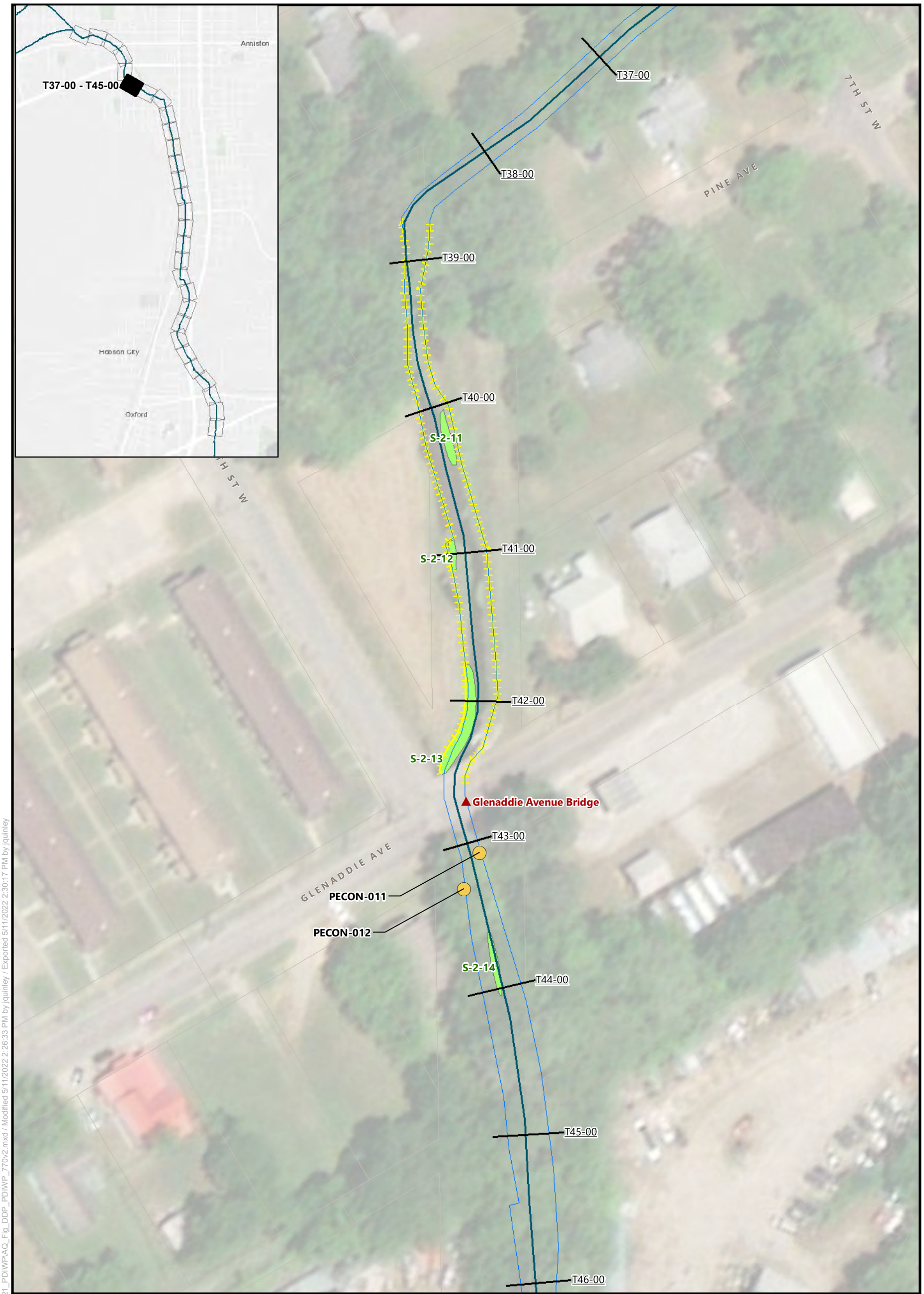
Anniston PCB Site
Anniston, Alabama
OU-1/OU-2 Predesign Investigation Work Plan

Snow Creek
Predesign Investigation Transects
T30-00 through T37-00

Solutia
Ingenuity Productivity Performance

Figure 3-9e

File Path: \\orcas\GISJobs\Solutia\nc_1718\Anniston_PCB_SiteMaps\2021_PDIWPAQ_Fig_DDP_PDIWP_770v2.mxd / Modified 5/11/2022 2:26:33 PM by jquinley / Exported 5/11/2022 2:30:09 PM by jquinley



Legend

- Known Structures along Snow Creek
- Previous Sediment Sample Location
- PDI Stations/Transects (100-foot spacing)
- Planned Natural Bank Stability Measures
- Snow Creek Centerline
- Snow Creek Shoreline
- Calhoun County Parcels

Snow Creek Sediment Deposits

- ≥ 3 mg/kg PCB (estimated concentration)

Notes:

1. Aerial imagery is from Esri basemaps service (accessed: 5/11/2022).

2. Transect station lines are spaced every 100 feet perpendicular to the Snow Creek centerline.

3. Snow Creek sediment deposits are based on the 1999 Resource Conservation Recovery Act Facility Investigation (RFI) geomorphological mapping of the creek.

mg/kg: milligrams per kilogram
PCB: polychlorinated biphenyl
PDI: predesign investigation

0 50 100 Feet

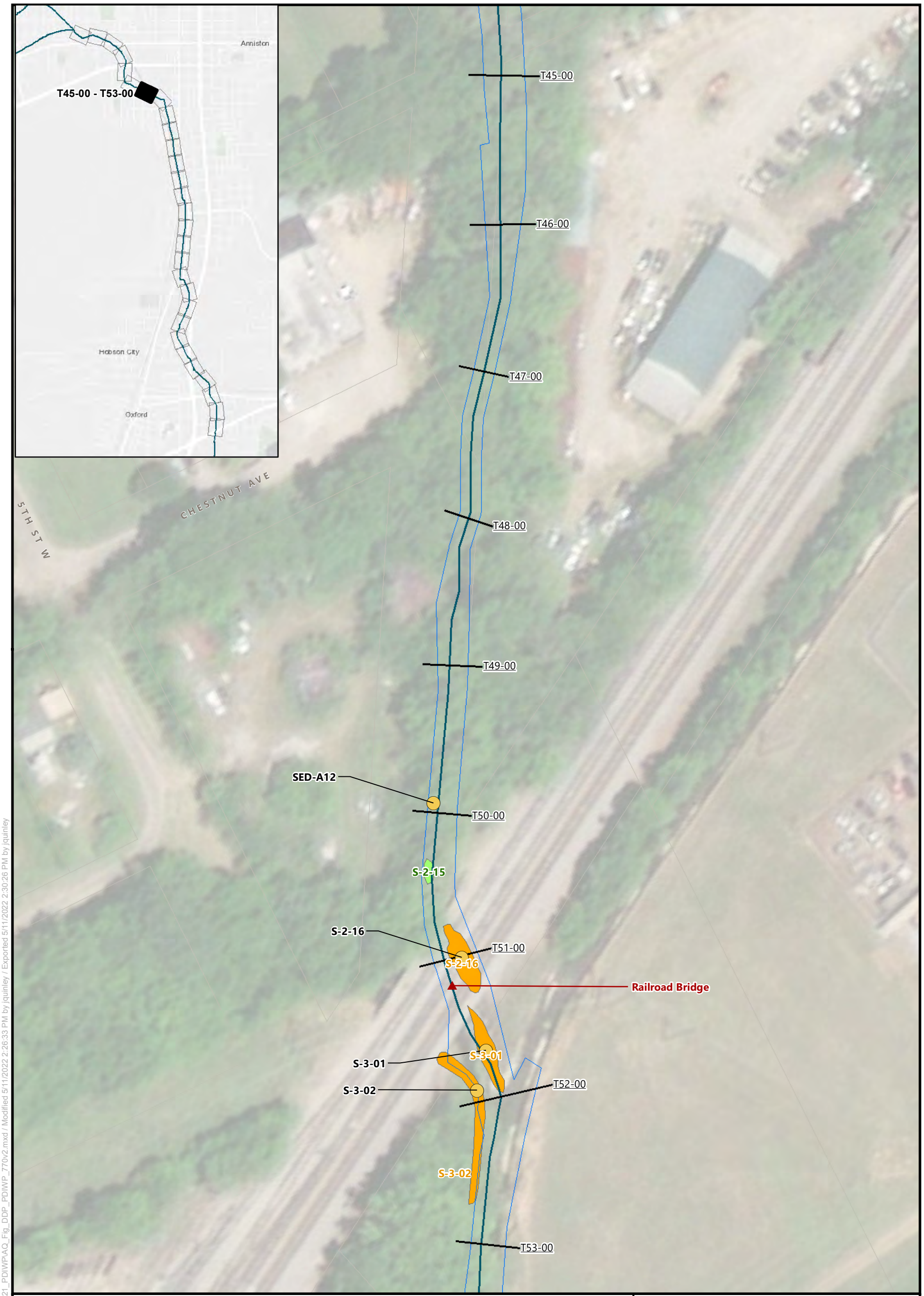
Anniston PCB Site
Anniston, Alabama
OU-1/OU-2 Predesign Investigation Work Plan

Snow Creek
Predesign Investigation Transects
T37-00 through T45-00

Solutia
Ingenuity Productivity Performance

Figure 3-9f

File Path: \\orcas\GIS\Jobs\Solutia\nc_1718\Anniston_PCB_SiteMaps\2021_PDIWPAQ_Fig_DDP_PDIWP_770v2.mxd / Modified 5/11/2022 2:26:33 PM by jquinley / Exported 5/11/2022 2:30:17 PM by jquinley



Legend

- Known Structures along Snow Creek
- Previous Sediment Sample Location
- PDI Stations/Transects (100-foot spacing)
- Snow Creek Centerline
- Snow Creek Shoreline
- Calhoun County Parcels

Snow Creek Sediment Deposits

- ≥ 3 mg/kg PCB (estimated concentration)
- ≥ 3 mg/kg PCB (average concentration)

Notes:

1. Aerial imagery is from Esri basemaps service (accessed: 5/11/2022).

2. Transect station lines are spaced every 100 feet perpendicular to the Snow Creek centerline.

3. Snow Creek sediment deposits are based on the 1999 Resource Conservation Recovery Act Facility Investigation (RFI) geomorphological mapping of the creek.

mg/kg: milligrams per kilogram
PCB: polychlorinated biphenyl
PDI: predesign investigation

0 50 100 Feet

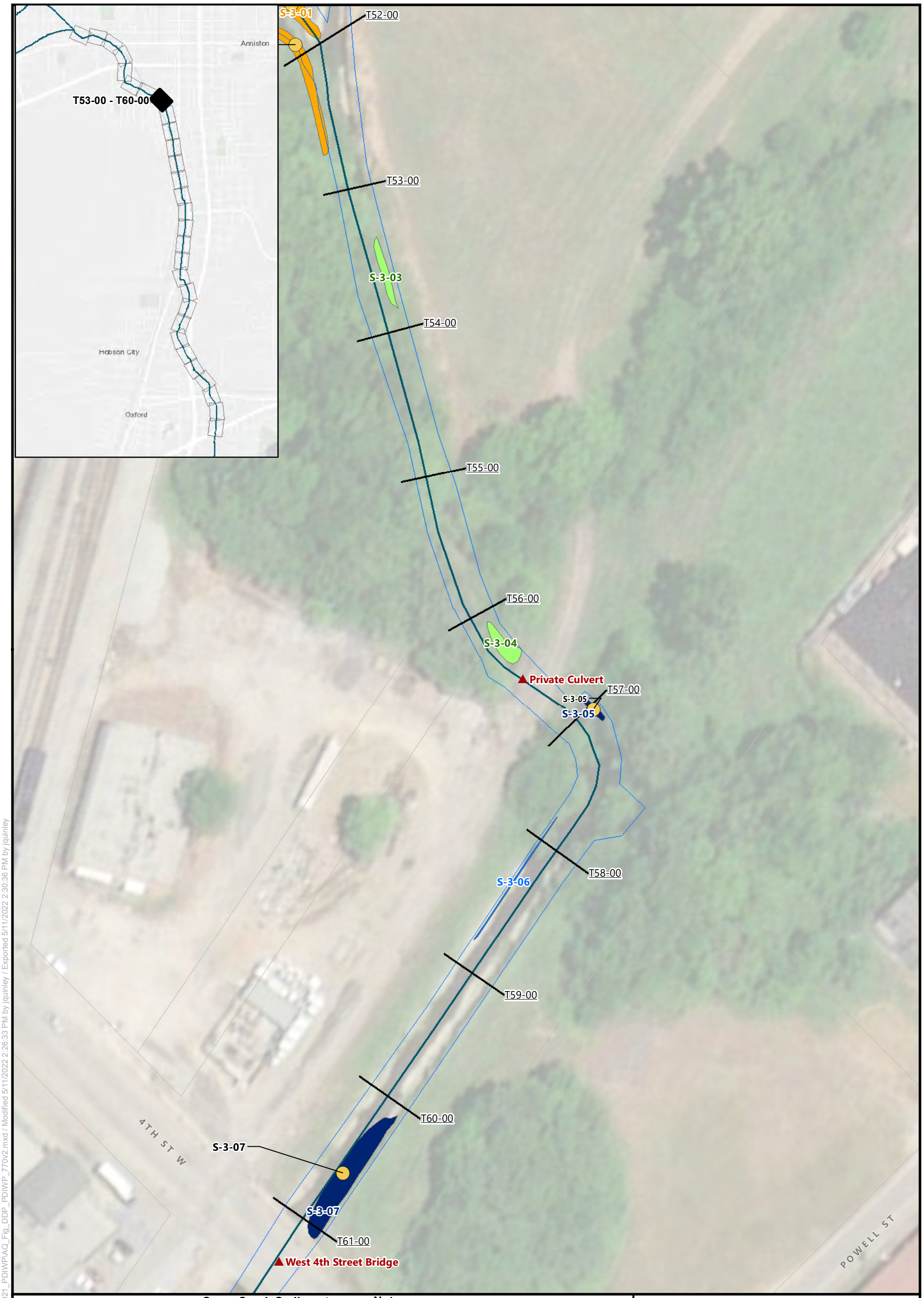
Anniston PCB Site
Anniston, Alabama
OU-1/OU-2 Predesign Investigation Work Plan

Snow Creek
Predesign Investigation Transects
T45-00 through T53-00

Solutia
Ingenuity Productivity Performance

Figure 3-9g

File Path: \\orcas\GIS\Jobs\Solutia\Inc_1718\Anniston_PCB_SiteMaps\2021_PDIWP\AQ_Fig_DDP_PDIWP_770v2.mxd / Modified 5/11/2022 2:26:33 PM by jquinley / Exported 5/11/2022 2:30:26 PM by jquinley



Legend

- Known Structures along Snow Creek
- Previous Sediment Sample Location
- PDI Stations/Transects (100-foot spacing)
- Snow Creek Centerline
- Snow Creek Shoreline
- Calhoun County Parcels

Snow Creek Sediment Deposits

- < 3 mg/kg PCB (estimated concentration)
- ≥ 3 mg/kg PCB (estimated concentration)
- < 3 mg/kg PCB (average concentration)
- ≥ 3 mg/kg PCB (average concentration)

Notes:

1. Aerial imagery is from Esri basemaps service (accessed: 5/11/2022).

2. Transect station lines are spaced every 100 feet perpendicular to the Snow Creek centerline.

3. Snow Creek sediment deposits are based on the 1999 Resource Conservation Recovery Act Facility Investigation (RFI) geomorphological mapping of the creek.

mg/kg: milligrams per kilogram
PCB: polychlorinated biphenyl
PDI: predesign investigation

0 50 100 Feet

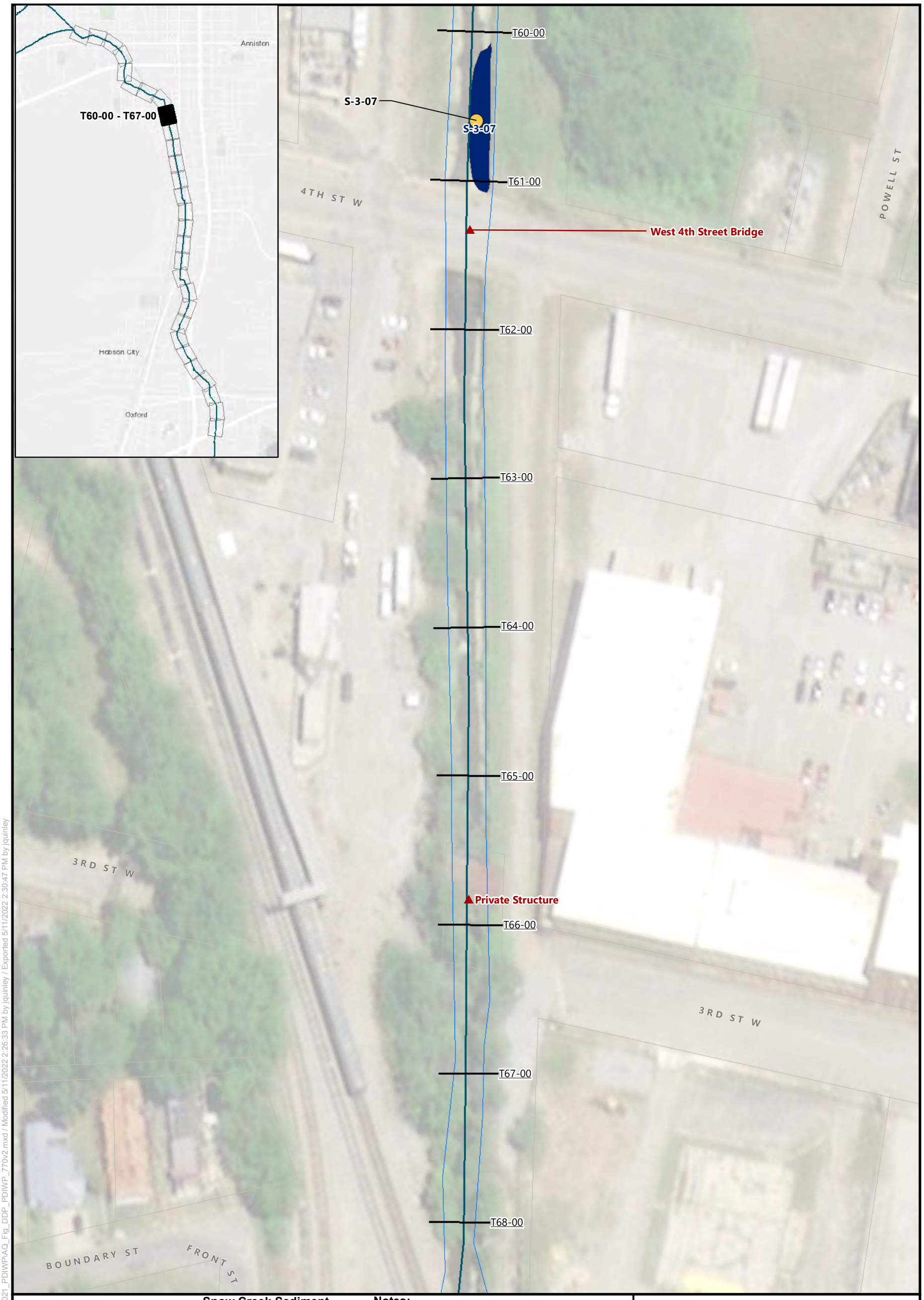
Anniston PCB Site
Anniston, Alabama
OU-1/OU-2 Predesign Investigation Work Plan

Snow Creek
Predesign Investigation Transects
T53-00 through T60-00

Solutia
Ingenuity Productivity Performance

Figure 3-9h

File Path: \\orcas\GIS\Jobs\Solutia\Inc_1718\Anniston_PCB_Site\Maps\2021_PDIWP\AQ_Fig_DDP_PDIWP_770v2.mxd / Modified 5/11/2022 2:26:33 PM by jquinley / Exported 5/11/2022 2:30:36 PM by jquinley



Legend

- Known Structures along Snow Creek
- Previous Sediment Sample Location
- PDI Stations/Transects (100-foot spacing)
- Snow Creek Centerline
- Snow Creek Shoreline
- Calhoun County Parcels

Snow Creek Sediment Deposits

- < 3 mg/kg PCB (average concentration)

Notes:

1. Aerial imagery is from Esri basemaps service (accessed: 5/11/2022).

2. Transect station lines are spaced every 100 feet perpendicular to the Snow Creek centerline.

3. Snow Creek sediment deposits are based on the 1999 Resource Conservation Recovery Act Facility Investigation (RFI) geomorphological mapping of the creek.

mg/kg: milligrams per kilogram
PCB: polychlorinated biphenyl
PDI: predesign investigation

0 50 100 Feet

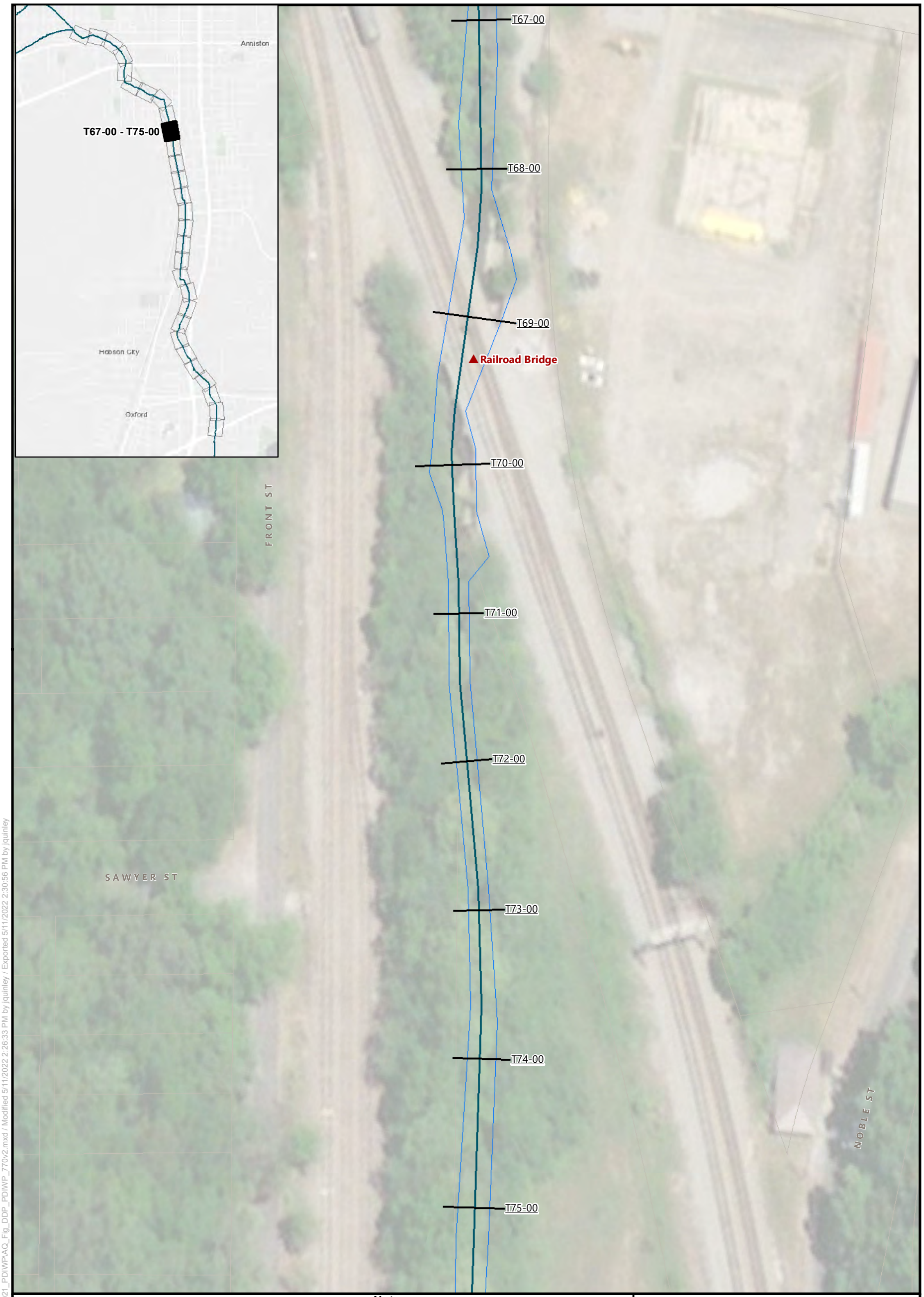
Anniston PCB Site
Anniston, Alabama
OU-1/OU-2 Predesign Investigation Work Plan

Snow Creek
Predesign Investigation Transects
T60-00 through T67-00

Solutia Ingenuity Productivity Performance

Figure 3-9i

File Path: \\orcas\GISJobs\Solutia\nc_1718\Anniston_PCB_SiteMaps\2021_PDIWPAQ_Fig_DDP_PDIWP_770v2.mxd / Modified 5/11/2022 2:26:33 PM by jquinley / Exported 5/11/2022 2:30:47 PM by jquinley



Legend

- Known Structures along Snow Creek
- PDI Stations/Transects (100-foot spacing)
- Snow Creek Centerline
- Snow Creek Shoreline
- Calhoun County Parcels

Notes:

1. Aerial imagery is from Esri basemaps service (accessed: 5/11/2022).

2. Transect station lines are spaced every 100 feet perpendicular to the Snow Creek centerline.

mg/kg: milligrams per kilogram
PCB: polychlorinated biphenyl
PDI: predesign investigation

0 50 100 Feet

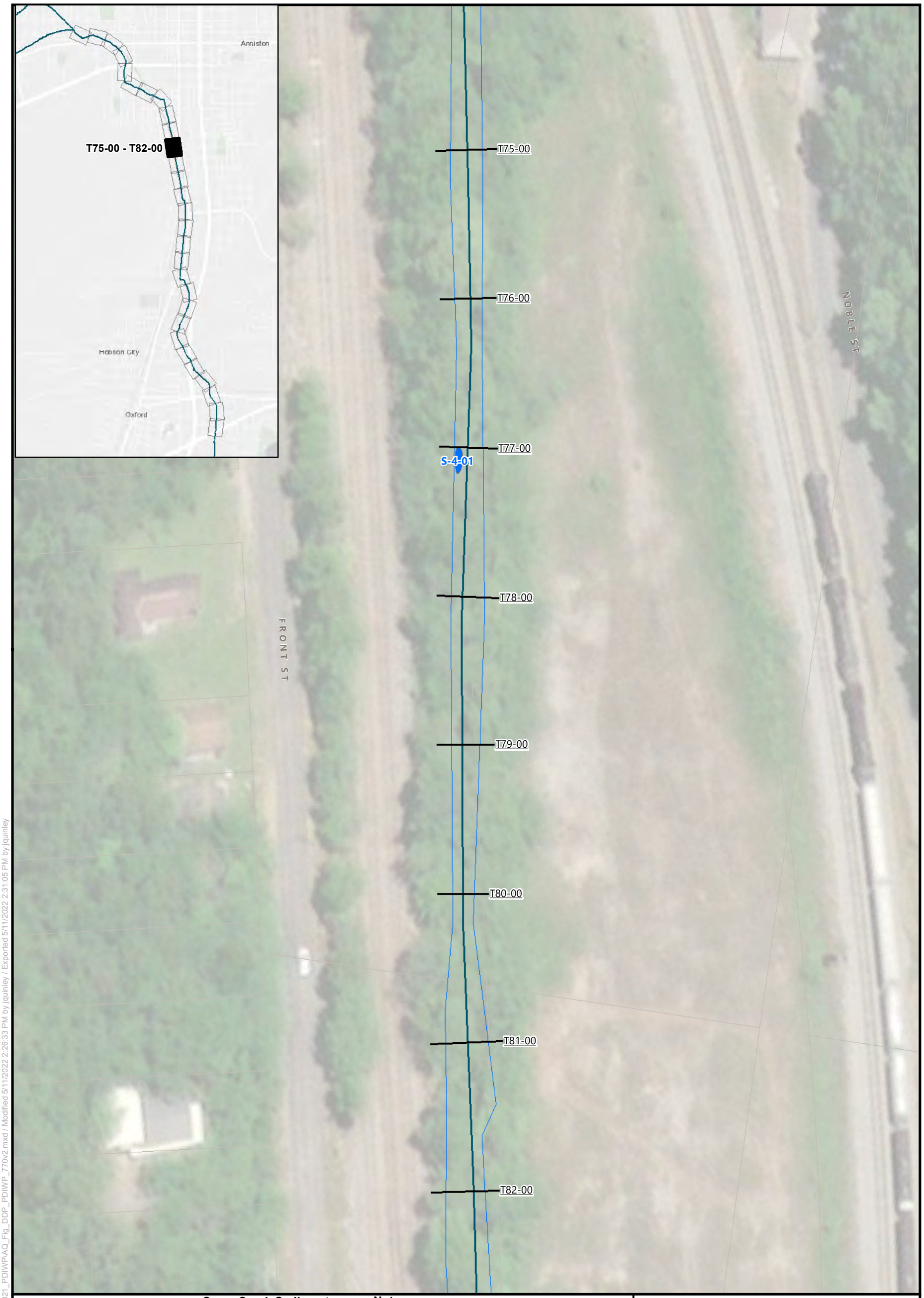
Anniston PCB Site
Anniston, Alabama
OU-1/OU-2 Predesign Investigation Work Plan

Snow Creek
Predesign Investigation Transects
T67-00 through T75-00

Solutia
Ingenuity Productivity Performance

Figure 3-9j

File Path: \\orcas\GISJobs\Solutia\nc_1718\Anniston_PCB_SiteMaps\2021_PDIWPAQ_Fig_DDP_PDIWP_770v2.mxd / Modified 5/11/2022 2:26:33 PM by jquinley / Exported 5/11/2022 2:30:56 PM by jquinley



Legend

- PDI Stations/Transects (100-foot spacing)
- Snow Creek Centerline
- Snow Creek Shoreline
- Calhoun County Parcels

Snow Creek Sediment Deposits

- < 3 mg/kg PCB (estimated concentration)

Notes:

1. Aerial imagery is from Esri basemaps service (accessed: 5/11/2022).

2. Transect station lines are spaced every 100 feet perpendicular to the Snow Creek centerline.

3. Snow Creek sediment deposits are based on the 1999 Resource Conservation Recovery Act Facility Investigation (RFI) geomorphological mapping of the creek.

mg/kg: milligrams per kilogram
PCB: polychlorinated biphenyl
PDI: predesign investigation

0 50 100 Feet

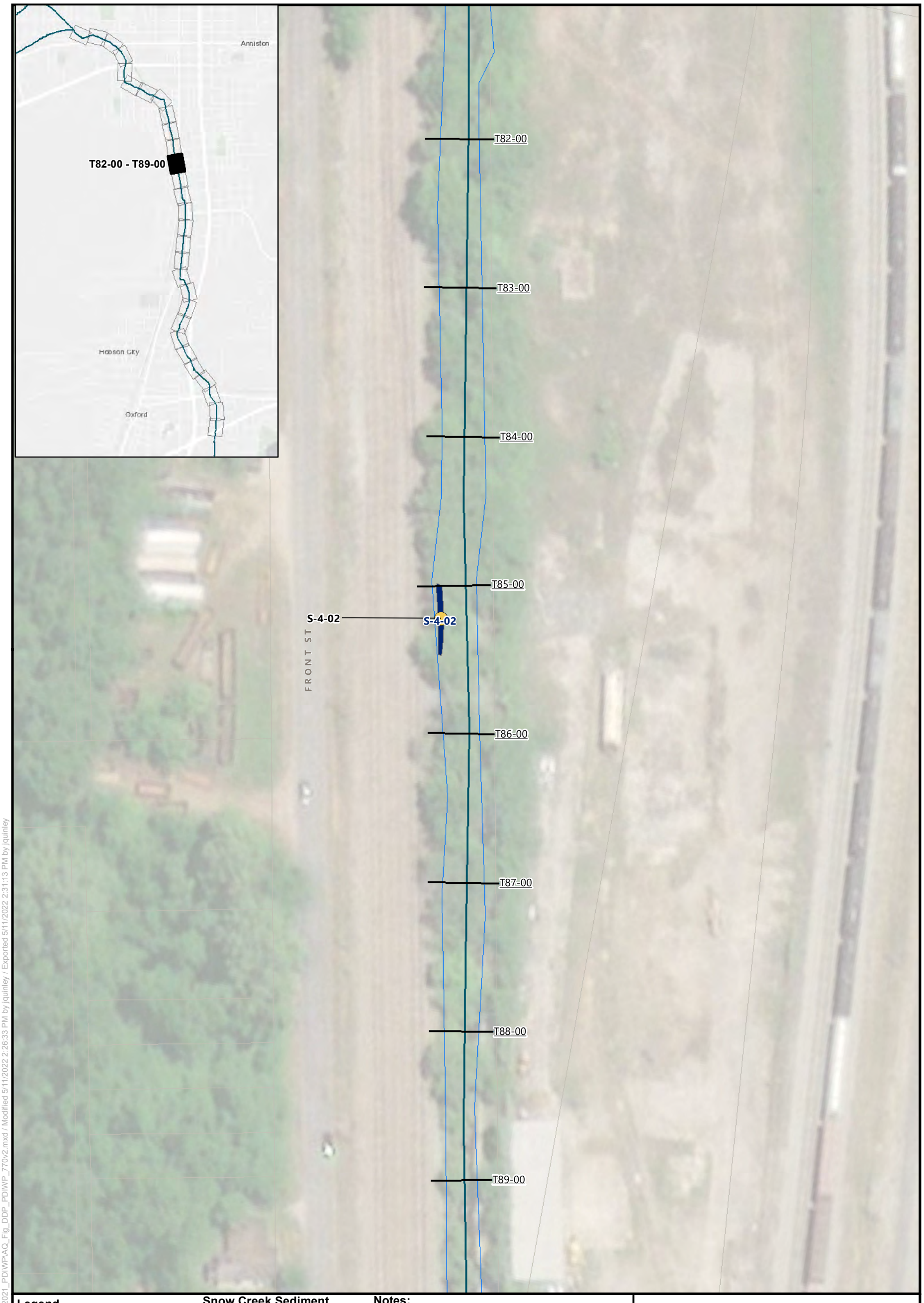
Anniston PCB Site
Anniston, Alabama
OU-1/OU-2 Predesign Investigation Work Plan

Snow Creek
Predesign Investigation Transects
T75-00 through T82-00

Solutia
Ingenuity Productivity Performance

Figure 3-9k

File Path: \\orcas\GISJobs\Solutia\nc_1718\Anniston_PCB_Site\Maps\2021_PDIWP\AQ_Fig_DDP_PDIWP_770v2.mxd / Modified 5/11/2022 2:26:33 PM by jquinley / Exported 5/11/2022 2:31:05 PM by jquinley



Legend

- Previous Sediment Sample Location
- PDI Stations/Transects (100-foot spacing)
- Snow Creek Centerline
- Snow Creek Shoreline
- Calhoun County Parcels

Snow Creek Sediment Deposits

- < 3 mg/kg PCB (average concentration)

Notes:

1. Aerial imagery is from Esri basemaps service (accessed: 5/11/2022).

2. Transect station lines are spaced every 100 feet perpendicular to the Snow Creek centerline.

3. Snow Creek sediment deposits are based on the 1999 Resource Conservation Recovery Act Facility Investigation (RFI) geomorphological mapping of the creek.

mg/kg: milligrams per kilogram
PCB: polychlorinated biphenyl
PDI: predesign investigation

0 50 100 Feet

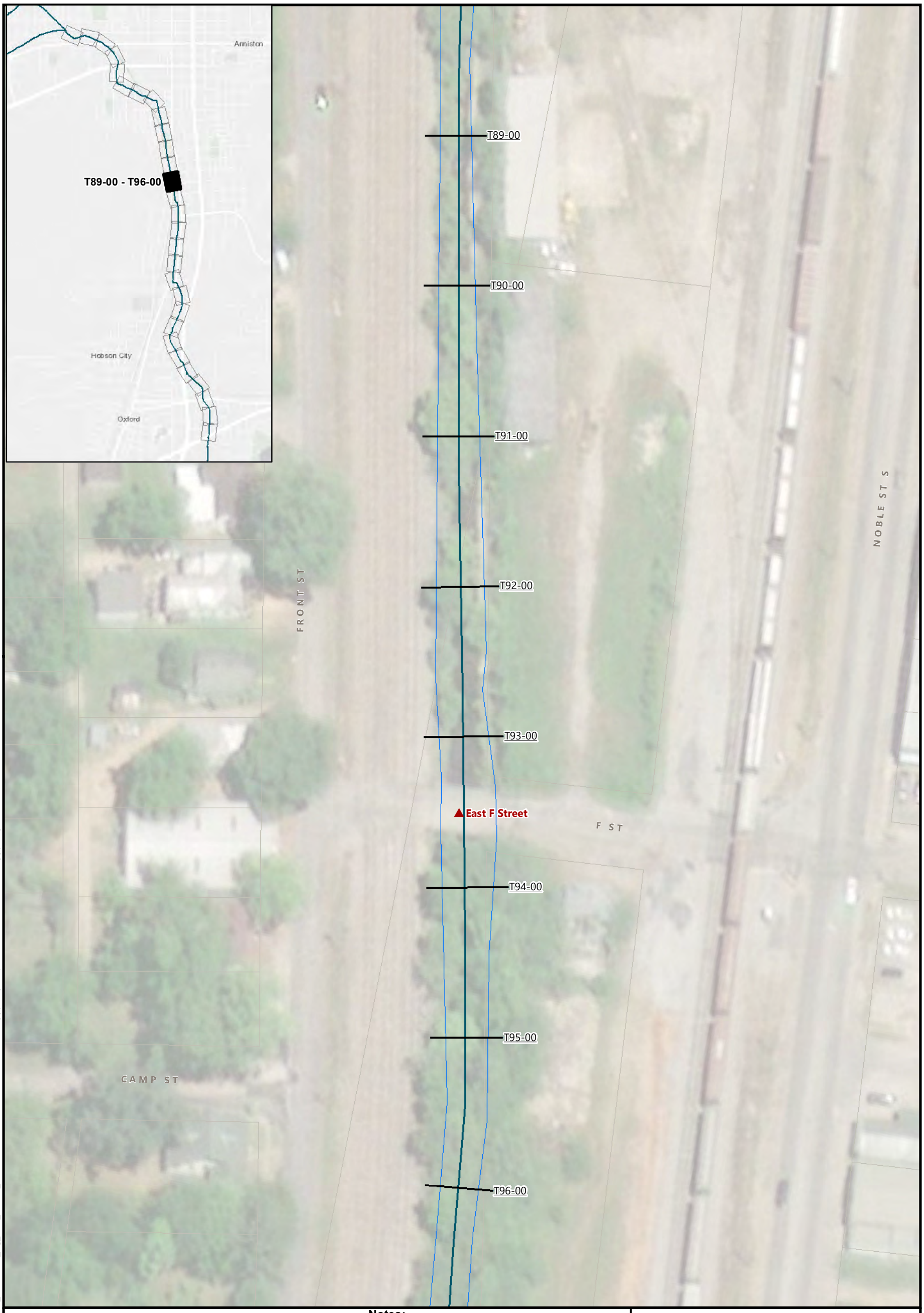
Anniston PCB Site
Anniston, Alabama
OU-1/OU-2 Predesign Investigation Work Plan

Snow Creek
Predesign Investigation Transects
T82-00 through T89-00

Solutia Ingenuity Productivity Performance

Figure 3-9l

File Path: \\orcas\GISJobs\SolutiaInc_1718\Anniston_PCB_Site\Maps\2021_PDIWPAQ_Fig_DDP_PDIWP_770v2.mxd / Modified 5/11/2022 2:26:33 PM by jquinley / Exported 5/11/2022 2:31:13 PM by jquinley



Legend

- Known Structures along Snow Creek
- PDI Stations/Transects (100-foot spacing)
- Snow Creek Centerline
- Snow Creek Shoreline
- Calhoun County Parcels

Notes:

1. Aerial imagery is from Esri basemaps service (accessed: 5/11/2022).

2. Transect station lines are spaced every 100 feet perpendicular to the Snow Creek centerline.

mg/kg: milligrams per kilogram
PCB: polychlorinated biphenyl
PDI: predesign investigation

0 50 100 Feet

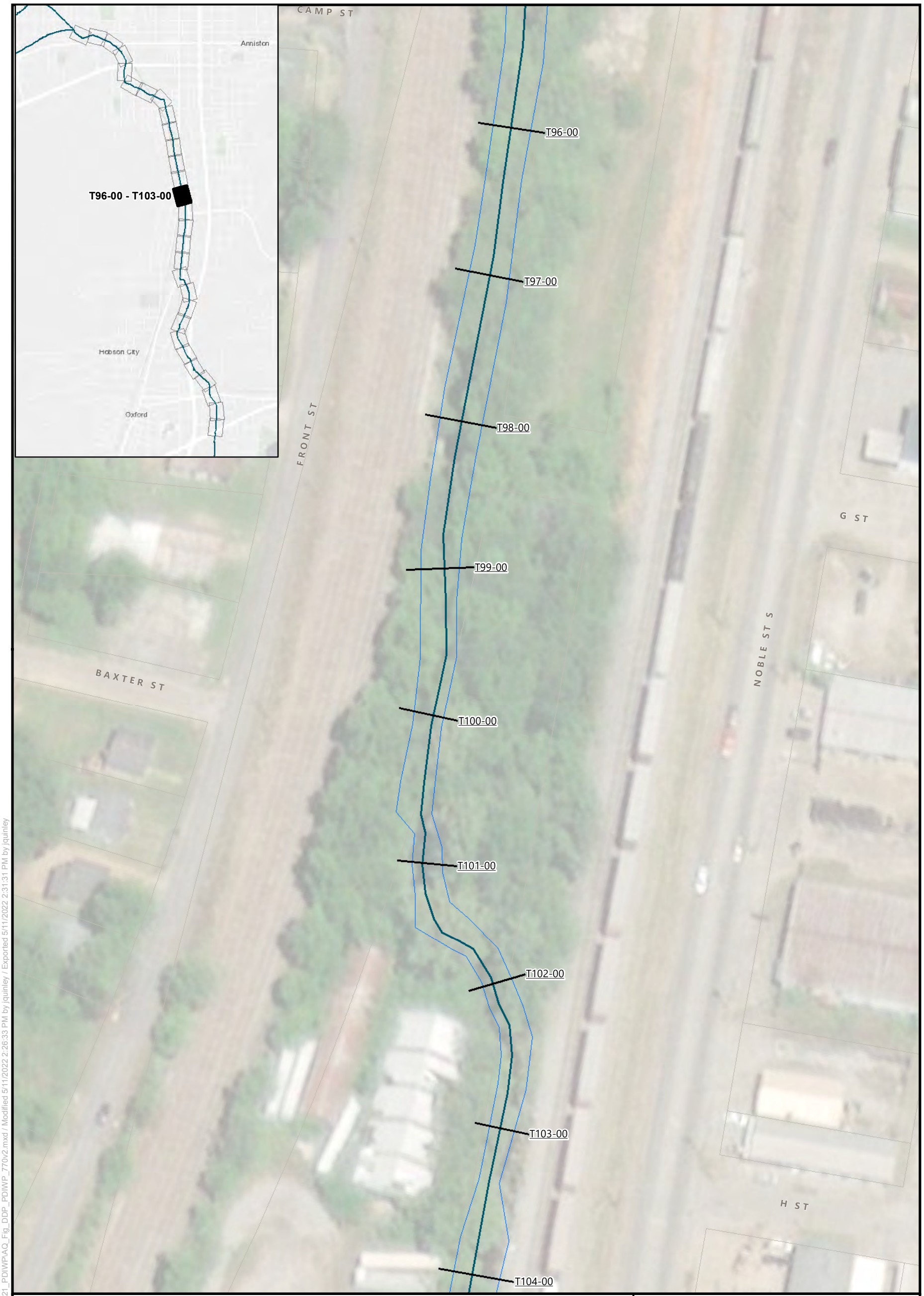
Anniston PCB Site
Anniston, Alabama
OU-1/OU-2 Predesign Investigation Work Plan

Snow Creek
Predesign Investigation Transects
T89-00 through T96-00

Solutia
Ingenuity Productivity Performance

Figure 3-9m

File Path: \\orcas\GIS\Jobs\SolutiaInc_1718\Anniston_PCB_SiteMaps\2021_PDIWP\AQ_Fig_DDP_PDIWP_770v2.mxd / Modified 5/11/2022 2:26:33 PM by jquinley / Exported 5/11/2022 2:31:22 PM by jquinley



Legend

- PDI Stations/Transects (100-foot spacing)
- Snow Creek Centerline
- Snow Creek Shoreline
- Calhoun County Parcels

Notes:

1. Aerial imagery is from Esri basemaps service (accessed: 5/11/2022).

2. Transect station lines are spaced every 100 feet perpendicular to the Snow Creek centerline.

mg/kg: milligrams per kilogram
PCB: polychlorinated biphenyl
PDI: predesign investigation

0 50 100 Feet

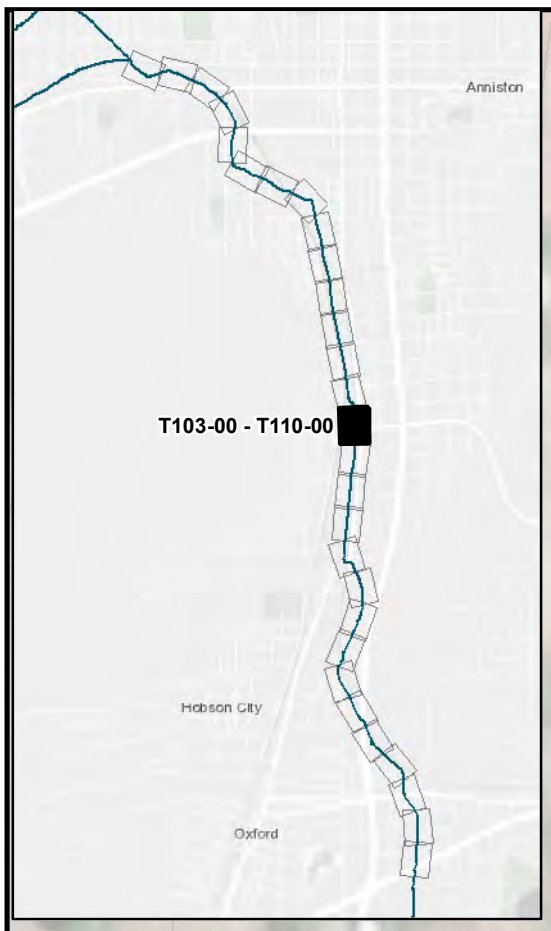
Anniston PCB Site
Anniston, Alabama
OU-1/OU-2 Predesign Investigation Work Plan

Snow Creek
Predesign Investigation Transects
T96-00 through T103-00

Solutia
Ingenuity Productivity Performance

Figure 3-9n

File Path: \\orcas\GIS\Jobs\Solutia\Inc_1718\Anniston_PCB_SiteMaps\2021_PDIWPAQ_Fig_DDP_PDIWP_770v2.mxd / Modified 5/11/2022 2:26:33 PM by jquinley / Exported 5/11/2022 2:31:31 PM by jquinley



T103-00
T104-00
T105-00
T106-00
T107-00
T108-00
T109-00
T110-00
T111-00

H ST
NOBLE ST S
GREENBRIER DEAR RD

Legend

- PDI Stations/Transects (100-foot spacing)
- Snow Creek Centerline
- Snow Creek Shoreline
- Calhoun County Parcels

Notes:

1. Aerial imagery is from Esri basemaps service (accessed: 5/11/2022).

2. Transect station lines are spaced every 100 feet perpendicular to the Snow Creek centerline.

mg/kg: milligrams per kilogram
PCB: polychlorinated biphenyl
PDI: predesign investigation

0 50 100 Feet

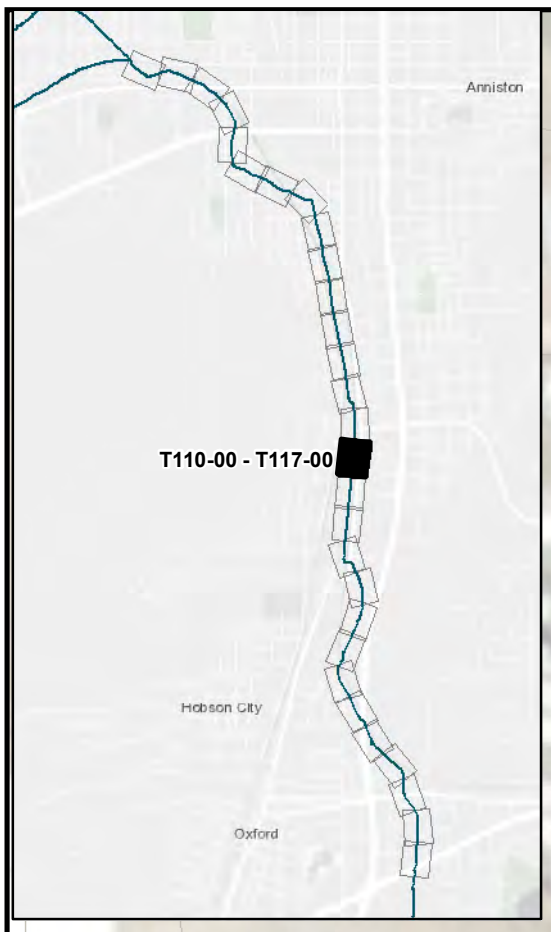
Anniston PCB Site
Anniston, Alabama
OU-1/OU-2 Predesign Investigation Work Plan

Snow Creek
Predesign Investigation Transects
T103-00 through T110-00

Solutia Ingenuity Productivity Performance

Figure 3-9o

File Path: \\orcas\GIS\Jobs\Solutia\Inc_1718\Anniston_PCB_SiteMaps\2021_PDIWPAQ_Fig_DDP_PDIWP_770v2.mxd / Modified 5/11/2022 2:26:33 PM by jquinley / Exported 5/11/2022 2:31:40 PM by jquinley



T110-00

T111-00

T112-00

T113-00

S-5-01

S-5-01

T114-00

NOBLE ST S

T115-00

T116-00

T117-00

T118-00

Legend

- Previous Sediment Sample Location
- PDI Stations/Transects (100-foot spacing)
- Snow Creek Centerline
- Snow Creek Shoreline
- Calhoun County Parcels

Snow Creek Sediment Deposits

- < 3 mg/kg PCB (average concentration)

Notes:

1. Aerial imagery is from Esri basemaps service (accessed: 5/11/2022).

2. Transect station lines are spaced every 100 feet perpendicular to the Snow Creek centerline.

3. Snow Creek sediment deposits are based on the 1999 Resource Conservation Recovery Act Facility Investigation (RFI) geomorphological mapping of the creek.

mg/kg: milligrams per kilogram
PCB: polychlorinated biphenyl
PDI: predesign investigation

0 50 100 Feet

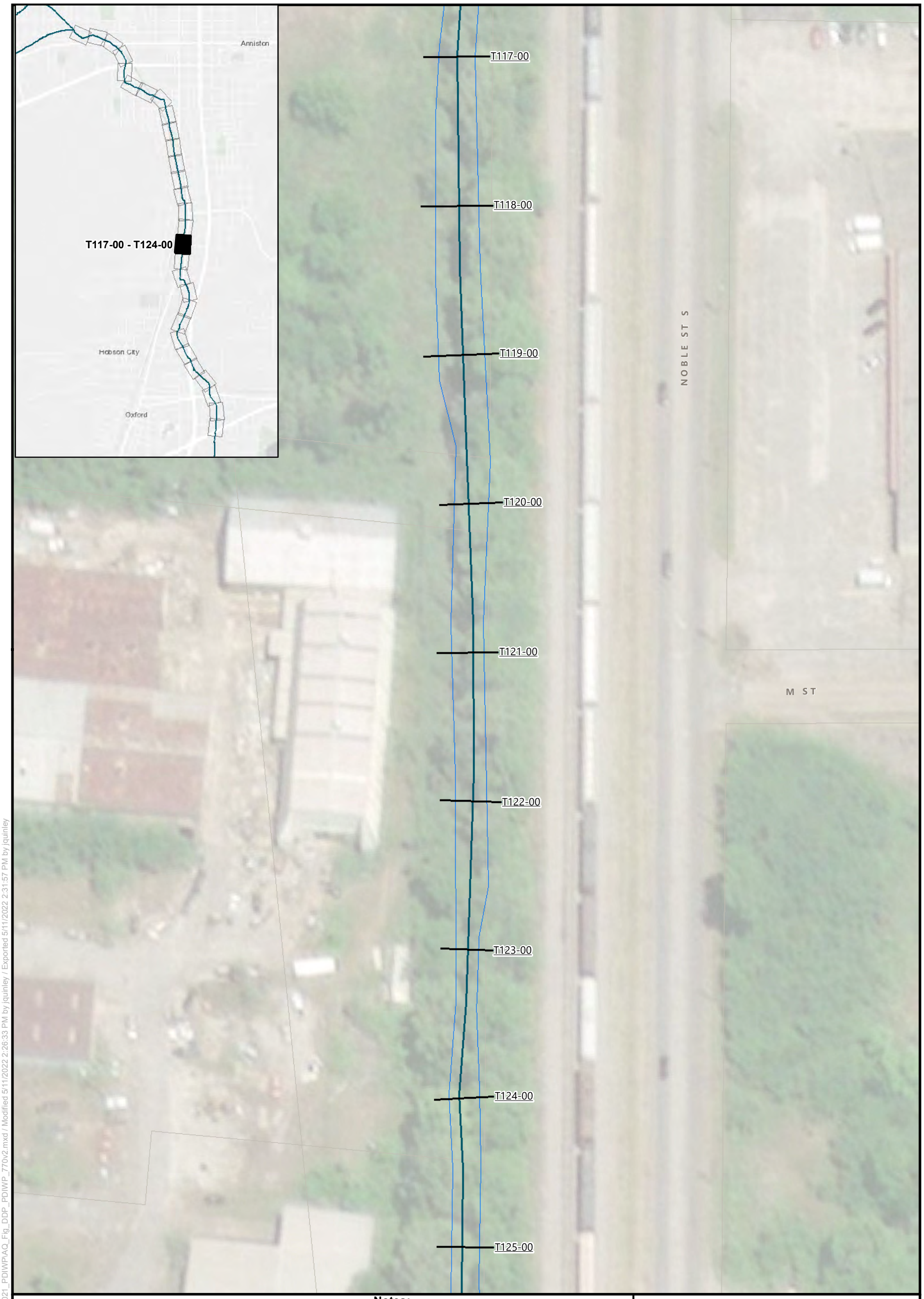
Anniston PCB Site
Anniston, Alabama
OU-1/OU-2 Predesign Investigation Work Plan

Snow Creek
Predesign Investigation Transects
T110-00 through T117-00

Solutia

Figure 3-9p

File Path: \\orcas\GISJobs\SolutiaInc_1718\Anniston_PCB_SiteMaps\2021_PDIWPAQ_Fig_DDP_PDIWP_770v2.mxd / Modified 5/11/2022 2:26:33 PM by jquinley / Exported 5/11/2022 2:31:48 PM by jquinley



Legend

- PDI Stations/Transects (100-foot spacing)
- Snow Creek Centerline
- Snow Creek Shoreline
- Calhoun County Parcels

Notes:

1. Aerial imagery is from Esri basemaps service (accessed: 5/11/2022).

2. Transect station lines are spaced every 100 feet perpendicular to the Snow Creek centerline.

mg/kg: milligrams per kilogram
PCB: polychlorinated biphenyl
PDI: predesign investigation

0 50 100 Feet

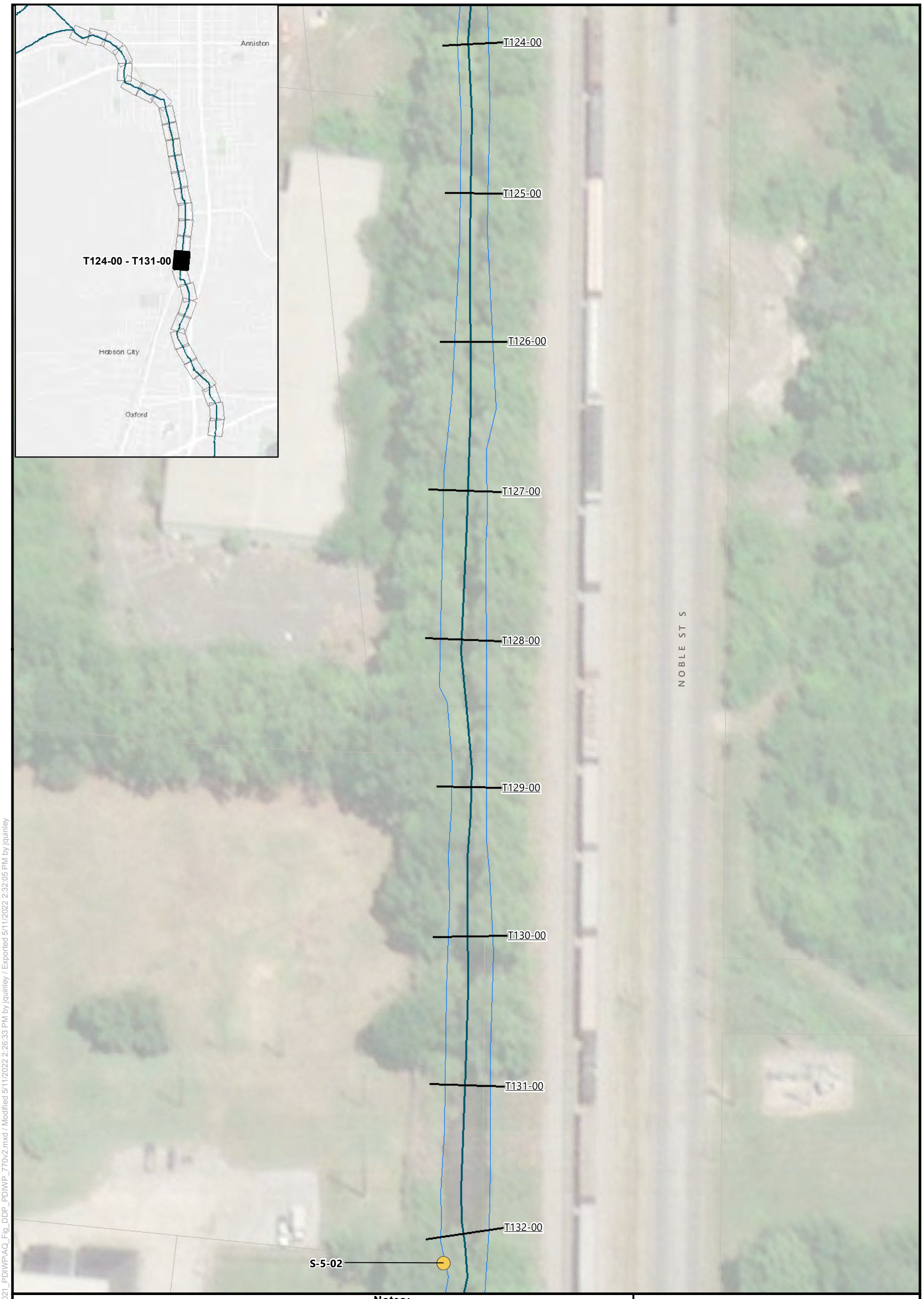
Anniston PCB Site
Anniston, Alabama
OU-1/OU-2 Predesign Investigation Work Plan

Snow Creek
Predesign Investigation Transects
T117-00 through T124-00

Solutia

Figure 3-9q

File Path: \\orcas\GIS\Jobs\Solutia\nc_1718\Anniston_PCB_SiteMaps\2021_PDIWP\AQ_Fig_DDP_PDIWP_770v2.mxd / Modified 5/11/2022 2:26:33 PM by jquinley / Exported 5/11/2022 2:31:57 PM by jquinley



Legend

- Previous Sediment Sample Location
- PDI Stations/Transects (100-foot spacing)
- Snow Creek Centerline
- Snow Creek Shoreline
- Calhoun County Parcels

Notes:

1. Aerial imagery is from Esri basemaps service (accessed: 5/11/2022).

2. Transect station lines are spaced every 100 feet perpendicular to the Snow Creek centerline.

mg/kg: milligrams per kilogram
PCB: polychlorinated biphenyl
PDI: predesign investigation

0 50 100 Feet

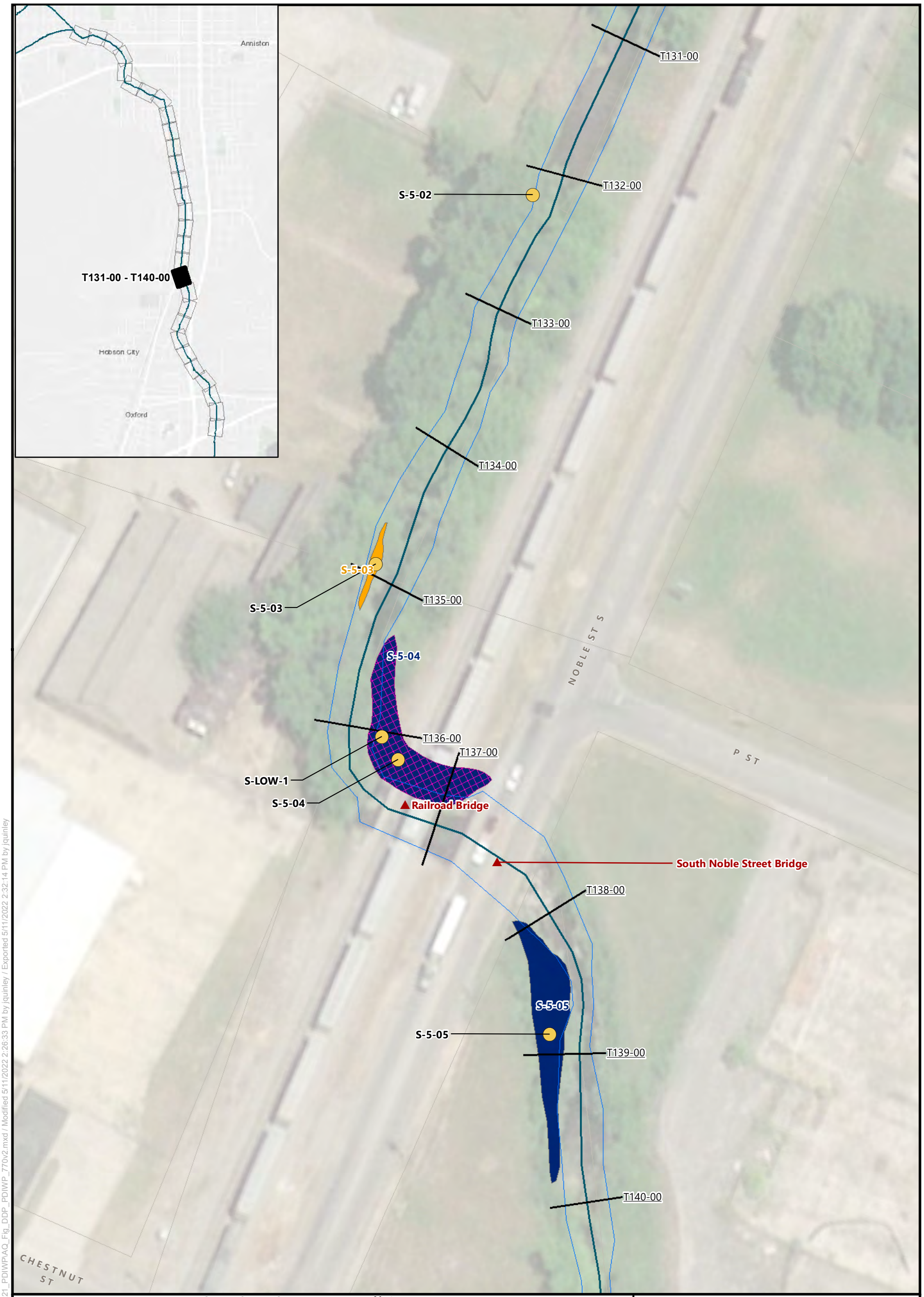
Anniston PCB Site
Anniston, Alabama
OU-1/OU-2 Predesign Investigation Work Plan

Snow Creek
Predesign Investigation Transects
T124-00 through T131-00

Solutia

Figure 3-9r

File Path: \\orcas\GISJobs\Solutia\nc_1718\Anniston_PCB_Site\Maps\2021_PDIWP\AQ_Fig_DDP_PDIWP_770v2.mxd / Modified 5/11/2022 2:26:33 PM by jquinley / Exported 5/11/2022 2:32:05 PM by jquinley



Legend

- Known Structures along Snow Creek
- Previous Sediment Sample Location
- PDI Stations/Transects (100-foot spacing)
- Snow Creek Centerline
- Snow Creek Shoreline
- Calhoun County Parcels

Snow Creek Sediment Deposits

- Deposit with Metals Exceedance and PCB <3 mg/kg
- < 3 mg/kg PCB (average concentration)
- ≥ 3 mg/kg PCB (average concentration)

Notes:

1. Aerial imagery is from Esri basemaps service (accessed: 5/11/2022).

2. Transect station lines are spaced every 100 feet perpendicular to the Snow Creek centerline.

3. Snow Creek sediment deposits are based on the 1999 Resource Conservation Recovery Act Facility Investigation (RFI) geomorphological mapping of the creek.

mg/kg: milligrams per kilogram
PCB: polychlorinated biphenyl
PDI: predesign investigation

0 50 100 Feet

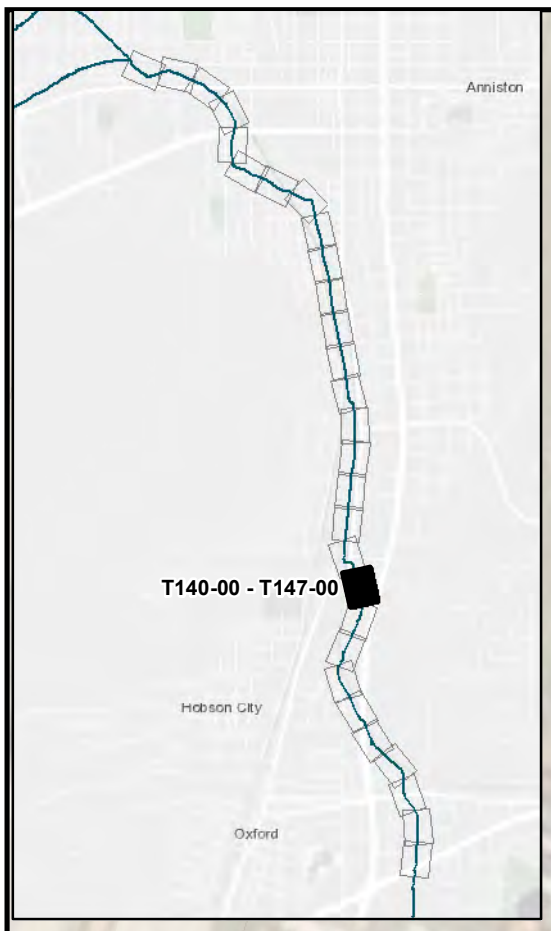
Anniston PCB Site
Anniston, Alabama
OU-1/OU-2 Predesign Investigation Work Plan

Snow Creek
Predesign Investigation Transects
T131-00 through T140-00

Solutia
Ingenuity Productivity Performance

Figure 3-9s

File Path: \\orcas\GIS\Jobs\Solutia\nc_1718\Anniston_PCB_SiteMaps\2021_PDIWPAAQ_Fig_DDP_PDIWP_770v2.mxd / Modified 5/11/2022 2:26:33 PM by jquinley / Exported 5/11/2022 2:32:14 PM by jquinley



S-5-05

T140-00

T141-00

T142-00

S-5-06

S-5-06

T143-00

S-5-07

T144-00

T145-00

T146-00

T147-00

QUINTARD AVE S

Legend

- Previous Sediment Sample Location
- PDI Stations/Transects (100-foot spacing)
- Snow Creek Centerline
- Snow Creek Shoreline
- Calhoun County Parcels

Snow Creek Sediment Deposits

- < 3 mg/kg PCB (estimated concentration)
- < 3 mg/kg PCB (average concentration)

Notes:

1. Aerial imagery is from Esri basemaps service (accessed: 5/11/2022).

2. Transect station lines are spaced every 100 feet perpendicular to the Snow Creek centerline.

3. Snow Creek sediment deposits are based on the 1999 Resource Conservation Recovery Act Facility Investigation (RFI) geomorphological mapping of the creek.

mg/kg: milligrams per kilogram
PCB: polychlorinated biphenyl
PDI: predesign investigation

0 50 100 Feet

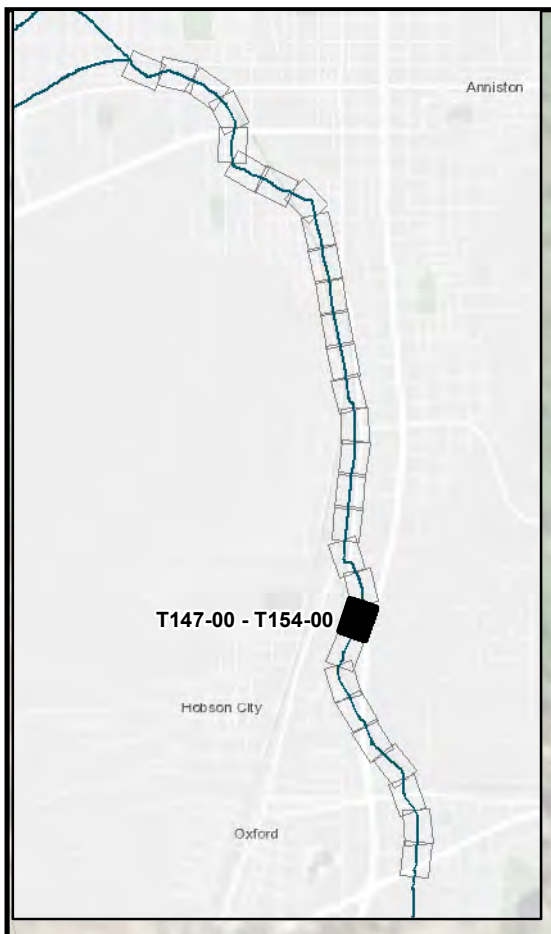
Anniston PCB Site
Anniston, Alabama
OU-1/OU-2 Predesign Investigation Work Plan

Snow Creek
Predesign Investigation Transects
T140-00 through T147-00

Solutia
Ingenuity Productivity Performance

Figure 3-9t

File Path: \\orcas\GISJobs\Solutia\nc_1718\Anniston_PCB_SiteMaps\2021_PDIWPAQ_Fig_DDP_PDIWP_770v2.mxd / Modified 5/11/2022 2:26:33 PM by jquinley / Exported 5/11/2022 2:32:24 PM by jquinley



T147-00
T148-00
S-5-08A
T149-00
S-5-08B
T150-00
T151-00
T152-00
T153-00
T154-00
S-5-09
QUINTARD AVE S

Legend

- PDI Stations/Transects (100-foot spacing)
- Snow Creek Centerline
- Snow Creek Shoreline
- Calhoun County Parcels

Snow Creek Sediment Deposits

- < 3 mg/kg PCB (estimated concentration)

Notes:

1. Aerial imagery is from Esri basemaps service (accessed: 5/11/2022).

2. Transect station lines are spaced every 100 feet perpendicular to the Snow Creek centerline.

3. Snow Creek sediment deposits are based on the 1999 Resource Conservation Recovery Act Facility Investigation (RFI) geomorphological mapping of the creek.

mg/kg: milligrams per kilogram
PCB: polychlorinated biphenyl
PDI: predesign investigation

0 50 100 Feet

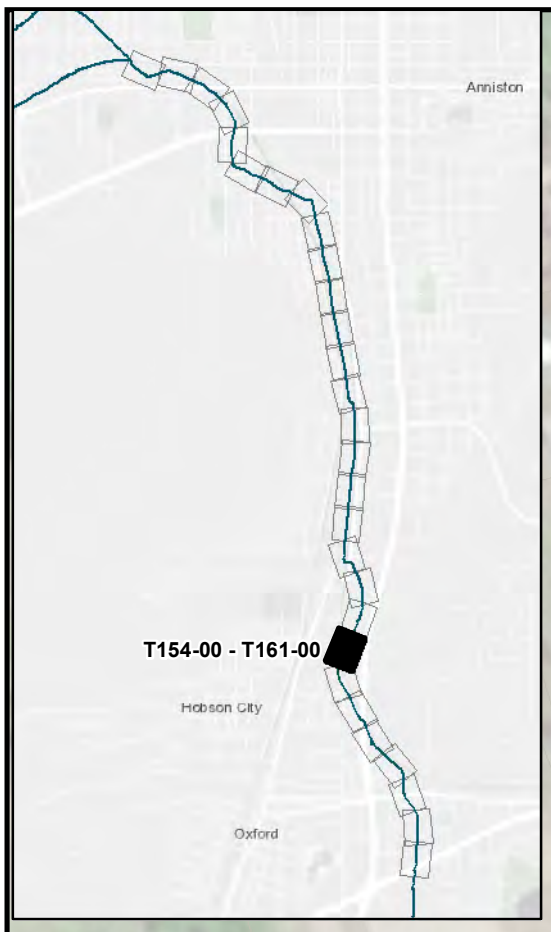
Anniston PCB Site
Anniston, Alabama
OU-1/OU-2 Predesign Investigation Work Plan

Snow Creek Predesign Investigation Transects T147-00 through T154-00

Solutia

Figure 3-9u

File Path: \\orcas\GISJobs\Solutia\nc_1718\Anniston_PCB_SiteMaps\2021_PDIWPAQ_Fig_DDP_PDIWP_770v2.mxd / Modified 5/11/2022 2:26:33 PM by jquinley / Exported 5/11/2022 2:32:33 PM by jquinley



T154-00
S-5-09
T155-00
T156-00
T157-00
T158-00
T159-00
S-5-10
T160-00
PECON-020
S-5-11A
S-5-11B
CA-25-9999-90
FE2-001-C
FE2-001-A
S-5-12A
Railroad Bridge
T161-00
S-5-12B
T162-00

Legend

- Known Structures along Snow Creek
- Previous Sediment Sample Location
- PDI Stations/Transects (100-foot spacing)
- Snow Creek Centerline
- Snow Creek Shoreline
- Calhoun County Parcels

Snow Creek Sediment Deposits

- Deposit with Metals Exceedance and PCB <3 mg/kg
- < 3 mg/kg PCB (estimated concentration)
- ≥ 3 mg/kg PCB (estimated concentration)

Notes:

1. Aerial imagery is from Esri basemaps service (accessed: 5/11/2022).

2. Transect station lines are spaced every 100 feet perpendicular to the Snow Creek centerline.

3. Snow Creek sediment deposits are based on the 1999 Resource Conservation Recovery Act Facility Investigation (RFI) geomorphological mapping of the creek.

mg/kg: milligrams per kilogram
PCB: polychlorinated biphenyl
PDI: predesign investigation

0 50 100 Feet

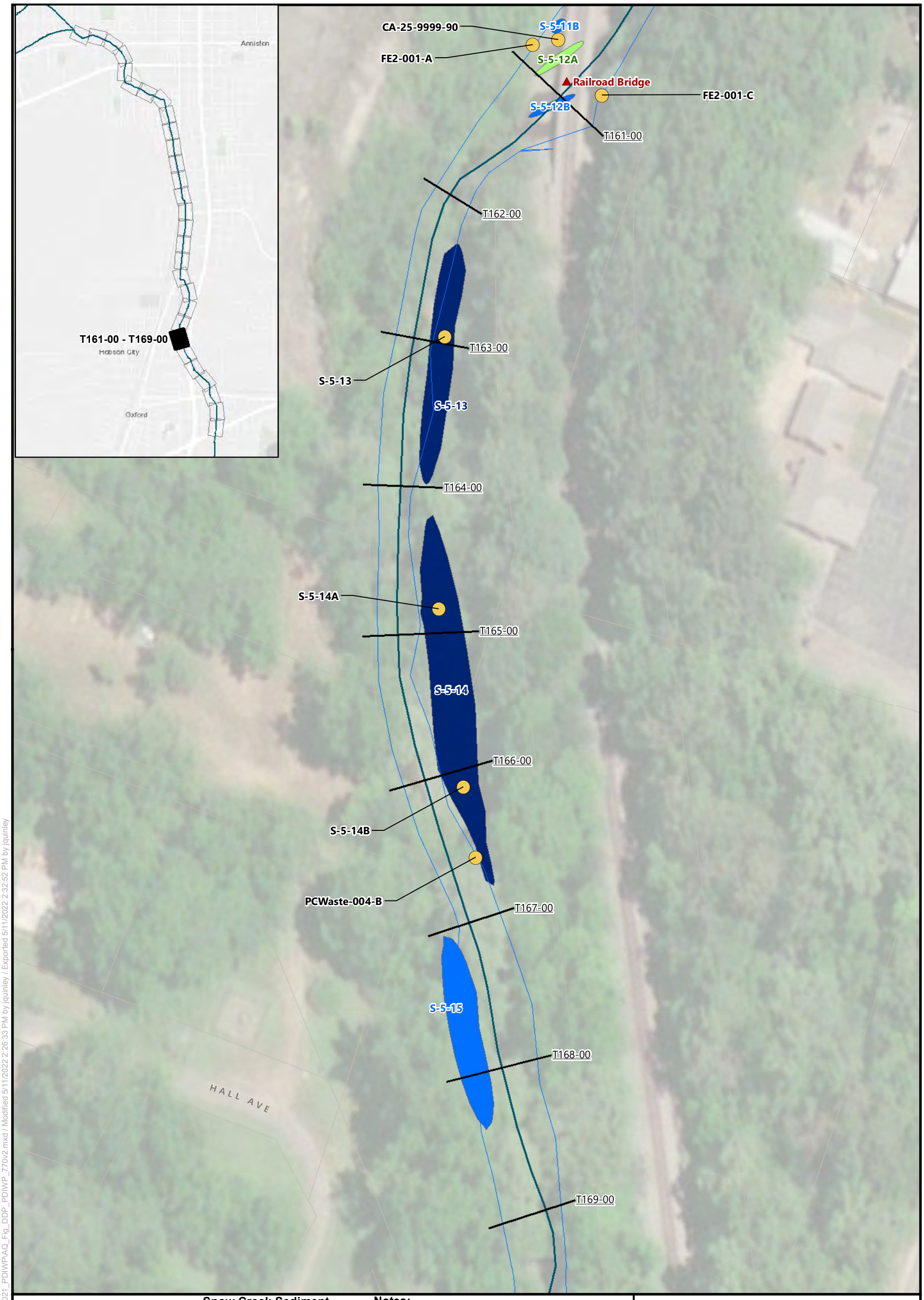
Anniston PCB Site
Anniston, Alabama
OU-1/OU-2 Predesign Investigation Work Plan

Snow Creek
Predesign Investigation Transects
T154-00 through T161-00

Solutia

Figure 3-9v

File Path: \\orcas\GIS\Jobs\Solutia\Inc_1718\Anniston_PCB_Site\Maps\2021_PDIWP\AQ_Fig_DDP_PDIWP_770v2.mxd / Modified 5/11/2022 2:26:33 PM by jquinley / Exported 5/11/2022 2:32:42 PM by jquinley



Legend

- Known Structures along Snow Creek
- Previous Sediment Sample Location
- PDI Stations/Transects (100-foot spacing)
- Snow Creek Centerline
- Snow Creek Shoreline
- Calhoun County Parcels

Snow Creek Sediment Deposits

- < 3 mg/kg PCB (estimated concentration)
- ≥ 3 mg/kg PCB (estimated concentration)
- < 3 mg/kg PCB (average concentration)

Notes:

1. Aerial imagery is from Esri basemaps service (accessed: 5/11/2022).

2. Transect station lines are spaced every 100 feet perpendicular to the Snow Creek centerline.

3. Snow Creek sediment deposits are based on the 1999 Resource Conservation Recovery Act Facility Investigation (RFI) geomorphological mapping of the creek.

mg/kg: milligrams per kilogram
PCB: polychlorinated biphenyl
PDI: predesign investigation

0 50 100 Feet

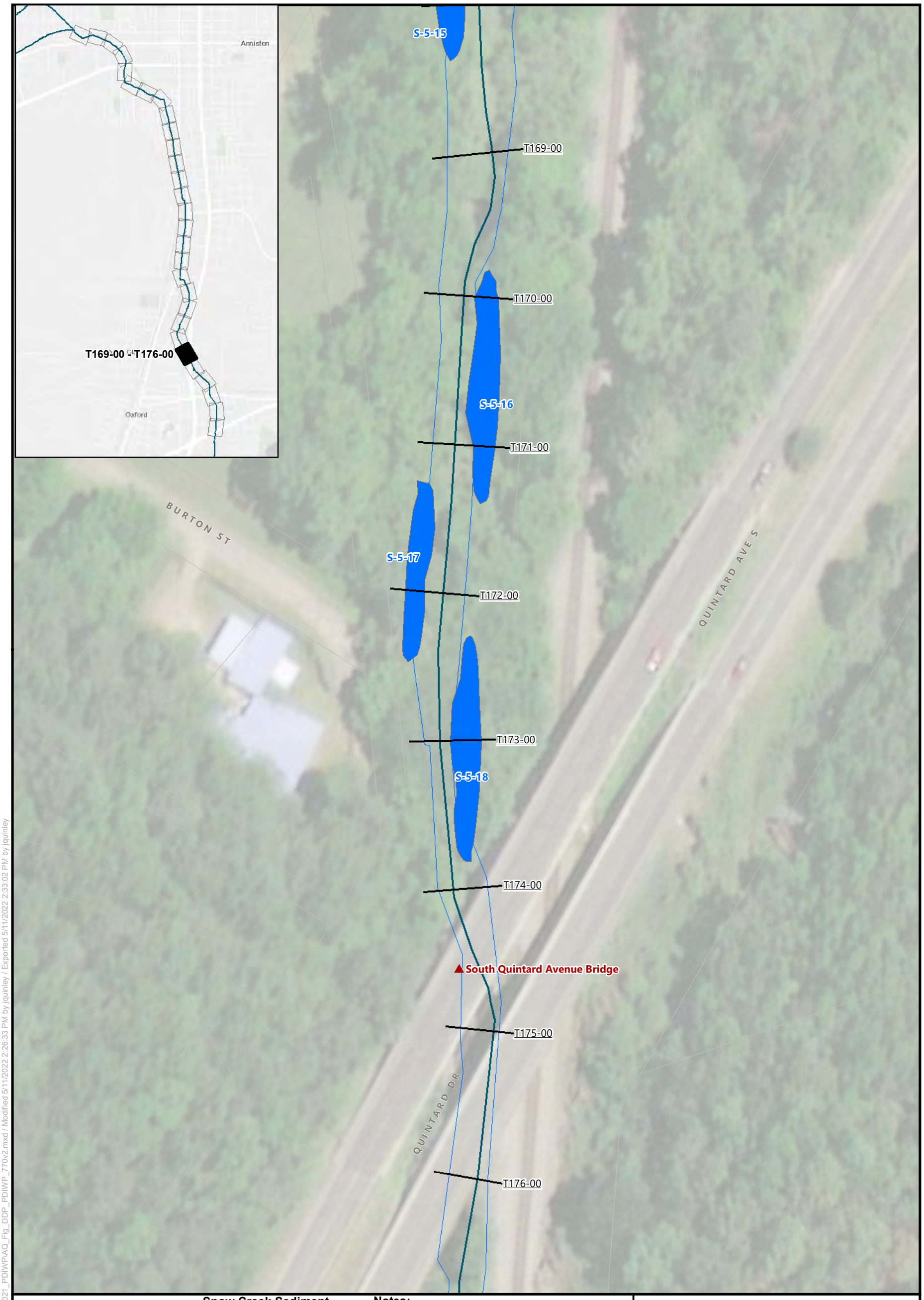
Anniston PCB Site
Anniston, Alabama
OU-1/OU-2 Predesign Investigation Work Plan

Snow Creek
Predesign Investigation Transects
T161-00 through T169-00

Solutia

Figure 3-9w

File Path: \\orcas\GIS\Jobs\Solutia\Inc_1718\Anniston_PCB_SiteMaps\2021_PDIWPAQ_Fig_DDP_PDIWP_770v2.mxd / Modified 5/11/2022 2:26:33 PM by jquinley / Exported 5/11/2022 2:32:52 PM by jquinley



Legend

- Known Structures along Snow Creek
- PDI Stations/Transects (100-foot spacing)
- Snow Creek Centerline
- Snow Creek Shoreline
- Calhoun County Parcels

Snow Creek Sediment Deposits

- < 3 mg/kg PCB (estimated concentration)

Notes:

1. Aerial imagery is from Esri basemaps service (accessed: 5/11/2022).

2. Transect station lines are spaced every 100 feet perpendicular to the Snow Creek centerline.

3. Snow Creek sediment deposits are based on the 1999 Resource Conservation Recovery Act Facility Investigation (RFI) geomorphological mapping of the creek.

mg/kg: milligrams per kilogram
PCB: polychlorinated biphenyl
PDI: predesign investigation

0 50 100 Feet

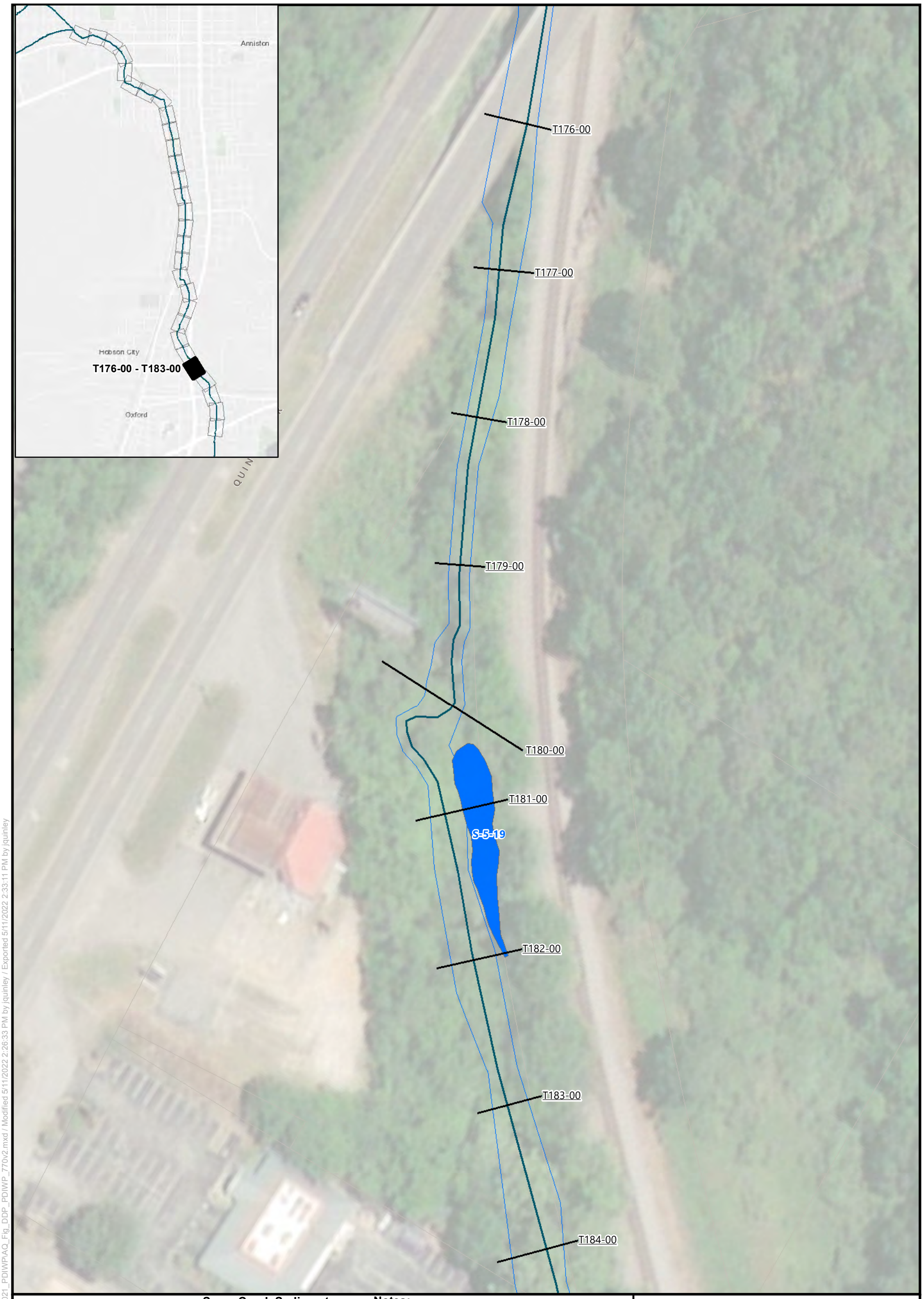
Anniston PCB Site
Anniston, Alabama
OU-1/OU-2 Predesign Investigation Work Plan

Snow Creek
Predesign Investigation Transects
T169-00 through T176-00

Solutia
Ingenuity Productivity Performance

Figure 3-9x

File Path: \\orcas\GISJobs\Solutia\nc_1718\Anniston_PCB_SiteMaps\2021_PDIWPAQ_Fig_DDP_PDIWP_770v2.mxd / Modified 5/11/2022 2:26:33 PM by jquinley / Exported 5/11/2022 2:33:02 PM by jquinley



Legend

- PDI Stations/Transects (100-foot spacing)
- Snow Creek Centerline
- Snow Creek Shoreline
- Calhoun County Parcels

Snow Creek Sediment Deposits

- < 3 mg/kg PCB (estimated concentration)

Notes:

1. Aerial imagery is from Esri basemaps service (accessed: 5/11/2022).

2. Transect station lines are spaced every 100 feet perpendicular to the Snow Creek centerline.

3. Snow Creek sediment deposits are based on the 1999 Resource Conservation Recovery Act Facility Investigation (RFI) geomorphological mapping of the creek.

mg/kg: milligrams per kilogram
PCB: polychlorinated biphenyl
PDI: predesign investigation

0 50 100 Feet

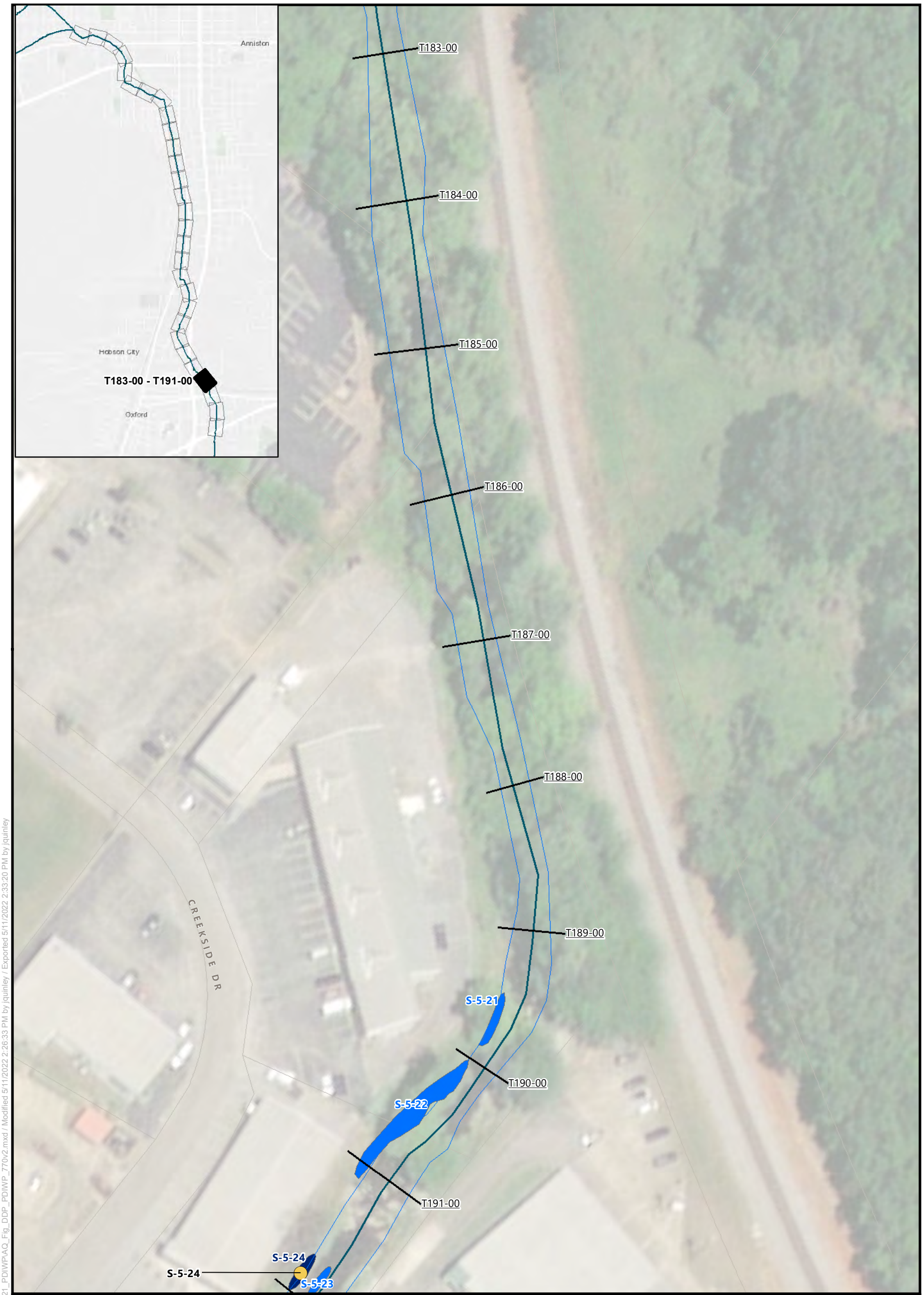
Anniston PCB Site
Anniston, Alabama
OU-1/OU-2 Predesign Investigation Work Plan

Snow Creek
Predesign Investigation Transects
T176-00 through T183-00

Solutia

Figure 3-9y

File Path: \\orcas\GIS\Jobs\Solutia\Inc_1718\Anniston_PCB_Site\Maps\2021_PDIWPAQ_Fig_DDP_PDIWP_770v2.mxd / Modified 5/11/2022 2:26:33 PM by jquinley / Exported 5/11/2022 2:33:11 PM by jquinley



Legend

- Previous Sediment Sample Location
- PDI Stations/Transects (100-foot spacing)
- Snow Creek Centerline
- Snow Creek Shoreline
- Calhoun County Parcels

Snow Creek Sediment Deposits

- < 3 mg/kg PCB (estimated concentration)
- < 3 mg/kg PCB (average concentration)

Notes:

1. Aerial imagery is from Esri basemaps service (accessed: 5/11/2022).

2. Transect station lines are spaced every 100 feet perpendicular to the Snow Creek centerline.

3. Snow Creek sediment deposits are based on the 1999 Resource Conservation Recovery Act Facility Investigation (RFI) geomorphological mapping of the creek.

mg/kg: milligrams per kilogram
PCB: polychlorinated biphenyl
PDI: predesign investigation

0 50 100 Feet

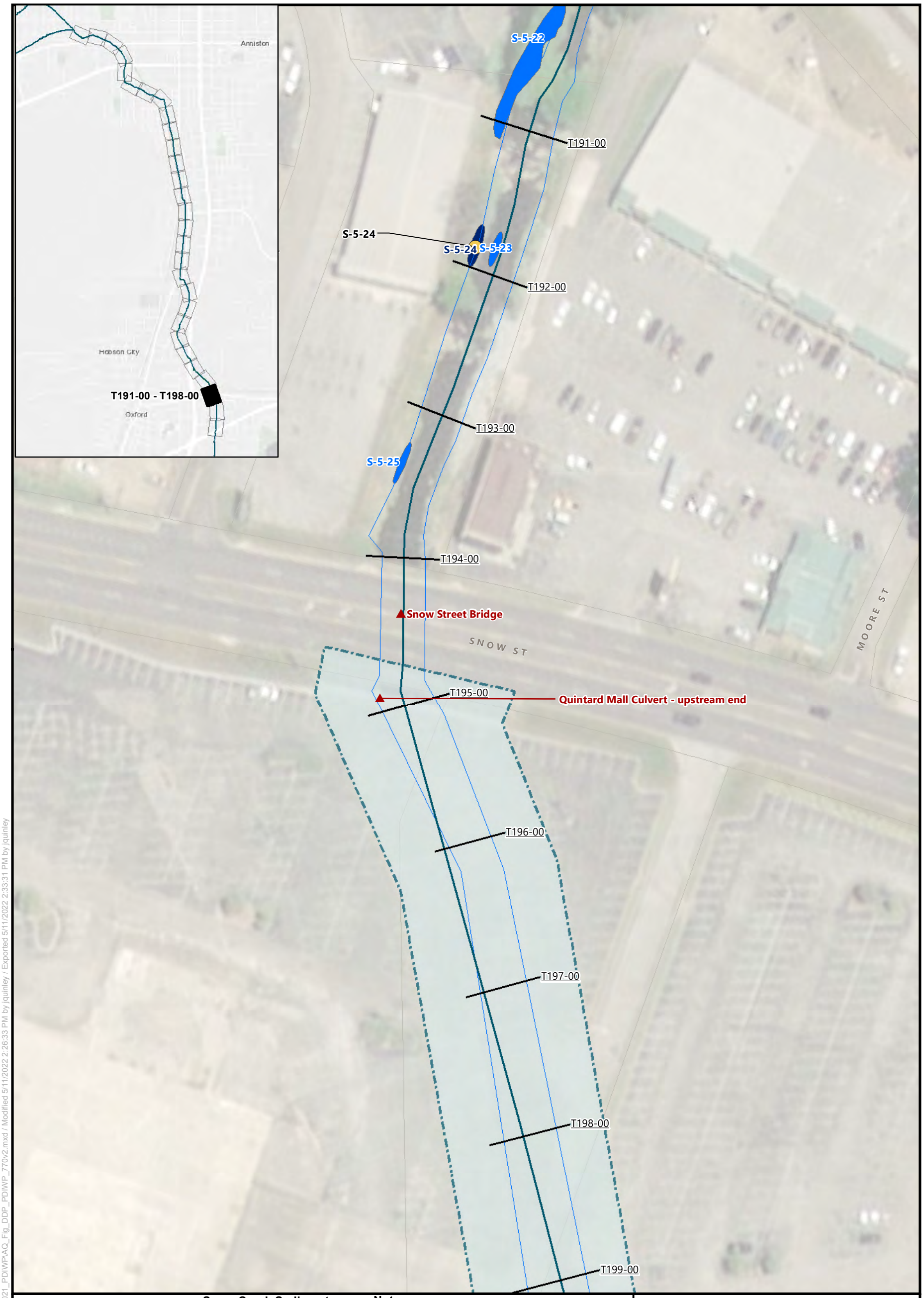
Anniston PCB Site
Anniston, Alabama
OU-1/OU-2 Predesign Investigation Work Plan

Snow Creek
Predesign Investigation Transects
T183-00 through T191-00

Solutia
Ingenuity Productivity Performance

Figure 3-9z

File Path: \\orcas\GIS\Jobs\Solutia\Inc_1718\Anniston_PCB_SiteMaps\2021_PDIWP\AQ_Fig_DDP_PDIWP_770v2.mxd / Modified 5/11/2022 2:26:33 PM by jquinley / Exported 5/11/2022 2:33:20 PM by jquinley



Legend

- Known Structures along Snow Creek
- Previous Sediment Sample Location
- PDI Stations/Transects (100-foot spacing)
- Snow Creek Centerline
- Snow Creek Shoreline
- Approximate Extent of Quintard Mall Culvert
- Calhoun County Parcels

Snow Creek Sediment Deposits

- < 3 mg/kg PCB (estimated concentration)
- < 3 mg/kg PCB (average concentration)

Notes:

1. Aerial imagery is from Esri basemaps service (accessed: 5/11/2022).

2. Transect station lines are spaced every 100 feet perpendicular to the Snow Creek centerline.

3. Snow Creek sediment deposits are based on the 1999 Resource Conservation Recovery Act Facility Investigation (RFI) geomorphological mapping of the creek.

mg/kg: milligrams per kilogram
PCB: polychlorinated biphenyl
PDI: predesign investigation

0 50 100 Feet

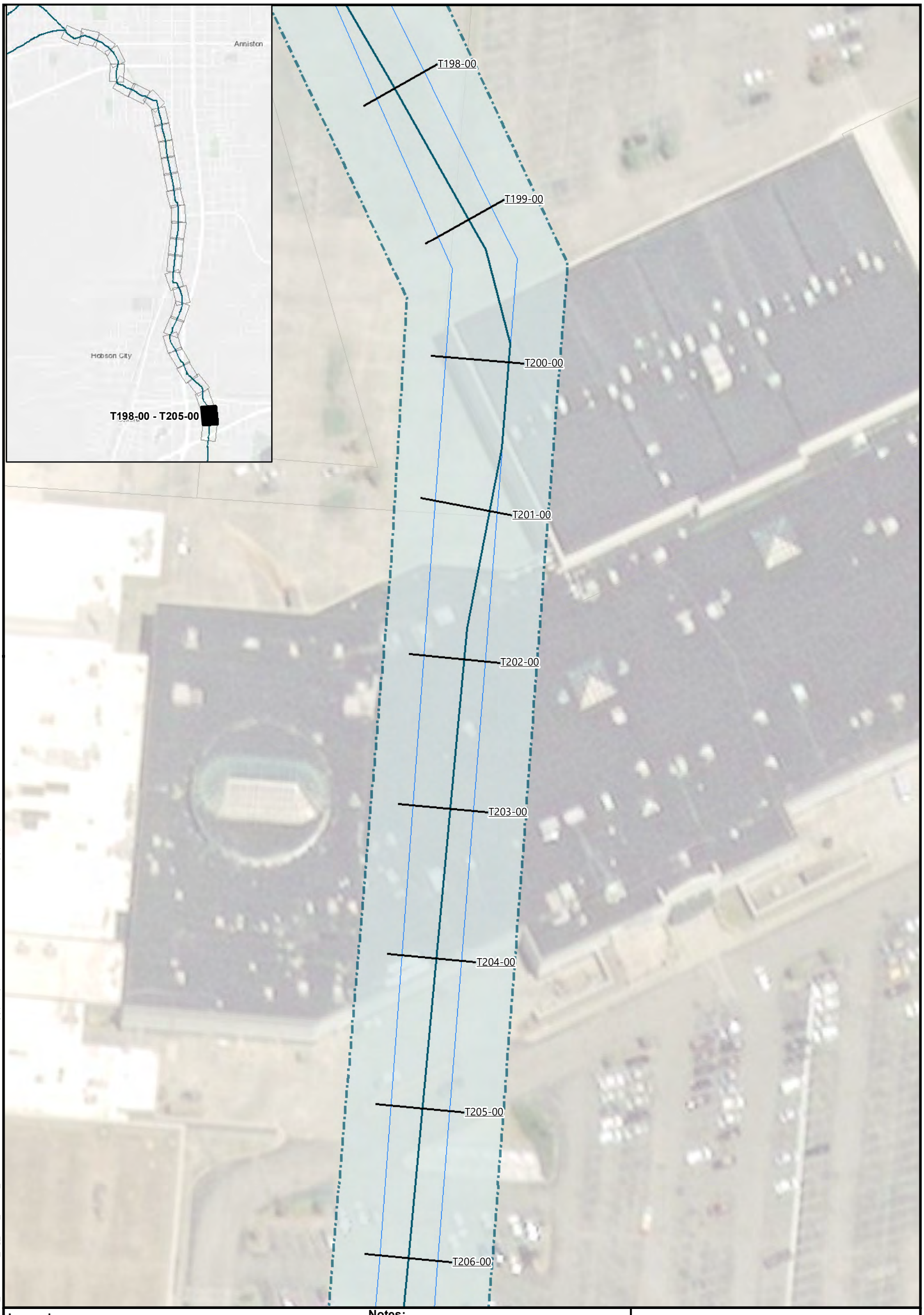
Anniston PCB Site
Anniston, Alabama
OU-1/OU-2 Predesign Investigation Work Plan

Snow Creek Predesign Investigation Transects T191-00 through T198-00

Solutia

Figure 3-9aa

File Path: \\orcas\GISJobs\Solutiainc_1718\Anniston_PCB_SiteMaps\2021_PDIWPAQ_Fig_DDP_PDIWP_770v2.mxd / Modified 5/11/2022 2:26:33 PM by jquinley / Exported 5/11/2022 2:33:31 PM by jquinley



Legend

- PDI Stations/Transects (100-foot spacing)
- Snow Creek Centerline
- Snow Creek Shoreline
- Approximate Extent of Quintard Mall Culvert
- Calhoun County Parcels

Notes:

1. Aerial imagery is from Esri basemaps service (accessed: 5/11/2022).

2. Transect station lines are spaced every 100 feet perpendicular to the Snow Creek centerline.

mg/kg: milligrams per kilogram
PCB: polychlorinated biphenyl
PDI: predesign investigation

0 50 100 Feet

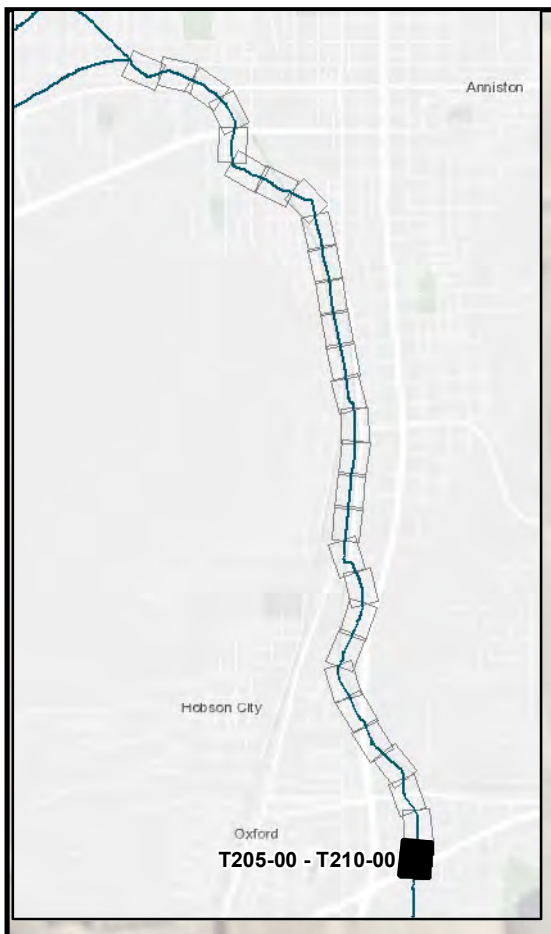
Anniston PCB Site
Anniston, Alabama
OU-1/OU-2 Predesign Investigation Work Plan

Snow Creek
Predesign Investigation Transects
T198-00 through T205-00

Solutia Ingenuity Productivity Performance

Figure 3-9bb

File Path: \\orcas\GISUobs\SolutiaInc_1718\Anniston_PCB_SiteMaps\2021_PDIWPAQ_Fig_DDP_PDIWP_770v2.mxd / Modified 5/11/2022 2:26:33 PM by jquinley / Exported 5/11/2022 2:33:41 PM by jquinley



T205-00

T206-00

T207-00

T208-00

T209-00

Quintard Mall Culvert - downstream end

T210-00

HAMRIC DR E

Highway 78 Bridge

ALI WAY

Legend

- Known Structures along Snow Creek
- PDI Stations/Transects (100-foot spacing)
- Snow Creek Centerline
- Snow Creek Shoreline
- Approximate Extent of Quintard Mall Culvert
- Calhoun County Parcels

Notes:

1. Aerial imagery is from Esri basemaps service (accessed: 5/11/2022).

2. Transect station lines are spaced every 100 feet perpendicular to the Snow Creek centerline.

3. Snow Creek sediment deposits are based on the 1999 Resource Conservation Recovery Act Facility Investigation (RFI) geomorphological mapping of the creek.

mg/kg: milligrams per kilogram
PCB: polychlorinated biphenyl
PDI: predesign investigation

0 50 100 Feet

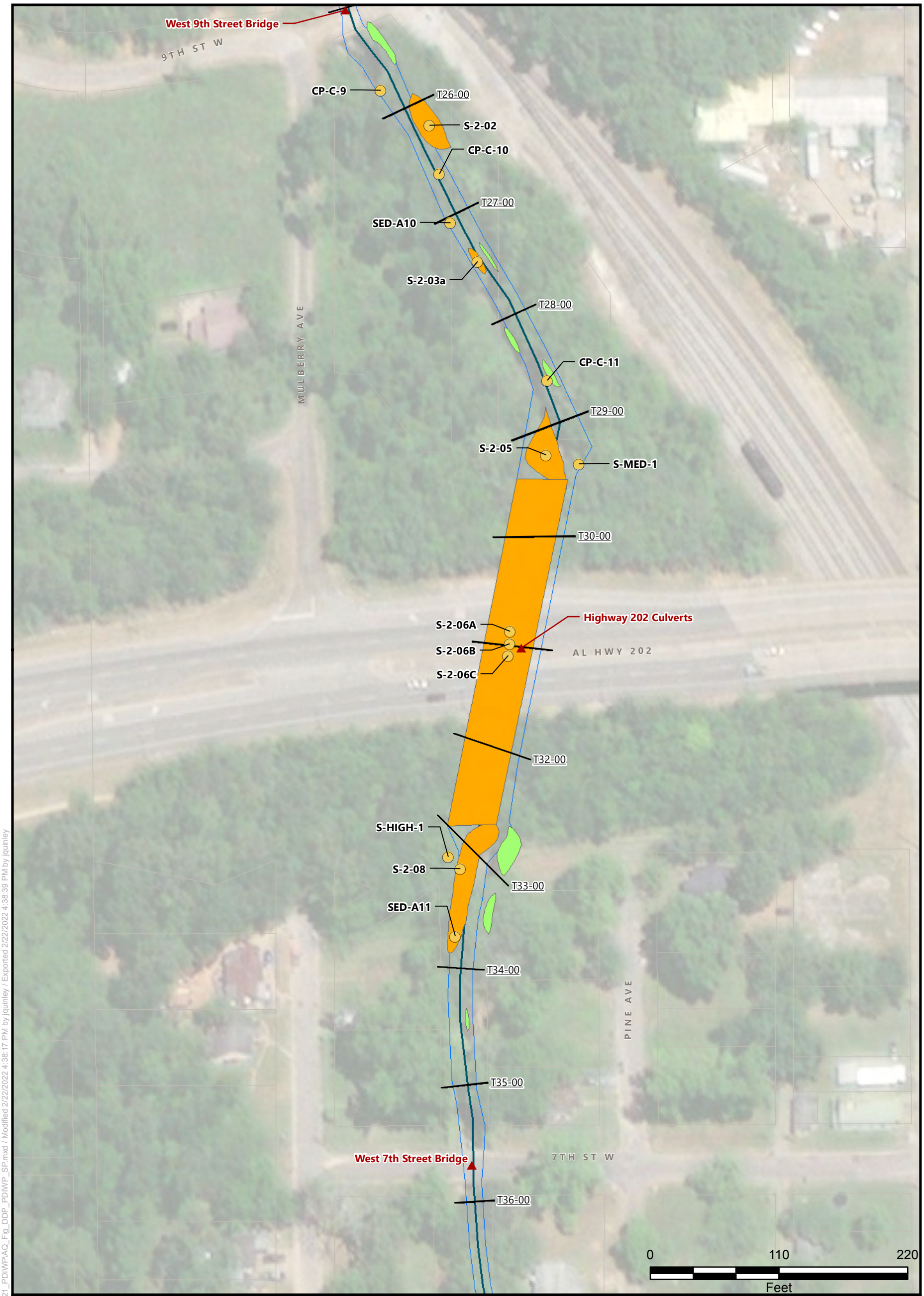
Anniston PCB Site
Anniston, Alabama
OU-1/OU-2 Predesign Investigation Work Plan

Snow Creek
Predesign Investigation Transects
T205-00 through T210-00

Solutia

Figure 3-9cc

File Path: \\orcas\GIS\Jobs\Solutia\Inc_1718\Anniston_PCB_SiteMaps\2021_PDIWPAQ_Fig_DDP_PDIWP_770v2.mxd / Modified 5/11/2022 2:26:33 PM by jquinley / Exported 5/11/2022 2:33:48 PM by jquinley



Legend

- Known Structures along Snow Creek
- Previous Sediment Sample Location
- PDI Stations/Transects (100-foot spacing)
- Snow Creek Centerline
- Snow Creek Shoreline
- Calhoun County Parcels

Snow Creek Sediment Deposits

- ≥ 3 mg/kg PCB (estimated concentration)
- ≥ 3 mg/kg PCB (average concentration)

Notes:

1. Aerial imagery is from Esri basemaps service (accessed: 2/22/2022)
2. Transect station lines are spaced every 100 feet perpendicular to the Snow Creek cernterline.
3. Snow Creek sediment deposits are based on the 1999 Resource Conservation Recovery Act Facility Investigation (RFI) geomorphological mapping of the creek.

mg/kg: milligrams per kilogram
PCB: polychlorinated biphenyl
PDI: predesign investigation

N

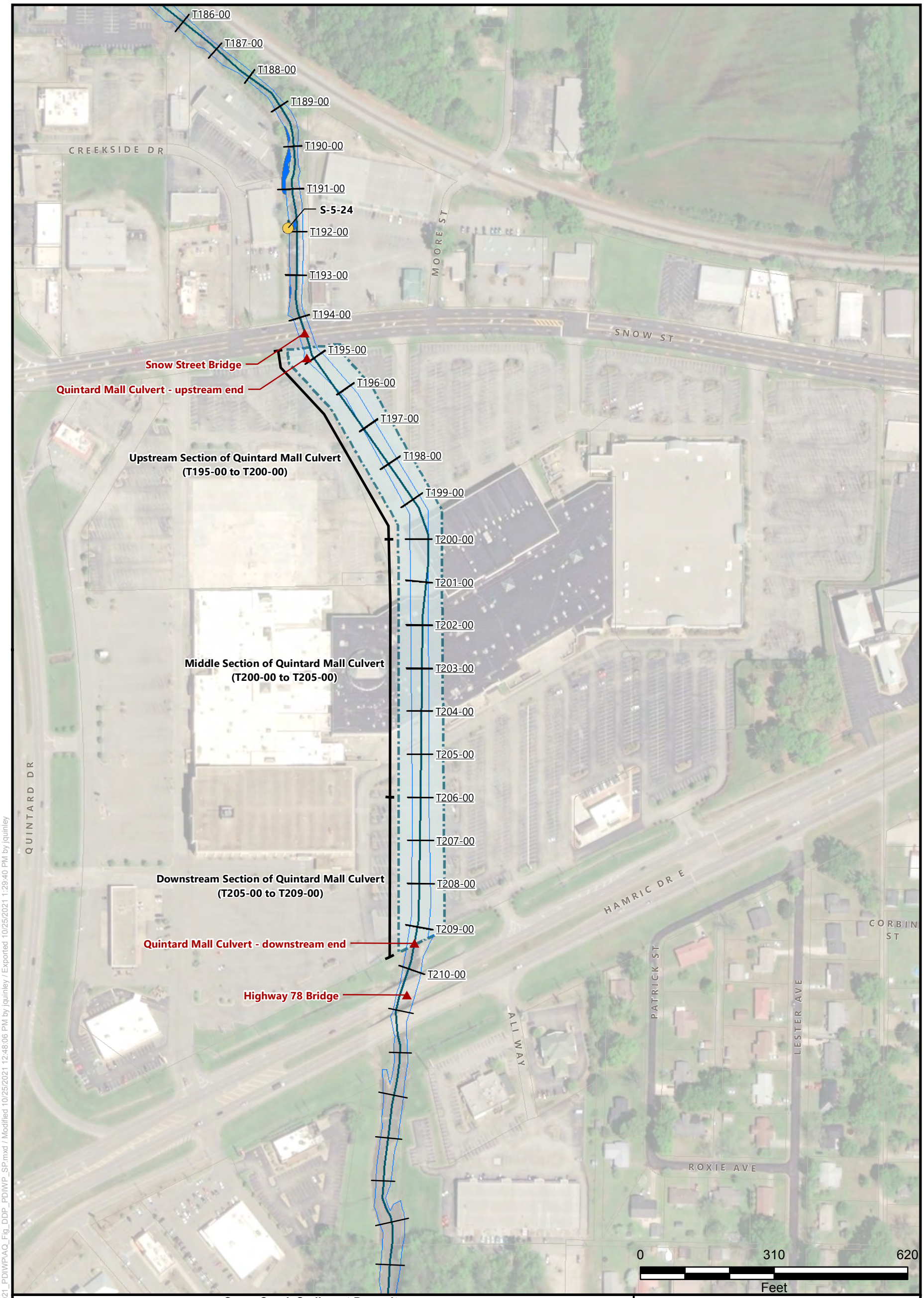
Anniston PCB Site
Anniston, Alabama
OU-1/OU-2 Predesign Investigation Work Plan

Highway 202 Culverts

Solutia
Ingenuity Productivity Performance

Figure 3-10

File Path: \\orcas\GISJobs\Solutia\nc_1718\Anniston_PCB_SiteMaps\2021_PDIWPAQ_Fig_DDP_PDIWP_SP.mxd / Modified 2/22/2022 4:38:17 PM by jquinley / Exported 2/22/2022 4:38:39 PM by jquinley



Legend

- Known Structures along Snow Creek
- Previous Sediment Sample Location
- PDI Stations/Transects (100-foot spacing)
- Snow Creek Centerline
- Snow Creek Shoreline
- Approximate Extent of Quintard Mall Culvert
- Calhoun County Parcels

Snow Creek Sediment Deposits

- < 3 mg/kg PCB (estimated concentration)
- < 3 mg/kg PCB (average concentration)

Notes:

1. Aerial imagery is from Esri basemaps service (accessed: 10/25/2021)
2. Transect station lines are spaced every 100 feet perpendicular to the Snow Creek cernterline.
3. Snow Creek sediment deposits are based on the 1999 Resource Conservation Recovery Act Facility Investigation (RFI) geomorphological mapping of the creek.

mg/kg: milligrams per kilogram
PCB: polychlorinated biphenyl
PDI: predesign investigation

N

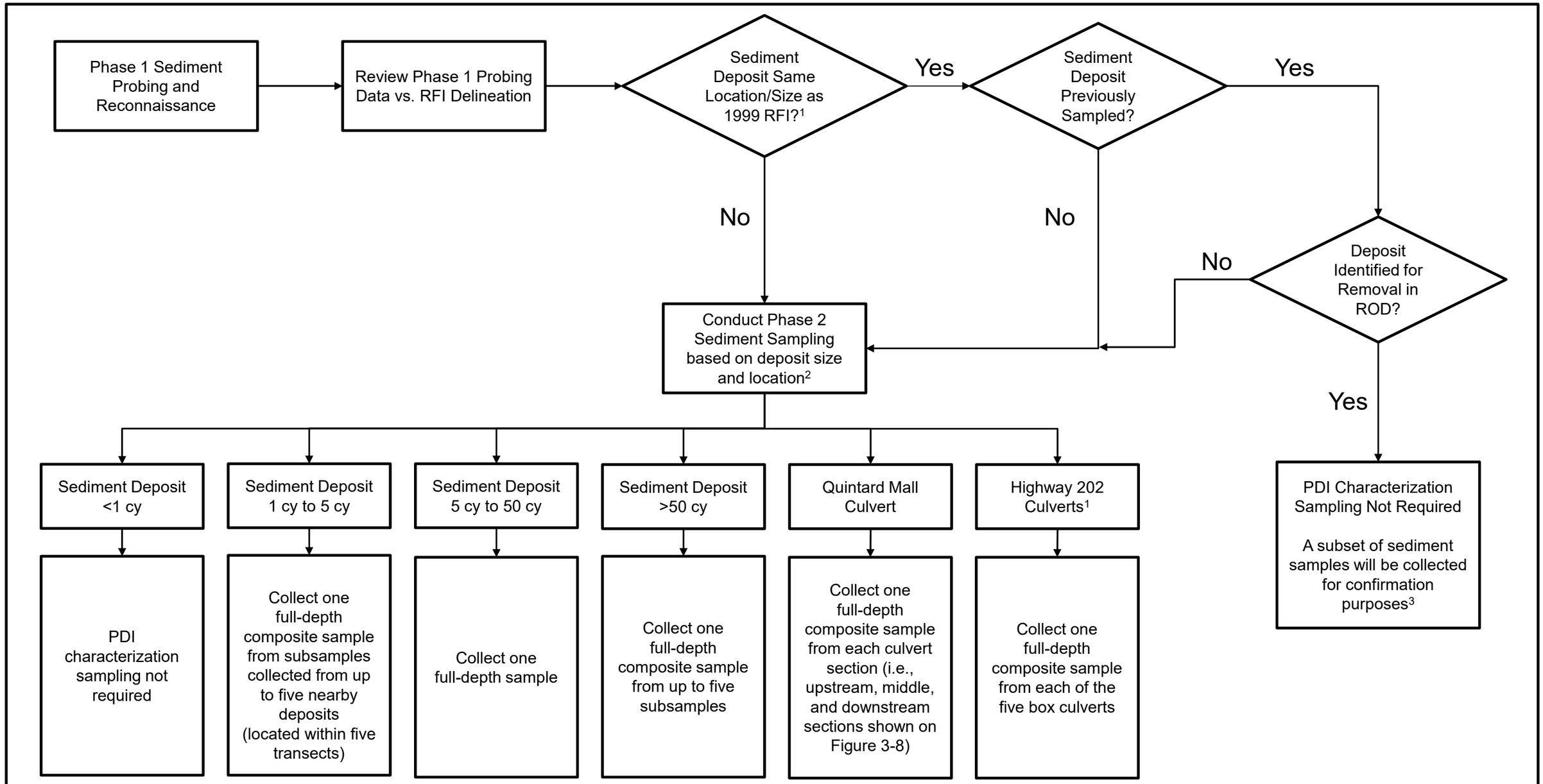
Anniston PCB Site
Anniston, Alabama
OU-1/OU-2 Predesign Investigation Work Plan

Quintard Mall Culvert

Solutia Ingenuity Productivity Performance

Figure 3-11

File Path: \\orcas\GIS\Jobs\Solutia\Inc_1718\Anniston_PCB_SiteMaps\2021_PDIWP\AQ_Fig_DDP_PDIWP_SP.mxd / Modified 10/25/2021 12:48:06 PM by jquinley / Exported 10/25/2021 1:29:40 PM by jquinley



Notes.

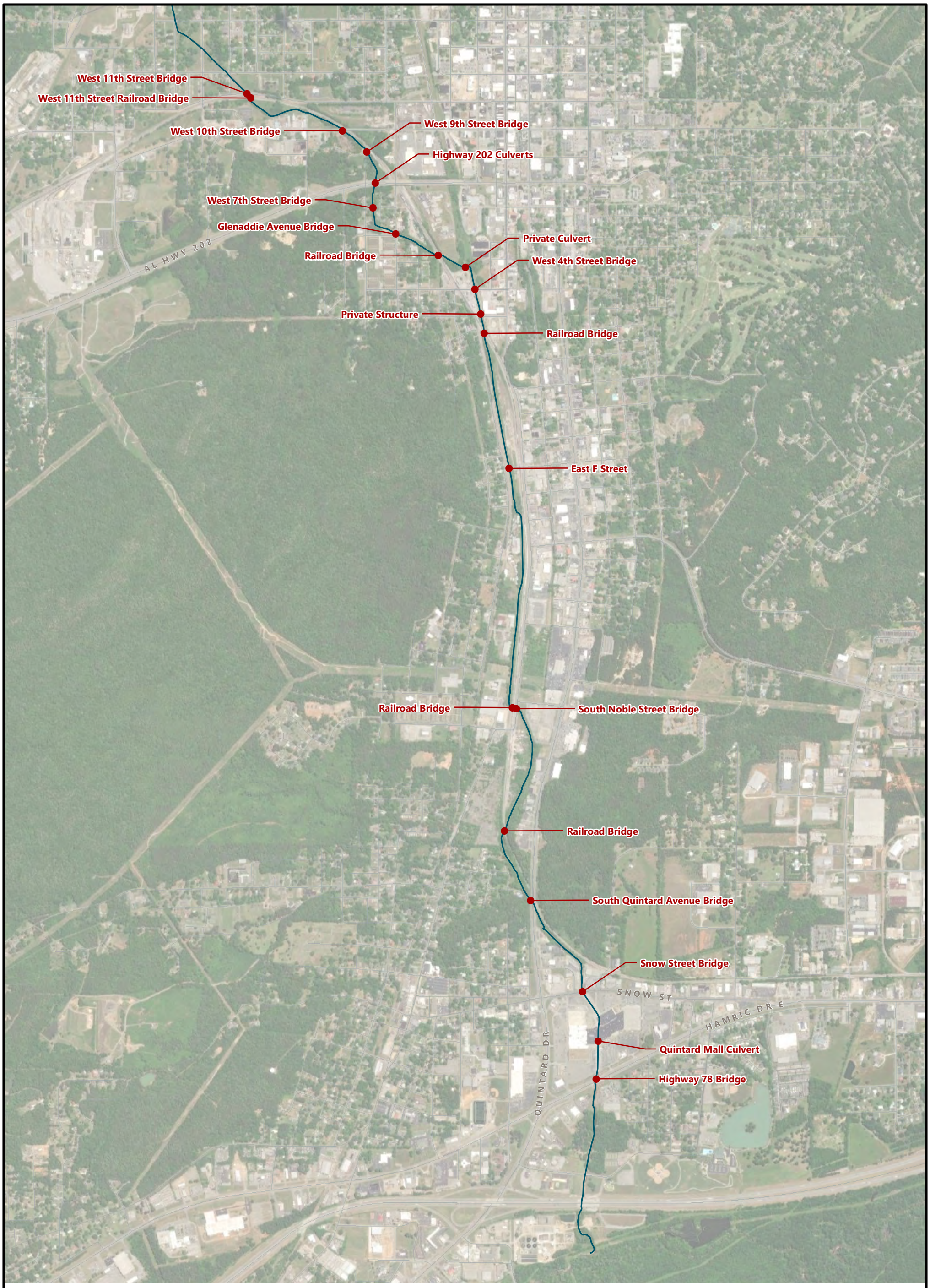
1. Sediment sampling will be performed within the Highway 202 culverts regardless of the sediment deposit location or size to evaluate the extent of polychlorinated biphenyls (PCBs) previously detected above 50 milligrams per kilogram within this portion of the creek.
2. Sediment samples will be submitted for laboratory analysis in accordance with Section 3.3.2 of the OU-1/OU-2 PDI Work Plan. Sampling for waste characterization and treatability testing will also be performed in accordance with Section 3.3.2 of the OU-1/OU-2 PDI Work Plan.
3. As described in Section 3.3.2.2 of the PDI Work Plan, for confirmational purposes, a subset of sediment samples (up to five samples) will be collected from sediment deposits that were previously characterized for removal and appear to be present at the same location and size as measured in the RFI.
4. cy: cubic yards
   PDI: predesign investigation
   RFI: Resource Conservation Recovery Act Facility Investigation
   ROD: Record of Decision

Anniston PCB Site
Anniston, Alabama
OU-1/OU-2 Predesign Investigation Work Plan

PDI Sediment Sampling Approach

Solutia

Figure 3-12



Legend

- Known Structures along Snow Creek
- Snow Creek Centerline

Notes

1. Aerial imagery provided by Esri basemap services

0 1,000 2,000 3,000 4,000

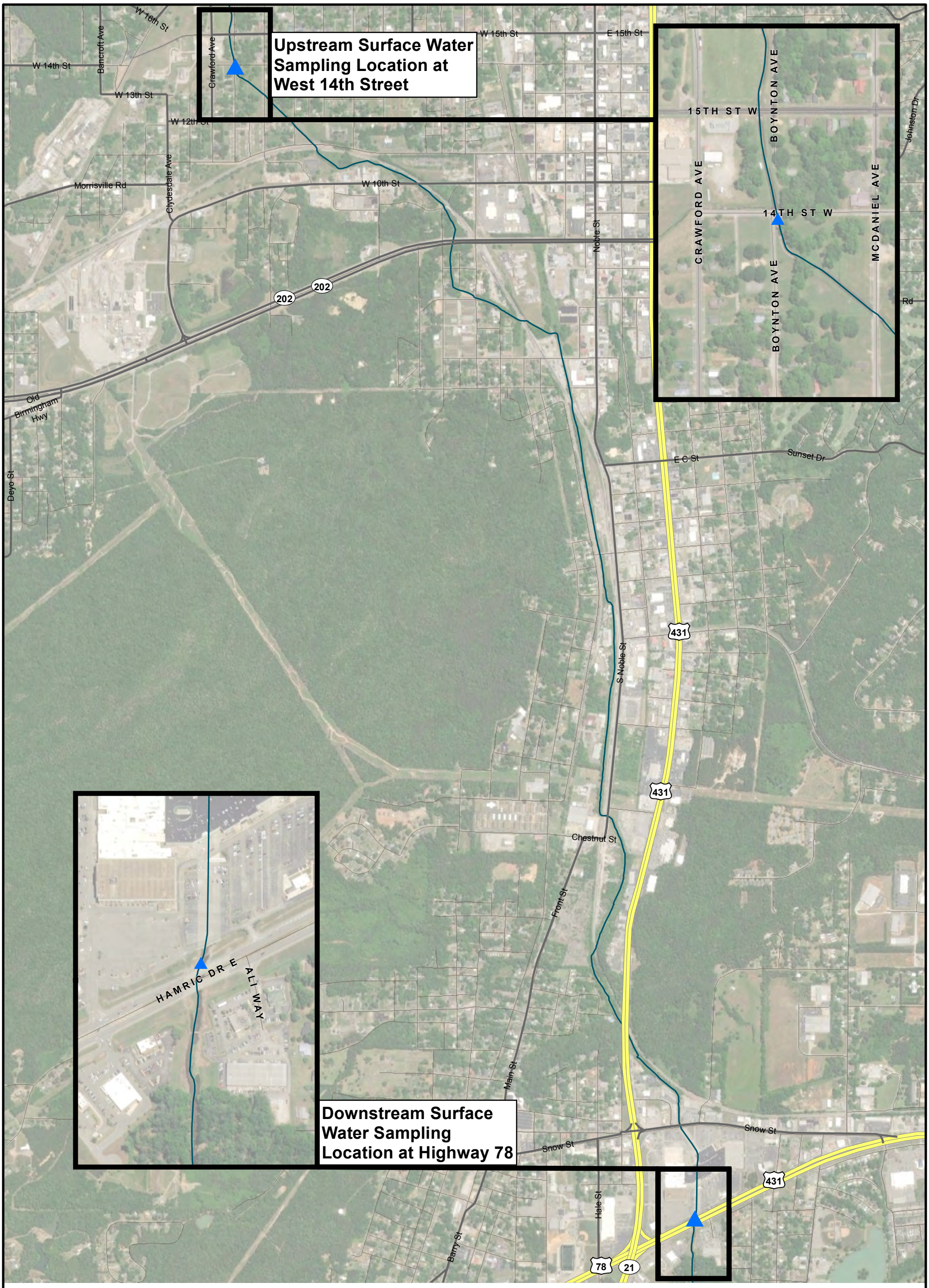
Feet

Anniston PCB Site
Anniston, Alabama
OU-1/OU-2 Predesign Investigation Work Plan

Known Structures along Snow Creek

Solutia

Figure
3-13



Legend

- Surface Water Sampling Location
- Snow Creek Centerline

Notes

1. Aerial imagery provided by Esri basemap services

0 1,000 2,000 3,000 4,000

Feet

Anniston PCB Site
Anniston, Alabama
OU-1/OU-2 Predesign Investigation Work Plan

Surface Water Sampling Locations along Snow Creek

Solutia

Figure
3-14

APPENDIX A
Uniform Federal Policy
Quality Assurance Project Plan

UFP-QAPP Worksheet #1 & #2: Title and Approval Page

Project Identifying Information

Site Name/Project Name:	Anniston PCB Site OU-1/OU-2
Site Location:	702 Clydesdale Ave., Anniston, Alabama 36201
Contract/Work Assignment Number:	Geosyntec Project Number #BR0579

Lead Organization:



June 8, 2022

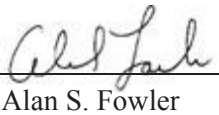
Gayle Macolly — Date
Anniston PCB Site Manager
Pharmacia LLC and Solutia Inc. (P/S)

Federal Regulatory Agency:

PAMELA SCULLY Digitally signed by PAMELA SCULLY
Date: 2022.07.05 11:56:24 -04'00'

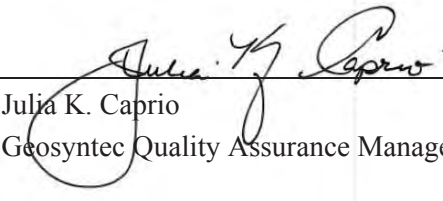
Pamela Scully, P.E. — Date
Remedial Project Manager
USEPA Region 4

Other Stakeholders:



June 8, 2022

Alan S. Fowler — Date
Geosyntec Project Manager



June 8, 2022

Julia K. Caprio — Date
Geosyntec Quality Assurance Manager

List plans and reports from previous investigations relevant to this project

BBL. 1999. *Supplemental RCRA Facility Investigation Work Plan for the Off-Site Portion of the RFI/CS Program*. Blasland, Bouck & Lee, Inc. February.

BBL. 2000. *Off-Site RCRA Facility Investigation (RFI) Report*. Blasland, Bouck & Lee, Inc. June 2000.

BBL. 2004. *Field Sampling Plan for the Non-Residential Properties of the Operable Unit 1/Operable Unit 2 Area of the Anniston PCB Site*. Blasland, Bouck & Lee, Inc. October.

BBL. 2005. *Addendum to Field Sampling Plan for the Non-Residential Properties of the Operable Unit 1/Operable Unit 2 Area of the Anniston PCB Site* (Addendum #1). Blasland, Bouck & Lee, Inc. November.

BBL. 2006. *Addendum to the Field Sampling Plan for the Non-Residential Properties of the Operable Unit 1/Operable Unit 2 Area of the Anniston PCB Site* (Addendum #2). Blasland, Bouck & Lee, Inc. May.

Environ. 2015. *Remedial Investigation Report for Operable Unit 1/Operable Unit 2 of the Anniston PCB Site*, Revision 3. February.

Golder. 2015. *NTC Removal Action Completion Report for Residential Properties in OU-1/OU-2*. November.

P/S. 2021a. *Remedial Design Work Plan*. Solutia, Inc., Anniston, Alabama. Prepared by Geosyntec Consultants Inc., GSI Environmental Inc., and Anchor QEA. August.

P/S. 2021b. *2020 Annual Groundwater Detection Monitoring and Corrective Action Effectiveness Report*. Solutia, Inc., Anniston, Alabama. Prepared by GSI Environmental Inc., Houston, Texas, April 28.

Ramboll Environ. 2016. *OU-1/OU-2 Feasibility Study Report, Anniston PCB Site*. March.

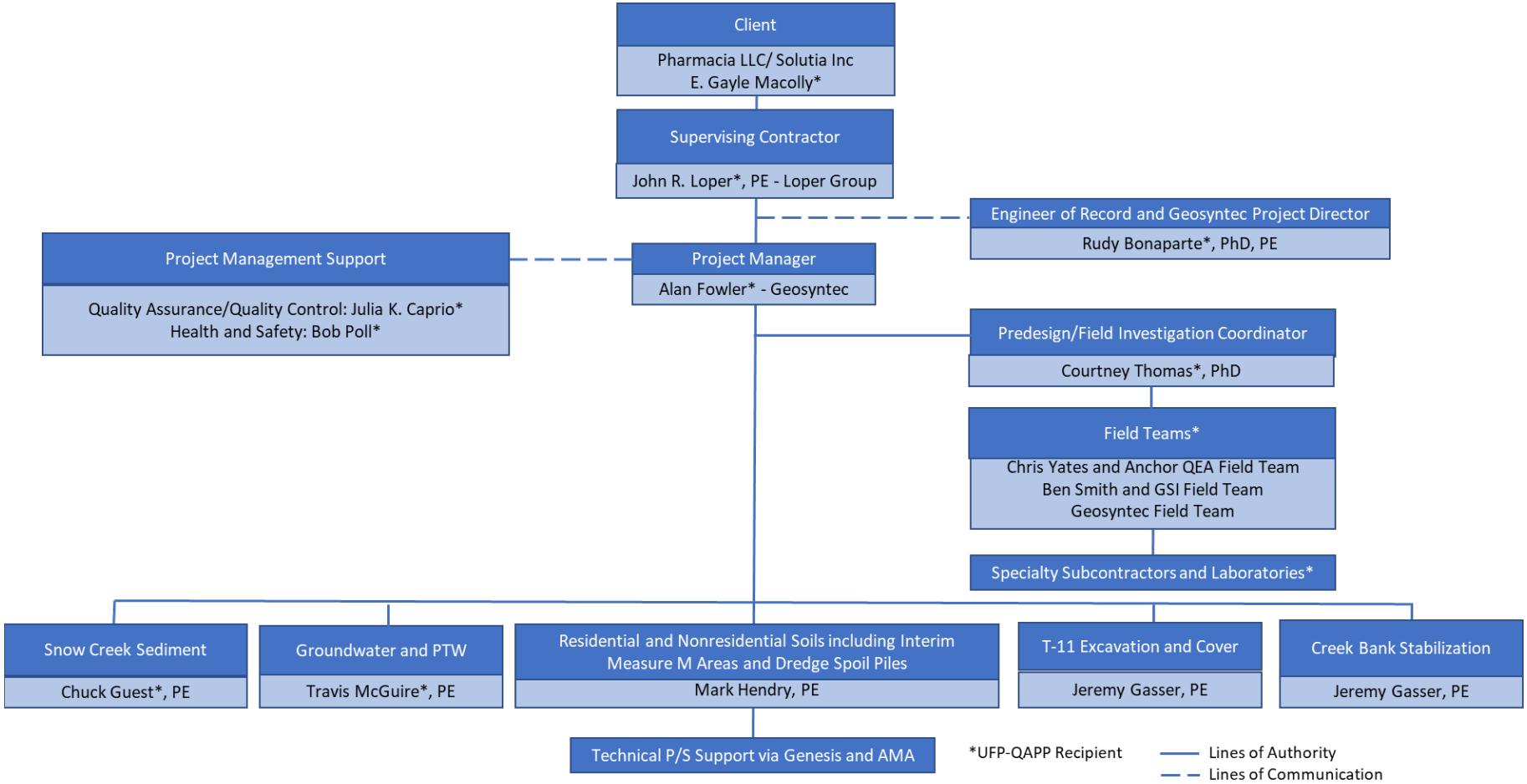
Solutia. 2003. *Removal Action Sampling Report West 9th Street Creek Anniston PCB Site*. Anniston, Alabama. April.

USEPA. 2006a. *Stipulation of the Parties Clarifying Partial Consent Decree between USEPA, Solutia Inc., and Pharmacia Corporation*. United States Environmental Protection Agency. July

USEPA. 2017. *Record of Decision, Operable Unit 1/Operable Unit 2, Anniston PCB Site, Anniston, Calhoun County, Alabama*. United States Environmental Protection Agency. November.

USEPA. 2021. *Remedial Design/Remedial Action Consent Decree.* United States of America v. Pharmacia LLC, and Solutia, Inc. Civil Action No. 1:02-CV-0749-KOB. U.S. District Court Northern District of Alabama Eastern Division. Effective date: March 26.

UFP-QAPP Worksheet #3 & #5: Project Organization and UFP-QAPP Distribution



PE: professional engineer
P/S: Pharmacia LLC and Solutia Inc.
PTW: principal threat waste

UFP-QAPP Worksheets #4, #7 & #8: Personnel Qualifications and Sign-Off Sheet

The final UFP-QAPP will be distributed to key project personnel listed in UFP-QAPP Worksheets #3 & #5, as well as to the Field Team Lead(s). A controlled electronic copy of the final UFP-QAPP will be made available to project personnel. Additionally, copies of applicable worksheets will be distributed to the analytical laboratories for reference during completion of project tasks to better ensure successful achievement of project data quality objectives (DQOs).

This worksheet identifies key project personnel for each organization performing tasks defined in this UFP-QAPP. The field supervisor, field team, and any subcontractors performing any hazardous waste on-site tasks are required to have current Occupational Safety and Health Administration (OSHA) HAZWOPER certifications (i.e., 40-hour and 8-hour annual refresher training) and also be current in medical monitoring clearance to work on hazardous waste sites. Additionally, the field supervisor will have completed the OSHA HAZWOPER 8-hour supervisor training.

Name	Project Title/Role	Organization	Responsibilities	Signature and Date of UFP-QAPP Read
Pamela Scully, PE	Remedial Project Manager	United States Environmental Protection Agency (USEPA)	• Project oversight and management • Project quality assurance and quality management • Community Involvement Plan, if determined to be necessary	
Gayle Macolly	Anniston PCB Site Manager	Pharmacia LLC/Solutia Inc. (P/S)	• Project oversight and management, and technical oversight of the Anniston PCB Site; communications with USEPA and the community in conjunction with USEPA and the Community Involvement Plan (if determined to be necessary)	
John Loper, PE	Supervising Contractor	The Loper Group	• Principal contractor retained by P/S to supervise and direct the implementation of the work; communications with P/S	
Alan Fowler	Project Manager	Geosyntec	• Initiate administrative actions with P/S, if budget or schedule changes. • Provide overall scheduling, supervision, and technical direction for sampling and related activities. • Coordinate site reviews and on-site inspections. • Maintain and organize project files, including field and analytical data. • Review and verify quality of field data during sampling and monitoring. • Maintain data acquisition documentation. • Ensure that all work is performed in accordance with USEPA policies and procedures and meets the requirements of the technical specifications, applicable codes and regulations, project contract, and scope. • Track budget and contract revisions and overall authority on documents. • Verify that work is performed in accordance with Geosyntec policies and procedures. • Lead approval authority for project documents. • Function as primary contact between the project team for information related to the project execution and decision-making. • Issue final reports.	
Rudy Bonaparte, PhD, PE (AL)	Engineer of Record and Project Director	Geosyntec	• Direct work to be performed in accordance with USEPA policies and procedures and so the work meets the requirements of the technical specifications, applicable codes and regulations, project contract, and scope. • Direct work to be performed in accordance with Geosyntec policies and procedures. • Provide senior leadership and direction.	

Name	Title	Organization	Responsibilities	Signature and Date of UFP-QAPP Read
Courtney L. Thomas, PhD	Field Team Lead	Geosyntec	• Provide field oversight of investigation activities and sample collection/field data acquisition. • Monitor quality and safety issues in the field and report deficiencies. • Review field forms for completeness and accuracy. • Organize field documents, including sampling forms and field data sheets. • Maintain daily field logs of work activities. • Verify proper calibration of sampling and field equipment. • Coordinate disposal investigation-derived waste (IDW). • Prepare and submit waste manifests and waste profiles.	
Julia K. Caprio	Quality Assurance Manager	Geosyntec	• Provide project quality assurance and quality control. • Assess final data usability, limitations, and completeness. • Perform field and laboratory audits as necessary. • Review field and laboratory nonconformance and corrective actions. • Provide data validation oversight.	
Kristoffer Henderson	Data Validator Team Lead	Geosyntec	• Perform data validation and verification according to Worksheets #34 through #36. • Conduct data usability assessment according to Worksheet #37. • Monitor corrective action documentation for adverse quality conditions and verify implementation of corrective actions. • Review definitive data with field records to apply data qualifiers. • Serve as main contact with analytical laboratory(ies) regarding data quality issues.	
John Andros	Analytical Laboratory Project Manager	Eurofins-TestAmerica Excel	• Perform analytical services for analytical samples collected. • Conduct quality assurance/quality control of laboratory procedures and analytical methods.	
Nader Rad	Geotechnical Laboratory Project Manager	Geotechnical Testing, Inc. (EGT)	• Perform geotechnical testing services for geotechnical samples collected. • Conduct quality assurance/quality control of laboratory procedures and geotechnical analytical methods.	

UFP-QAPP Worksheet #6: Communication Pathways

Communication Drivers	Responsible Affiliation	Name	Phone Number/E-mail	Procedure
Review of the UFP-QAPP	Pharmacia LLC and Solutia Inc. (P/S) Loper Group Geosyntec	P/S: Gayle Macolly Supervising Contractor: John R. Loper, PE Geosyntec Project Manager (PM): Alan Fowler	(256) 770-1299 egmaco@eastman.com (281) 635-2509 jloper@lopergroup.com (617) 834-5847 afowler@geosyntec.com	Review UFP-QAPP for submission to the United States Environmental Protection Agency (USEPA) Remedial Project Manager (RPM) for approval.
Final Approval of UFP-QAPP or Amendments	USEPA	USEPA RPM: Pamela Scully	(404) 562-8935 scully.pamela@epa.gov	Final approved UFP-QAPP or amendments received from P/S for USEPA RPM approval.
Approval of Amendments to the UFP-QAPP	Geosyntec	Geosyntec PM: Alan Fowler	(617) 834-5847 afowler@geosyntec.com	Obtain initial approval from P/S prior to completing amendments.
Review of Amendments to the UFP-QAPP	P/S Loper Group Geosyntec	P/S: Gayle Macolly Supervising Contractor: John R. Loper, PE Geosyntec PM: Alan Fowler	(256) 770-1299 egmaco@eastman.com (281) 635-2509 jloper@lopergroup.com (617) 834-5847 afowler@geosyntec.com	Review amendments to the UFP-QAPP for submission to the USEPA RPM for approval.
Final Approval of UFP-QAPP Amendments	USEPA	USEPA RPM: Pamela Scully	(404) 562-8935 scully.pamela@epa.gov	Final approved UFP-QAPP Amendments received from P/S for USEPA RPM approval.

Communication Drivers	Responsible Affiliation	Name	Phone Number/E-mail	Procedure
Approval of Activities Deviating from the UFP-QAPP	Loper Group Geosyntec	Supervising Contractor: John R. Loper, PE Geosyntec PM: Alan Fowler	(281) 635-2509 jloper@lopergroup.com (617) 834-5847 afowler@geosyntec.com	Obtain initial approval from P/S prior to deviating from the UFP-QAPP.
Review of Activities Deviating from the UFP-QAPP	P/S	P/S: Gayle Macolly	(256) 770-1299 egmaco@eastman.com	Review deviations to the UFP-QAPP for submission to the USEPA RPM for approval.
Document Control	P/S Loper Group Geosyntec	P/S: Gayle Macolly Supervising Contractor: John R. Loper, PE Geosyntec PM: Alan Fowler	(256) 770-1299 egmaco@eastman.com (281) 635-2509 jloper@lopergroup.com (617) 834-5847 afowler@geosyntec.com	All reports and formal correspondence will be reviewed by the Geosyntec PM prior to being submitted to P/S. Documents prepared by Geosyntec PM for submittal to USEPA will be reviewed by the Quality Assurance (QA) Manager or other team member prior to submittal to P/S for submission to the USEPA RPM.

Communication Drivers	Responsible Affiliation	Name	Phone Number/E-mail	Procedure
Stop Work and Initiation of Corrective Action	All staff	All staff	All staff	Any member of the team has the authority to stop work based on concerns related to quality control (QC), H&S, or other serious deficiencies that may compromise the integrity of the long-term monitoring program or the safety of the field crew. The Geosyntec PM will communicate work stoppages to P/S within 24 hours. P/S will notify the USEPA RPM, as needed.
	P/S	P/S: Gayle Macolly	(256) 770-1299 egmaco@eastman.com	
	Loper Group	Supervising Contractor: John R. Loper, PE	(281) 635-2509 jloper@lopergroup.com	
	Geosyntec	Geosyntec PM: Alan Fowler	(617) 834-5847 afowler@geosyntec.com	

Communication Drivers	Responsible Affiliation	Name	Phone Number/E-mail	Procedure
Reporting of Health and Safety (H&S) Issues	P/S Loper Group Geosyntec	P/S: Gayle Macolly Supervising Contractor: John R. Loper, PE Geosyntec PM: Alan Fowler	(256) 770-1299 egmaco@eastman.com (281) 635-2509 jloper@lopergroup.com (617) 834-5847 afowler@geosyntec.com	H&S issues involving an injury, a near miss, or a condition that may result in an incident must be reported to the H&S Manager immediately. The H&S Manager will forward this information to the Geosyntec PM using telephone and email as soon as possible. The Geosyntec PM will notify P/S or designee as soon as possible. P/S will notify the USEPA RPM, as needed, of any serious H&S incident/issues. Incidents/issues that are not serious may be forwarded to the USEPA RPM through P/S monthly in the monthly progress reports.
Reporting Issues Related to Statement of Work (SOW) Requirements	P/S Loper Group Geosyntec	P/S: Gayle Macolly Supervising Contractor: John R. Loper, PE Geosyntec PM: Alan Fowler	(256) 770-1299 egmaco@eastman.com (281) 635-2509 jloper@lopergroup.com (617) 834-5847 afowler@geosyntec.com	All issues that may impact data quality objectives (DQOs) will be reported to the Geosyntec PM and P/S immediately.

Communication Drivers	Responsible Affiliation	Name	Phone Number/E-mail	Procedure
Community Relations, Including Community Involvement Plan (if needed at later date)	USEPA	USEPA RPM: Pamela Scully	(404) 562-8935 scully.pamela@epa.gov	All community relations will be reported to P/S, and P/S will coordinate with the USEPA RPM and community involvement coordinator (if assigned at a later date), as needed.
		USEPA Community Involvement Coordinator: Stephanie Brown	(404) 562-8450 brown.stephanie@epa.gov	
	P/S	P/S: Gayle Macolly	(256) 770-1299 egmaco@eastman.com	
		P/S Community Outreach Coordinator: Carol Kirk	(256) 231-8447 ckirk@eastman.com	
	Loper Group	Supervising Contractor: John R. Loper, PE	(281) 635-2509 jloper@lopergroup.com	
	Geosyntec	Geosyntec PM: Alan Fowler	(617) 834-5847 afowler@geosyntec.com	
Schedule Changes	P/S	P/S: Gayle Macolly	(256) 770-1299 egmaco@eastman.com	Changes to USEPA-approved schedules (e.g., field sampling schedule) will be communicated to the Geosyntec PM, who will in turn communicate changes to P/S for discussion with the USEPA RPM.
	Loper Group	Supervising Contractor: John R. Loper, PE	(281) 635-2509 jloper@lopergroup.com	
	Geosyntec	Geosyntec PM: Alan Fowler	(617) 834-5847 afowler@geosyntec.com	

Communication Drivers	Responsible Affiliation	Name	Phone Number/E-mail	Procedure
Data Release	P/S Loper Group Geosyntec	P/S: Gayle Macolly Supervising Contractor: John R. Loper, PE Geosyntec PM: Alan Fowler QA Manager: Julia Caprio	(256) 770-1299 egmaco@eastman.com (281) 635-2509 jloper@lopergroup.com (617) 834-5847 afowler@geosyntec.com (865)291-4696 jklenscaprio@geosyntec.com	Data (in format of standard regional electronic data deliverable [EDD] and laboratory reports) will be reviewed by the QA Manager and Geosyntec PM prior to being provided to P/S for release to the USEPA RPM. All data records will be delivered to USEPA in accordance with the Consent Decree.
Notification of Delays or Changes to Field Work	P/S Loper Group Geosyntec	P/S: Gayle Macolly Supervising Contractor: John R. Loper, PE Geosyntec PM: Alan Fowler Field Team Lead (FTL): Courtney Thomas	(256) 770-1299 egmaco@eastman.com (281) 635-2509 jloper@lopergroup.com (617) 834-5847 afowler@geosyntec.com (713)408-6885 clthomas@geosyntec.com	Delays or changes to the approved work plan will require approval by the Geosyntec PM and P/S. P/S will communicate delays or changes to USEPA, as needed.

Communication Drivers	Responsible Affiliation	Name	Phone Number/E-mail	Procedure
Real-Time Changes to Sample Collection or Analysis Procedures	P/S Loper Group Geosyntec Eurofins-TestAmerica Excel Geotechnical Testing (EGT)	P/S: Gayle Macolly Supervising Contractor: John R. Loper, PE Geosyntec PM: Alan Fowler FTL: Courtney Thomas Eurofins-TestAmerica Lab PM: John Andros EGT Lab PM: Nader Rad	(256) 770-1299 egmaco@eastman.com (281) 635-2509 jloper@lopergroup.com (617) 834-5847 afowler@geosyntec.com (713) 408-6885 clthomas@geosyntec.com (404) 944-4744 john.andros@eurofinset.com (770) 910-7537 nrad@excelgeotesting.com	Conditions requiring variation to sampling and analysis procedures will be reported to the FTL within 24 hours of the condition requiring the modification. The FTL will notify the Geosyntec PM. These conditions will require approval by the Geosyntec PM and P/S and will be reported by P/S, to the USEPA RPM, as needed.
Quality Issues within Laboratory	Geosyntec Eurofins-TestAmerica EGT	Geosyntec PM: Alan Fowler QA Manager: Julia Caprio Eurofins-TestAmerica Lab PM: John Andros EGT Lab PM: Nader Rad	(617) 834-5847 afowler@geosyntec.com (865) 291-4696 jklenscaprio@geosyntec.com (404) 944-4744 john.andros@eurofinset.com (770) 910-7537 nrad@excelgeotesting.com	Eurofins-TestAmerica and EGT lab PMs will notify QA Manager and Geosyntec PM to determine corrective actions.

Communication Drivers	Responsible Affiliation	Name	Phone Number/E-mail	Procedure
Reporting Issues Related to Data Quality, Including Inability to Meet Reporting Limits	Eurofins-TestAmerica EGT Geosyntec	Eurofins-TestAmerica Lab PM: John Andros EGT Lab PM: Nader Rad QA Manager: Julia Caprio	(404) 944-4744 john.andros@eurofinset.com (770) 910-7537 nrad@excelgeotesting.com (865)291-4696 jklenscaprio@geosyntec.com	Problems with data quality will be reported to the Laboratory PM and the QA Manager within 24 hours of laboratory results.

Communication Drivers	Responsible Affiliation	Name	Phone Number/E-mail	Procedure
Corrective Actions	Geosyntec	QA Manager: Julia Caprio	(865)291-4696 jklenscaprio@geosyntec.com	Corrective action responses may include the following: • Field Safety Audit completed by FTL, Geosyntec PM, and/or Health and Safety Manager • On-Site Field Audit completed by FTL and/or QA Manager • Technical System Internal Audit or Field Sampling Procedure completed by QA Manager • Off-Site Laboratory Technical Systems Audit completed by Laboratory Personnel • Data Quality Assessment completed by QA Manager • Project Documentation Audit completed by QA Manager
		Health and Safety Manager: Bob Poll	(813)379-4420 bpoll@geosyntec.com	
		Geosyntec PM: Alan Fowler	(617) 834-5847 afowler@geosyntec.com	
		FTL: Courtney Thomas	(713)408-6885 clthomas@geosyntec.com	
	Eurofins-TestAmerica	Eurofins-TestAmerica Lab PM: John Andros	(404) 944-4744 john.andros@eurofinset.com	
	EGT	EGT Lab PM: Nader Rad	(770) 910-7537 nrad@excelgeotesting.com	
Quality Issues of Laboratory-Reported Data	Geosyntec	Lead Validator: Kristoffer Henderson	(865) 291-4705 khenderson@geosyntec.com	Lead Validator will notify QA Manager and Geosyntec PM of data validation and usability assessment issues.
		QA Manager: Julia Caprio	(865)291-4696 jklenscaprio@geosyntec.com	
		Geosyntec PM: Alan Fowler	(617) 834-5847 afowler@geosyntec.com	
Reporting	Geosyntec	Geosyntec PM: Alan Fowler	(617) 834-5847 afowler@geosyntec.com	Lead report preparation following each sampling event

UFP-QAPP Worksheet #9: Project Scoping Session Participants Sheet

Project Name: Anniston PCB Site **Project Manager: Alan Fowler**	**Site Name: Anniston PCB Site** **Site Location: Anniston, Alabama**

Date of Session: July 29, 2021
Scoping Session Purpose: Predesign investigation data gap identification

Name	Affiliation	E-mail Address	Project Role
Gayle Macolly	Pharmacia LLC./ Solutia Inc. (P/S)	egmaco@eastman.com	P/S Project Coordinator
John Loper	The Loper Group	jloper@lopergroup.com	Supervising Contractor
Thomas Loper	The Loper Group	tloper@lopergroup.com	Technical support resource
Michael Price	Genesis Project, Inc.	mprice@genproject.com	Residential Removal Manager
John Schell	TEA, Inc.	jschell@teainconline.com	Technical support resource
Chuck Guest	Anchor QEA	cguest@anchorqea.com	Project Manager for Anchor QEA
Chris Yates	Anchor QEA	cyates@anchorqea.com	Field Lead for Anchor QEA
Travis McGuire	GSI	tmmcguire@gsi-net.com	GSI Project Manager
Ben Smith	GSI	wbsmith@gsi-net.com	Field Lead for GSI
Kenneth Walker	GSI	klwalker@gsi-net.com	Field sampling support for GSI
Alan Fowler	Geosyntec	afowler@geosyntec.com	Geosyntec Project Manager
Rudy Bonaparte	Geosyntec	rbonaparte@geosyntec.com	Engineer of Record and Geosyntec Project Director

Decisions reached during meetings on date listed above:

- July 29, 2021, virtual meeting/conference call with key members of the remedial design (RD) team to identify data gaps based on an assessment of data needs and a review of existing data.
- Data gaps identified in the July 29, 2019, meeting were used to scope and plan the in-person Predesign Investigation Work Plan (PDIWP) and Site-walk meeting that was scheduled for August 25–26, 2021, in Anniston, Alabama.

Project Name: Anniston PCB Site Project Manager: Alan Fowler	Site Name: Anniston PCB Site Site Location: Anniston, Alabama

Date of Session: August 25 and 26, 2021

Scoping Session Purpose: Site Walk and Predesign Investigation Work Plan (PDIWP) Planning Meeting

Name	Affiliation	E-mail Address	Project Role
Gayle Macolly	Pharmacia LLC./ Solutia Inc. (P/S)	egmaco@eastman.com	P/S Project Coordinator
John Loper	The Loper Group	jloper@lopergroup.com	Supervising Contractor
Thomas Loper	The Loper Group	tloper@lopergroup.com	Technical support resource
Michael Price	Genesis Project, Inc.	mprice@genproject.com	Residential Removal Manager
Josh Threadgill	Genesis Project, Inc.	jthreadgill@genproject.com	Construction Manager
John Schell	TEA, Inc.	jschell@teainconline.com	Technical support resource
Chuck Guest	Anchor QEA	cguest@anchorqea.com	Project Manager for Anchor QEA
Chris Yates	Anchor QEA	cyates@anchorqea.com	Field Lead for Anchor QEA
Travis McGuire	GSI	tmmcguire@gsi-net.com	GSI Project Manager
Ben Smith	GSI	wbsmith@gsi-net.com	Field Lead for GSI
Alan Fowler	Geosyntec	afowler@geosyntec.com	Geosyntec Project Manager
Rudy Bonaparte	Geosyntec	rbonaparte@geosyntec.com	Engineer of Record and Geosyntec Project Director
Mark Hendry	Geosyntec	mhendry@geosyntec.com	Engineering support for Geosyntec
Jeremy Gasser	Geosyntec	jgasser@geosyntec.com	Engineering support for Geosyntec
Courtney Thomas	Geosyntec	cthomas@geosyntec.com	Field Team Lead

Decisions reached during meetings on date listed above:

- RD team meeting to review the planned predesign investigation (PDI) activities to address data gaps discussed during the July 29, 2021, meeting held via teleconference.
- August 25–26, 2021, in-person meeting provided the RD team with a boots-on-the-ground opportunity to conduct a field review of the remediation areas identified in the Operable Unit 1/Operable Unit 2 (OU-1/OU-2) Remedial Design/Remedial Action Consent Decree Statement of Work (RD/RA CD SOW). This was especially important

because some RD team members either had not physically been to the Site or had only worked on other OUs and had not viewed the portions of the Anniston PCB Site that will be remediated under the OU-/OU-2 RD/RA CD.

- The site-specific knowledge gained during the August 25–26, 2021, Site walk and the data gaps identified during the July 29, 2021, virtual meeting were used to finalize the outline for the PDIWP and the approach to prepare this UFP-QAPP.
- Analytical and geotechnical laboratory reports and electronic data deliverables will be submitted to USEPA in accordance with the Consent Decree (USEPA 2021).

UFP-QAPP Worksheet #10: Problem Definition

Problem Statement: The remedy the United States Environmental Protection Agency (USEPA) selected for Operable Unit 1 and Operable Unit 2 (OU-2/OU-2) includes multiple components for soil, groundwater, and sediment as described in the Remedial Design Work Plan (Pharmacia LLC and Solutia Inc. [P/S] 2021a) based on the presence of polychlorinated biphenyls (PCBs) and non-PCB constituents in these media at concentrations greater than their respective goals from the OU-1/OU-2 Record of Decision (ROD).

Project Description: The work to be conducted as part of a predesign investigation (PDI) and treatability studies (TSs) includes the following:

Floodplain and Creek Bank Soil Sampling

The existing floodplain soil and creek bank data were reviewed relative to the data needed to prepare a remedial design (RD) that meets the requirements of the OU-1/OU-2 ROD to identify data gaps. Data gaps were not identified for residential soil because all removal actions have been conducted for residential properties in OU-1/OU-2 where access has been granted and the properties have been identified as suitable for removal. No data gaps were identified for the high-activity special use areas because these areas have been sampled and removal actions completed where required under the Non-Time-Critical Removal Action Agreement.

Comparing the data needed to complete the RD with the existing data revealed data gaps for several other categories of floodplain soil and the creek bank areas identified in the OU-1/OU-2 ROD. The results of this process are presented below for the low-activity special use areas, interim measure (IM) areas, dredge spoil piles, nonresidential soils, and creek bank areas.

- **Low-activity special use areas:** PCB concentration data for subsurface soil in the low-activity special use area are needed to compare with the remedial goal of 97 milligram per kilogram (mg/kg). While these data could be collected as confirmation samples during remedial action (RA) construction, most of these low-activity areas are easy to access , and the data can be efficiently collected during the PDI and used to scope the required excavation depths as part of the RD. Land use conditions at the low-activity special use areas will be reviewed during the PDI prior to sampling to confirm they are consistent with the Stipulation's definition of a low-activity special use area and are suitable for removal (e.g., are not heavily vegetated or wooded; like low-activity special use area with property identification number [PPIN] 20717). Documentation of current land use will be collected for all low-activity special use areas identified in the OU-1/OU-2 ROD. This information will be used to confirm that the land use initially identified as part of the investigations conducted under the Stipulation have not changed. For example, if a low-activity area has transitioned to industrial use, relevant information (ownership, zoning, the removal or addition of built-structures, etc.) will be gathered to document this change in land use. If no changes to the properties have taken place since the initial surveys were conducted under the Stipulation, the plan is

to conduct the sampling as described in Section 3.1.2.1 of the OU-1/OU-2 PDI Work Plan (PDIWP) for locations shown on Figures 3-2a through 3-2l of OU-1/OU-2 PDIWP. Any changes in land use will be discussed with USEPA and may result in modifying the sampling program. Consistent with the OU-1/OU-2 ROD, subsurface soil sampling will be conducted only for low-activity special use areas that are suitable for removal. For example, overgrown and paved areas will not be sampled.

- **IM areas:** PCB concentrations in subsurface soil for the specific IM areas identified in the OU-1/OU-2 ROD are needed to compare with the remedial goal of 97 mg/kg. These IM areas are accessible, and the data can be efficiently collected as part of the PDI. The Hall Street IM area is also accessible for conducting physical inspections of the cover integrity as part of the PDI. PCB concentrations in surface and subsurface soil for the drainage ditch located west and east of the PB-RR-37 location will be collected for comparison with the surface remedial goals for surface and subsurface soil of 21 mg/kg and 97 mg/kg, respectively. Surface soil at the downstream end of the eastern portion of this ditch that have the potential to erode and migrate to Snow Creek will also be compared to the remedial goal for sediment of 3 mg/kg. Proposed sample locations are shown on Figures 3-3a-f of OU-1/OU-2 PDIWP.
- **Dredge spoil piles:** PCB concentration data are needed to assess whether remediation of dredge spoil pile SC-2 is required. Dredge spoil SC-1 and dredge spoil pile SC-2 are located sufficiently away from Snow Creek such that soils located at the outside edge of the piles are not potential sources to Snow Creek. Soil PCB concentrations beneath the dredge spoil piles required for removal will be assessed through post-removal samples collected during the RA construction phase of the project. The dredge spoil piles are in remote areas with difficult physical access. Collecting samples beneath the piles during RA construction will limit the disturbance of existing vegetation because the equipment needed to sample beneath these piles is like the equipment that will be used for their excavation. Since dredge spoil pile SC-8 is located adjacent to and within the borders of Snow Creek, this dredge spoil pile is being addressed as part of the Snow Creek sediment RD. The available analytical testing results for these dredge spoil piles are shown on Figure 2-5a of the OU-1/OU-2 PDIWP for dredge spoil pile SC-1 and on Figure 2-5b of the OU-1/OU-2 PDIWP for dredge spoil piles SC-7 and SC-8.
- **Nonresidential soil:** PCB concentrations in subsurface soil for the nonresidential soil areas identified in the OU-1/OU-2 ROD as candidate removal areas are needed to compare with the remedial goals of 97 mg/kg. For the PB-RR-37 area, confirmation of surface soil PCB concentrations will be conducted as an initial activity and pending the results, the subsurface soils may be analyzed. Additionally, PCB concentrations at PB-RR-37 will be profiled from ground surface to the groundwater table to evaluate potential leaching of PCBs to groundwater. Surface and subsurface soils from the ditch upstream and downstream of PB-RR-37 will also be evaluated relative to the applicable goals. The surface soil PCB results from samples collected from the portion of the ditch closest to Snow Creek will also be assessed relative to potential erosion into Snow Creek. Geotechnical data are also needed to design the excavation of principal threat waste (PTW) soil at the T-11 area and for the broader T-11 area that will be capped with clean backfill and a low-permeability cover. These data will assist in assessing the potential for differential settlement of the post-cap surface if groundwater treatment

equipment is constructed on the T-11 side of Snow Creek. Geotechnical data are also needed to design long-term access to the T-11 area that may include a bridge or pipe bridge. The nonresidential areas selected for remediation in the OU-1/OU-2 ROD and included in the Statement of Work (SOW) are shown on Figure 3-4a through 3-4h of the OU-1/OU-2 PDIWP.

- **Creek bank areas:** Geotechnical data are needed to assess creek bank stabilization options along Snow Creek at and immediately downstream of its confluence with the 11th Street Ditch.

Specific data quality objectives (DQOs) for the OU-1/OU-2 PDI program are as follows for soil:

- SO-DQO 1: Refine the current understanding of PCBs for dredge spoil pile SC-2 to evaluate whether PCB concentrations exceed the remedial goal of 3 mg/kg. Once the dredge spoil piles identified for remediation are removed, surface soil and subsurface soil beneath the dredge spoil piles will be sampled and assessed for PCBs based on the ROD requirements of 21 mg/kg for surface soil and 97 mg/kg for subsurface soil.
- SO-DQO 2: Refine the current understanding of subsurface soil PCB concentrations in floodplain soil areas, including confirmed low-activity special use areas, certain IM areas, and the nonresidential areas, for comparison with the OU-1/OU-2 ROD remedial goal of 97 mg/kg.
- SO-DQO 3: Gather geotechnical characterization data from the T-11 area to support the detailed design of a low-permeability cap and creek bank stabilization measures.
- SO-DQO 4: Evaluate whether representative soil PCB concentrations at the PB-RR-37 area, including soils along the drainage ditch upstream and downstream of this area, are above the ROD requirements of 21 mg/kg for surface soil and 97 mg/kg for subsurface soils. For locations adjacent to Snow Creek, concentrations will be compared to the OU-1/OU-2 ROD remedial goal of 3 mg/kg, and evaluated relative to their ability to erode and migrate into Snow Creek. If PCB are detected in subsurface soil proximate to the groundwater table in the PB-RR-37, discussions will be held with USEPA regarding the potential installation of a groundwater monitoring well to assess if groundwater PCB concentrations in this area exceed the remedial goal of 0.5 µg/L.
- SO-DQO 5: Evaluate whether portions of the Hall Street IM cover soils require augmentation to meet the IM design requirements.

The PDI efforts are focused on the following activities for floodplain and creek bank soils to address these DQOs:

- For the dredge spoil piles, sampling will be focused on SC-2 to evaluate PCB concentrations relative to the remedial goal of 3 mg/kg.
- For low-activity special use areas, subsurface soil will be sampled at the same surface soil locations associated with the *Stipulation of the Parties Clarifying Partial Consent Decree between USEPA, Solutia Inc., and Pharmacia Corporation* (USEPA 2006a) and the OU-1/OU-2 ROD requirement of 97 mg/kg. Composite samples will be collected from each low-activity special use area. Material from five sample locations will make up one composite sample.

- For IM areas (excluding the Hall Street IM area), soil will be sampled relative to the subsurface remedial goal of 97 mg/kg by collecting 5-point composites of soil material. For the Hall Street IM area, the area will be visually inspected to evaluate whether portions of the IM cover soils require augmentation to meet the IM design requirements.
- For the nonresidential areas, subsurface soil will be sampled using incremental sampling methodology (ISM) for the candidate removal areas greater than 0.5 acres identified in the OU-1/OU-2 ROD for comparison with the subsurface remedial goal of 97 mg/kg. 5-point composites will be collected for nonresidential areas smaller than 0.5 acres. One 5-point composite will be collected per 0.25-acre area.
- For the PB-RR-37 area, historical results of collocated soil samples (PB-RR-37 and PB-RR-40) exhibited PCB concentrations differing by more than 3 orders of magnitude (3,700J mg/kg and 2.3J mg/kg, respectively). These variable concentrations suggest significant heterogeneity in surface soil PCB concentrations. Confirmation soil sampling will be conducted to determine a representative PCB concentration in soils. Subsurface soil sampling at PB-RR-37 will also be conducted to the elevation of the groundwater table and if PCBs are detected in soils at the groundwater interface, the potential installation of a groundwater well at this location will be discussed with USEPA.
- For the T-11 area, a geotechnical characterization will be completed to support the development of the RD for excavation and low-permeability capping in this area. The geotechnical investigation will also be used to evaluate potential bridge or pipe bridge options to access the T-11 area during and after engineering design aspects of the low-permeability cap, the groundwater treatment plant, and bridge foundations. Results of the geotechnical investigations along the banks of Snow Creek in the T-11 area will also be used along with physical inspection results to develop the RD for the select creek bank areas identified in the OU-1/OU-2 ROD.

Groundwater Sampling and TS

Groundwater investigations were conducted in OU-1/OU-2 prior to the remedial investigation (RI) as part of previous activities conducted at and adjacent to OU-3 and under the non-time-critical residential removal program. Groundwater was also investigated as part of the RI. Details of investigations and their results relevant to the groundwater RD for the Eastside Properties and T-11 area are provided in the following subsections. Section 2.1.1.6 of the PDIWP discusses existing data for the PB-RR-37 area, where the need for groundwater RD activities will be determined based on findings of the PDI.

- **Eastside Properties**: As discussed in the OU-1/OU-2 ROD, PCB concentrations in soil in portions of the Eastside Properties have been detected at concentrations over 500 mg/kg, indicating the potential presence of PTW. Between 1997 and 2012, IMs were implemented to reduce potential exposure to and migration of PCB-impacted soil. The IMs generally included soil caps where total PCB concentrations were less than 500 mg/kg and geomembrane caps where PCB concentrations exceeded 500 mg/kg. Existing soil characterization data for the Eastside Properties (shown on Figures 2-14a through 2 14c of the PDIWP) were used to evaluate potential locations for a new groundwater monitoring

well that met the following criteria: (1) an area with low or no PCB impacts in shallow soil to minimize potential drag-down of PCBs from soil to groundwater during well installation and (2) an area close to and downgradient of areas within the Eastside Properties with high PCB concentrations in soil.

- **T-11 Area**: Groundwater samples collected from monitoring well T-11 (Figure 2-12 of the PDIWP) as part of the OU-1/OU 2 RI in 2008 exhibited total PCB concentrations in unfiltered samples ranging from 3.3 micrograms per liter (µg/L) (Aroclors) to 17.3 µg/L (homologs), with a nondetect result (Aroclors) and a concentration of 10.4 µg/L (homologs) reported following redevelopment of the monitoring well that was completed due to high turbidity noted in the initial samples. Additional samples collected from T-11 in 2012 exhibited total PCB concentrations in the unfiltered sample between 12 µg/L based on Aroclors and 30 µg/L based on the sum of PCB homologs. As noted in the ROD, the distribution of PCBs in the floodplain area surrounding T-11 appeared to be unique for the Site, characterized by the presence of the lower-chlorinated PCB compounds (e.g., Aroclor 1221, and monochlorobiphenyl and dichlorobiphenyl homolog groups).

  Existing hydrogeologic data related to T-11 groundwater includes information on the dominant soil type observed during well installation, groundwater elevation data over time, and aquifer slug testing data. The soils encountered at T-11 consisted of an "orange to red, stiff to soft clay" that were present from ground surface to the termination depth of 21 feet below ground surfaces (bgs). Based on the proximity of T-11 to the surface water of Snow Creek, the well was installed to monitor an interval of "minor moisture" observed from 6 to 10 feet bgs. The well is constructed with a 5-foot screen placed from 8.5 to 13.5 feet bgs. The water table at T-11 was observed at approximately 7 feet bgs following installation. Recent depth-to-water measurements have been consistent with the initial observation, corresponding to a static water elevation of approximately 687.7 feet above mean sea level. The hydraulic conductivity reported for T-11 based on slug testing was 5.93 feet per day, corresponding to 2×10^{-3} centimeters per second.

Specific DQOs for the OU-1/OU-2 PDI program are as follow for groundwater:

- GW-DQO 1: Evaluate whether representative groundwater PCB concentrations at the Eastside Properties (and potentially location PB-RR-37) exceed the remedial goal of 0.5 µg/L.
- GW-DQO 2: Gather hydrogeologic data at the T-11 area to support the detailed design of a groundwater extraction-and-treatment system.
- GW-DQO 3: Evaluate potentially efficient and cost-effective granular activated carbon (GAC) media for PCB treatment.
- GW-DQO 4: Estimate the time for PCB breakthrough in a full-scale GAC system.

Groundwater samples will be collected to support the design of the T-11 area treatment system and the evaluation of potential PTW that will be conducted for the Eastside Properties area. Groundwater in the PB-RR-37 area may be sampled depending on the results of the soil investigation to be completed in the PB-RR-37 area. As part of the groundwater TS, bench-scale

testing will be conducted to evaluate groundwater treatment using granular activated carbon filtration.

Sediment Sampling and TS

Most of the existing sediment data for the OU-1/OU-2 portion of Snow Creek were generated as part of the 1999 Resource Conservation and Recovery Act Facility Investigation (RFI). Due to the dynamic nature of the creek system, there is the potential for sediment deposits to have moved or for new sediment deposits to have formed over the past 20-plus years. In addition, the culvert beneath Quintard Mall and other portions of Snow Creek where property access was not granted were not characterized during the 1999 RFI.

The sediment PDI will be conducted to address the following data gaps:

- Additional chemical and physical data are needed to provide updated information to refine the sediment remedial extents and volumes and to determine off-site disposal requirements.
- Treatability testing is needed to evaluate potential options for dewatering sediment that will be removed from the creek.

Specific DQOs for the OU-1/OU-2 PDI program are as follows for sediment:

- SED-DQO 1: Provide an updated delineation of the current locations, extents, and depths of sediment deposits within the OU-1/OU-2 portion of Snow Creek as compared to the deposits depicted in the OU-1/OU-2 FS to refine removal volume estimates.
- SED-DQO 2: Refine the current understanding of sediment in the OU-1/OU-2 portion of Snow Creek that exceed the remedial criteria listed in the ROD to define the lateral and vertical limits of sediment removal.
- SED-DQO 3: Evaluate potentially efficient and cost-effective methods for dewatering and stabilizing sediment excavated from Snow Creek.
- SED-DQO 4: Evaluate treatment technologies that may be necessary to meet the requirements for discharging the supernatant that drains from sediment excavated from the creek.

SED-DQOs 1 and 2 will be addressed through field investigations. SED-DQO 3 and SED-DQO 4 will be addressed through a bench-scale TS.

Reconnaissance and probing activities will be completed to identity the current locations of sediment deposits and measure their dimensions to quantify the volume of sediment deposits in Snow Creek. Once reconnaissance and probing activities are completed, the deposits will be sampled to further define the chemical and physical characteristics of Snow Creek sediment to support the RD of the OU-1/OU-2 portion of Snow Creek. As part of the sediment TS, bench-scale testing will be conducted to evaluate methods for dewatering and stabilizing sediment excavated from Snow Creek and treatment technologies that may be necessary to meet the requirements for discharging the supernatant that drains from sediment excavated from Snow Creek.

Surface Water Sampling

The USEPA-selected remedy includes monitoring surface water at the upstream and downstream ends of OU-1/OU-2. Surface water sampling will be conducted as part of the PDI to establish a baseline for comparing post-remedy conditions. Since the ROD specified remedial goals for surface water are based on the USEPA's chronic aquatic life freshwater ambient water quality criteria (AWQC), the monitoring will be conducted during base flow conditions that are present 90% of the time.

The specific DQO for the surface water sampling as part of the OU-1/OU-2 PDI program is as follows:

- SW-DQO 1: Provide baseline surface water data that can be used for comparison with post-remedy surface water monitoring data.

OU-1/OU-2-Wide Evaluations

Data needs and data gaps were identified for floodplain and creek bank soil, groundwater, and Snow Creek sediment, as well as site-wide evaluations for utilities, structures, and topo-bathymetry that need to be addressed to complete the OU-1/OU-2 RD as defined in the ROD. Descriptions of these data needs and data gaps are provided below.

As part of the OU-1/OU-2 RD process, existing conditions of utilities, structures (e.g., bridges, roadways, outfalls, and culverts), surface elevations, and the presence of cultural and archeological resources need to be evaluated for inclusion on design drawings and for any preventative measures that need to be implemented to protect the integrity of the utilities and structures.

The PDI will be conducted to address the following data gaps:

- Information is needed regarding structures immediately adjacent to target remedial areas to confirm that the remediation work will not affect the integrity of the structures.
- Current project base mapping is needed to support the development of the RD and contract drawings.
- Updated topography and bathymetry data are needed to support the design for the soil and sediment remediation and for use in hydraulic modeling of potential flooding impacts associated with potential remediation within and near the creek and the Highway 202 culvert area.

The DQOs for these site wide evaluations (i.e., utility [US], structural [STR], and base mapping [BM]) activities as part of OU-1/OU-2 investigation are as follows:

- US-DQO 1: Identify utilities located within or adjacent to the proposed remedial areas and gather information for those features to design the remedial action and potential mitigative measures to prevent damaging them.
- STR-DQO 1: Identify structures, slopes, and utilities located within or adjacent to the proposed remedial areas and gather information for those features to design the remedial action and potential mitigative measures to prevent damaging them.
- BM-DQO 1: Update the project base mapping to support development of the remedial design and contract drawings.

- BM-DQO 2: Gather topography and bathymetry data to support the design for the sediment remediation and for use in hydraulic modeling of potential flooding impacts associated with potential remediation within and near the Highway 202 culverts.

The OU-1/OU-2-wide evaluations are common elements needed to complete the RDs for soil, groundwater, and sediment. The following tasks will be completed OU-1/OU-2-wide:

- A geophysical survey will be conducted to evaluate whether there are buried structures, pipes, utilities, or other features within the OU-1/OU-2 RD/RA areas.
- The Highway 202 and Quintard Mall culverts and other structures adjacent to OU-1/OU-2 RD/RA areas will be evaluated to assess whether special considerations will be required during the RA.
- Topographical surveys of OU-1/OU-2 will be conducted to create a survey-level base map for the RD.
- An assessment of cultural and archeological resources will be conducted to evaluate the presence of historical or archeological resources.

Proposed Analytical Methods to Characterize Contamination and Environmental Indicators

Samples will be analyzed by the analytical methods listed in the PDIWP. Further details regarding analysis are provided in Worksheets #12, #15, and #18.

UFP-QAPP Worksheet #11: Project/Data Quality Objectives

Data quality objectives (DQOs) are used to establish performance or acceptance criteria that will serve as the basis for collecting data of sufficient quality and quantity to support the goals of this study. The DQO process is a seven-step process; the seven steps are enumerated below. The DQOs included in this UFP-QAPP, the Predesign Investigation Work Plan (PDIWP), and Treatability Study Work Plans (TSWPs) included as Appendix B and C to the PDIWP, were developed to support the scopes of work listed in those work plans.

1. **State the problem**

The Site includes residential and nonresidential (i.e., commercial/industrial, and public) properties located in and around Anniston, Oxford, Hobson City, and areas of Calhoun and Talladega Counties in Alabama. The Site is divided into operable units (OUs). OU-1/OU-2 is a combination of residential and nonresidential properties around Solutia's Anniston Facility (the Facility) and downstream along Snow Creek to Highway 78. The Facility and its adjacent closed landfills (i.e., the South Landfill and West End Landfill) comprise OU-3. OU-4 includes Choccolocco Creek and its 100-year floodplain downstream of its confluence with Snow Creek and the portion of Snow Creek downstream of Highway 78 to its confluence with Choccolocco Creek.

The remedy the United States Environmental Protection Agency (USEPA) selected for OU-1/OU-2 includes multiple components for soil, groundwater, and sediment. The Record of Decision (ROD) identified eight categories or types of remedies based on the range of remedial alternatives presented in the OU-1/OU-2 Feasibility Study (Ramboll Environ 2016), and Pharmacia LLC and Solutia, Inc. (P/S) and USEPA agreed that P/S would implement the selected remedy for seven of these categories. The seven categories of remedial alternatives for OU-1/OU-2 identified in the CD and the selected remedy for each category are as follows:

- **Residential soils:** The selected remedy for residential soil is removal and on-site or off-site disposal of surface soil (i.e., soil from ground surface to 12 inches below ground surface [bgs]) with polychlorinated biphenyl (PCB) concentrations greater than or equal to 1 milligram per kilogram (mg/kg) but less than 10 mg/kg and removal and off-site disposal of surface and subsurface soil (i.e., soil deeper than 12 inches bgs) with PCB concentrations greater than or equal to 10 mg/kg. The selected remedy also includes long-term soil management for (1) properties with residual PCBs greater than or equal to 1 mg/kg, (2) properties with PCB concentrations in soil greater than or equal to 1 mg/kg that have been designated as "unsuitable for removal," and (3) properties that potentially have residual PCBs beneath a structure (i.e., a building, shed, or paved area) that limits exposure.
- **Special use properties:** For high-activity special use properties, the selected remedy includes removal and on-site or off-site disposal of surface soil with PCB concentrations greater than or equal to 1 mg/kg but less than 10 mg/kg and removal

and off-site disposal of subsurface soil with PCB concentrations greater than or equal to 10 mg/kg. For low-activity special use properties, the selected remedy includes removal and on-site or off-site disposal of surface soil with PCB concentrations greater than or equal to 1 mg/kg but less than 10 mg/kg and removal and off-site disposal of surface soil with PCB concentrations greater than or equal to 10 mg/kg and subsurface soil with PCB concentrations greater than or equal to 97 mg/kg. The selected remedy also includes long-term soil management for (1) properties where residual PCBs are at concentrations greater than or equal to 1 mg/kg, (2) properties with PCB concentrations in soil greater than or equal to 1 mg/kg that have been designated as "unsuitable for removal," and (3) properties that potentially have residual PCBs beneath a structure (i.e., a building, shed, or paved area) that limits exposure.

- **Interim measure (IM) areas:** The IM component of the OU-1/OU-2 remedy includes maintaining the existing IMs and excavating soil around the Northside Area, Eastside Area, and the Eastside Drainageway (including the drainage ditch to the north and PB RR-37 area) IMs to meet the surface soil remedial goal for PCBs of 21 mg/kg and subsurface soil remedial goal for PCBs of 97 mg/kg. Excavated soil will be disposed of off-site. The OU-1/OU-2 remedy also includes assessing the potential for principal threat waste (PTW) to be present in the portion of OU-1/OU-2 that is located just east of OU-3. The presence of PTW will be characterized by groundwater with PCBs at concentrations greater than the State maximum contaminant level and the Federal Safe Drinking Water Act maximum contaminant level. If PTW is found to be present, the specific approach to address the PTW material will be developed in coordination with USEPA during the RD.
- **Dredge spoil piles:** The remedy includes removal and off-site disposal of four dredge spoil piles located near or adjacent to Snow Creek. The CD includes predesign investigation (PDI) sampling for dredge spoil pile SC-2, which was not characterized during the remedial investigation (RI) to assess whether PCB concentrations are greater than or equal to the threshold concentration of 3 mg/kg for dredge spoil pile removal in OU-1/OU-2 under the selected remedy. The surface soil located directly beneath the dredge spoil piles will also be evaluated for conformance with the surface soil remedial goal of 21 mg/kg. The subsurface soil will be evaluated using the remedial goal of 97 mg/kg. Long-term soil management for areas underlying dredge spoil piles with residual PCBs greater than or equal to 1 mg/kg is also part of the remedy.
- **Nonresidential soils:** For nonresidential soil, the remedy includes removal and off-site disposal of surface soil with PCB concentrations greater than or equal to 21 mg/kg, removal and off-site disposal for subsurface soil with PCB concentrations greater than or equal to 97 mg/kg, and long-term soil management for residual soil with PCB concentrations greater than or equal to 1 mg/kg. The remedy also includes the removal and off-site disposal of nonresidential soil from several small areas where non-PCB constituents are present at concentrations greater than their respective remedial goals from the OU-1/OU-2 ROD.

- **Soil and groundwater at T-11:** For the T-11 area, the remedy includes removal and off-site disposal of soil from the upper 18-inch horizon across the broader T-11 area and the removal and off-site disposal of soil at depth directly surrounding groundwater well T-11; placement of a low-permeability cap over the broader T-11 area; the extraction, treatment, and discharge of groundwater from new groundwater wells installed in the T-11 area; and land use controls (e.g., deed restrictions or environmental covenant) that prohibit future groundwater use.
- **Sediment and creek bank soils:** For sediment and creek bank soil, the remedy includes stabilization of select creek bank areas and the removal and off-site disposal of sediment with PCB concentrations greater than or equal to 3 mg/kg. The remedy also includes the removal and off-site disposal of several sediment deposits containing non-PCB constituents at concentrations greater than their respective remedial goals from the OU-1/OU-2 ROD. The remedy for creek bank areas along Snow Creek includes stabilization of the specific reaches of the creek identified in the OU-1/OU-2 ROD.

As detailed in the PDIWP, additional investigation will be completed to support the remedial design.

2. Identify the Goals of the Study

The field investigation and treatability studies (TSs) detailed in the PDIWP will be conducted to support the remedial design. The PDI and TS activities are critical to developing a remedial design that is protective of human health and the environment over the long term and will receive regulatory approval. Study questions and DQOs for the PDI field investigations and TSs are included below.

The following study questions will be addressed through the PDI:

Principal Study Question	Alternative Actions	Decision Statement
What is the spatial extent of PCB concentrations in floodplain soil areas, including dredge spoil piles, low-activity special use areas, certain IM areas, and nonresidential areas in comparison to the OU-1/OU-2 ROD remedial goals?	Conduct excavation for soils above OU-1/OU-2 ROD remedial goals.	Evaluate whether soils are above the OU-1/OU-2 ROD remedial goals and complete excavation.
	No action.	
What are the geotechnical properties of soils within the T-11 area?	Complete excavation and low-permeability cap design for the T-11 area to support placement of groundwater treatment equipment.	Evaluate the potential for differential settlements and collect design parameters of low-permeability cover surfaces in the T-11 area to support placement of groundwater treatment equipment in this area.
	No action.	

Principal Study Question	Alternative Actions	Decision Statement
What are the geotechnical properties of creek banks along Snow Creek at and immediately downstream of its confluence with the 11th Street Ditch?	Complete design of creek bank stability measures.	Evaluate the appropriate design parameters for creek bank stabilization measures.
	No action.	
Do portions of the Hall Street IM area cover soils require augmentation to meet the IM design requirements?	Complete augmentation of cover soils to meet the IM design requirements.	Evaluate whether portions of the Hall Street IM cover soils require augmentation to meet the IM design requirements and complete augmentation.
	No action.	
Do PCB concentrations in groundwater at the Eastside Properties exceed OU-1/OU-2 ROD remedial goals?	Install new monitoring well for routine sampling and testing concurrently with the OU-3 annual groundwater monitoring program.	Evaluate if groundwater concentrations are in exceedance of OU-1/OU-2 ROD remedial goals and complete installation of new monitoring well in the Eastside Properties area.
	No further monitoring.	
What is the appropriate dosage and type of granular activated carbon to be used for treatment of PCBs in groundwater?	Complete design of treatment system using granular activated carbon.	Evaluate if GAC is an effective groundwater treatment remedy and collect design parameters for treatment of PCB-impacted groundwater.
	Complete additional treatability studies if granular activated carbon is not effective for groundwater treatment.	
What are the current locations, extents, and depths of sediment deposits within the OU-1/OU-2 portion of Snow Creek? If sediment deposit extents and depths have changed, what are the concentrations of site constituents within these sediment deposits?	Complete removal of sediment deposits above OU-1/OU-2 ROD remedial goals.	Evaluate extent of sediment deposits within Snow Creek that require removal.
	No action.	
What are applicable treatment technologies and design parameters for sediment dewatering and stabilization of excavated sediment from Snow Creek?	Complete design based on treatability study results.	Evaluate the appropriate design parameters for sediment dewatering and stabilization of excavated sediment and treatment of filtrate.
	Complete additional treatability studies if treatment technologies evaluated are not effective for sediment dewatering/stabilization or filtrate treatment.	

Specific DQOs for the OU-1/OU-2 PDI program are as follows for soil:

- SO-DQO 1: Refine the current understanding of PCBs for dredge spoil pile SC-2 to evaluate whether PCB concentrations exceed the remedial goal of 3 mg/kg. Once the dredge spoil piles identified for remediation are removed, surface soil and subsurface soil beneath the dredge spoil piles will be sampled and assessed for PCBs based on the ROD requirements of 21 mg/kg for surface soil and 97 mg/kg for subsurface soil.
- SO-DQO 2: Refine the current understanding of subsurface soil PCB concentrations in floodplain soil areas, including confirmed low-activity special use areas, certain IM areas, and the nonresidential areas, for comparison with the OU-1/OU-2 ROD remedial goal of 97 mg/kg.
- SO-DQO 3: Gather geotechnical characterization data from the T-11 area to support the detailed design of a low-permeability cap and creek bank stabilization measures.
- SO-DQO 4: Evaluate whether representative soil PCB concentrations at the PB-RR-37 area, including soil along the drainage ditch upstream and downstream of this area, are above the ROD requirements of 21 mg/kg for surface soil and 97 mg/kg for subsurface soils. For locations adjacent to Snow Creek, concentrations will be compared to the OU-1/OU-2 ROD remedial goal of 3 mg/kg, and evaluated relative to their ability to erode and migrate into Snow Creek. If PCB are detected in subsurface soil proximate to the groundwater table in the PB-RR-37, discussions will be held with USEPA relative to the potential installation of a groundwater monitoring well to assess if groundwater PCB concentrations in this area exceed the remedial goal of 0.5 µg/L.
- SO-DQO 5: Evaluate whether portions of the Hall Street IM cover soils require augmentation to meet the IM design requirements.

Specific DQOs for the OU-1/OU-2 PDI program are as follow for groundwater:

- GW-DQO 1: Evaluate whether representative groundwater PCB concentrations at the Eastside Properties (and potentially location PB-RR-37) exceed the remedial goal of 0.5 µg/L.
- GW-DQO 2: Gather hydrogeologic data at the T-11 area to support the detailed design of a groundwater extraction-and-treatment system.
- GW-DQO 3: Evaluate potentially efficient and cost-effective GAC media for PCB treatment.
- GW-DQO 4: Estimate the time for PCB breakthrough in a full-scale GAC system.

GW-DQOs 1 and 2 will be addressed through field investigations. GW-DQO 3 and GW-DQO 4 will be addressed through a bench-scale TS.

Specific DQOs for the OU-1/OU-2 PDI program are as follows for sediment:

- SED-DQO 1: Provide an updated delineation of the current locations, extents, and depths of sediment deposits within the OU-1/OU-2 portion of Snow Creek as compared to the deposits depicted in the OU-1/OU-2 FS to refine removal volume estimates.
- SED-DQO 2: Refine the current understanding of sediment in the OU-1/OU-2 portion of Snow Creek that exceed the remedial criteria listed in the ROD to define the lateral and vertical limits of sediment removal.
- SED-DQO 3: Evaluate potentially efficient and cost-effective methods for dewatering and stabilizing sediment excavated from Snow Creek.
- SED-DQO 4: Evaluate treatment technologies that may be necessary to meet the requirements for discharging the supernatant that drains from sediment excavated from the creek.

SED-DQOs 1 and 2 will be addressed through field investigations. SED-DQO 3 and SED-DQO 4 will be addressed through a bench-scale TS.

TSs are being conducted under the PDIWP to support the development of the preliminary (30%) remedial design. The TSs are being conducted for groundwater and sediment and are summarized in Sections 4.1 and 4.2 of the PDIWP. Supporting details are provided for the groundwater and sediment in stand-alone TS work plans that are provided in Appendices B and C of the PDIWP.

The specific DQO for the surface water sampling as part of the OU-1/OU-2 PDI program is as follows:

- SW-DQO 1: Provide baseline surface water data that can be used for comparison with post-remedy surface water monitoring data.

In addition to sampling environmental media, the PDI includes utility (US), structural (STR), and base mapping (BM) activities as part of OU-1/OU-2 investigation. Specific DQOs for these evaluations are as follows:

- US-DQO 1: Identify utilities located within or adjacent to the proposed remedial areas and gather information for those features to design the remedial action and potential mitigative measures to prevent damaging them.

- STR-DQO 1: Identify structures, slopes, and utilities located within or adjacent to the proposed remedial areas and gather information for those features to design the remedial action and potential mitigative measures to prevent damaging them.

- BM-DQO 1: Update the project base mapping to support development of the remedial design and contract drawings.

- BM-DQO 2: Gather topography and bathymetry data to support the design for the sediment remediation and for use in hydraulic modeling of potential flooding impacts associated with potential remediation within and near the Highway 202 culverts.

3. **Identify Information Inputs**

Sampling locations, intervals, matrices, and analytical methods to achieve the objectives of the PDIWP are presented in Worksheet #18 and the PDIWP. Specifically, for each media, the inputs to address the goal are as follows:

- *Soil*: Soil samples will be collected for chemical and geotechnical characterization to support the design of the remedy for OU-1/OU-2. Composite and ISM soil sampling methods will be used. See Worksheets #17 and #18 for additional detail.
- *Groundwater*: Groundwater samples will be collected for chemical characterization, completion of a TS, and evaluation of hydrogeologic properties of the groundwater-bearing unit to support the design of a treatment system for OU-1/OU-2. See Worksheets #17 and #18 for additional details.
- *Sediment*: Sediment samples will be collected for chemical and physical characterization and completion of a TS to support the design of the remedy for OU-1/OU-2. See Worksheets #17 and #18 for additional details.
- *Surface Water*: Surface water samples will be collected for chemical characterization to establish a baseline for comparison of post-remedy monitoring. See Worksheets #17 and #18 for additional details.

In addition to collection of environmental samples, utility, structures, and topo-bathymetric data will be collected to support the design for soil and sediment removal activities.

4. Define the Boundaries of the Study

Boundaries of the Study and Potential Constraints: The approximate spatial boundaries of the PDI study area for the low-activity special use areas are shown on Figures 2-1a through 2-1l of the PDIWP. Spatial boundaries for PDI efforts to address the dredge spoil piles are shown on Figures 2-5a and 2-5b. Spatial boundaries for PDI activities to address IM areas including soil and groundwater are shown on Figures 2-3a through 2-3e and Figures 2-14a through 2-14c of the PDIWP. The spatial boundaries for PDIs to address nonresidential soil and groundwater at the T-11 area are shown on Figure 2-12 of the PDIWP. Spatial boundaries to address Snow Creek sediment are shown on Figure 2-8 of the PDIWP and include the upstream limit of Snow Creek beginning at the northeastern end of EU5 and flowing south to the downstream end of EU28. The geographic limits of the creek bank PDIs are shown on Figures 2-5a and 2-5b of the PDIWP. The PDI field work is expected to be conducted as described in the PDIWP and Worksheet #14/16 during 2022.

Target Population of Interest: The target populations of interest consist of the soil, groundwater, and sediment from within OU-1/OU-2 as shown in the PDIWP figures identified above.

5. **Develop the Analytical Approach**

The PDI is intended to collect data that support the goals and questions of the study (listed above). Data will be collected and compared against regulatory criteria listed in Worksheet #15. Data will be presented in a PDI Evaluation Report, and additional sampling may be planned for collection to achieve the goals listed above. Data will be collected from soil, groundwater, sediment, and surface water and will be evaluated using the following analytical approaches:

- *Soil samples*: Discrete and ISM soil samples will be collected and analyzed for the parameters listed in the PDIWP and Worksheet #15. Results will be used to 1) develop an understanding of contaminant distribution laterally and vertically in areas of investigation prior to implementing the removal activities in floodplain soil areas, including confirmed low-activity special use areas, certain IM areas, and the nonresidential areas, 2) evaluate the potential for PCBs to leach from soil to groundwater at the Eastside Properties and the PB-RR-37 area, and 3) design a low-permeability cap and groundwater treatment area within the T-11 area. Soil sampling will consist of the following:
    - A single five-point composite soil sample will be collected from the dredge spoil pile SC-2 from the 0–1-foot horizon.
    - Five-point composite subsurface soil samples will be collected in confirmed low-activity special use areas from the same locations that were used for surface soil sampling under the Stipulation where surface soil concentrations were found to be greater than or equal to 1 mg/kg. Composite samples will be collected from three soil depth intervals of 1 to 2 feet bgs, 2 to 3 feet bgs, and 3 to 4 feet bgs. Composite samples collected from the soil depths of 2 to 3 feet bgs and 3 to 4 feet bgs will be archived at the analytical laboratory pending analysis of the composite sample collected from 1 to 2 feet bgs. The subsequent depth interval sample will be analyzed if PCB concentrations detected in the analyzed interval are above the PCB remedial goal for subsurface soils (97 mg/kg). For example, if PCB concentrations in the 1-to-2-foot-bgs composite sample interval are detected above the remedial goal for subsurface soils (97 mg/kg), the composite sample collected from the 2–3-foot depth interval will be analyzed.
    - Five-point composite subsurface soil samples will be collected in IM areas and nonresidential areas where the size of the contiguous area to be sampled is equal to or less than 0.5 acres. All of the IM areas evaluated under this PDI are less than 0.5 acres and will be sampled by collecting one 5-point composite per 0.25-acre area. Composite samples will be collected as described above from three soil depth intervals of 1 to 2 feet bgs, 2 to 3 feet bgs, and 3 to 4 feet bgs. Composite samples collected from the soil depths of 2 to 3 feet bgs and 3 to 4 feet bgs will be archived at the analytical laboratory pending analysis of the composite sample collected from 1 to 2 feet bgs. The subsequent depth interval sample will be analyzed if PCB concentrations detected in the analyzed interval are above the PCB remedial goal for subsurface soils (97 mg/kg).

- A five-point composite soil samples from the drainage ditch upstream and downstream of PB-RR-37. Composite samples will be collected as described above from four sample depths: 0 to 1 foot bgs, 1 to 2 feet bgs, 2 to 3 feet bgs, and 3 to 4 feet bgs. Additionally, during collection of the 5-point composite samples, individual split samples (aliquots) for the two locations closest to Snow Creek will be collected from 0 to 1 foot bgs and analyzed for comparison to 3 mg/kg. The composite PCB result for samples collected from this ditch will be compared to the nonresidential remedial goal of 21 mg/kg. Samples collected from the soil depths of 1–2 feet and below will be archived at the analytical laboratory pending analysis of samples collected from 0–1 feet bgs.
- The subsurface soil in nonresidential areas that are larger than 0.5 acres will be sampled using ISM in accordance with the Interstate Technology Regulatory Council (ITRC) Technical Guidance: Incremental Sampling Methodology Update (ISM-2) guidance document (ITRC 2020) and Attachment A-12 (Incremental Sampling Methodology Procedures). ISM is a probabilistic sampling method used to evaluate the representative concentration of a constituent in an area. It is designed to provide an unbiased and statistically valid estimate of the mean concentration of analyzed constituents within each sample area of the IM areas. ISM was developed to decrease the effect of soil sample variability due to heterogeneity and provides a reasonably unbiased estimate of mean concentration in a defined soil volume, referred to as a decision unit (DU) (ITRC 2020). Each IM and nonresidential area will be sampled as three DUs based on sample depth: 1 to 2 feet bgs, 2 to 3 feet bgs, and 3 to 4 feet bgs.
- Surface and subsurface soil samples will be collected from depth intervals from a discrete location proximate to PB-RR-37. Samples will be collected at 1-foot depth intervals from ground surface until the soil–groundwater interface is reached based on field observations (approximately 15 to 25 feet bgs). The last depth interval will be located immediately above the observed groundwater level.
- Four geotechnical borings will be advanced at locations within the T-11 area where the RA excavation and bank stabilization will be completed.

- *Groundwater samples*: Groundwater samples will be analyzed for the parameters listed in the PDIWP and Worksheet #15. Analytical results will be compared to applicable remedial goals specified in the ROD. These data will be used to assess the impact of PCB leaching from soil to groundwater in the Eastside Properties area and PB-RR-37 Area. Additionally as part of the groundwater TS, bench-scale testing will be conducted to evaluate groundwater treatment using granular activated carbon filtration. Groundwater sampling will consist of the following activities:
  - Two new groundwater monitoring wells will be installed in the Eastside Properties area for sampling.
  - Groundwater samples will be collected from well T-11

- *Sediment samples*: Sediment probing will be completed to identify the current locations of sediment deposits and measure the deposits' dimensions to quantify the volume of sediment in Snow Creek. New sediment deposits or sediment deposits in a different location or size than identified and characterized in the Off-Site Resource Conservation and Recovery Act (RCRA) Facility Investigation (RFI) Program evaluation (Blasland, Bouck & Lee, Inc. [BBL] 1999) will be sampled for parameters listed in the PDIWP and Worksheet #15. Results will supplement existing sediment data, support the delineation of sediment that has PCBs and other constituent concentrations above the applicable remedial goals specified in the ROD, and establish waste disposal requirements. As part of the sediment TS, bench-scale testing will be conducted to evaluate methods for dewatering and stabilizing sediment excavated from Snow Creek and treatment technologies that may be necessary to meet the requirements for discharging the supernatant that drains from sediment excavated from Snow Creek.
- *Surface water samples*: Surface water samples will be collected for the parameters listed in the PDIWP and Worksheet #15. Results will be used to establish a baseline data set that can be used for comparison with post-remedy surface water monitoring data. Surface water will be collected from two locations (i.e., the upstream and downstream ends of Snow Creek in OU-1/OU-2).

6. **Specify Performance Criteria**

Data collected as part of the PDI will be assessed as inputs to the remedial design. Following data validation, the data will be entered into the project database. Data will be summarized on figures and tables. Comparisons between and among sample locations may be presented to assess distributions of the data based on available data sets.

The measurement performance criteria for data associated with the target population and specific analyses include considerations for precision, accuracy, representativeness, completeness, comparability, and sensitivity (PARCCS). To meet PARCCS requirements, quality control (QC) criteria are provided in the standard field and laboratory methods. These criteria include the following:

- Field duplicates, laboratory duplicates, and matrix spike/matrix spike duplicate (MS/MSD) samples to assess precision
- MS, laboratory control samples (LCS), calibration results, and field and method blanks to assess accuracy and bias
- Field sampling design and sample collection standard operating procedures (SOPs) to measure representativeness
- Standard methods and the consistent use of field and laboratory SOPs to achieve comparability and method detection limit (MDL) studies, calibration, and method blanks to determine and assess sensitivity

Consistent sampling protocols, applicable documentation, sample handling procedures, and measurement system procedures will be used during field and laboratory activities associated with the PDIWP. Worksheet #12 provides the measurement performance criteria associated with the field and laboratory QC parameters and samples associated with the criteria. Data that are generated from the sample handling system described in this UFP-QAPP and whose associated QC results meet the criteria described in the applicable method will be considered usable for supporting the goals of the PDIWP.

7. **Develop the Plan for Obtaining Data**

The PDIWP is based on the requirements of the OU-1/OU-2 *Remedial Design/Remedial Action Consent Decree* (USEPA 2021). The basis of the sampling design and rationale for the PDI and TS tasks are presented in the PDIWP, along with the sampling locations for each matrix and associated analytical suites. This includes the specific PDIWP figures identified above, Table 3-1, Section 3 of the PDIWP, and Worksheet #18 of this UFP-QAPP.

UFP-QAPP Worksheet #12a: Measurement Performance Criteria for Water Matrices with Volatiles

Matrix	Aqueous (toxicity characteristic leaching procedure [TCLP] leachate, aqueous water quality control [QC] samples, equipment/source/trip blanks)
Analytical Group	Volatile Organic Compounds (United States Environmental Protection Agency [USEPA] SW-846 Method 8260C)
Anticipated Concentration Level	Low to Moderate

Analytical Method/Standard Operating Procedure (SOP) Reference	Data Quality Indicators (DQIs)[1]	Measurement Performance Criteria	QC Sample and/or Activity Used to Assess Measurement Performance	QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S&A)
USEPA SW-846 Method 8260C/SA-VO-004	Precision; Comparability	Relative percent difference ≤ 35 percent (%) when detections for both primary and duplicate samples are ≥ Limit of Quantitation (LOQ*)	Field duplicates	S
	Accuracy/Bias	Statistically derived control limits (CLs) in accordance with SOPs in Attachment B	Surrogate spikes, laboratory control samples, matrix spikes	A
	Accuracy/Bias	No target compounds > LOQ	Method blank, instrument blank, trip blank	S&A
	Representativeness/ Completeness	Samples collected as described in Predesign Investigation Work Plan (PDIWP) and sampling SOPs	Field measurements	S
	Sensitivity	Statistically derived CLs in accordance with SOPs in Attachment B	Laboratory fortified blank at LOQ	A
	Completeness	Samples collected and analyzed as described in PDIWP; Data completeness > 90%	Data completeness check	S&A

[1] Data quality indicators are defined in Worksheet #37.
*LOQ is considered comparable with the reporting limit.

UFP-QAPP Worksheet #12b: Measurement Performance Criteria for Water Matrices with Semivolatiles

Matrix	Aqueous (toxicity characteristic leaching procedure [TCLP] leachate, aqueous water quality control [QC] samples, equipment/source blanks)
Analytical Group	Semivolatile Organic Compounds (United States Environmental Protection Agency [USEPA] SW-846 Method 8270D)
Anticipated Concentration Level	Low to Moderate

Analytical Method/Standard Operating Procedure (SOP) Reference	Data Quality Indicators (DQIs)[1]	Measurement Performance Criteria	QC Sample and/or Activity Used to Assess Measurement Performance	QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S&A)
USEPA SW-846 Method 8270D/SA-SM-003	Precision; Comparability	Relative percent difference ≤ 35% when detections for both primary and duplicate samples are ≥ Limit of Quantitation (LOQ)	Field duplicates	S
	Accuracy/Bias	Statistically derived control limits (CLs) in accordance with SOPs in Attachment B	Surrogate spikes, laboratory control samples, matrix spikes	A
	Accuracy/Bias	No target compounds > LOQ	Method blank, instrument blank	A
	Representativeness/ Completeness	Samples collected as described in the Predesign Investigation Work Plan (PDIWP) and sampling SOPs	Field measurements	S
	Sensitivity	Statistically derived CLs in accordance with SOPs in Attachment B	Laboratory fortified blank at LOQ	A
	Completeness	Samples collected and analyzed as described in the PDIWP; Data completeness > 90%	Data completeness check	S&A

[1] Data quality indicators are defined in Worksheet #37.

UFP-QAPP Worksheet #12c: Measurement Performance Criteria for Water Matrices with Polychlorinated Biphenyl Aroclors, Homologs, and Congeners

Matrix	Aqueous (groundwater, porewater performance reference compounds, sediment filtrate, toxicity characteristic leaching procedure [TCLP] leachate quality control [QC] samples, equipment/source blanks)
Analytical Group	Polychlorinated Biphenyls (United States Environmental Protection Agency [USEPA] SW-846 Methods 8082A and 680)
Anticipated Concentration Level	Low to High

Analytical Method/Standard Operating Procedure (SOP) Reference	Data Quality Indicators (DQIs)[1]	Measurement Performance Criteria	QC Sample and/or Activity Used to Assess Measurement Performance	QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S&A)
USEPA SW-846 Method 8082A/BR-GC-005 or PT-GC-005 and Method 680/BR-MS-010	Precision; Comparability	Relative percent difference ≤ 35% when detections for both primary and duplicate samples are ≥ Limit of Quantitation (LOQ)	Field duplicates	S
	Accuracy/Bias	Statistically derived control limits (CLs) in accordance with SOPs in Attachment B	Surrogate spikes, laboratory control samples, matrix spikes	A
	Accuracy/Bias	No target compounds > LOQ	Method blank, instrument blank	A
	Representativeness/ Completeness	Samples collected as described in the Predesign Investigation Work Plan (PDIWP) and sampling SOPs	Field measurements	S
	Sensitivity	Statistically derived CLs in accordance with SOPs in Attachment B	Laboratory fortified blank at LOQ	A
	Completeness	Samples collected and analyzed as described in the PDIWP; Data completeness > 90%	Data completeness check	S&A

[1] Data quality indicators are defined in Worksheet #37.

UFP-QAPP Worksheet #12d: Measurement Performance Criteria for Solid Matrices with Polychlorinated Biphenyl Aroclors and Homologs

Matrix	Solids (soil and sediment)
Analytical Group	Polychlorinated Biphenyls (United States Environmental Protection Agency [USEPA] SW-846 Methods 8082A and 680)
Anticipated Concentration Level	Low to High

Analytical Method/Standard Operating Procedure (SOP) Reference	Data Quality Indicators (DQIs)[1]	Measurement Performance Criteria	Quality Control (QC) Sample and/or Activity Used to Assess Measurement Performance	QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S&A)
USEPA SW-846 Method 8082A/BR-GC-005 or PT-GC-005 and Method 680/BR-MS-010	Precision; Comparability	Relative percent difference ≤ 50% when detections for both primary and duplicate samples are ≥ Limit of Quantitation (LOQ)	Field duplicates	S
	Accuracy/Bias	Statistically derived control limits (CLs) in accordance with SOPs in Attachment B	Surrogate spikes, laboratory control samples, matrix spikes	A
	Accuracy/Bias	No target compounds > LOQ	Method blank, instrument blank	A
	Representativeness/ Completeness	Samples collected from distinct intervals as described in the predesign investigation work plan (PDIWP) and sampling SOPs	Field measurements	S
	Sensitivity	Statistically derived CLs in accordance with SOPs in Attachment B	Laboratory fortified blank at LOQ	A
	Completeness	Samples collected and analyzed as described in the PDIWP; Data completeness > 90%	Data completeness check	S&A

[1] Data quality indicators are defined in Worksheet #37.

UFP-QAPP Worksheet #12e: Measurement Performance Criteria for Water Matrices with Metals/Cations

Matrix	Aqueous (groundwater, sediment filtrate, toxicity characteristic leaching procedure leachate, quality control [QC] samples, equipment/source blanks)
Analytical Group	Metals/Cations (United States Environmental Protection Agency [USEPA] SW-846 Method 6010C)
Anticipated Concentration Level	Low to Moderate

Analytical Method/Standard Operating Procedure (SOP) Reference	Data Quality Indicators (DQIs)[1]	Measurement Performance Criteria	QC Sample and/or Activity Used to Assess Measurement Performance	QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S&A)
USEPA SW-846 Method 6010D/SA-ME-070	Precision; Comparability	Relative percent difference ≤ 35% when detections for both primary and duplicate samples are ≥ Limit of Quantitation (LOQ)	Field duplicates	S
	Accuracy/Bias	Statistically derived control limits (CLs) in accordance with SOPs in Attachment B	Laboratory control samples, matrix spikes	A
	Accuracy/Bias	No target compounds > LOQ	Method blank, instrument blank	A
	Representativeness/ Completeness	Samples as described in the Predesign Investigation Work Plan (PDIWP) and sampling SOPs	Field measurements	S
	Sensitivity	Statistically derived CLs in accordance with SOPs in Attachment B	Laboratory fortified blank at LOQ	A
	Completeness	Samples collected and analyzed as described in the PDIWP; Data completeness > 90%	Data completeness check	S&A

[1] Data quality indicators are defined in Worksheet #37.

UFP-QAPP Worksheet #12f: Measurement Performance Criteria for Solid Matrices with Metals

Matrix	Solids (soils and sediment)
Analytical Group	Metals/Cations (United States Environmental Protection Agency [USEPA] SW-846 Method 6010C)
Anticipated Concentration Level	Low to Moderate

Analytical Method/Standard Operating Procedure (SOP) Reference	Data Quality Indicators (DQIs)[1]	Measurement Performance Criteria	Quality Control (QC) Sample and/or Activity Used to Assess Measurement Performance	QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S&A)
USEPA SW-846 Method 6010D/SA-ME-070	Precision; Comparability	Relative percent difference ≤ 50% when detections for both primary and duplicate samples are ≥ Limit of Quantitation (LOQ)	Field duplicates	S
	Accuracy/Bias	Statistically derived control limits (CLs) in accordance with SOPs in Attachment B	Laboratory control samples, matrix spikes	A
	Accuracy/Bias	No target compounds > LOQ	Method blank, instrument blank	A
	Representativeness/ Completeness	Samples as described in the Predesign Investigation Work Plan (PDIWP) and sampling SOPs	Field measurements	S
	Sensitivity	Statistically derived CLs in accordance with SOPs in Attachment B	Laboratory fortified blank at LOQ	A
	Completeness	Samples collected and analyzed as described in the PDIWP; Data completeness > 90%	Data completeness check	S&A

[1] Data quality indicators are defined in Worksheet #37.

UFP-QAPP Worksheet #12g: Measurement Performance Criteria for Water Matrices with Mercury

Matrix	Aqueous (sediment filtrate, toxicity characteristic leaching procedure leachate. quality control [QC] samples, equipment/ source blanks)
Analytical Group	Mercury (United States Environmental Protection Agency [USEPA] Method 7470A)
Anticipated Concentration Level	Low to Moderate

Analytical Method/Standard Operating Procedure (SOP) Reference	Data Quality Indicators (DQIs)[1]	Measurement Performance Criteria	QC Sample and/or Activity Used to Assess Measurement Performance	QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S&A)
USEPA SW-846 Method 7470A/ PT-MT-005	Precision; Comparability	Relative percent difference ≤ 35% when detections for both primary and duplicate samples are ≥ Limit of Quantitation (LOQ)	Field Duplicates	S
	Accuracy/Bias	Statistically derived control limits (CLs) in accordance with SOPs in Attachment B	Laboratory control samples, matrix spikes	A
	Accuracy/Bias	No target compounds> LOQ	Method blank, instrument blank	A
	Representativeness/ Completeness	Samples collected as described in the Predesign Investigation Work Plan (PDIWP) and sampling SOPs	Field measurements	S
	Sensitivity	Statistically derived CLs in accordance with SOPs in Attachment B	Laboratory fortified blank at LOQ	A
	Completeness	Samples collected and analyzed as described in the PDIWP; Data completeness > 90%	Data completeness check	S&A

[1] Data quality indicators are defined in Worksheet #37.

UFP-QAPP Worksheet #12h: Measurement Performance Criteria for Solid Matrices with Mercury

Matrix	Solids (soil and sediment)
Analytical Group	Mercury (United States Environmental Protection Agency [USEPA] Method 7471B)
Anticipated Concentration Level	Low to Moderate

Analytical Method/Standard Operating Procedure (SOP) Reference	Data Quality Indicators (DQIs)[1]	Measurement Performance Criteria	Quality Control (QC) Sample and/or Activity Used to Assess Measurement Performance	QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S&A)
USEPA Method 7471B/ SA-ME-028	Precision; Comparability	Relative percent difference ≤ 50 percent (%) when detections for both primary and duplicate samples are ≥ Limit of Quantitation (LOQ)	Field duplicates	S
	Accuracy/Bias	Statistically derived control limits (CLs) in accordance with SOPs in Attachment B	Laboratory control samples, matrix spikes	A
	Accuracy/Bias	No target compounds > LOQ	Method blank, instrument blank	A
	Representativeness/ Completeness	Soil samples collected from distinct intervals as described in the Predesign Investigation Work Plan (PDIWP) and sampling SOPs	Field measurements	S
	Sensitivity	Statistically derived CLs in accordance with SOPs in Attachment B	Laboratory fortified blank at LOQ	A
	Completeness	Samples collected and analyzed as described in the PDIWP; Data completeness > 90%	Data completeness check	S&A

[1] Data quality indicators are defined in Worksheet #37.

UFP-QAPP Worksheet #12i: Measurement Performance Criteria for Soil/Water Matrices for General Chemistry Parameters

Matrix	Aqueous (groundwater, sediment filtrate, aqueous water quality control [QC] samples, equipment/source blanks)/Soil (soil and sediment)
Analytical Group	General Chemistry Parameters (Alkalinity, Anions, Hardness, Total Dissolved Solids, Total Suspended Solids. Total Dissolved Solids, Total Organic Carbon, and Dissolved Organic Carbon)
Anticipated Concentration Level	Low to Moderate

Analytical Methods/Standard Operating Procedure (SOP) References	Data Quality Indicators (DQIs)[1]	Measurement Performance Criteria	QC Sample and/or Activity Used to Assess Measurement Performance	QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S&A)
United States Environmental Protection Agency (USEPA) Methods 300.0/SA-GE-115, 9060A/SA-GE-204 and Standard Methods 2320B/SA-GW-193, 2340B/SA-GE-201, 2540C/PT-WC-043, 2540D/PT-WC-045; Lloyd Khan/BR-WC-008	Precision; Comparability	Relative percent difference (RPD) ≤ 35% for water matrices and RPD ≤50% for solid matrices when detections for both primary and duplicate samples are ≥ Limit of Quantitation (LOQ)	Field duplicates	S
	Accuracy/Bias	Statistically derived control limits (CLs) in accordance with SOPs in Attachment B	Laboratory control samples, matrix spikes	A
	Accuracy/Bias	No target compounds > LOQ	Method blank, instrument blank	A
	Representativeness; Completeness	Samples collected and analyzed as described in the Predesign Investigation Work Plan; Data completeness > 90%	Field measurements; data completeness check	S&A

[1] Data quality indicators are defined in Worksheet #37.

UFP-QAPP Worksheet #12j: Measurement Performance Criteria for Water Matrices with Hexavalent Chromium

Matrix	Aqueous (surface water, quality control [QC] samples, equipment/source blanks)
Analytical Group	Hexavalent chromium (United States Environmental Protection Agency [USEPA] SW-846 Method 7196A)
Anticipated Concentration Level	Low to Moderate

Analytical Method/Standard Operating Procedure (SOP) Reference	Data Quality Indicators (DQIs)[1]	Measurement Performance Criteria	QC Sample and/or Activity Used to Assess Measurement Performance	QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S&A)
USEPA SW-846 Method 7196A/SA-GE-001	Accuracy/Bias	Statistically derived control limits (CLs) in accordance with SOPs in Attachment B	Laboratory control samples, matrix spikes	A
	Accuracy/Bias	No target compounds > LOQ	Method blank, instrument blank	A
	Representativeness/ Completeness	Samples as described in the Predesign Investigation Work Plan (PDIWP) and sampling SOPs	Field measurements	S
	Sensitivity	Statistically derived CLs in accordance with SOPs in Attachment B	Laboratory fortified blank at LOQ	A
	Completeness	Samples collected and analyzed as described in the PDIWP; Data completeness > 90%	Data completeness check	S&A

[1] Data quality indicators are defined in Worksheet #37.

UFP-QAPP Worksheet #13: Secondary Data Criteria and Limitations

Secondary Data	Data Source (Originating Organization, Report Title, and Date)	Data Generator(s) (Originating Org., Data Types, Data Generation/ Collection Dates)	How Data Will Be Used	Limitations on Data Use
Historical soil, groundwater, and sediment data	Blasland, Bouck & Lee, Inc. (BBL), Off-Site Resource Conservation and Recovery Act (RCRA) Facility Investigation (RFI) Program Report, 2000	BBL, environmental media, 1999	Provide information for decision-making for subsequent Site characterization activities.	No known limitations
	BBL, Nonresidential Properties Field Sampling Plan (FSP), 2004 and associated addenda (BBL 2005 and 2006)	BBL, environmental media, 2004 to 2006	Provide information for decision-making for subsequent Site characterization activities.	No known limitations
	Arcadis BBL, Preliminary Site Characterization Summary Report for OU-1/OU-2, 2007	BBL, environmental media, 2007	Provide information for decision-making for subsequent Site characterization activities.	No known limitations
	Golder, Operable Unit 1/Operable Unit 2 Third Non-Residential Groundwater Investigation for the Anniston PCB Site, 2012	Golder, groundwater, 2012	Provide information for decision-making for subsequent Site characterization activities.	No known limitations
Historical soil, groundwater, and sediment data	Golder, NTC Removal Action Completion Report for Residential Properties in OU-1/OU-2, 2015	Golder, environmental media. 2015	Provide information for decision-making for subsequent Site characterization activities.	No known limitations

Secondary Data	Data Source (Originating Organization, Report Title, and Date)	Data Generator(s) (Originating Org., Data Types, Data Generation/ Collection Dates)	How Data Will Be Used	Limitations on Data Use
Historical soil, groundwater, and sediment data (cont.)	United States Environmental Protection Agency (USEPA) electronic data transfer, 2000	USEPA, sediment samples, 2000	Provide information for decision-making for subsequent Site characterization activities.	No known limitations
	CDM, Anniston PCB Site Operable Units 1 and 2 Baseline Risk Assessment, 2009	CDM, environmental media, 2009	Provide information for decision-making for subsequent Site characterization activities.	No known limitations
	USEPA, Remedial Design/Remedial Action Consent Decree for Portions of the Record of Decision for Operable Unit 1/Operable Unit 2, 2021	USEPA	Provide information for decision-making for subsequent Site characterization activities needed to satisfy requirements of Consent Decree.	No known limitations
	USEPA, Record of Decision for Operable Unit 1/Operable Unit 2, Anniston PCB Site, 2017	USEPA	Provide information for decision-making for subsequent Site characterization activities needed to satisfy requirements of ROD.	No known limitations
	Pharmacia LLC/Solutia Inc. (P/S), Supplemental Work Plan for the Off-Site Portion of the RFI/CS Program, 1999	P/S, sediment, surface water, fish tissue, 1999	Provide information for decision-making for subsequent Site characterization activities.	No known limitations

Secondary Data	Data Source (Originating Organization, Report Title, and Date)	Data Generator(s) (Originating Org., Data Types, Data Generation/ Collection Dates)	How Data Will Be Used	Limitations on Data Use
Historical soil, groundwater, and sediment data (cont.)	P/S, RFI/CS Report, 2002	P/S, groundwater, surface water, soil, 1998	Provide information for decision-making for subsequent Site characterization activities.	No known limitations
	P/S, Remedial Investigation Report for OU-1/OU-2, 2015	P/S, environmental media, 2010–2015	Provide information for decision-making for subsequent Site characterization activities.	No known limitations
	P/S. Feasibility Study for OU-1/OU-2, 2016	P/S. environmental media,	Provide information for decision-making for subsequent Site characterization activities needed to satisfy requirements to complete design activities.	No known limitations

UFP-QAPP Worksheets #14 and #16: Project Tasks & Schedule

Preliminary Tasks

Before subsurface drilling activities begin, utilities will be located as described in the Predesign Investigation Work Plan (PDIWP).

Sampling Tasks

Sampling tasks are described in the PDIWP and Worksheet #17.

Analysis Tasks

Soil, groundwater, and sediment samples will be collected and documented as described in the PDIWP. Samples will be sent to analytical laboratories for analysis in accordance with Worksheets #18, #19, and #30.

Soil and soil leachates will be analyzed using the following methods depending on scope of work to satisfy data quality objectives (DQOs):

- Polychlorinated biphenyl (PCB) Aroclors (United States Environmental Protection Agency [USEPA] SW-846 8082A)
- PCB homologs (USEPA Method 680)
- Moisture content (ASTM International [ASTM] D2216)
- Particle size (ASTM D6913/D7928)
- Atterberg (Liquid and Plastic) limits (ASTM D4318)
- Unconsolidated undrained (UU) triaxial (ASTM D2850)
- Consolidated undrained (CU) triaxial (ASTM D4767)
- Toxicity characteristic leaching procedure (TCLP) metals (USEPA SW-846 6010C/7471B/7470A), volatile organic compounds (VOCs; USEPA SW-846 8260C), and semivolatile organic compounds (SVOCs; USEPA SW-846 8270D)
- Corrosivity (USEPA SW-846 9045D)
- Ignitability (USEPA Chapter 7, Section 7.1)
- Reactivity – cyanide (USEPA SW-846 Method 9010C) and sulfide (USEPA SW-846 9030B/9034)

Sediment and sediment filtrates from the sediment treatability study will be analyzed using the following methods depending on scope of work to satisfy DQOs:

- PCBs Aroclors (USEPA SW-846 8082A)

- Metals (barium, chromium [total], cobalt, lead, manganese, mercury [total], nickel, and vanadium only) (USEPA SW-846 6010C/7471B)
- Total organic carbon (TOC; Lloyd Kahn)
- Specific gravity (ASTM D854)
- Moisture content (ASTM D2216)
- Particle size (ASTM D6913/D7928)
- Atterberg (Liquid and Plastic) limits (ASTM D4318)
- Dry density (ASTM D2937)
- Organic content (ASTM D2974)
- Unconfined compression strength (ASTM D2166)
- Paint filter test (USEPA Method 9095B)
- Total suspended solids (SM2540D)
- Oil and Grease (USEPA Method 1664B)

Groundwater will be analyzed using the following methods:

- PCBs Aroclors (USEPA SW-846 8082A)
- PCB homologs (USEPA Method 680)
- Total and dissolved organic carbon (USEPA 9060A)
- Total and dissolved cations (calcium, magnesium, sodium, potassium, manganese, iron; USEPA Method 6010C)
- Total and dissolved anions (sulfate, chloride, nitrate; USEPA Method 300.0)
- Alkalinity (SM2320B)
- Hardness by calculation (USEPA Method 2340B)
- Total suspended solids (SM2540D)
- Total dissolved solids (SM2540C)
- TCLP metals (USEPA SW-846 6010C/7470A), VOCs (USEPA SW-846 8260C), and SVOCs (USEPA SW-846 8270D)
- Corrosivity (USEPA SW-846 9045D)
- Ignitability (USEPA Chapter 7, Section 7.1)
- Reactivity – cyanide (USEPA SW-846 Method 9010C) and sulfide (USEPA SW-846 9030B/9034)

Surface water will be analyzed using the following methods:

- PCB homologs (USEPA Method 680)
- PCB congeners (USEPA Method 1668A)
- Metals (lead and total chromium only) (USEPA Method 6010C)
- Hexavalent chromium (USEPA Method 7196A)

Quality Control Tasks

All applicable quality control (QC) protocols will be performed according to the listed method and the associated standard operating procedures (SOPs). Specified QC criteria will be met and documented.

Secondary Data

Previous studies and reports. See Worksheet #13.

Data Management Tasks

Data are generated from three primary pathways: (i) data derived from field activities; (ii) laboratory analytical data; and (iii) validated data. Data from all three pathways are entered into an electronic format in accordance with the project protocols.

Data generated during field activities will be recorded using a field logbook and/or field forms. The Field Team Lead reviews these forms for completeness and accuracy. Pertinent data from the field forms are entered into the project database. Hard copy field records are stored in a secure project file or scanned and stored on the project server.

Data generated during laboratory analysis are recorded as electronic reports in portable document format (PDF) format, and in electronic data deliverables (EDDs) after the samples have been analyzed. These data are then submitted for data validation. Data validation is performed in accordance with Worksheets #33, #34, #35, #36, and #37. Data and chain-of-custody forms are stored securely. Laboratories that generated the data archive data for a minimum of 5 years. Data are archived in electronic format.

Documentation and Records

In association with sample collection, field personnel are required to document pertinent data, including date, time, location (coordinates), field personnel, weather conditions, instrument identification, and any other factors that may affect data quality. Chain-of-custody procedures will be followed for all samples. All hardcopy data (field notebooks; photos; hard copies of chain-of-custody forms; etc.) are housed at the Geosyntec office (Portsmouth, New Hampshire) and/or scanned and stored on the project server. These records will be retained for at least 10 years after USEPA's Certification of Work Completion of the Statement of Work (SOW) as specified in the Consent Decree Section XIX.

Assessment/Audit Tasks

Review of SOPs as applicable relating to field and laboratory measurements, data validation, and project activities is required prior to project start. Audit records of the laboratories are maintained by the laboratory and available upon request.

Data Review Tasks

Peer and senior review of documentation will occur during data analysis and reporting. Senior and peer reviews are documented. Laboratory data will undergo data validation and verification.

Schedule

Activities	Organization	Anticipated Date(s) of Initiation	Anticipated Date of Completion	Deliverable	Deliverable Due Date
Preparation of draft Predesign Investigation Work Plan (PDIWP) and UFP-QAPP	Pharmacia LLC and Solutia Inc. (P/S), Geosyntec, GSI, and Anchor QEA	September 2021	November 2021	Draft PDIWP and UFP-QAPP with Attachments	November 9, 2021
Final PDIWP and UFP-QAPP after United States Environmental Protection Agency (USEPA) review of draft *(60 days following receipt of USEPA comments unless an extension is granted)*	P/S, Geosyntec, GSI, and Anchor QEA	May 2022	June 2022	Final PDIWP and UFP-QAPP with Attachments	June 8, 2022
Implementation of predesign investigation (PDI) activities *(schedule depends on property access that will be ongoing after submittal of the draft PDIWP and subcontractor availability)*	P/S, Geosyntec, GSI, and Anchor QEA	Summer 2022	Summer–Fall 2022	N/A	N/A
Monthly Progress Reports (initiated following USEPA's approval of the Remedial Design Work Plan and will continue monthly as a combined report for the Site as a whole)	P/S	N/A	TBD	Progress Reports, submitted at month's end	Monthly
PDI Evaluation Report *(60 days following completion of data validation for the PDI data collected under the PDIWP)*	P/S, Geosyntec, GSI, and Anchor QEA	Fall 2022	Spring 2023	Final Report	TBD

UFP-QAPP Worksheet #15: Reference Limits and Evaluation Table

Analyte	CAS Number	Analytical Laboratory	Units	Laboratory Specified		RCRA Permit Concentration Limits[1]		OU-1/OU-2 ROD RGs Groundwater		OU-1/OU-2 ROD RGs Surface Water	
				RL	MDL	Value	Laboratory MDL < Selected Screening Value	Value	Laboratory MDL < Selected Screening Value	Value	Laboratory MDL < Selected Screening Value
Polychlorinated biphenyl (PCB) Aroclors by USEPA SW-846 Method 8082A											
Aroclor 1016	12674-11-2	Eurofins-TestAmerica (Burlington, VT)	µg/L	0.5	0.087	0.5	Yes	0.5	Yes	--	Not Applicable
Aroclor 1221	11104-28-2	Eurofins-TestAmerica (Burlington, VT)	µg/L	0.5	0.097	0.5	Yes	0.5	Yes	--	Not Applicable
Aroclor 1232	11141-16-5	Eurofins-TestAmerica (Burlington, VT)	µg/L	0.5	0.056	0.5	Yes	0.5	Yes	--	Not Applicable
Aroclor 1242	53469-21-9	Eurofins-TestAmerica (Burlington, VT)	µg/L	0.5	0.048	0.5	Yes	0.5	Yes	--	Not Applicable
Aroclor 1248	12672-29-6	Eurofins-TestAmerica (Burlington, VT)	µg/L	0.5	0.059	0.5	Yes	0.5	Yes	--	Not Applicable
Aroclor 1254	11097-69-1	Eurofins-TestAmerica (Burlington, VT)	µg/L	0.5	0.089	0.5	Yes	0.5	Yes	--	Not Applicable
Aroclor 1260	11096-82-5	Eurofins-TestAmerica (Burlington, VT)	µg/L	0.5	0.12	0.5	Yes	0.5	Yes	--	Not Applicable
Aroclor 1262	37324-23-5	Eurofins-TestAmerica (Burlington, VT)	µg/L	0.5	0.084	0.5	Yes	0.5	Yes	--	Not Applicable
Aroclor 1268	11100-14-4	Eurofins-TestAmerica (Burlington, VT)	µg/L	0.5	0.054	0.5	Yes	0.5	Yes	--	Not Applicable
PCBs, Total	1336-36-3	Eurofins-TestAmerica (Burlington, VT)	µg/L	0.5	0.048	--	Not Applicable	--	Not Applicable	--	Not Applicable
PCB homologs by USEPA SW-846 Method 8082A											
Mono - CB	27323-18-8	Eurofins-TestAmerica (Burlington, VT)	µg/L	0.1	0.0022	--	Not Applicable	0.5	Yes	0.014	Yes
Di - CB	25512-42-9	Eurofins-TestAmerica (Burlington, VT)	µg/L	0.1	0.0021	--	Not Applicable	0.5	Yes	0.014	Yes
Tri - CB	25323-68-6	Eurofins-TestAmerica (Burlington, VT)	µg/L	0.1	0.011	--	Not Applicable	0.5	Yes	0.014	Yes
Tetra - CB	26914-33-0	Eurofins-TestAmerica (Burlington, VT)	µg/L	0.2	0.0099	--	Not Applicable	0.5	Yes	0.014	Yes
Penta - CB	25429-29-2	Eurofins-TestAmerica (Burlington, VT)	µg/L	0.2	0.0097	--	Not Applicable	0.5	Yes	0.014	Yes
Hexa - CB	26601-64-9	Eurofins-TestAmerica (Burlington, VT)	µg/L	0.2	0.0036	--	Not Applicable	0.5	Yes	0.014	Yes
Hepta - CB	28655-71-2	Eurofins-TestAmerica (Burlington, VT)	µg/L	0.3	0.0043	--	Not Applicable	0.5	Yes	0.014	Yes
Octa - CB	55722-26-4	Eurofins-TestAmerica (Burlington, VT)	µg/L	0.3	0.0061	--	Not Applicable	0.5	Yes	0.014	Yes
Nona - CB	53742-07-7	Eurofins-TestAmerica (Burlington, VT)	µg/L	0.5	0.0063	--	Not Applicable	0.5	Yes	0.014	Yes
Deca - CB	2051-24-3	Eurofins-TestAmerica (Burlington, VT)	µg/L	0.5	0.011	--	Not Applicable	0.5	Yes	--	Not Applicable
PCB congeners by USEPA SW-846 Method 1668A											
PCB-1	2051-60-7	Eurofins-TestAmerica (Knoxville, TN)	ng/L	0.04	0.0091	--	Not Applicable	--	Not Applicable	--	Not Applicable
PCB-2	2051-61-8	Eurofins-TestAmerica (Knoxville, TN)	ng/L	0.04	0.0097	--	Not Applicable	--	Not Applicable	--	Not Applicable
PCB-3	2051-62-9	Eurofins-TestAmerica (Knoxville, TN)	ng/L	0.04	0.0097	--	Not Applicable	--	Not Applicable	--	Not Applicable
PCB-4	13029-08-8	Eurofins-TestAmerica (Knoxville, TN)	ng/L	0.06	0.021	--	Not Applicable	--	Not Applicable	--	Not Applicable
PCB-5	16605-91-7	Eurofins-TestAmerica (Knoxville, TN)	ng/L	0.04	0.014	--	Not Applicable	--	Not Applicable	--	Not Applicable
PCB-6	25569-80-6	Eurofins-TestAmerica (Knoxville, TN)	ng/L	0.04	0.0068	--	Not Applicable	--	Not Applicable	--	Not Applicable
PCB-7	33284-50-3	Eurofins-TestAmerica (Knoxville, TN)	ng/L	0.04	0.0086	--	Not Applicable	--	Not Applicable	--	Not Applicable
PCB-8	34883-43-7	Eurofins-TestAmerica (Knoxville, TN)	ng/L	0.06	0.0092	--	Not Applicable	--	Not Applicable	--	Not Applicable
PCB-9	34883-39-1	Eurofins-TestAmerica (Knoxville, TN)	ng/L	0.04	0.0096	--	Not Applicable	--	Not Applicable	--	Not Applicable
PCB-10	33146-45-1	Eurofins-TestAmerica (Knoxville, TN)	ng/L	0.04	0.009	--	Not Applicable	--	Not Applicable	--	Not Applicable
PCB-11	2050-67-1	Eurofins-TestAmerica (Knoxville, TN)	ng/L	0.06	0.034	--	Not Applicable	--	Not Applicable	--	Not Applicable
PCB-12	2974-92-7	Eurofins-TestAmerica (Knoxville, TN)	ng/L	0.08	0.016	--	Not Applicable	--	Not Applicable	--	Not Applicable
PCB-13	2974-90-5	Eurofins-TestAmerica (Knoxville, TN)	ng/L	0.08	0.016	--	Not Applicable	--	Not Applicable	--	Not Applicable

UFP-QAPP Worksheet #15: Reference Limits and Evaluation Table

Analyte	CAS Number	Analytical Laboratory	Units	Laboratory Specified		RCRA Permit Concentration Limits[1]		OU-1/OU-2 ROD RGs Groundwater		OU-1/OU-2 ROD RGs Surface Water	
				RL	MDL	Value	Laboratory MDL < Selected Screening Value	Value	Laboratory MDL < Selected Screening Value	Value	Laboratory MDL < Selected Screening Value
PCB-14	34883-41-5	Eurofins-TestAmerica (Knoxville, TN)	ng/L	0.04	0.0081	--	Not Applicable	--	Not Applicable	--	Not Applicable
PCB-15	2050-68-2	Eurofins-TestAmerica (Knoxville, TN)	ng/L	0.04	0.0061	--	Not Applicable	--	Not Applicable	--	Not Applicable
PCB-16	38444-78-9	Eurofins-TestAmerica (Knoxville, TN)	ng/L	0.04	0.0091	--	Not Applicable	--	Not Applicable	--	Not Applicable
PCB-17	37680-66-3	Eurofins-TestAmerica (Knoxville, TN)	ng/L	0.04	0.0098	--	Not Applicable	--	Not Applicable	--	Not Applicable
PCB-18	37680-65-2	Eurofins-TestAmerica (Knoxville, TN)	ng/L	0.08	0.016	--	Not Applicable	--	Not Applicable	--	Not Applicable
PCB-19	38444-73-4	Eurofins-TestAmerica (Knoxville, TN)	ng/L	0.04	0.0093	--	Not Applicable	--	Not Applicable	--	Not Applicable
PCB-20	38444-84-7	Eurofins-TestAmerica (Knoxville, TN)	ng/L	0.08	0.012	--	Not Applicable	--	Not Applicable	--	Not Applicable
PCB-21	55702-46-0	Eurofins-TestAmerica (Knoxville, TN)	ng/L	0.08	0.032	--	Not Applicable	--	Not Applicable	--	Not Applicable
PCB-22	38444-85-8	Eurofins-TestAmerica (Knoxville, TN)	ng/L	0.04	0.0078	--	Not Applicable	--	Not Applicable	--	Not Applicable
PCB-23	55720-44-0	Eurofins-TestAmerica (Knoxville, TN)	ng/L	0.04	0.0075	--	Not Applicable	--	Not Applicable	--	Not Applicable
PCB-24	55702-45-9	Eurofins-TestAmerica (Knoxville, TN)	ng/L	0.04	0.012	--	Not Applicable	--	Not Applicable	--	Not Applicable
PCB-25	55712-37-3	Eurofins-TestAmerica (Knoxville, TN)	ng/L	0.04	0.0069	--	Not Applicable	--	Not Applicable	--	Not Applicable
PCB-26	38444-81-4	Eurofins-TestAmerica (Knoxville, TN)	ng/L	0.08	0.013	--	Not Applicable	--	Not Applicable	--	Not Applicable
PCB-27	38444-76-7	Eurofins-TestAmerica (Knoxville, TN)	ng/L	0.04	0.0079	--	Not Applicable	--	Not Applicable	--	Not Applicable
PCB-28	7012-37-5	Eurofins-TestAmerica (Knoxville, TN)	ng/L	0.08	0.012	--	Not Applicable	--	Not Applicable	--	Not Applicable
PCB-29	15862-07-4	Eurofins-TestAmerica (Knoxville, TN)	ng/L	0.08	0.013	--	Not Applicable	--	Not Applicable	--	Not Applicable
PCB-30	35693-92-6	Eurofins-TestAmerica (Knoxville, TN)	ng/L	0.08	0.016	--	Not Applicable	--	Not Applicable	--	Not Applicable
PCB-31	16606-02-3	Eurofins-TestAmerica (Knoxville, TN)	ng/L	0.04	0.0088	--	Not Applicable	--	Not Applicable	--	Not Applicable
PCB-32	38444-77-8	Eurofins-TestAmerica (Knoxville, TN)	ng/L	0.04	0.0095	--	Not Applicable	--	Not Applicable	--	Not Applicable
PCB-33	38444-86-9	Eurofins-TestAmerica (Knoxville, TN)	ng/L	0.08	0.032	--	Not Applicable	--	Not Applicable	--	Not Applicable
PCB-34	37680-68-5	Eurofins-TestAmerica (Knoxville, TN)	ng/L	0.04	0.0061	--	Not Applicable	--	Not Applicable	--	Not Applicable
PCB-35	37680-69-6	Eurofins-TestAmerica (Knoxville, TN)	ng/L	0.04	0.007	--	Not Applicable	--	Not Applicable	--	Not Applicable
PCB-36	38444-87-0	Eurofins-TestAmerica (Knoxville, TN)	ng/L	0.04	0.0074	--	Not Applicable	--	Not Applicable	--	Not Applicable
PCB-37	38444-90-5	Eurofins-TestAmerica (Knoxville, TN)	ng/L	0.04	0.0075	--	Not Applicable	--	Not Applicable	--	Not Applicable
PCB-38	53555-66-1	Eurofins-TestAmerica (Knoxville, TN)	ng/L	0.04	0.0081	--	Not Applicable	--	Not Applicable	--	Not Applicable
PCB-39	38444-88-1	Eurofins-TestAmerica (Knoxville, TN)	ng/L	0.04	0.0079	--	Not Applicable	--	Not Applicable	--	Not Applicable
PCB-40	38444-93-8	Eurofins-TestAmerica (Knoxville, TN)	ng/L	0.12	0.018	--	Not Applicable	--	Not Applicable	--	Not Applicable
PCB-41	52663-59-9	Eurofins-TestAmerica (Knoxville, TN)	ng/L	0.12	0.018	--	Not Applicable	--	Not Applicable	--	Not Applicable
PCB-42	36559-22-5	Eurofins-TestAmerica (Knoxville, TN)	ng/L	0.04	0.0065	--	Not Applicable	--	Not Applicable	--	Not Applicable
PCB-43	70362-46-8	Eurofins-TestAmerica (Knoxville, TN)	ng/L	0.08	0.012	--	Not Applicable	--	Not Applicable	--	Not Applicable
PCB-44	41464-39-5	Eurofins-TestAmerica (Knoxville, TN)	ng/L	0.12	0.057	--	Not Applicable	--	Not Applicable	--	Not Applicable
PCB-45	70362-45-7	Eurofins-TestAmerica (Knoxville, TN)	ng/L	0.08	0.016	--	Not Applicable	--	Not Applicable	--	Not Applicable
PCB-46	41464-47-5	Eurofins-TestAmerica (Knoxville, TN)	ng/L	0.04	0.0077	--	Not Applicable	--	Not Applicable	--	Not Applicable
PCB-47	2437-79-8	Eurofins-TestAmerica (Knoxville, TN)	ng/L	0.12	0.057	--	Not Applicable	--	Not Applicable	--	Not Applicable

UFP-QAPP Worksheet #15: Reference Limits and Evaluation Table

Analyte	CAS Number	Analytical Laboratory	Units	Laboratory Specified		RCRA Permit Concentration Limits[1]		OU-1/OU-2 ROD RGs Groundwater		OU-1/OU-2 ROD RGs Surface Water	
				RL	MDL	Value	Laboratory MDL < Selected Screening Value	Value	Laboratory MDL < Selected Screening Value	Value	Laboratory MDL < Selected Screening Value
PCB-48	70362-47-9	Eurofins-TestAmerica (Knoxville, TN)	ng/L	0.04	0.0076	--	Not Applicable	--	Not Applicable	--	Not Applicable
PCB-49	41464-40-8	Eurofins-TestAmerica (Knoxville, TN)	ng/L	0.08	0.012	--	Not Applicable	--	Not Applicable	--	Not Applicable
PCB-50	62796-65-0	Eurofins-TestAmerica (Knoxville, TN)	ng/L	0.08	0.012	--	Not Applicable	--	Not Applicable	--	Not Applicable
PCB-51	68194-04-7	Eurofins-TestAmerica (Knoxville, TN)	ng/L	0.08	0.016	--	Not Applicable	--	Not Applicable	--	Not Applicable
PCB-52	35693-99-3	Eurofins-TestAmerica (Knoxville, TN)	ng/L	0.04	0.0098	--	Not Applicable	--	Not Applicable	--	Not Applicable
PCB-53	41464-41-9	Eurofins-TestAmerica (Knoxville, TN)	ng/L	0.08	0.012	--	Not Applicable	--	Not Applicable	--	Not Applicable
PCB-54	15968-05-5	Eurofins-TestAmerica (Knoxville, TN)	ng/L	0.04	0.0096	--	Not Applicable	--	Not Applicable	--	Not Applicable
PCB-55	74338-24-2	Eurofins-TestAmerica (Knoxville, TN)	ng/L	0.04	0.0072	--	Not Applicable	--	Not Applicable	--	Not Applicable
PCB-56	41464-43-1	Eurofins-TestAmerica (Knoxville, TN)	ng/L	0.04	0.01	--	Not Applicable	--	Not Applicable	--	Not Applicable
PCB-57	70424-67-8	Eurofins-TestAmerica (Knoxville, TN)	ng/L	0.04	0.0074	--	Not Applicable	--	Not Applicable	--	Not Applicable
PCB-58	41464-49-7	Eurofins-TestAmerica (Knoxville, TN)	ng/L	0.04	0.0078	--	Not Applicable	--	Not Applicable	--	Not Applicable
PCB-59	74472-33-6	Eurofins-TestAmerica (Knoxville, TN)	ng/L	0.12	0.017	--	Not Applicable	--	Not Applicable	--	Not Applicable
PCB-60	33025-41-1	Eurofins-TestAmerica (Knoxville, TN)	ng/L	0.04	0.0071	--	Not Applicable	--	Not Applicable	--	Not Applicable
PCB-61	33284-53-6	Eurofins-TestAmerica (Knoxville, TN)	ng/L	0.16	0.022	--	Not Applicable	--	Not Applicable	--	Not Applicable
PCB-62	54230-22-7	Eurofins-TestAmerica (Knoxville, TN)	ng/L	0.12	0.017	--	Not Applicable	--	Not Applicable	--	Not Applicable
PCB-63	74472-34-7	Eurofins-TestAmerica (Knoxville, TN)	ng/L	0.04	0.0054	--	Not Applicable	--	Not Applicable	--	Not Applicable
PCB-64	52663-58-8	Eurofins-TestAmerica (Knoxville, TN)	ng/L	0.04	0.007	--	Not Applicable	--	Not Applicable	--	Not Applicable
PCB-65	33284-54-7	Eurofins-TestAmerica (Knoxville, TN)	ng/L	0.12	0.057	--	Not Applicable	--	Not Applicable	--	Not Applicable
PCB-66	32598-10-0	Eurofins-TestAmerica (Knoxville, TN)	ng/L	0.04	0.0097	--	Not Applicable	--	Not Applicable	--	Not Applicable
PCB-67	73575-53-8	Eurofins-TestAmerica (Knoxville, TN)	ng/L	0.04	0.0078	--	Not Applicable	--	Not Applicable	--	Not Applicable
PCB-68	73575-52-7	Eurofins-TestAmerica (Knoxville, TN)	ng/L	0.04	0.014	--	Not Applicable	--	Not Applicable	--	Not Applicable
PCB-69	60233-24-1	Eurofins-TestAmerica (Knoxville, TN)	ng/L	0.08	0.012	--	Not Applicable	--	Not Applicable	--	Not Applicable
PCB-70	32598-11-1	Eurofins-TestAmerica (Knoxville, TN)	ng/L	0.16	0.022	--	Not Applicable	--	Not Applicable	--	Not Applicable
PCB-71	41464-46-4	Eurofins-TestAmerica (Knoxville, TN)	ng/L	0.12	0.018	--	Not Applicable	--	Not Applicable	--	Not Applicable
PCB-72	41464-42-0	Eurofins-TestAmerica (Knoxville, TN)	ng/L	0.04	0.006	--	Not Applicable	--	Not Applicable	--	Not Applicable
PCB-73	74338-23-1	Eurofins-TestAmerica (Knoxville, TN)	ng/L	0.08	0.012	--	Not Applicable	--	Not Applicable	--	Not Applicable
PCB-74	32690-93-0	Eurofins-TestAmerica (Knoxville, TN)	ng/L	0.16	0.022	--	Not Applicable	--	Not Applicable	--	Not Applicable
PCB-75	32598-12-2	Eurofins-TestAmerica (Knoxville, TN)	ng/L	0.12	0.017	--	Not Applicable	--	Not Applicable	--	Not Applicable
PCB-76	70362-48-0	Eurofins-TestAmerica (Knoxville, TN)	ng/L	0.16	0.022	--	Not Applicable	--	Not Applicable	--	Not Applicable
PCB-77	32598-13-3	Eurofins-TestAmerica (Knoxville, TN)	ng/L	0.04	0.007	--	Not Applicable	--	Not Applicable	--	Not Applicable
PCB-78	70362-49-1	Eurofins-TestAmerica (Knoxville, TN)	ng/L	0.04	0.0071	--	Not Applicable	--	Not Applicable	--	Not Applicable
PCB-79	41464-48-6	Eurofins-TestAmerica (Knoxville, TN)	ng/L	0.04	0.008	--	Not Applicable	--	Not Applicable	--	Not Applicable
PCB-80	33284-52-5	Eurofins-TestAmerica (Knoxville, TN)	ng/L	0.04	0.006	--	Not Applicable	--	Not Applicable	--	Not Applicable
PCB-81	70362-50-4	Eurofins-TestAmerica (Knoxville, TN)	ng/L	0.04	0.0081	--	Not Applicable	--	Not Applicable	--	Not Applicable

UFP-QAPP Worksheet #15: Reference Limits and Evaluation Table

Analyte	CAS Number	Analytical Laboratory	Units	Laboratory Specified		RCRA Permit Concentration Limits[1]		OU-1/OU-2 ROD RGs Groundwater		OU-1/OU-2 ROD RGs Surface Water	
				RL	MDL	Value	Laboratory MDL < Selected Screening Value	Value	Laboratory MDL < Selected Screening Value	Value	Laboratory MDL < Selected Screening Value
PCB-82	52663-62-4	Eurofins-TestAmerica (Knoxville, TN)	ng/L	0.04	0.011	--	Not Applicable	--	Not Applicable	--	Not Applicable
PCB-83	60145-20-2	Eurofins-TestAmerica (Knoxville, TN)	ng/L	0.08	0.015	--	Not Applicable	--	Not Applicable	--	Not Applicable
PCB-84	52663-60-2	Eurofins-TestAmerica (Knoxville, TN)	ng/L	0.04	0.012	--	Not Applicable	--	Not Applicable	--	Not Applicable
PCB-85	65510-45-4	Eurofins-TestAmerica (Knoxville, TN)	ng/L	0.12	0.029	--	Not Applicable	--	Not Applicable	--	Not Applicable
PCB-86	55312-69-1	Eurofins-TestAmerica (Knoxville, TN)	ng/L	0.24	0.061	--	Not Applicable	--	Not Applicable	--	Not Applicable
PCB-87	38380-02-8	Eurofins-TestAmerica (Knoxville, TN)	ng/L	0.24	0.061	--	Not Applicable	--	Not Applicable	--	Not Applicable
PCB-88	55215-17-3	Eurofins-TestAmerica (Knoxville, TN)	ng/L	0.08	0.02	--	Not Applicable	--	Not Applicable	--	Not Applicable
PCB-89	73575-57-2	Eurofins-TestAmerica (Knoxville, TN)	ng/L	0.04	0.01	--	Not Applicable	--	Not Applicable	--	Not Applicable
PCB-90	68194-07-0	Eurofins-TestAmerica (Knoxville, TN)	ng/L	0.12	0.024	--	Not Applicable	--	Not Applicable	--	Not Applicable
PCB-91	68194-05-8	Eurofins-TestAmerica (Knoxville, TN)	ng/L	0.08	0.02	--	Not Applicable	--	Not Applicable	--	Not Applicable
PCB-92	52663-61-3	Eurofins-TestAmerica (Knoxville, TN)	ng/L	0.04	0.0077	--	Not Applicable	--	Not Applicable	--	Not Applicable
PCB-93	73575-56-1	Eurofins-TestAmerica (Knoxville, TN)	ng/L	0.08	0.023	--	Not Applicable	--	Not Applicable	--	Not Applicable
PCB-94	73575-55-0	Eurofins-TestAmerica (Knoxville, TN)	ng/L	0.04	0.0091	--	Not Applicable	--	Not Applicable	--	Not Applicable
PCB-95	38379-99-6	Eurofins-TestAmerica (Knoxville, TN)	ng/L	0.04	0.0095	--	Not Applicable	--	Not Applicable	--	Not Applicable
PCB-96	73575-54-9	Eurofins-TestAmerica (Knoxville, TN)	ng/L	0.04	0.01	--	Not Applicable	--	Not Applicable	--	Not Applicable
PCB-97	41464-51-1	Eurofins-TestAmerica (Knoxville, TN)	ng/L	0.24	0.061	--	Not Applicable	--	Not Applicable	--	Not Applicable
PCB-98	60233-25-2	Eurofins-TestAmerica (Knoxville, TN)	ng/L	0.08	0.018	--	Not Applicable	--	Not Applicable	--	Not Applicable
PCB-99	38380-01-7	Eurofins-TestAmerica (Knoxville, TN)	ng/L	0.08	0.015	--	Not Applicable	--	Not Applicable	--	Not Applicable
PCB-100	39485-83-1	Eurofins-TestAmerica (Knoxville, TN)	ng/L	0.08	0.023	--	Not Applicable	--	Not Applicable	--	Not Applicable
PCB-101	37680-73-2	Eurofins-TestAmerica (Knoxville, TN)	ng/L	0.12	0.024	--	Not Applicable	--	Not Applicable	--	Not Applicable
PCB-102	68194-06-9	Eurofins-TestAmerica (Knoxville, TN)	ng/L	0.08	0.018	--	Not Applicable	--	Not Applicable	--	Not Applicable
PCB-103	60145-21-3	Eurofins-TestAmerica (Knoxville, TN)	ng/L	0.04	0.0091	--	Not Applicable	--	Not Applicable	--	Not Applicable
PCB-104	56558-16-8	Eurofins-TestAmerica (Knoxville, TN)	ng/L	0.04	0.009	--	Not Applicable	--	Not Applicable	--	Not Applicable
PCB-105	32598-14-4	Eurofins-TestAmerica (Knoxville, TN)	ng/L	0.04	0.0093	--	Not Applicable	--	Not Applicable	--	Not Applicable
PCB-106	70424-69-0	Eurofins-TestAmerica (Knoxville, TN)	ng/L	0.04	0.008	--	Not Applicable	--	Not Applicable	--	Not Applicable
PCB-107	70424-68-9	Eurofins-TestAmerica (Knoxville, TN)	ng/L	0.04	0.0091	--	Not Applicable	--	Not Applicable	--	Not Applicable
PCB-108	70362-41-3	Eurofins-TestAmerica (Knoxville, TN)	ng/L	0.08	0.013	--	Not Applicable	--	Not Applicable	--	Not Applicable
PCB-109	74472-35-8	Eurofins-TestAmerica (Knoxville, TN)	ng/L	0.24	0.061	--	Not Applicable	--	Not Applicable	--	Not Applicable
PCB-110	38380-03-9	Eurofins-TestAmerica (Knoxville, TN)	ng/L	0.08	0.02	--	Not Applicable	--	Not Applicable	--	Not Applicable
PCB-111	39635-32-0	Eurofins-TestAmerica (Knoxville, TN)	ng/L	0.04	0.0091	--	Not Applicable	--	Not Applicable	--	Not Applicable
PCB-112	74472-36-9	Eurofins-TestAmerica (Knoxville, TN)	ng/L	0.04	0.012	--	Not Applicable	--	Not Applicable	--	Not Applicable
PCB-113	68194-10-5	Eurofins-TestAmerica (Knoxville, TN)	ng/L	0.12	0.024	--	Not Applicable	--	Not Applicable	--	Not Applicable
PCB-114	74472-37-0	Eurofins-TestAmerica (Knoxville, TN)	ng/L	0.04	0.0073	--	Not Applicable	--	Not Applicable	--	Not Applicable
PCB-115	74472-38-1	Eurofins-TestAmerica (Knoxville, TN)	ng/L	0.08	0.02	--	Not Applicable	--	Not Applicable	--	Not Applicable

UFP-QAPP Worksheet #15: Reference Limits and Evaluation Table

Analyte	CAS Number	Analytical Laboratory	Units	Laboratory Specified		RCRA Permit Concentration Limits[1]		OU-1/OU-2 ROD RGs Groundwater		OU-1/OU-2 ROD RGs Surface Water	
				RL	MDL	Value	Laboratory MDL < Selected Screening Value	Value	Laboratory MDL < Selected Screening Value	Value	Laboratory MDL < Selected Screening Value
PCB-116	18259-05-7	Eurofins-TestAmerica (Knoxville, TN)	ng/L	0.12	0.029	--	Not Applicable	--	Not Applicable	--	Not Applicable
PCB-117	68194-11-6	Eurofins-TestAmerica (Knoxville, TN)	ng/L	0.12	0.029	--	Not Applicable	--	Not Applicable	--	Not Applicable
PCB-118	31508-00-6	Eurofins-TestAmerica (Knoxville, TN)	ng/L	0.04	0.016	--	Not Applicable	--	Not Applicable	--	Not Applicable
PCB-119	56558-17-9	Eurofins-TestAmerica (Knoxville, TN)	ng/L	0.24	0.061	--	Not Applicable	--	Not Applicable	--	Not Applicable
PCB-120	68194-12-7	Eurofins-TestAmerica (Knoxville, TN)	ng/L	0.04	0.012	--	Not Applicable	--	Not Applicable	--	Not Applicable
PCB-121	56558-18-0	Eurofins-TestAmerica (Knoxville, TN)	ng/L	0.04	0.0096	--	Not Applicable	--	Not Applicable	--	Not Applicable
PCB-122	76842-07-4	Eurofins-TestAmerica (Knoxville, TN)	ng/L	0.04	0.0079	--	Not Applicable	--	Not Applicable	--	Not Applicable
PCB-123	65510-44-3	Eurofins-TestAmerica (Knoxville, TN)	ng/L	0.04	0.01	--	Not Applicable	--	Not Applicable	--	Not Applicable
PCB-124	70424-70-3	Eurofins-TestAmerica (Knoxville, TN)	ng/L	0.08	0.013	--	Not Applicable	--	Not Applicable	--	Not Applicable
PCB-125	74472-39-2	Eurofins-TestAmerica (Knoxville, TN)	ng/L	0.24	0.061	--	Not Applicable	--	Not Applicable	--	Not Applicable
PCB-126	57465-28-8	Eurofins-TestAmerica (Knoxville, TN)	ng/L	0.04	0.0086	--	Not Applicable	--	Not Applicable	--	Not Applicable
PCB-127	39635-33-1	Eurofins-TestAmerica (Knoxville, TN)	ng/L	0.04	0.0065	--	Not Applicable	--	Not Applicable	--	Not Applicable
PCB-128	38380-07-3	Eurofins-TestAmerica (Knoxville, TN)	ng/L	0.08	0.017	--	Not Applicable	--	Not Applicable	--	Not Applicable
PCB-129	55215-18-4	Eurofins-TestAmerica (Knoxville, TN)	ng/L	0.16	0.03	--	Not Applicable	--	Not Applicable	--	Not Applicable
PCB-130	52663-66-8	Eurofins-TestAmerica (Knoxville, TN)	ng/L	0.04	0.0072	--	Not Applicable	--	Not Applicable	--	Not Applicable
PCB-131	61798-70-7	Eurofins-TestAmerica (Knoxville, TN)	ng/L	0.04	0.0092	--	Not Applicable	--	Not Applicable	--	Not Applicable
PCB-132	38380-05-1	Eurofins-TestAmerica (Knoxville, TN)	ng/L	0.04	0.0073	--	Not Applicable	--	Not Applicable	--	Not Applicable
PCB-133	35694-04-3	Eurofins-TestAmerica (Knoxville, TN)	ng/L	0.04	0.0089	--	Not Applicable	--	Not Applicable	--	Not Applicable
PCB-134	52704-70-8	Eurofins-TestAmerica (Knoxville, TN)	ng/L	0.08	0.011	--	Not Applicable	--	Not Applicable	--	Not Applicable
PCB-135	52744-13-5	Eurofins-TestAmerica (Knoxville, TN)	ng/L	0.08	0.024	--	Not Applicable	--	Not Applicable	--	Not Applicable
PCB-136	38411-22-2	Eurofins-TestAmerica (Knoxville, TN)	ng/L	0.04	0.0096	--	Not Applicable	--	Not Applicable	--	Not Applicable
PCB-137	35694-06-5	Eurofins-TestAmerica (Knoxville, TN)	ng/L	0.04	0.009	--	Not Applicable	--	Not Applicable	--	Not Applicable
PCB-138	35065-28-2	Eurofins-TestAmerica (Knoxville, TN)	ng/L	0.16	0.03	--	Not Applicable	--	Not Applicable	--	Not Applicable
PCB-139	56030-56-9	Eurofins-TestAmerica (Knoxville, TN)	ng/L	0.08	0.016	--	Not Applicable	--	Not Applicable	--	Not Applicable
PCB-140	59291-64-4	Eurofins-TestAmerica (Knoxville, TN)	ng/L	0.08	0.016	--	Not Applicable	--	Not Applicable	--	Not Applicable
PCB-141	52712-04-6	Eurofins-TestAmerica (Knoxville, TN)	ng/L	0.04	0.01	--	Not Applicable	--	Not Applicable	--	Not Applicable
PCB-142	41411-61-4	Eurofins-TestAmerica (Knoxville, TN)	ng/L	0.04	0.011	--	Not Applicable	--	Not Applicable	--	Not Applicable
PCB-143	68194-15-0	Eurofins-TestAmerica (Knoxville, TN)	ng/L	0.08	0.011	--	Not Applicable	--	Not Applicable	--	Not Applicable
PCB-144	68194-14-9	Eurofins-TestAmerica (Knoxville, TN)	ng/L	0.04	0.012	--	Not Applicable	--	Not Applicable	--	Not Applicable
PCB-145	74472-40-5	Eurofins-TestAmerica (Knoxville, TN)	ng/L	0.04	0.011	--	Not Applicable	--	Not Applicable	--	Not Applicable
PCB-146	51908-16-8	Eurofins-TestAmerica (Knoxville, TN)	ng/L	0.04	0.0076	--	Not Applicable	--	Not Applicable	--	Not Applicable
PCB-147	68194-13-8	Eurofins-TestAmerica (Knoxville, TN)	ng/L	0.08	0.017	--	Not Applicable	--	Not Applicable	--	Not Applicable
PCB-148	74472-41-6	Eurofins-TestAmerica (Knoxville, TN)	ng/L	0.04	0.0095	--	Not Applicable	--	Not Applicable	--	Not Applicable
PCB-149	38380-04-0	Eurofins-TestAmerica (Knoxville, TN)	ng/L	0.08	0.017	--	Not Applicable	--	Not Applicable	--	Not Applicable

UFP-QAPP Worksheet #15: Reference Limits and Evaluation Table

Analyte	CAS Number	Analytical Laboratory	Units	Laboratory Specified		RCRA Permit Concentration Limits[1]		OU-1/OU-2 ROD RGs Groundwater		OU-1/OU-2 ROD RGs Surface Water	
				RL	MDL	Value	Laboratory MDL < Selected Screening Value	Value	Laboratory MDL < Selected Screening Value	Value	Laboratory MDL < Selected Screening Value
PCB-150	68194-08-1	Eurofins-TestAmerica (Knoxville, TN)	ng/L	0.04	0.014	--	Not Applicable	--	Not Applicable	--	Not Applicable
PCB-151	52663-63-5	Eurofins-TestAmerica (Knoxville, TN)	ng/L	0.08	0.024	--	Not Applicable	--	Not Applicable	--	Not Applicable
PCB-152	68194-09-2	Eurofins-TestAmerica (Knoxville, TN)	ng/L	0.04	0.0065	--	Not Applicable	--	Not Applicable	--	Not Applicable
PCB-153	35065-27-1	Eurofins-TestAmerica (Knoxville, TN)	ng/L	0.08	0.016	--	Not Applicable	--	Not Applicable	--	Not Applicable
PCB-154	60145-22-4	Eurofins-TestAmerica (Knoxville, TN)	ng/L	0.04	0.012	--	Not Applicable	--	Not Applicable	--	Not Applicable
PCB-155	33979-03-2	Eurofins-TestAmerica (Knoxville, TN)	ng/L	0.04	0.007	--	Not Applicable	--	Not Applicable	--	Not Applicable
PCB-156	38380-08-4	Eurofins-TestAmerica (Knoxville, TN)	ng/L	0.08	0.018	--	Not Applicable	--	Not Applicable	--	Not Applicable
PCB-157	69782-90-7	Eurofins-TestAmerica (Knoxville, TN)	ng/L	0.08	0.018	--	Not Applicable	--	Not Applicable	--	Not Applicable
PCB-158	74472-42-7	Eurofins-TestAmerica (Knoxville, TN)	ng/L	0.04	0.0084	--	Not Applicable	--	Not Applicable	--	Not Applicable
PCB-159	39635-35-3	Eurofins-TestAmerica (Knoxville, TN)	ng/L	0.04	0.0084	--	Not Applicable	--	Not Applicable	--	Not Applicable
PCB-160	41411-62-5	Eurofins-TestAmerica (Knoxville, TN)	ng/L	0.16	0.03	--	Not Applicable	--	Not Applicable	--	Not Applicable
PCB-161	74472-43-8	Eurofins-TestAmerica (Knoxville, TN)	ng/L	0.04	0.0073	--	Not Applicable	--	Not Applicable	--	Not Applicable
PCB-162	39635-34-2	Eurofins-TestAmerica (Knoxville, TN)	ng/L	0.04	0.0094	--	Not Applicable	--	Not Applicable	--	Not Applicable
PCB-163	74472-44-9	Eurofins-TestAmerica (Knoxville, TN)	ng/L	0.16	0.03	--	Not Applicable	--	Not Applicable	--	Not Applicable
PCB-164	74472-45-0	Eurofins-TestAmerica (Knoxville, TN)	ng/L	0.04	0.0096	--	Not Applicable	--	Not Applicable	--	Not Applicable
PCB-165	74472-46-1	Eurofins-TestAmerica (Knoxville, TN)	ng/L	0.04	0.0073	--	Not Applicable	--	Not Applicable	--	Not Applicable
PCB-166	41411-63-6	Eurofins-TestAmerica (Knoxville, TN)	ng/L	0.08	0.017	--	Not Applicable	--	Not Applicable	--	Not Applicable
PCB-167	52663-72-6	Eurofins-TestAmerica (Knoxville, TN)	ng/L	0.04	0.0077	--	Not Applicable	--	Not Applicable	--	Not Applicable
PCB-168	59291-65-5	Eurofins-TestAmerica (Knoxville, TN)	ng/L	0.08	0.016	--	Not Applicable	--	Not Applicable	--	Not Applicable
PCB-169	32774-16-6	Eurofins-TestAmerica (Knoxville, TN)	ng/L	0.04	0.0085	--	Not Applicable	--	Not Applicable	--	Not Applicable
PCB-170	35065-30-6	Eurofins-TestAmerica (Knoxville, TN)	ng/L	0.04	0.0089	--	Not Applicable	--	Not Applicable	--	Not Applicable
PCB-171	52663-71-5	Eurofins-TestAmerica (Knoxville, TN)	ng/L	0.08	0.018	--	Not Applicable	--	Not Applicable	--	Not Applicable
PCB-172	52663-74-8	Eurofins-TestAmerica (Knoxville, TN)	ng/L	0.04	0.0083	--	Not Applicable	--	Not Applicable	--	Not Applicable
PCB-173	68194-16-1	Eurofins-TestAmerica (Knoxville, TN)	ng/L	0.08	0.018	--	Not Applicable	--	Not Applicable	--	Not Applicable
PCB-174	38411-25-5	Eurofins-TestAmerica (Knoxville, TN)	ng/L	0.04	0.0088	--	Not Applicable	--	Not Applicable	--	Not Applicable
PCB-175	40186-70-7	Eurofins-TestAmerica (Knoxville, TN)	ng/L	0.04	0.0079	--	Not Applicable	--	Not Applicable	--	Not Applicable
PCB-176	52663-65-7	Eurofins-TestAmerica (Knoxville, TN)	ng/L	0.04	0.008	--	Not Applicable	--	Not Applicable	--	Not Applicable
PCB-177	52663-70-4	Eurofins-TestAmerica (Knoxville, TN)	ng/L	0.04	0.0092	--	Not Applicable	--	Not Applicable	--	Not Applicable
PCB-178	52663-67-9	Eurofins-TestAmerica (Knoxville, TN)	ng/L	0.04	0.011	--	Not Applicable	--	Not Applicable	--	Not Applicable
PCB-179	52663-64-6	Eurofins-TestAmerica (Knoxville, TN)	ng/L	0.04	0.0071	--	Not Applicable	--	Not Applicable	--	Not Applicable
PCB-180	35065-29-3	Eurofins-TestAmerica (Knoxville, TN)	ng/L	0.08	0.018	--	Not Applicable	--	Not Applicable	--	Not Applicable
PCB-181	74472-47-2	Eurofins-TestAmerica (Knoxville, TN)	ng/L	0.04	0.0086	--	Not Applicable	--	Not Applicable	--	Not Applicable
PCB-182	60145-23-5	Eurofins-TestAmerica (Knoxville, TN)	ng/L	0.04	0.008	--	Not Applicable	--	Not Applicable	--	Not Applicable
PCB-183	52663-69-1	Eurofins-TestAmerica (Knoxville, TN)	ng/L	0.08	0.016	--	Not Applicable	--	Not Applicable	--	Not Applicable

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Analyte	CAS Number	Analytical Laboratory	Units	Laboratory Specified		RCRA Permit Concentration Limits[1]		OU-1/OU-2 ROD RGs Groundwater		OU-1/OU-2 ROD RGs Surface Water	
				RL	MDL	Value	Laboratory MDL < Selected Screening Value	Value	Laboratory MDL < Selected Screening Value	Value	Laboratory MDL < Selected Screening Value
PCB-184	74472-48-3	Eurofins-TestAmerica (Knoxville, TN)	ng/L	0.04	0.0079	--	Not Applicable	--	Not Applicable	--	Not Applicable
PCB-185	52712-05-7	Eurofins-TestAmerica (Knoxville, TN)	ng/L	0.08	0.016	--	Not Applicable	--	Not Applicable	--	Not Applicable
PCB-186	74472-49-4	Eurofins-TestAmerica (Knoxville, TN)	ng/L	0.04	0.008	--	Not Applicable	--	Not Applicable	--	Not Applicable
PCB-187	52663-68-0	Eurofins-TestAmerica (Knoxville, TN)	ng/L	0.04	0.009	--	Not Applicable	--	Not Applicable	--	Not Applicable
PCB-188	74487-85-7	Eurofins-TestAmerica (Knoxville, TN)	ng/L	0.04	0.01	--	Not Applicable	--	Not Applicable	--	Not Applicable
PCB-189	39635-31-9	Eurofins-TestAmerica (Knoxville, TN)	ng/L	0.04	0.008	--	Not Applicable	--	Not Applicable	--	Not Applicable
PCB-190	41411-64-7	Eurofins-TestAmerica (Knoxville, TN)	ng/L	0.04	0.0077	--	Not Applicable	--	Not Applicable	--	Not Applicable
PCB-191	74472-50-7	Eurofins-TestAmerica (Knoxville, TN)	ng/L	0.04	0.0078	--	Not Applicable	--	Not Applicable	--	Not Applicable
PCB-192	74472-51-8	Eurofins-TestAmerica (Knoxville, TN)	ng/L	0.04	0.0078	--	Not Applicable	--	Not Applicable	--	Not Applicable
PCB-193	69782-91-8	Eurofins-TestAmerica (Knoxville, TN)	ng/L	0.08	0.018	--	Not Applicable	--	Not Applicable	--	Not Applicable
PCB-194	35694-08-7	Eurofins-TestAmerica (Knoxville, TN)	ng/L	0.04	0.011	--	Not Applicable	--	Not Applicable	--	Not Applicable
PCB-195	52663-78-2	Eurofins-TestAmerica (Knoxville, TN)	ng/L	0.04	0.009	--	Not Applicable	--	Not Applicable	--	Not Applicable
PCB-196	42740-50-1	Eurofins-TestAmerica (Knoxville, TN)	ng/L	0.04	0.0095	--	Not Applicable	--	Not Applicable	--	Not Applicable
PCB-197	33091-17-7	Eurofins-TestAmerica (Knoxville, TN)	ng/L	0.04	0.009	--	Not Applicable	--	Not Applicable	--	Not Applicable
PCB-198	68194-17-2	Eurofins-TestAmerica (Knoxville, TN)	ng/L	0.08	0.018	--	Not Applicable	--	Not Applicable	--	Not Applicable
PCB-199	52663-75-9	Eurofins-TestAmerica (Knoxville, TN)	ng/L	0.08	0.018	--	Not Applicable	--	Not Applicable	--	Not Applicable
PCB-200	52663-73-7	Eurofins-TestAmerica (Knoxville, TN)	ng/L	0.04	0.008	--	Not Applicable	--	Not Applicable	--	Not Applicable
PCB-201	40186-71-8	Eurofins-TestAmerica (Knoxville, TN)	ng/L	0.04	0.0091	--	Not Applicable	--	Not Applicable	--	Not Applicable
PCB-202	2136-99-4	Eurofins-TestAmerica (Knoxville, TN)	ng/L	0.04	0.0096	--	Not Applicable	--	Not Applicable	--	Not Applicable
PCB-203	52663-76-0	Eurofins-TestAmerica (Knoxville, TN)	ng/L	0.04	0.0089	--	Not Applicable	--	Not Applicable	--	Not Applicable
PCB-204	74472-52-9	Eurofins-TestAmerica (Knoxville, TN)	ng/L	0.04	0.0086	--	Not Applicable	--	Not Applicable	--	Not Applicable
PCB-205	74472-53-0	Eurofins-TestAmerica (Knoxville, TN)	ng/L	0.04	0.011	--	Not Applicable	--	Not Applicable	--	Not Applicable
PCB-206	40186-72-9	Eurofins-TestAmerica (Knoxville, TN)	ng/L	0.04	0.01	--	Not Applicable	--	Not Applicable	--	Not Applicable
PCB-207	52663-79-3	Eurofins-TestAmerica (Knoxville, TN)	ng/L	0.04	0.009	--	Not Applicable	--	Not Applicable	--	Not Applicable
PCB-208	52663-77-1	Eurofins-TestAmerica (Knoxville, TN)	ng/L	0.04	0.0097	--	Not Applicable	--	Not Applicable	--	Not Applicable
DCB Decachlorobiphenyl	2051-24-3	Eurofins-TestAmerica (Knoxville, TN)	ng/L	0.04	0.013	--	Not Applicable	--	Not Applicable	--	Not Applicable
Semi-volatile Organic Compounds by USEPA SW-846 Method 8270											
Acenaphthene	83-32-9	Eurofins-TestAmerica (Pittsburgh, PA)	µg/L	1.9	0.65	--	Not Applicable	--	Not Applicable	--	Not Applicable
Acenaphthylene	208-96-8	Eurofins-TestAmerica (Pittsburgh, PA)	µg/L	1.9	0.65	--	Not Applicable	--	Not Applicable	--	Not Applicable
Acetophenone	98-86-2	Eurofins-TestAmerica (Pittsburgh, PA)	µg/L	10	1.59	--	Not Applicable	--	Not Applicable	--	Not Applicable
Anthracene	120-12-7	Eurofins-TestAmerica (Pittsburgh, PA)	µg/L	1.9	0.49	--	Not Applicable	--	Not Applicable	--	Not Applicable
Atrazine	1912-24-9	Eurofins-TestAmerica (Pittsburgh, PA)	µg/L	10	6.33	--	Not Applicable	--	Not Applicable	--	Not Applicable
Benzaldehyde	100-52-7	Eurofins-TestAmerica (Pittsburgh, PA)	µg/L	10	5.38	--	Not Applicable	--	Not Applicable	--	Not Applicable
Benzo[a]anthracene	56-55-3	Eurofins-TestAmerica (Pittsburgh, PA)	µg/L	1.9	0.75	--	Not Applicable	--	Not Applicable	--	Not Applicable
Benzo[a]pyrene	50-32-8	Eurofins-TestAmerica (Pittsburgh, PA)	µg/L	1.9	0.53	--	Not Applicable	--	Not Applicable	--	Not Applicable

UFP-QAPP Worksheet #15: Reference Limits and Evaluation Table

Analyte	CAS Number	Analytical Laboratory	Units	Laboratory Specified		RCRA Permit Concentration Limits[1]		OU-1/OU-2 ROD RGs Groundwater		OU-1/OU-2 ROD RGs Surface Water	
				RL	MDL	Value	Laboratory MDL < Selected Screening Value	Value	Laboratory MDL < Selected Screening Value	Value	Laboratory MDL < Selected Screening Value
Benzo[b]fluoranthene	205-99-2	Eurofins-TestAmerica (Pittsburgh, PA)	µg/L	1.9	0.97	--	Not Applicable	--	Not Applicable	--	Not Applicable
Benzo[g,h,i]perylene	191-24-2	Eurofins-TestAmerica (Pittsburgh, PA)	µg/L	1.9	0.69	--	Not Applicable	--	Not Applicable	--	Not Applicable
Benzo[k]fluoranthene	207-08-9	Eurofins-TestAmerica (Pittsburgh, PA)	µg/L	1.9	0.88	--	Not Applicable	--	Not Applicable	--	Not Applicable
1,1'-Biphenyl	92-52-4	Eurofins-TestAmerica (Pittsburgh, PA)	µg/L	10	1.42	--	Not Applicable	--	Not Applicable	--	Not Applicable
Bis(2-chloroethoxy)methane	111-91-1	Eurofins-TestAmerica (Pittsburgh, PA)	µg/L	10	1.52	--	Not Applicable	--	Not Applicable	--	Not Applicable
Bis(2-chloroethyl)ether	111-44-4	Eurofins-TestAmerica (Pittsburgh, PA)	µg/L	1.9	0.4	--	Not Applicable	--	Not Applicable	--	Not Applicable
Bis(2-ethylhexyl) phthalate	117-81-7	Eurofins-TestAmerica (Pittsburgh, PA)	µg/L	100	62.3	--	Not Applicable	--	Not Applicable	--	Not Applicable
4-Bromophenyl phenyl ether	101-55-3	Eurofins-TestAmerica (Pittsburgh, PA)	µg/L	10	3.19	--	Not Applicable	--	Not Applicable	--	Not Applicable
Butyl benzyl phthalate	85-68-7	Eurofins-TestAmerica (Pittsburgh, PA)	µg/L	10	4.62	--	Not Applicable	--	Not Applicable	--	Not Applicable
Caprolactam	105-60-2	Eurofins-TestAmerica (Pittsburgh, PA)	µg/L	50	4.7	--	Not Applicable	--	Not Applicable	--	Not Applicable
Carbazole	86-74-8	Eurofins-TestAmerica (Pittsburgh, PA)	µg/L	1.9	0.51	--	Not Applicable	--	Not Applicable	--	Not Applicable
4-Chloroaniline	106-47-8	Eurofins-TestAmerica (Pittsburgh, PA)	µg/L	10	3.76	--	Not Applicable	--	Not Applicable	--	Not Applicable
4-Chloro-3-methylphenol	59-50-7	Eurofins-TestAmerica (Pittsburgh, PA)	µg/L	10	2.78	--	Not Applicable	--	Not Applicable	--	Not Applicable
2-Chloronaphthalene	91-58-7	Eurofins-TestAmerica (Pittsburgh, PA)	µg/L	1.9	0.59	--	Not Applicable	--	Not Applicable	--	Not Applicable
2-Chlorophenol	95-57-8	Eurofins-TestAmerica (Pittsburgh, PA)	µg/L	10	1.29	--	Not Applicable	--	Not Applicable	--	Not Applicable
4-Chlorophenyl phenyl ether	7005-72-3	Eurofins-TestAmerica (Pittsburgh, PA)	µg/L	10	2.21	--	Not Applicable	--	Not Applicable	--	Not Applicable
Chrysene	218-01-9	Eurofins-TestAmerica (Pittsburgh, PA)	µg/L	1.9	0.81	--	Not Applicable	--	Not Applicable	--	Not Applicable
Dibenz(a,h)anthracene	53-70-3	Eurofins-TestAmerica (Pittsburgh, PA)	µg/L	1.9	0.72	--	Not Applicable	--	Not Applicable	--	Not Applicable
Dibenzofuran	132-64-9	Eurofins-TestAmerica (Pittsburgh, PA)	µg/L	10	1.9	--	Not Applicable	--	Not Applicable	--	Not Applicable
3,3'-Dichlorobenzidine	91-94-1	Eurofins-TestAmerica (Pittsburgh, PA)	µg/L	10	5.83	--	Not Applicable	--	Not Applicable	--	Not Applicable
2,4-Dichlorophenol	120-83-2	Eurofins-TestAmerica (Pittsburgh, PA)	µg/L	1.9	0.51	--	Not Applicable	--	Not Applicable	--	Not Applicable
Diethyl phthalate	84-66-2	Eurofins-TestAmerica (Pittsburgh, PA)	µg/L	10	5.67	--	Not Applicable	--	Not Applicable	--	Not Applicable
2,4-Dimethylphenol	105-67-9	Eurofins-TestAmerica (Pittsburgh, PA)	µg/L	10	1.67	--	Not Applicable	--	Not Applicable	--	Not Applicable
Dimethyl phthalate	131-11-3	Eurofins-TestAmerica (Pittsburgh, PA)	µg/L	10	2	--	Not Applicable	--	Not Applicable	--	Not Applicable
Di-n-butyl phthalate	84-74-2	Eurofins-TestAmerica (Pittsburgh, PA)	µg/L	10	7.43	--	Not Applicable	--	Not Applicable	--	Not Applicable
4,6-Dinitro-2-methylphenol	534-52-1	Eurofins-TestAmerica (Pittsburgh, PA)	µg/L	50	14.7	--	Not Applicable	--	Not Applicable	--	Not Applicable
2,4-Dinitrophenol	51-28-5	Eurofins-TestAmerica (Pittsburgh, PA)	µg/L	100	15.3	--	Not Applicable	--	Not Applicable	--	Not Applicable
2,4-Dinitrotoluene	121-14-2	Eurofins-TestAmerica (Pittsburgh, PA)	µg/L	10	3.53	--	Not Applicable	--	Not Applicable	--	Not Applicable
2,6-Dinitrotoluene	606-20-2	Eurofins-TestAmerica (Pittsburgh, PA)	µg/L	10	1.73	--	Not Applicable	--	Not Applicable	--	Not Applicable
Di-n-octyl phthalate	117-84-0	Eurofins-TestAmerica (Pittsburgh, PA)	µg/L	10	6.85	--	Not Applicable	--	Not Applicable	--	Not Applicable
Fluoranthene	206-44-0	Eurofins-TestAmerica (Pittsburgh, PA)	µg/L	1.9	0.6	--	Not Applicable	--	Not Applicable	--	Not Applicable
Fluorene	86-73-7	Eurofins-TestAmerica (Pittsburgh, PA)	µg/L	1.9	0.69	--	Not Applicable	--	Not Applicable	--	Not Applicable
Hexachlorobenzene	118-74-1	Eurofins-TestAmerica (Pittsburgh, PA)	µg/L	1.9	0.56	--	Not Applicable	--	Not Applicable	--	Not Applicable
Hexachlorobutadiene	87-68-3	Eurofins-TestAmerica (Pittsburgh, PA)	µg/L	1.9	0.69	--	Not Applicable	--	Not Applicable	--	Not Applicable

UFP-QAPP Worksheet #15: Reference Limits and Evaluation Table

Analyte	CAS Number	Analytical Laboratory	Units	Laboratory Specified		RCRA Permit Concentration Limits[1]		OU-1/OU-2 ROD RGs Groundwater		OU-1/OU-2 ROD RGs Surface Water	
				RL	MDL	Value	Laboratory MDL < Selected Screening Value	Value	Laboratory MDL < Selected Screening Value	Value	Laboratory MDL < Selected Screening Value
Hexachlorocyclopentadiene	77-47-4	Eurofins-TestAmerica (Pittsburgh, PA)	µg/L	10	4.97	--	Not Applicable	--	Not Applicable	--	Not Applicable
Hexachloroethane	67-72-1	Eurofins-TestAmerica (Pittsburgh, PA)	µg/L	10	1.33	--	Not Applicable	--	Not Applicable	--	Not Applicable
Indeno[1,2,3-cd]pyrene	193-39-5	Eurofins-TestAmerica (Pittsburgh, PA)	µg/L	1.9	0.85	--	Not Applicable	--	Not Applicable	--	Not Applicable
Isophorone	78-59-1	Eurofins-TestAmerica (Pittsburgh, PA)	µg/L	10	1.88	--	Not Applicable	--	Not Applicable	--	Not Applicable
2-Methylnaphthalene	91-57-6	Eurofins-TestAmerica (Pittsburgh, PA)	µg/L	1.9	0.62	--	Not Applicable	--	Not Applicable	--	Not Applicable
2-Methylphenol	95-48-7	Eurofins-TestAmerica (Pittsburgh, PA)	µg/L	10	3	--	Not Applicable	--	Not Applicable	--	Not Applicable
Methylphenol, 3 & 4	106-44-5	Eurofins-TestAmerica (Pittsburgh, PA)	µg/L	10	3.72	--	Not Applicable	--	Not Applicable	--	Not Applicable
Naphthalene	91-20-3	Eurofins-TestAmerica (Pittsburgh, PA)	µg/L	1.9	0.59	--	Not Applicable	--	Not Applicable	--	Not Applicable
2-Nitroaniline	88-74-4	Eurofins-TestAmerica (Pittsburgh, PA)	µg/L	50	5.48	--	Not Applicable	--	Not Applicable	--	Not Applicable
3-Nitroaniline	99-09-2	Eurofins-TestAmerica (Pittsburgh, PA)	µg/L	50	4.37	--	Not Applicable	--	Not Applicable	--	Not Applicable
4-Nitroaniline	100-01-6	Eurofins-TestAmerica (Pittsburgh, PA)	µg/L	50	3.63	--	Not Applicable	--	Not Applicable	--	Not Applicable
Nitrobenzene	98-95-3	Eurofins-TestAmerica (Pittsburgh, PA)	µg/L	20	5	--	Not Applicable	--	Not Applicable	--	Not Applicable
2-Nitrophenol	88-75-5	Eurofins-TestAmerica (Pittsburgh, PA)	µg/L	10	1.93	--	Not Applicable	--	Not Applicable	--	Not Applicable
4-Nitrophenol	100-02-7	Eurofins-TestAmerica (Pittsburgh, PA)	µg/L	50	9.4	128	Yes	--	Not Applicable	--	Not Applicable
N-Nitrosodi-n-propylamine	621-64-7	Eurofins-TestAmerica (Pittsburgh, PA)	µg/L	1.9	0.71	--	Not Applicable	--	Not Applicable	--	Not Applicable
N-Nitrosodiphenylamine	86-30-6	Eurofins-TestAmerica (Pittsburgh, PA)	µg/L	10	1.19	--	Not Applicable	--	Not Applicable	--	Not Applicable
2,2'-oxybis[1-chloropropane]	108-60-1	Eurofins-TestAmerica (Pittsburgh, PA)	µg/L	1.9	0.58	--	Not Applicable	--	Not Applicable	--	Not Applicable
Pentachlorophenol	87-86-5	Eurofins-TestAmerica (Pittsburgh, PA)	µg/L	50	8.47	1	No	--	Not Applicable	--	Not Applicable
Phenanthrene	85-01-8	Eurofins-TestAmerica (Pittsburgh, PA)	µg/L	1.9	0.55	--	Not Applicable	--	Not Applicable	--	Not Applicable
Phenol	108-95-2	Eurofins-TestAmerica (Pittsburgh, PA)	µg/L	10	4.87	--	Not Applicable	--	Not Applicable	--	Not Applicable
Pyrene	129-00-0	Eurofins-TestAmerica (Pittsburgh, PA)	µg/L	1.9	0.54	--	Not Applicable	--	Not Applicable	--	Not Applicable
1,2,4,5-Tetrachlorobenzene	95-94-3	Eurofins-TestAmerica (Pittsburgh, PA)	µg/L	10	1.5	--	Not Applicable	--	Not Applicable	--	Not Applicable
2,3,4,6-Tetrachlorophenol	58-90-2	Eurofins-TestAmerica (Pittsburgh, PA)	µg/L	10	3.26	--	Not Applicable	--	Not Applicable	--	Not Applicable
2,4,6-Trichlorophenol	88-06-2	Eurofins-TestAmerica (Pittsburgh, PA)	µg/L	10	2.24	47	Yes	--	Not Applicable	--	Not Applicable
Reactivity (Cyanide and Sulfide) by USEPA SW-846 9010C and USEPA SW-846 9030B/9034											
Cyanide, Total	57-12-5	Eurofins-TestAmerica (Pittsburgh, PA)	µg/L	10	8	--	Not Applicable	--	Not Applicable	--	Not Applicable
Sulfide	18496-25-8	Eurofins-TestAmerica (Pittsburgh, PA)	mg/l	3	2.085	--	Not Applicable	--	Not Applicable	--	Not Applicable
Total Dissolved Solids by SM 2540C											
Total Dissolved Solids	--	Eurofins-TestAmerica (Pittsburgh, PA)	mg/l	10	--	--	Not Applicable	--	Not Applicable	--	Not Applicable
Total Suspended Solids by SM 2540D											
Total Suspended Solids	--	Eurofins-TestAmerica (Pittsburgh, PA)	mg/l	2	--	--	Not Applicable	--	Not Applicable	--	Not Applicable
Total Organic Carbon by USEPA SW-846 9060A											
Total Organic Carbon	--	Eurofins-TestAmerica (Pittsburgh, PA)	mg/l	1	--	--	Not Applicable	--	Not Applicable	--	Not Applicable
Dissolved Organic Carbon	--	Eurofins-TestAmerica (Pittsburgh, PA)	mg/l	1	--	--	Not Applicable	--	Not Applicable	--	Not Applicable
Oil and Grease by Method 1664B											
Oil and Grease	--	Eurofins-TestAmerica (Pittsburgh, PA)	mg/l	5	4.3	--	Not Applicable	--	Not Applicable	--	Not Applicable

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Analyte	CAS Number	Analytical Laboratory	Units	Laboratory Specified		RCRA Permit Concentration Limits[1]		OU-1/OU-2 ROD RGs Groundwater		OU-1/OU-2 ROD RGs Surface Water	
				RL	MDL	Value	Laboratory MDL < Selected Screening Value	Value	Laboratory MDL < Selected Screening Value	Value	Laboratory MDL < Selected Screening Value
Anions by Method 300.0											
Bromide	24959-67-9	Eurofins-TestAmerica (Pittsburgh, PA)	mg/l	0.1	--	--	Not Applicable	--	Not Applicable	--	Not Applicable
Chloride	16887-00-6	Eurofins-TestAmerica (Pittsburgh, PA)	mg/l	1	--	--	Not Applicable	--	Not Applicable	--	Not Applicable
Fluoride	16984-48-8	Eurofins-TestAmerica (Pittsburgh, PA)	mg/l	0.1	--	--	Not Applicable	--	Not Applicable	--	Not Applicable
Nitrate-N	14797-55-8	Eurofins-TestAmerica (Pittsburgh, PA)	mg/l	0.1	--	--	Not Applicable	--	Not Applicable	--	Not Applicable
Nitrite-N	14797-65-0	Eurofins-TestAmerica (Pittsburgh, PA)	mg/l	0.05	--	--	Not Applicable	--	Not Applicable	--	Not Applicable
Ortho-Phosphorous	7664-38-2	Eurofins-TestAmerica (Pittsburgh, PA)	mg/l	0.5	--	--	Not Applicable	--	Not Applicable	--	Not Applicable
Sulfate	14808-79-8	Eurofins-TestAmerica (Pittsburgh, PA)	mg/l	1	--	--	Not Applicable	--	Not Applicable	--	Not Applicable
Mercury by Method 7470A											
Mercury	7439-97-6	Eurofins-TestAmerica (Savannah, GA)	µg/L	0.2	0.08	2	Yes	--	Not Applicable	--	Not Applicable
Metals by Method 6010C											
Aluminum	7429-90-5	Eurofins-TestAmerica (Savannah, GA)	µg/L	200	54	--	Not Applicable	--	Not Applicable	--	Not Applicable
Antimony	7440-36-0	Eurofins-TestAmerica (Savannah, GA)	µg/L	20	9	--	Not Applicable	--	Not Applicable	--	Not Applicable
Arsenic	7440-38-2	Eurofins-TestAmerica (Savannah, GA)	µg/L	20	6.4	--	Not Applicable	--	Not Applicable	--	Not Applicable
Barium	7440-39-3	Eurofins-TestAmerica (Savannah, GA)	µg/L	10	4.4	--	Not Applicable	--	Not Applicable	--	Not Applicable
Beryllium	7440-41-7	Eurofins-TestAmerica (Savannah, GA)	µg/L	4	0.3	--	Not Applicable	--	Not Applicable	--	Not Applicable
Cadmium	7440-43-9	Eurofins-TestAmerica (Savannah, GA)	µg/L	5	0.44	--	Not Applicable	--	Not Applicable	--	Not Applicable
Calcium	7440-70-2	Eurofins-TestAmerica (Savannah, GA)	µg/L	500	165	--	Not Applicable	--	Not Applicable	--	Not Applicable
Chromium	7440-47-3	Eurofins-TestAmerica (Savannah, GA)	µg/L	10	1.1	--	Not Applicable	--	Not Applicable	74	Yes
Cobalt	7440-48-4	Eurofins-TestAmerica (Savannah, GA)	µg/L	10	1.4	694	Yes	--	Not Applicable	--	Not Applicable
Copper	7440-50-8	Eurofins-TestAmerica (Savannah, GA)	µg/L	20	3.2	--	Not Applicable	--	Not Applicable	--	Not Applicable
Iron	7439-89-6	Eurofins-TestAmerica (Savannah, GA)	µg/L	100	20	--	Not Applicable	--	Not Applicable	--	Not Applicable
Lead	7439-92-1	Eurofins-TestAmerica (Savannah, GA)	µg/L	10	6.6	--	Not Applicable	--	Not Applicable	2.5	No
Magnesium	7439-95-4	Eurofins-TestAmerica (Savannah, GA)	µg/L	500	28	--	Not Applicable	--	Not Applicable	--	Not Applicable
Manganese	7439-96-5	Eurofins-TestAmerica (Savannah, GA)	µg/L	10	1.3	--	Not Applicable	--	Not Applicable	--	Not Applicable
Nickel	7440-02-0	Eurofins-TestAmerica (Savannah, GA)	µg/L	40	3.3	--	Not Applicable	--	Not Applicable	--	Not Applicable
Potassium	7440-09-7	Eurofins-TestAmerica (Savannah, GA)	µg/L	1000	67	--	Not Applicable	--	Not Applicable	--	Not Applicable
Selenium	7782-49-2	Eurofins-TestAmerica (Savannah, GA)	µg/L	20	10	--	Not Applicable	--	Not Applicable	--	Not Applicable
Silver	7440-22-4	Eurofins-TestAmerica (Savannah, GA)	µg/L	10	1.5	--	Not Applicable	--	Not Applicable	--	Not Applicable
Sodium	7440-23-5	Eurofins-TestAmerica (Savannah, GA)	µg/L	2000	480	--	Not Applicable	--	Not Applicable	--	Not Applicable
Thallium	7440-28-0	Eurofins-TestAmerica (Savannah, GA)	µg/L	25	8	--	Not Applicable	--	Not Applicable	--	Not Applicable
Vanadium	7440-62-2	Eurofins-TestAmerica (Savannah, GA)	µg/L	10	1	--	Not Applicable	--	Not Applicable	--	Not Applicable
Zinc	7440-66-6	Eurofins-TestAmerica (Savannah, GA)	µg/L	20	8.7	--	Not Applicable	--	Not Applicable	--	Not Applicable
Hexavalent Chromium by USEPA SW-846 Method 7196A											
Hexavalent Chromium	18540-29-9	Eurofins-TestAmerica (Savannah, GA)	µg/L	10	3	--	Not Applicable	--	Not Applicable	11	Yes
Semivolatile Organic Compounds by Method 8270D											

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				RL	MDL	Value	Laboratory MDL < Selected Screening Value	Value	Laboratory MDL < Selected Screening Value	Value	Laboratory MDL < Selected Screening Value
1,1'-Biphenyl	92-52-4	Eurofins-TestAmerica (Savannah, GA)	µg/L	10	4.6	--	Not Applicable	--	Not Applicable	--	Not Applicable
2,4,5-Trichlorophenol	95-95-4	Eurofins-TestAmerica (Savannah, GA)	µg/L	10	3.3	--	Not Applicable	--	Not Applicable	--	Not Applicable
2,4,6-Trichlorophenol	88-06-2	Eurofins-TestAmerica (Savannah, GA)	µg/L	10	2.2	47	Yes	--	Not Applicable	--	Not Applicable
2,4-Dichlorophenol	120-83-2	Eurofins-TestAmerica (Savannah, GA)	µg/L	10	3.7	--	Not Applicable	--	Not Applicable	--	Not Applicable
2,4-Dimethylphenol	105-67-9	Eurofins-TestAmerica (Savannah, GA)	µg/L	10	3	--	Not Applicable	--	Not Applicable	--	Not Applicable
2,4-Dinitrophenol	51-28-5	Eurofins-TestAmerica (Savannah, GA)	µg/L	50	16	--	Not Applicable	--	Not Applicable	--	Not Applicable
2,4-Dinitrotoluene	121-14-2	Eurofins-TestAmerica (Savannah, GA)	µg/L	10	2.3	--	Not Applicable	--	Not Applicable	--	Not Applicable
2,6-Dinitrotoluene	606-20-2	Eurofins-TestAmerica (Savannah, GA)	µg/L	10	2.8	--	Not Applicable	--	Not Applicable	--	Not Applicable
2-Chloronaphthalene	91-58-7	Eurofins-TestAmerica (Savannah, GA)	µg/L	10	4.8	--	Not Applicable	--	Not Applicable	--	Not Applicable
2-Chlorophenol	95-57-8	Eurofins-TestAmerica (Savannah, GA)	µg/L	10	3.2	--	Not Applicable	--	Not Applicable	--	Not Applicable
2-Methylnaphthalene	91-57-6	Eurofins-TestAmerica (Savannah, GA)	µg/L	10	4.6	--	Not Applicable	--	Not Applicable	--	Not Applicable
2-Methylphenol	95-48-7	Eurofins-TestAmerica (Savannah, GA)	µg/L	10	1.8	--	Not Applicable	--	Not Applicable	--	Not Applicable
2-Nitroaniline	88-74-4	Eurofins-TestAmerica (Savannah, GA)	µg/L	50	2.9	--	Not Applicable	--	Not Applicable	--	Not Applicable
2-Nitrophenol	88-75-5	Eurofins-TestAmerica (Savannah, GA)	µg/L	10	3.9	--	Not Applicable	--	Not Applicable	--	Not Applicable
3 & 4 Methylphenol	15831-10-4	Eurofins-TestAmerica (Savannah, GA)	µg/L	10	1.4	--	Not Applicable	--	Not Applicable	--	Not Applicable
3,3'-Dichlorobenzidine	91-94-1	Eurofins-TestAmerica (Savannah, GA)	µg/L	60	10	--	Not Applicable	--	Not Applicable	--	Not Applicable
3-Nitroaniline	99-09-2	Eurofins-TestAmerica (Savannah, GA)	µg/L	50	3	--	Not Applicable	--	Not Applicable	--	Not Applicable
4,6-Dinitro-2-methylphenol	534-52-1	Eurofins-TestAmerica (Savannah, GA)	µg/L	50	5.3	--	Not Applicable	--	Not Applicable	--	Not Applicable
4-Bromophenyl phenyl ether	101-55-3	Eurofins-TestAmerica (Savannah, GA)	µg/L	10	4.7	--	Not Applicable	--	Not Applicable	--	Not Applicable
4-Chloro-3-methylphenol	59-50-7	Eurofins-TestAmerica (Savannah, GA)	µg/L	10	2.5	--	Not Applicable	--	Not Applicable	--	Not Applicable
4-Chloroaniline	106-47-8	Eurofins-TestAmerica (Savannah, GA)	µg/L	20	1	--	Not Applicable	--	Not Applicable	--	Not Applicable
4-Chlorophenyl phenyl ether	7005-72-3	Eurofins-TestAmerica (Savannah, GA)	µg/L	10	5.3	--	Not Applicable	--	Not Applicable	--	Not Applicable
4-Nitroaniline	100-01-6	Eurofins-TestAmerica (Savannah, GA)	µg/L	50	3	--	Not Applicable	--	Not Applicable	--	Not Applicable
4-Nitrophenol	100-02-7	Eurofins-TestAmerica (Savannah, GA)	µg/L	50	8.1	128	Yes	--	Not Applicable	--	Not Applicable
Acenaphthene	83-32-9	Eurofins-TestAmerica (Savannah, GA)	µg/L	10	4.4	--	Not Applicable	--	Not Applicable	--	Not Applicable
Acenaphthylene	208-96-8	Eurofins-TestAmerica (Savannah, GA)	µg/L	10	3.6	--	Not Applicable	--	Not Applicable	--	Not Applicable
Acetophenone	98-86-2	Eurofins-TestAmerica (Savannah, GA)	µg/L	10	2.2	--	Not Applicable	--	Not Applicable	--	Not Applicable
Anthracene	120-12-7	Eurofins-TestAmerica (Savannah, GA)	µg/L	10	3.6	--	Not Applicable	--	Not Applicable	--	Not Applicable
Atrazine	1912-24-9	Eurofins-TestAmerica (Savannah, GA)	µg/L	10	2.2	--	Not Applicable	--	Not Applicable	--	Not Applicable
Benzaldehyde	100-52-7	Eurofins-TestAmerica (Savannah, GA)	µg/L	10	4.8	--	Not Applicable	--	Not Applicable	--	Not Applicable
Benzo[a]anthracene	56-55-3	Eurofins-TestAmerica (Savannah, GA)	µg/L	10	4.2	--	Not Applicable	--	Not Applicable	--	Not Applicable
Benzo[a]pyrene	50-32-8	Eurofins-TestAmerica (Savannah, GA)	µg/L	10	2.8	--	Not Applicable	--	Not Applicable	--	Not Applicable
Benzo[b]fluoranthene	205-99-2	Eurofins-TestAmerica (Savannah, GA)	µg/L	10	3.1	--	Not Applicable	--	Not Applicable	--	Not Applicable
Benzo[g,h,i]perylene	191-24-2	Eurofins-TestAmerica (Savannah, GA)	µg/L	10	3	--	Not Applicable	--	Not Applicable	--	Not Applicable

UFP-QAPP Worksheet #15: Reference Limits and Evaluation Table

Analyte	CAS Number	Analytical Laboratory	Units	Laboratory Specified		RCRA Permit Concentration Limits[1]		OU-1/OU-2 ROD RGs Groundwater		OU-1/OU-2 ROD RGs Surface Water	
				RL	MDL	Value	Laboratory MDL < Selected Screening Value	Value	Laboratory MDL < Selected Screening Value	Value	Laboratory MDL < Selected Screening Value
Benzo[k]fluoranthene	207-08-9	Eurofins-TestAmerica (Savannah, GA)	µg/L	10	2.9	--	Not Applicable	--	Not Applicable	--	Not Applicable
bis (2-chloroisopropyl) ether	108-60-1	Eurofins-TestAmerica (Savannah, GA)	µg/L	10	3.1	--	Not Applicable	--	Not Applicable	--	Not Applicable
Bis(2-chloroethoxy)methane	111-91-1	Eurofins-TestAmerica (Savannah, GA)	µg/L	10	1.8	--	Not Applicable	--	Not Applicable	--	Not Applicable
Bis(2-chloroethyl)ether	111-44-4	Eurofins-TestAmerica (Savannah, GA)	µg/L	10	3.6	--	Not Applicable	--	Not Applicable	--	Not Applicable
Bis(2-ethylhexyl) phthalate	117-81-7	Eurofins-TestAmerica (Savannah, GA)	µg/L	10	3	--	Not Applicable	--	Not Applicable	--	Not Applicable
Butyl benzyl phthalate	85-68-7	Eurofins-TestAmerica (Savannah, GA)	µg/L	10	2.8	--	Not Applicable	--	Not Applicable	--	Not Applicable
Caprolactam	105-60-2	Eurofins-TestAmerica (Savannah, GA)	µg/L	10	2.8	--	Not Applicable	--	Not Applicable	--	Not Applicable
Carbazole	86-74-8	Eurofins-TestAmerica (Savannah, GA)	µg/L	10	1.8	--	Not Applicable	--	Not Applicable	--	Not Applicable
Chrysene	218-01-9	Eurofins-TestAmerica (Savannah, GA)	µg/L	10	3.8	--	Not Applicable	--	Not Applicable	--	Not Applicable
Dibenz(a,h)anthracene	53-70-3	Eurofins-TestAmerica (Savannah, GA)	µg/L	10	3	--	Not Applicable	--	Not Applicable	--	Not Applicable
Dibenzofuran	132-64-9	Eurofins-TestAmerica (Savannah, GA)	µg/L	10	1.9	--	Not Applicable	--	Not Applicable	--	Not Applicable
Diethyl phthalate	84-66-2	Eurofins-TestAmerica (Savannah, GA)	µg/L	10	2.2	--	Not Applicable	--	Not Applicable	--	Not Applicable
Dimethyl phthalate	131-11-3	Eurofins-TestAmerica (Savannah, GA)	µg/L	10	2.1	--	Not Applicable	--	Not Applicable	--	Not Applicable
Di-n-butyl phthalate	84-74-2	Eurofins-TestAmerica (Savannah, GA)	µg/L	10	2.3	--	Not Applicable	--	Not Applicable	--	Not Applicable
Di-n-octyl phthalate	117-84-0	Eurofins-TestAmerica (Savannah, GA)	µg/L	10	2.8	--	Not Applicable	--	Not Applicable	--	Not Applicable
Fluoranthene	206-44-0	Eurofins-TestAmerica (Savannah, GA)	µg/L	10	1.6	--	Not Applicable	--	Not Applicable	--	Not Applicable
Fluorene	86-73-7	Eurofins-TestAmerica (Savannah, GA)	µg/L	10	3.4	--	Not Applicable	--	Not Applicable	--	Not Applicable
Hexachlorobenzene	118-74-1	Eurofins-TestAmerica (Savannah, GA)	µg/L	10	1.6	--	Not Applicable	--	Not Applicable	--	Not Applicable
Hexachlorobutadiene	87-68-3	Eurofins-TestAmerica (Savannah, GA)	µg/L	10	4	--	Not Applicable	--	Not Applicable	--	Not Applicable
Hexachlorocyclopentadiene	77-47-4	Eurofins-TestAmerica (Savannah, GA)	µg/L	10	3.5	--	Not Applicable	--	Not Applicable	--	Not Applicable
Hexachloroethane	67-72-1	Eurofins-TestAmerica (Savannah, GA)	µg/L	10	3.6	--	Not Applicable	--	Not Applicable	--	Not Applicable
Indeno[1,2,3-cd]pyrene	193-39-5	Eurofins-TestAmerica (Savannah, GA)	µg/L	10	4.3	--	Not Applicable	--	Not Applicable	--	Not Applicable
Isophorone	78-59-1	Eurofins-TestAmerica (Savannah, GA)	µg/L	10	2	--	Not Applicable	--	Not Applicable	--	Not Applicable
Naphthalene	91-20-3	Eurofins-TestAmerica (Savannah, GA)	µg/L	10	3.5	--	Not Applicable	--	Not Applicable	--	Not Applicable
Nitrobenzene	98-95-3	Eurofins-TestAmerica (Savannah, GA)	µg/L	10	3.5	--	Not Applicable	--	Not Applicable	--	Not Applicable
N-Nitrosodi-n-propylamine	621-64-7	Eurofins-TestAmerica (Savannah, GA)	µg/L	10	2.4	--	Not Applicable	--	Not Applicable	--	Not Applicable
N-Nitrosodiphenylamine	86-30-6	Eurofins-TestAmerica (Savannah, GA)	µg/L	10	2.9	--	Not Applicable	--	Not Applicable	--	Not Applicable
Pentachlorophenol	87-86-5	Eurofins-TestAmerica (Savannah, GA)	µg/L	50	9.9	1	No	--	Not Applicable	--	Not Applicable
Phenanthrene	85-01-8	Eurofins-TestAmerica (Savannah, GA)	µg/L	10	4.3	--	Not Applicable	--	Not Applicable	--	Not Applicable
Phenol	108-95-2	Eurofins-TestAmerica (Savannah, GA)	µg/L	10	1.5	--	Not Applicable	--	Not Applicable	--	Not Applicable
Pyrene	129-00-0	Eurofins-TestAmerica (Savannah, GA)	µg/L	10	3.7	--	Not Applicable	--	Not Applicable	--	Not Applicable
Volatile Organic Compounds by Method 8260C											
1,1,1-Trichloroethane	71-55-6	Eurofins-TestAmerica (Savannah, GA)	µg/L	1	0.21	--	Not Applicable	--	Not Applicable	--	Not Applicable
1,1,2,2-Tetrachloroethane	79-34-5	Eurofins-TestAmerica (Savannah, GA)	µg/L	1	0.4	--	Not Applicable	--	Not Applicable	--	Not Applicable
1,1,2-Trichloro-1,2,2-trifluoroethane	76-13-1	Eurofins-TestAmerica (Savannah, GA)	µg/L	1	0.23	--	Not Applicable	--	Not Applicable	--	Not Applicable

UFP-QAPP Worksheet #15: Reference Limits and Evaluation Table

Analyte	CAS Number	Analytical Laboratory	Units	Laboratory Specified		RCRA Permit Concentration Limits[1]		OU-1/OU-2 ROD RGs Groundwater		OU-1/OU-2 ROD RGs Surface Water	
				RL	MDL	Value	Laboratory MDL < Selected Screening Value	Value	Laboratory MDL < Selected Screening Value	Value	Laboratory MDL < Selected Screening Value
1,1,2-Trichloroethane	79-00-5	Eurofins-TestAmerica (Savannah, GA)	µg/L	1	0.32	--	Not Applicable	--	Not Applicable	--	Not Applicable
1,1-Dichloroethane	75-34-3	Eurofins-TestAmerica (Savannah, GA)	µg/L	1	0.33	--	Not Applicable	--	Not Applicable	--	Not Applicable
1,1-Dichloroethene	75-35-4	Eurofins-TestAmerica (Savannah, GA)	µg/L	1	0.33	--	Not Applicable	--	Not Applicable	--	Not Applicable
1,2,4-Trichlorobenzene	120-82-1	Eurofins-TestAmerica (Savannah, GA)	µg/L	5	0.53	--	Not Applicable	--	Not Applicable	--	Not Applicable
1,2-Dibromo-3-Chloropropane	96-12-8	Eurofins-TestAmerica (Savannah, GA)	µg/L	5	1.8	--	Not Applicable	--	Not Applicable	--	Not Applicable
1,2-Dichlorobenzene	95-50-1	Eurofins-TestAmerica (Savannah, GA)	µg/L	1	0.31	612	Yes	--	Not Applicable	--	Not Applicable
1,2-Dichloroethane	107-06-2	Eurofins-TestAmerica (Savannah, GA)	µg/L	1	0.25	--	Not Applicable	--	Not Applicable	--	Not Applicable
1,2-Dichloropropane	78-87-5	Eurofins-TestAmerica (Savannah, GA)	µg/L	1	0.22	--	Not Applicable	--	Not Applicable	--	Not Applicable
1,3-Dichlorobenzene	541-73-1	Eurofins-TestAmerica (Savannah, GA)	µg/L	1	0.31	--	Not Applicable	--	Not Applicable	--	Not Applicable
1,4-Dichlorobenzene	106-46-7	Eurofins-TestAmerica (Savannah, GA)	µg/L	1	0.31	77	Yes	--	Not Applicable	--	Not Applicable
2-Butanone (MEK)	78-93-3	Eurofins-TestAmerica (Savannah, GA)	µg/L	10	6.4	--	Not Applicable	--	Not Applicable	--	Not Applicable
2-Hexanone	591-78-6	Eurofins-TestAmerica (Savannah, GA)	µg/L	10	3.2	--	Not Applicable	--	Not Applicable	--	Not Applicable
4-Methyl-2-pentanone (MIBK)	108-10-1	Eurofins-TestAmerica (Savannah, GA)	µg/L	10	2.7	--	Not Applicable	--	Not Applicable	--	Not Applicable
Acetone	67-64-1	Eurofins-TestAmerica (Savannah, GA)	µg/L	10	3.7	--	Not Applicable	--	Not Applicable	--	Not Applicable
Benzene	71-43-2	Eurofins-TestAmerica (Savannah, GA)	µg/L	1	0.27	--	Not Applicable	--	Not Applicable	--	Not Applicable
Bromoform	75-25-2	Eurofins-TestAmerica (Savannah, GA)	µg/L	1	0.59	--	Not Applicable	--	Not Applicable	--	Not Applicable
Bromomethane	74-83-9	Eurofins-TestAmerica (Savannah, GA)	µg/L	5	3.7	--	Not Applicable	--	Not Applicable	--	Not Applicable
Carbon disulfide	75-15-0	Eurofins-TestAmerica (Savannah, GA)	µg/L	2	0.43	--	Not Applicable	--	Not Applicable	--	Not Applicable
Carbon tetrachloride	56-23-5	Eurofins-TestAmerica (Savannah, GA)	µg/L	1	0.3	--	Not Applicable	--	Not Applicable	--	Not Applicable
Chlorobenzene	108-90-7	Eurofins-TestAmerica (Savannah, GA)	µg/L	1	0.15	102	Yes	--	Not Applicable	--	Not Applicable
Chlorodibromomethane	124-48-1	Eurofins-TestAmerica (Savannah, GA)	µg/L	1	0.39	--	Not Applicable	--	Not Applicable	--	Not Applicable
Chloroethane	75-00-3	Eurofins-TestAmerica (Savannah, GA)	µg/L	5	4.6	--	Not Applicable	--	Not Applicable	--	Not Applicable
Chloroform	67-66-3	Eurofins-TestAmerica (Savannah, GA)	µg/L	1	0.27	--	Not Applicable	--	Not Applicable	--	Not Applicable
Chloromethane	74-87-3	Eurofins-TestAmerica (Savannah, GA)	µg/L	1	0.54	--	Not Applicable	--	Not Applicable	--	Not Applicable
cis-1,2-Dichloroethene	156-59-2	Eurofins-TestAmerica (Savannah, GA)	µg/L	1	0.25	--	Not Applicable	--	Not Applicable	--	Not Applicable
cis-1,3-Dichloropropene	10061-01-5	Eurofins-TestAmerica (Savannah, GA)	µg/L	1	0.26	--	Not Applicable	--	Not Applicable	--	Not Applicable
Cyclohexane	110-82-7	Eurofins-TestAmerica (Savannah, GA)	µg/L	1	0.28	--	Not Applicable	--	Not Applicable	--	Not Applicable
Dichlorobromomethane	75-27-4	Eurofins-TestAmerica (Savannah, GA)	µg/L	1	0.25	--	Not Applicable	--	Not Applicable	--	Not Applicable
Dichlorodifluoromethane	75-71-8	Eurofins-TestAmerica (Savannah, GA)	µg/L	1	0.36	--	Not Applicable	--	Not Applicable	--	Not Applicable
Ethylbenzene	100-41-4	Eurofins-TestAmerica (Savannah, GA)	µg/L	1	0.2	--	Not Applicable	--	Not Applicable	--	Not Applicable
Ethylene Dibromide	106-93-4	Eurofins-TestAmerica (Savannah, GA)	µg/L	1	0.33	--	Not Applicable	--	Not Applicable	--	Not Applicable
Isopropylbenzene	98-82-8	Eurofins-TestAmerica (Savannah, GA)	µg/L	1	0.26	--	Not Applicable	--	Not Applicable	--	Not Applicable
Methyl acetate	79-20-9	Eurofins-TestAmerica (Savannah, GA)	µg/L	5	3.2	--	Not Applicable	--	Not Applicable	--	Not Applicable
Methyl tert-butyl ether	1634-04-4	Eurofins-TestAmerica (Savannah, GA)	µg/L	5	0.81	--	Not Applicable	--	Not Applicable	--	Not Applicable

UFP-QAPP Worksheet #15: Reference Limits and Evaluation Table

Analyte	CAS Number	Analytical Laboratory	Units	Laboratory Specified		RCRA Permit Concentration Limits[1]		OU-1/OU-2 ROD RGs Groundwater		OU-1/OU-2 ROD RGs Surface Water	
				RL	MDL	Value	Laboratory MDL < Selected Screening Value	Value	Laboratory MDL < Selected Screening Value	Value	Laboratory MDL < Selected Screening Value
Methylcyclohexane	108-87-2	Eurofins-TestAmerica (Savannah, GA)	µg/L	1	0.33	--	Not Applicable	--	Not Applicable	--	Not Applicable
Methylene Chloride	75-09-2	Eurofins-TestAmerica (Savannah, GA)	µg/L	5	3.2	--	Not Applicable	--	Not Applicable	--	Not Applicable
Styrene	100-42-5	Eurofins-TestAmerica (Savannah, GA)	µg/L	1	0.27	--	Not Applicable	--	Not Applicable	--	Not Applicable
Tetrachloroethene	127-18-4	Eurofins-TestAmerica (Savannah, GA)	µg/L	0.5	0.35	--	Not Applicable	--	Not Applicable	--	Not Applicable
Toluene	108-88-3	Eurofins-TestAmerica (Savannah, GA)	µg/L	1	0.25	--	Not Applicable	--	Not Applicable	--	Not Applicable
trans-1,2-Dichloroethene	156-60-5	Eurofins-TestAmerica (Savannah, GA)	µg/L	1	0.34	--	Not Applicable	--	Not Applicable	--	Not Applicable
trans-1,3-Dichloropropene	10061-02-6	Eurofins-TestAmerica (Savannah, GA)	µg/L	1	0.23	--	Not Applicable	--	Not Applicable	--	Not Applicable
Trichloroethene	79-01-6	Eurofins-TestAmerica (Savannah, GA)	µg/L	1	0.2	--	Not Applicable	--	Not Applicable	--	Not Applicable
Trichlorofluoromethane	75-69-4	Eurofins-TestAmerica (Savannah, GA)	µg/L	1	0.33	--	Not Applicable	--	Not Applicable	--	Not Applicable
Vinyl chloride	75-01-4	Eurofins-TestAmerica (Savannah, GA)	µg/L	1	0.4	--	Not Applicable	--	Not Applicable	--	Not Applicable
Xylenes, Total	1330-20-7	Eurofins-TestAmerica (Savannah, GA)	µg/L	1	0.23	--	Not Applicable	--	Not Applicable	--	Not Applicable

Notes:

1. Listed for point of compliance monitoring in RCRA permit # ALD 004 019 048

CAS: Chemical Abstracts Service
GA: Georgia
MDL: method detection limit
µg/L: micrograms per liter
mg/l: milligrams per liter
ng/L: nanograms per liter
OU-1: Operable Unit 1
OU-2: Operable Unit 2
PA: Pennsylvania
RCRA: Resource Conservation and Recovery Act
RG: remedial goal
RL: reporting limit
ROD: record of decision
TN: Tennessee
USEPA: United States Environmental Protection Agency
VT: Vermont

UFP-QAPP Worksheet #15: Reference Limits and Evaluation Table

Analyte	CAS Number	Analytical Laboratory	Units	Laboratory Specified		OU-1/OU-2 ROD RGs Soil		OU-1/OU-2 ROD RGs Sediment	
				RL	MDL	Value	Laboratory MDL < Selected Screening Value	Value	Laboratory MDL < Selected Screening Value
Polychlorinated biphenyl (PCB) Aroclors by EPA SW-846 Method 8082A									
Aroclor 1016	12674-11-2	Eurofins-TestAmerica (Burlington, VT)	mg/kg	0.017	0.0027	1	Yes	3	Yes
Aroclor 1221	11104-28-2	Eurofins-TestAmerica (Burlington, VT)	mg/kg	0.017	0.0042	1	Yes	3	Yes
Aroclor 1232	11141-16-5	Eurofins-TestAmerica (Burlington, VT)	mg/kg	0.017	0.0033	1	Yes	3	Yes
Aroclor 1242	53469-21-9	Eurofins-TestAmerica (Burlington, VT)	mg/kg	0.017	0.0017	1	Yes	3	Yes
Aroclor 1248	12672-29-6	Eurofins-TestAmerica (Burlington, VT)	mg/kg	0.017	0.0027	1	Yes	3	Yes
Aroclor 1254	11097-69-1	Eurofins-TestAmerica (Burlington, VT)	mg/kg	0.017	0.0043	1	Yes	3	Yes
Aroclor 1260	11096-82-5	Eurofins-TestAmerica (Burlington, VT)	mg/kg	0.017	0.0032	1	Yes	3	Yes
Aroclor 1262	37324-23-5	Eurofins-TestAmerica (Burlington, VT)	mg/kg	0.017	0.0027	1	Yes	3	Yes
Aroclor 1268	11100-14-4	Eurofins-TestAmerica (Burlington, VT)	mg/kg	0.017	0.0033	1	Yes	3	Yes
PCBs, Total	1336-36-3	Eurofins-TestAmerica (Burlington, VT)	mg/kg	0.017	0.0017	--	Not Applicable	--	Not Applicable
PCB Homologs by EPA SW-846 Method 680									
Mono - CB	27323-18-8	Eurofins-TestAmerica (Burlington, VT)	mg/kg	0	0	--	Not Applicable	--	Not Applicable
Di - CB	25512-42-9	Eurofins-TestAmerica (Burlington, VT)	mg/kg	0	0	--	Not Applicable	--	Not Applicable
Tri - CB	25323-68-6	Eurofins-TestAmerica (Burlington, VT)	mg/kg	0	0	--	Not Applicable	--	Not Applicable
Tetra - CB	26914-33-0	Eurofins-TestAmerica (Burlington, VT)	mg/kg	0	0	--	Not Applicable	--	Not Applicable
Penta - CB	25429-29-2	Eurofins-TestAmerica (Burlington, VT)	mg/kg	0	0	--	Not Applicable	--	Not Applicable
Hexa - CB	26601-64-9	Eurofins-TestAmerica (Burlington, VT)	mg/kg	0	0	--	Not Applicable	--	Not Applicable
Hepta - CB	28655-71-2	Eurofins-TestAmerica (Burlington, VT)	mg/kg	0	0	--	Not Applicable	--	Not Applicable
Octa - CB	55722-26-4	Eurofins-TestAmerica (Burlington, VT)	mg/kg	0	0	--	Not Applicable	--	Not Applicable
Nona - CB	53742-07-7	Eurofins-TestAmerica (Burlington, VT)	mg/kg	0	0	--	Not Applicable	--	Not Applicable
Deca - CB	2051-24-3	Eurofins-TestAmerica (Burlington, VT)	mg/kg	0	0	--	Not Applicable	--	Not Applicable
Total Organic Carbon (TOC) by Lloyd Khan									
TOC	7440-440-0	Eurofins-TestAmerica (Burlington, VT)	mg/kg	1000	815	--	Not Applicable	--	Not Applicable
Mercury by Method 7471B									
Mercury	7439-97-6	Eurofins-TestAmerica (Savannah, GA)	mg/kg	0.025	0.008	--	Not Applicable	1	Yes
Metals by Method 6010C									
Aluminum	7429-90-5	Eurofins-TestAmerica (Savannah, GA)	mg/kg	20	6.8	--	Not Applicable	--	Not Applicable
Antimony	7440-36-0	Eurofins-TestAmerica (Savannah, GA)	mg/kg	2	0.87	--	Not Applicable	--	Not Applicable
Arsenic	7440-38-2	Eurofins-TestAmerica (Savannah, GA)	mg/kg	2	1.2	382	Yes	--	Not Applicable
Barium	7440-39-3	Eurofins-TestAmerica (Savannah, GA)	mg/kg	1	0.41	--	Not Applicable	322	Yes
Beryllium	7440-41-7	Eurofins-TestAmerica (Savannah, GA)	mg/kg	0.4	0.071	--	Not Applicable	--	Not Applicable

UFP-QAPP Worksheet #15: Reference Limits and Evaluation Table

Analyte	CAS Number	Analytical Laboratory	Units	Laboratory Specified		OU-1/OU-2 ROD RGs Soil		OU-1/OU-2 ROD RGs Sediment	
				RL	MDL	Value	Laboratory MDL < Selected Screening Value	Value	Laboratory MDL < Selected Screening Value
Cadmium	7440-43-9	Eurofins-TestAmerica (Savannah, GA)	mg/kg	0.5	0.1	--	Not Applicable	--	Not Applicable
Calcium	7440-70-2	Eurofins-TestAmerica (Savannah, GA)	mg/kg	50	7.4	--	Not Applicable	--	Not Applicable
Chromium	7440-47-3	Eurofins-TestAmerica (Savannah, GA)	mg/kg	1	0.17	568	Yes	111	Yes
Cobalt	7440-48-4	Eurofins-TestAmerica (Savannah, GA)	mg/kg	1	0.33	--	Not Applicable	59	Yes
Copper	7440-50-8	Eurofins-TestAmerica (Savannah, GA)	mg/kg	2.5	0.5	--	Not Applicable	--	Not Applicable
Iron	7439-89-6	Eurofins-TestAmerica (Savannah, GA)	mg/kg	20	12	--	Not Applicable	--	Not Applicable
Lead	7439-92-1	Eurofins-TestAmerica (Savannah, GA)	mg/kg	1	0.61	--	Not Applicable	128	Yes
Magnesium	7439-95-4	Eurofins-TestAmerica (Savannah, GA)	mg/kg	50	9.1	--	Not Applicable	--	Not Applicable
Manganese	7439-96-5	Eurofins-TestAmerica (Savannah, GA)	mg/kg	1	0.2	--	Not Applicable	1100	Yes
Nickel	7440-02-0	Eurofins-TestAmerica (Savannah, GA)	mg/kg	4	0.9	--	Not Applicable	46	Yes
Potassium	7440-09-7	Eurofins-TestAmerica (Savannah, GA)	mg/kg	100	11	--	Not Applicable	--	Not Applicable
Selenium	7782-49-2	Eurofins-TestAmerica (Savannah, GA)	mg/kg	2.5	1.1	--	Not Applicable	--	Not Applicable
Silver	7440-22-4	Eurofins-TestAmerica (Savannah, GA)	mg/kg	1	0.32	--	Not Applicable	--	Not Applicable
Sodium	7440-23-5	Eurofins-TestAmerica (Savannah, GA)	mg/kg	200	55	--	Not Applicable	--	Not Applicable
Thallium	7440-28-0	Eurofins-TestAmerica (Savannah, GA)	mg/kg	2.5	0.78	--	Not Applicable	--	Not Applicable
Vanadium	7440-62-2	Eurofins-TestAmerica (Savannah, GA)	mg/kg	1	0.16	--	Not Applicable	41	Yes
Zinc	7440-66-6	Eurofins-TestAmerica (Savannah, GA)	mg/kg	2	0.58	--	Not Applicable	--	Not Applicable
Semivolatile Organic Compounds by Method 8270D									
1,1'-Biphenyl	92-52-4	Eurofins-TestAmerica (Savannah, GA)	µg/kg	1700	1700	--	Not Applicable	--	Not Applicable
2,4,5-Trichlorophenol	95-95-4	Eurofins-TestAmerica (Savannah, GA)	µg/kg	330	35	--	Not Applicable	--	Not Applicable
2,4,6-Trichlorophenol	88-06-2	Eurofins-TestAmerica (Savannah, GA)	µg/kg	330	29	--	Not Applicable	--	Not Applicable
2,4-Dichlorophenol	120-83-2	Eurofins-TestAmerica (Savannah, GA)	µg/kg	330	35	--	Not Applicable	--	Not Applicable
2,4-Dimethylphenol	105-67-9	Eurofins-TestAmerica (Savannah, GA)	µg/kg	330	44	--	Not Applicable	--	Not Applicable
2,4-Dinitrophenol	51-28-5	Eurofins-TestAmerica (Savannah, GA)	µg/kg	1700	830	--	Not Applicable	--	Not Applicable
2,4-Dinitrotoluene	121-14-2	Eurofins-TestAmerica (Savannah, GA)	µg/kg	330	49	--	Not Applicable	--	Not Applicable
2,6-Dinitrotoluene	606-20-2	Eurofins-TestAmerica (Savannah, GA)	µg/kg	330	42	--	Not Applicable	--	Not Applicable
2-Chloronaphthalene	91-58-7	Eurofins-TestAmerica (Savannah, GA)	µg/kg	330	35	--	Not Applicable	--	Not Applicable
2-Chlorophenol	95-57-8	Eurofins-TestAmerica (Savannah, GA)	µg/kg	330	40	--	Not Applicable	--	Not Applicable
2-Methylnaphthalene	91-57-6	Eurofins-TestAmerica (Savannah, GA)	µg/kg	330	38	--	Not Applicable	--	Not Applicable
2-Methylphenol	95-48-7	Eurofins-TestAmerica (Savannah, GA)	µg/kg	330	27	--	Not Applicable	--	Not Applicable
2-Nitroaniline	88-74-4	Eurofins-TestAmerica (Savannah, GA)	µg/kg	1700	45	--	Not Applicable	--	Not Applicable
2-Nitrophenol	88-75-5	Eurofins-TestAmerica (Savannah, GA)	µg/kg	330	41	--	Not Applicable	--	Not Applicable
3 & 4 Methylphenol	15831-10-4	Eurofins-TestAmerica (Savannah, GA)	µg/kg	330	43	--	Not Applicable	--	Not Applicable

UFP-QAPP Worksheet #15: Reference Limits and Evaluation Table

Analyte	CAS Number	Analytical Laboratory	Units	Laboratory Specified		OU-1/OU-2 ROD RGs Soil		OU-1/OU-2 ROD RGs Sediment	
				RL	MDL	Value	Laboratory MDL < Selected Screening Value	Value	Laboratory MDL < Selected Screening Value
3,3'-Dichlorobenzidine	91-94-1	Eurofins-TestAmerica (Savannah, GA)	µg/kg	660	28	--	Not Applicable	--	Not Applicable
3-Nitroaniline	99-09-2	Eurofins-TestAmerica (Savannah, GA)	µg/kg	1700	46	--	Not Applicable	--	Not Applicable
4,6-Dinitro-2-methylphenol	534-52-1	Eurofins-TestAmerica (Savannah, GA)	µg/kg	1700	170	--	Not Applicable	--	Not Applicable
4-Bromophenyl phenyl ether	101-55-3	Eurofins-TestAmerica (Savannah, GA)	µg/kg	330	36	--	Not Applicable	--	Not Applicable
4-Chloro-3-methylphenol	59-50-7	Eurofins-TestAmerica (Savannah, GA)	µg/kg	330	35	--	Not Applicable	--	Not Applicable
4-Chloroaniline	106-47-8	Eurofins-TestAmerica (Savannah, GA)	µg/kg	660	52	--	Not Applicable	--	Not Applicable
4-Chlorophenyl phenyl ether	7005-72-3	Eurofins-TestAmerica (Savannah, GA)	µg/kg	330	44	--	Not Applicable	--	Not Applicable
4-Nitroaniline	100-01-6	Eurofins-TestAmerica (Savannah, GA)	µg/kg	1700	49	--	Not Applicable	--	Not Applicable
4-Nitrophenol	100-02-7	Eurofins-TestAmerica (Savannah, GA)	µg/kg	1700	330	--	Not Applicable	--	Not Applicable
Acenaphthene	83-32-9	Eurofins-TestAmerica (Savannah, GA)	µg/kg	330	41	--	Not Applicable	--	Not Applicable
Acenaphthylene	208-96-8	Eurofins-TestAmerica (Savannah, GA)	µg/kg	330	36	--	Not Applicable	--	Not Applicable
Acetophenone	98-86-2	Eurofins-TestAmerica (Savannah, GA)	µg/kg	330	28	--	Not Applicable	--	Not Applicable
Anthracene	120-12-7	Eurofins-TestAmerica (Savannah, GA)	µg/kg	330	25	--	Not Applicable	--	Not Applicable
Atrazine	1912-24-9	Eurofins-TestAmerica (Savannah, GA)	µg/kg	330	23	--	Not Applicable	--	Not Applicable
Benzaldehyde	100-52-7	Eurofins-TestAmerica (Savannah, GA)	µg/kg	330	58	--	Not Applicable	--	Not Applicable
Benzo[a]anthracene	56-55-3	Eurofins-TestAmerica (Savannah, GA)	µg/kg	330	27	--	Not Applicable	--	Not Applicable
Benzo[a]pyrene	50-32-8	Eurofins-TestAmerica (Savannah, GA)	µg/kg	330	52	--	Not Applicable	--	Not Applicable
Benzo[b]fluoranthene	205-99-2	Eurofins-TestAmerica (Savannah, GA)	µg/kg	330	38	--	Not Applicable	--	Not Applicable
Benzo[g,h,i]perylene	191-24-2	Eurofins-TestAmerica (Savannah, GA)	µg/kg	330	22	--	Not Applicable	--	Not Applicable
Benzo[k]fluoranthene	207-08-9	Eurofins-TestAmerica (Savannah, GA)	µg/kg	330	65	--	Not Applicable	--	Not Applicable
bis (2-chloroisopropyl) ether	108-60-1	Eurofins-TestAmerica (Savannah, GA)	µg/kg	330	30	--	Not Applicable	--	Not Applicable
Bis(2-chloroethoxy)methane	111-91-1	Eurofins-TestAmerica (Savannah, GA)	µg/kg	330	39	--	Not Applicable	--	Not Applicable
Bis(2-chloroethyl)ether	111-44-4	Eurofins-TestAmerica (Savannah, GA)	µg/kg	330	45	--	Not Applicable	--	Not Applicable
Bis(2-ethylhexyl) phthalate	117-81-7	Eurofins-TestAmerica (Savannah, GA)	µg/kg	330	29	--	Not Applicable	--	Not Applicable
Butyl benzyl phthalate	85-68-7	Eurofins-TestAmerica (Savannah, GA)	µg/kg	330	26	--	Not Applicable	--	Not Applicable
Caprolactam	105-60-2	Eurofins-TestAmerica (Savannah, GA)	µg/kg	330	66	--	Not Applicable	--	Not Applicable
Carbazole	86-74-8	Eurofins-TestAmerica (Savannah, GA)	µg/kg	330	30	--	Not Applicable	--	Not Applicable
Chrysene	218-01-9	Eurofins-TestAmerica (Savannah, GA)	µg/kg	330	21	--	Not Applicable	--	Not Applicable
Dibenz(a,h)anthracene	53-70-3	Eurofins-TestAmerica (Savannah, GA)	µg/kg	330	39	--	Not Applicable	--	Not Applicable
Dibenzofuran	132-64-9	Eurofins-TestAmerica (Savannah, GA)	µg/kg	330	33	--	Not Applicable	--	Not Applicable
Diethyl phthalate	84-66-2	Eurofins-TestAmerica (Savannah, GA)	µg/kg	330	37	--	Not Applicable	--	Not Applicable
Di-n-butyl phthalate	84-74-2	Eurofins-TestAmerica (Savannah, GA)	µg/kg	330	30	--	Not Applicable	--	Not Applicable
Di-n-octyl phthalate	117-84-0	Eurofins-TestAmerica (Savannah, GA)	µg/kg	330	29	--	Not Applicable	--	Not Applicable

UFP-QAPP Worksheet #15: Reference Limits and Evaluation Table

Analyte	CAS Number	Analytical Laboratory	Units	Laboratory Specified		OU-1/OU-2 ROD RGs Soil		OU-1/OU-2 ROD RGs Sediment	
				RL	MDL	Value	Laboratory MDL < Selected Screening Value	Value	Laboratory MDL < Selected Screening Value
Fluoranthene	206-44-0	Eurofins-TestAmerica (Savannah, GA)	µg/kg	330	32	--	Not Applicable	--	Not Applicable
Fluorene	86-73-7	Eurofins-TestAmerica (Savannah, GA)	µg/kg	330	36	--	Not Applicable	--	Not Applicable
Hexachlorobenzene	118-74-1	Eurofins-TestAmerica (Savannah, GA)	µg/kg	330	39	--	Not Applicable	--	Not Applicable
Hexachlorobutadiene	87-68-3	Eurofins-TestAmerica (Savannah, GA)	µg/kg	330	36	--	Not Applicable	--	Not Applicable
Hexachlorocyclopentadiene	77-47-4	Eurofins-TestAmerica (Savannah, GA)	µg/kg	330	41	--	Not Applicable	--	Not Applicable
Hexachloroethane	67-72-1	Eurofins-TestAmerica (Savannah, GA)	µg/kg	330	28	--	Not Applicable	--	Not Applicable
Indeno[1,2,3-cd]pyrene	193-39-5	Eurofins-TestAmerica (Savannah, GA)	µg/kg	330	28	21000	Yes	--	Not Applicable
Isophorone	78-59-1	Eurofins-TestAmerica (Savannah, GA)	µg/kg	330	33	--	Not Applicable	--	Not Applicable
Naphthalene	91-20-3	Eurofins-TestAmerica (Savannah, GA)	µg/kg	330	30	--	Not Applicable	--	Not Applicable
Nitrobenzene	98-95-3	Eurofins-TestAmerica (Savannah, GA)	µg/kg	330	26	--	Not Applicable	--	Not Applicable
N-Nitrosodi-n-propylamine	621-64-7	Eurofins-TestAmerica (Savannah, GA)	µg/kg	330	32	--	Not Applicable	--	Not Applicable
N-Nitrosodiphenylamine	86-30-6	Eurofins-TestAmerica (Savannah, GA)	µg/kg	330	33	--	Not Applicable	--	Not Applicable
Pentachlorophenol	87-86-5	Eurofins-TestAmerica (Savannah, GA)	µg/kg	1700	330	--	Not Applicable	--	Not Applicable
Phenanthrene	85-01-8	Eurofins-TestAmerica (Savannah, GA)	µg/kg	330	27	--	Not Applicable	--	Not Applicable
Phenol	108-95-2	Eurofins-TestAmerica (Savannah, GA)	µg/kg	330	34	--	Not Applicable	--	Not Applicable
Pyrene	129-00-0	Eurofins-TestAmerica (Savannah, GA)	µg/kg	330	27	--	Not Applicable	--	Not Applicable
Volatile Organic Compounds by Method 8260C									
1,1,1-Trichloroethane	71-55-6	Eurofins-TestAmerica (Savannah, GA)	µg/kg	5	0.59	--	Not Applicable	--	Not Applicable
1,1,2,2-Tetrachloroethane	79-34-5	Eurofins-TestAmerica (Savannah, GA)	µg/kg	5	1.6	--	Not Applicable	--	Not Applicable
1,1,2-Trichloro-1,2,2-trifluoroethane	76-13-1	Eurofins-TestAmerica (Savannah, GA)	µg/kg	5	1.3	--	Not Applicable	--	Not Applicable
1,1,2-Trichloroethane	79-00-5	Eurofins-TestAmerica (Savannah, GA)	µg/kg	5	1.3	--	Not Applicable	--	Not Applicable
1,1-Dichloroethane	75-34-3	Eurofins-TestAmerica (Savannah, GA)	µg/kg	5	1.1	--	Not Applicable	--	Not Applicable
1,1-Dichloroethene	75-35-4	Eurofins-TestAmerica (Savannah, GA)	µg/kg	5	1.5	--	Not Applicable	--	Not Applicable
1,2,4-Trichlorobenzene	120-82-1	Eurofins-TestAmerica (Savannah, GA)	µg/kg	5	0.89	--	Not Applicable	--	Not Applicable
1,2-Dibromo-3-Chloropropane	96-12-8	Eurofins-TestAmerica (Savannah, GA)	µg/kg	10	4.4	--	Not Applicable	--	Not Applicable
1,2-Dichlorobenzene	95-50-1	Eurofins-TestAmerica (Savannah, GA)	µg/kg	5	1.3	--	Not Applicable	--	Not Applicable
1,2-Dichloroethane	107-06-2	Eurofins-TestAmerica (Savannah, GA)	µg/kg	5	1.1	--	Not Applicable	--	Not Applicable
1,2-Dichloropropane	78-87-5	Eurofins-TestAmerica (Savannah, GA)	µg/kg	5	0.86	--	Not Applicable	--	Not Applicable
1,3-Dichlorobenzene	541-73-1	Eurofins-TestAmerica (Savannah, GA)	µg/kg	5	1.6	--	Not Applicable	--	Not Applicable
1,4-Dichlorobenzene	106-46-7	Eurofins-TestAmerica (Savannah, GA)	µg/kg	5	0.74	--	Not Applicable	--	Not Applicable
2-Butanone (MEK)	78-93-3	Eurofins-TestAmerica (Savannah, GA)	µg/kg	25	2.4	--	Not Applicable	--	Not Applicable
2-Hexanone	591-78-6	Eurofins-TestAmerica (Savannah, GA)	µg/kg	25	3.3	--	Not Applicable	--	Not Applicable
4-Methyl-2-pentanone (MIBK)	108-10-1	Eurofins-TestAmerica (Savannah, GA)	µg/kg	25	4.2	--	Not Applicable	--	Not Applicable

UFP-QAPP Worksheet #15: Reference Limits and Evaluation Table

Analyte	CAS Number	Analytical Laboratory	Units	Laboratory Specified		OU-1/OU-2 ROD RGs Soil		OU-1/OU-2 ROD RGs Sediment	
				RL	MDL	Value	Laboratory MDL < Selected Screening Value	Value	Laboratory MDL < Selected Screening Value
Acetone	67-64-1	Eurofins-TestAmerica (Savannah, GA)	µg/kg	50	11	--	Not Applicable	--	Not Applicable
Benzene	71-43-2	Eurofins-TestAmerica (Savannah, GA)	µg/kg	5	0.73	--	Not Applicable	--	Not Applicable
Bromoform	75-25-2	Eurofins-TestAmerica (Savannah, GA)	µg/kg	5	1.5	--	Not Applicable	--	Not Applicable
Bromomethane	74-83-9	Eurofins-TestAmerica (Savannah, GA)	µg/kg	5	1.5	--	Not Applicable	--	Not Applicable
Carbon disulfide	75-15-0	Eurofins-TestAmerica (Savannah, GA)	µg/kg	5	1.1	--	Not Applicable	--	Not Applicable
Carbon tetrachloride	56-23-5	Eurofins-TestAmerica (Savannah, GA)	µg/kg	5	0.83	--	Not Applicable	--	Not Applicable
Chlorobenzene	108-90-7	Eurofins-TestAmerica (Savannah, GA)	µg/kg	5	0.96	--	Not Applicable	--	Not Applicable
Chlorodibromomethane	124-48-1	Eurofins-TestAmerica (Savannah, GA)	µg/kg	5	1.7	--	Not Applicable	--	Not Applicable
Chloroethane	75-00-3	Eurofins-TestAmerica (Savannah, GA)	µg/kg	5	2.7	--	Not Applicable	--	Not Applicable
Chloroform	67-66-3	Eurofins-TestAmerica (Savannah, GA)	µg/kg	5	1.1	--	Not Applicable	--	Not Applicable
Chloromethane	74-87-3	Eurofins-TestAmerica (Savannah, GA)	µg/kg	5	1	--	Not Applicable	--	Not Applicable
cis-1,2-Dichloroethene	156-59-2	Eurofins-TestAmerica (Savannah, GA)	µg/kg	5	1.4	--	Not Applicable	--	Not Applicable
cis-1,3-Dichloropropene	10061-01-5	Eurofins-TestAmerica (Savannah, GA)	µg/kg	5	0.83	--	Not Applicable	--	Not Applicable
Cyclohexane	110-82-7	Eurofins-TestAmerica (Savannah, GA)	µg/kg	5	1.3	--	Not Applicable	--	Not Applicable
Dichlorobromomethane	75-27-4	Eurofins-TestAmerica (Savannah, GA)	µg/kg	5	0.97	--	Not Applicable	--	Not Applicable
Dichlorodifluoromethane	75-71-8	Eurofins-TestAmerica (Savannah, GA)	µg/kg	5	0.94	--	Not Applicable	--	Not Applicable
Ethylbenzene	100-41-4	Eurofins-TestAmerica (Savannah, GA)	µg/kg	5	1.3	--	Not Applicable	--	Not Applicable
Ethylene Dibromide	106-93-4	Eurofins-TestAmerica (Savannah, GA)	µg/kg	5	1.5	--	Not Applicable	--	Not Applicable
Isopropylbenzene	98-82-8	Eurofins-TestAmerica (Savannah, GA)	µg/kg	5	1.9	--	Not Applicable	--	Not Applicable
Methyl acetate	79-20-9	Eurofins-TestAmerica (Savannah, GA)	µg/kg	25	5	--	Not Applicable	--	Not Applicable
Methyl tert-butyl ether	1634-04-4	Eurofins-TestAmerica (Savannah, GA)	µg/kg	5	1	--	Not Applicable	--	Not Applicable
Methylcyclohexane	108-87-2	Eurofins-TestAmerica (Savannah, GA)	µg/kg	5	0.86	--	Not Applicable	--	Not Applicable
Methylene Chloride	75-09-2	Eurofins-TestAmerica (Savannah, GA)	µg/kg	5	0.98	--	Not Applicable	--	Not Applicable
Styrene	100-42-5	Eurofins-TestAmerica (Savannah, GA)	µg/kg	5	0.93	--	Not Applicable	--	Not Applicable
Tetrachloroethene	127-18-4	Eurofins-TestAmerica (Savannah, GA)	µg/kg	5	1.9	--	Not Applicable	--	Not Applicable
Toluene	108-88-3	Eurofins-TestAmerica (Savannah, GA)	µg/kg	5	0.84	--	Not Applicable	--	Not Applicable
trans-1,2-Dichloroethene	156-60-5	Eurofins-TestAmerica (Savannah, GA)	µg/kg	5	0.63	--	Not Applicable	--	Not Applicable
trans-1,3-Dichloropropene	10061-02-6	Eurofins-TestAmerica (Savannah, GA)	µg/kg	5	0.87	--	Not Applicable	--	Not Applicable

UFP-QAPP Worksheet #15: Reference Limits and Evaluation Table

Analyte	CAS Number	Analytical Laboratory	Units	Laboratory Specified		OU-1/OU-2 ROD RGs Soil		OU-1/OU-2 ROD RGs Sediment	
				RL	MDL	Value	Laboratory MDL < Selected Screening Value	Value	Laboratory MDL < Selected Screening Value
Trichloroethene	79-01-6	Eurofins-TestAmerica (Savannah, GA)	µg/kg	5	1.3	--	Not Applicable	--	Not Applicable
Trichlorofluoromethane	75-69-4	Eurofins-TestAmerica (Savannah, GA)	µg/kg	5	1.2	--	Not Applicable	--	Not Applicable
Vinyl chloride	75-01-4	Eurofins-TestAmerica (Savannah, GA)	µg/kg	5	1.5	--	Not Applicable	--	Not Applicable
Xylenes, Total	1330-20-7	Eurofins-TestAmerica (Savannah, GA)	µg/kg	10	1.1	--	Not Applicable	--	Not Applicable

Notes:

1. Listed for point of compliance monitoring in RCRA permit # ALD 004 019 048.

CAS: Chemical Abstracts Service
GA: Georgia
MDL: method detection limit
mg/kg: milligrams per kilogram
µg/kg: micrograms per kilogram
OU-1: Operable Unit 1
OU-2: Operable Unit 2
RCRA: Resource Conservation and Recovery Act
RG: remeidal goal
RL: reporting limit
ROD: record of decision
USEPA: United States Environmental Protection Agency
VT: Vermont

UFP-QAPP Worksheet #17: Sampling Design and Rationale

Soil, groundwater, and sediment samples will be collected for laboratory analysis during the implementation of the Predesign Investigation Work Plan (PDIWP). Sampling rationale is summarized below and is provided in the PDIWP.

Sampling locations are shown in PDIWP figures. Samples of each media will be collected and analyzed according to the PDIWP.

The purpose of the assessment is to obtain additional information needed to complete the remedial design through the investigation of polychlorinated biphenyls (PCBs) and other Site compounds in these media. The data will be used to (i) evaluate the presence or absence of target compounds in media; (ii) provide additional information regarding the nature and extent of target compounds in media; (iii) evaluate potential treatment technologies for groundwater; and (iv) evaluate potential options for dewatering sediment.

Media sampled to accomplish these objectives will include soil, groundwater, sediment, and surface water, and these media will be sampled using conventional techniques. Media will be collected to achieve the above-stated objectives and the following additional objectives:

- Soil borings will be advanced to provide an updated understanding of Site geology and contaminant distribution.
- Groundwater samples will be collected to evaluate the presence of target compounds in groundwater and support the design of a treatment system. Additionally, monitoring wells will be installed to evaluate the groundwater flow regime and to support the design of a groundwater extraction system.
- Sediment samples will be collected to provide an updated delineation of location, physical extents, and contaminant distribution of sediment deposits.
- Surface water samples will be collected to generate a baseline dataset that can be used for comparison with post-remedy surface water monitoring data.

Sample Naming Convention

Each sample bottle will be labeled with the following information: unique sample identification (ID), date and time of sample collection, project name, and sampler's initials. Indelible ink will be used to record information on the sample label. Field documentation will accurately trace sample IDs to corresponding sample locations.

Samples that are collected in the field will be identified with a unique alpha-numeric sample ID that will abide by the following format:

XXX-WWA-D0-D1-YYYYMMDD

Where:

- "XXX" is a two- or three-letter description of the sample matrix.
- "WW" is a two-digit number describing the sample location. Numbers begin at "01" and continue upwards.
- "A" is used to indicate whether a monitoring well sample is shallow or deep. If this designation is not relevant to the sample, A will be omitted from the sample name.
- D0-D1: "D0" indicates sample start depth, and "D1" indicates sample end depth. All sampling depths are presumed to be measured in units of feet with one decimal. If D0 and/or D1 are not relevant to the sample, they will be omitted from the sample name.
    - "YYYYMMDD" is an 8-digit number representing the year, month, and date of sampling.

For example, the groundwater sample collected from the 01 shallow monitoring well from 10 feet below ground surface on July 4, 2021, would be assigned the sample ID: "MW-01S-10-20210704." A soil sample collected from SBS-05 from a start depth of 3 feet below ground surface and an end depth of 5 feet below ground surface would be given a sample ID of "SBS-05-3.0-5.0-YYYYMMDD."

Quality Assurance/Quality Control (QA/QC) Sample IDs

QA/QC samples to be collected include duplicate samples, equipment blanks, trip blanks, matrix spikes, and matrix spike duplicates (MS/MSD). Data about the source of the duplicate samples will be documented in field notes, but not provided to the laboratory.

QA/QC samples that are collected in the field will be identified with a unique alpha-numeric sample ID that will abide by the following format:

XXX-QC-BB-YYYYMMDD

Where:

- "XXX" is the two- or three-letter description of the sample matrix described above
- "QC" is the QC sample designation:

QC	QC Sample Type
DUP	Duplicate or Split Sample
EB	Equipment Blank Sample
TB	Trip Blank Sample

- "BB" is the is a numerical code starting at 01 and increasing based on the number of those samples collected on the day designated in the ID.
    - "YYYYMMDD" is an 8-digit number representing the year, month, and date of sampling.

For example, a duplicate sample collected at MW-01S on July 5, 2021, would be labeled "MW-DUP-01-20210705."

MS/MSD samples will follow a different convention from other QC samples: they will be labeled with the unique sample ID, as described above for each sample, with the suffix "-MS" or "-MSD" appended to the end of the sample ID. For example, an MSD sample collected from 10 feet deep from the 01 soil boring location on-site on July 5, 2021, would be labeled, "SBS-01-10-20210705-MSD."

Field Procedures

The following factors will be considered to meet the objectives of this field effort:

- Decontamination of Non-dedicated Equipment: Non-dedicated equipment will be decontaminated as described in the PDIWP.
- Complete and Appropriate Documentation: Field notes and other documentation will be maintained as described in the PDIWP.
- Sampling Equipment Construction Material: The material from which sampling equipment is constructed can affect sample analytical results. Materials must not contaminate the sample being collected, and reusable equipment must be able to be readily cleaned so that samples are not cross-contaminated.
- Sample Containers and Volumes: The sample volume obtained will be sufficient to supply the laboratory with adequate material to perform all required analyses and QC. Container types and required sample volumes are specified in Worksheet #19.
- Sample Preservation: Samples will be preserved to maintain their integrity as specified in Worksheet #19.
- Sample Holding Times: The elapsed time between sample collection and initiation of laboratory analyses will be consistent with the analytical methods per matrix and is specified in Worksheet #19.
- Sample Chain of Custody: Chain-of-custody procedures will be maintained from the point of sampling to final analysis as described in the PDIWP.

Inspection/Acceptance for Field Supplies and Consumables

Critical field supplies and consumables include the following:

- Sample bottleware
- Decontamination fluids (e.g., Liquinox®, nitric acid, acetone, deionized water, and distilled water)
- Personal protective equipment (PPE)
- Calibration standards for field measurement equipment
- General sampling consumables (ice, plastic bags, paper towels, aluminum foil, etc.).

For bottleware, the acceptance criteria will entail an inspection upon receipt to confirm the presence of appropriate preservatives (e.g., hydrochloric acid or nitric acid). For decontamination fluids, the field staff shall verify that the fluids meet the necessary requirements for concentration and quality grade (e.g., reagent-grade acetone). Calibration standards will be used within the specified expiration dates. PPE will be inspected to confirm integrity and ensure that the appropriate sizes are available as required by sampling team members.

Non-direct Measurements

In limited cases, non-direct measurement sources and tools will be used for project tasks. These sources and tools include the following:

- Geographic information system (GIS) base data, including aerial photographs, topographic quadrangles, and land use/land control data
- Regional literature describing the local geologic setting and geochemical environment
- Site characterization reports prepared under previous regulatory frameworks
- Analytical or numerical models to support fate and transport assessments, which may include both computational packages and internal databases for key site setting or transport parameters, such as climatic parameters

These non-direct measurement sources will play an important role in meeting project data quality objectives (DQOs). The acceptance criteria for these measurement sources are as follows:

- Where possible, GIS base data will be acquired from public data sources, such as the United States Department of Agriculture (USDA), Natural Resources Conservation Service (NRCS), or Geospatial Data Gateway (http://datagateway.nrcs.usda.gov/). These public sources provide a standardized approach to data quality review and georeferencing, with thorough documentation in accompanying metadata. Before these data are used in the Site GIS, the metadata shall be reviewed to ensure that the appropriate coordinate translation is applied.

- Regional literature will be sought from peer-reviewed public sources, such as United States Geological Survey (USGS) reports, to ensure a standard level of data review.
    - Prior Site characterization reports that have been reviewed by the United States Environmental Protection Agency (USEPA) will be used as the primary source for historical data.

Field Documentation

Field personnel will record the field sampling, field analysis, and sample chain-of-custody information. This documentation consists of a record that allows reconstruction of field events to aid in the data review and evaluation process. Documents, records, and information relating to the performance of the field work will be retained in the project files in accordance with Worksheet #29.

The following are various forms of documentation to be maintained throughout field activities:

- Daily Production Documentation: A field notebook consisting of a waterproof, bound notebook that will contain a record of activities performed at the Site. Field data sheets may also be used to record observations and sample collection information.
- Sampling Information: Detailed notes will be made as to the sampling location, physical observations, and weather conditions (as appropriate).
- Chain of Custody: Chain-of-custody forms will provide the record of responsibility for sample collection, transport, and submittal to the laboratory. Chain-of-custody forms will be completed for all samples collected at the Site. In the event that the samples are relinquished by the designated sampling person to other sampling or field personnel, the chain-of-custody form will be signed and dated by the appropriate personnel to document the sample transfer. The original chain-of-custody form will accompany the samples to the laboratory, and copies will be forwarded to the project files. Persons will have custody of samples when the samples are in their physical possession, in their view after being in their possession, or in their physical possession and secured so they cannot be tampered with. In addition, when samples are secured in a restricted area accessible only to authorized personnel, they will be deemed to be in the custody of such authorized personnel.
    - Field Equipment, Calibration, and Maintenance Logs: Calibration and maintenance of field instrumentation will be documented for each piece of field equipment.

Sample Handling and Custody

The first step in proper sample handling and custody is observance of analytical holding times, which can vary from 24 hours to 180 days depending upon the analytical method(s) selected for the samples. Knowledge of required holding times will directly affect scheduling of sample

collection, packing, and shipping activities. The sample container, volume, preservation, and holding times applicable to each analytical method are shown in Worksheet #19.

Field sample custody ensures that samples are not tampered with between the time of sample collection and the time of receipt at the analytical laboratory. Chain-of-custody procedures serve at least two essential purposes:

- They provide a formalized mechanism to assign responsibility for sample integrity.
- They provide objective, physical evidence of the procession history and integrity of each sample—collection through analysis to data reporting—supporting the validity of Site investigation data.

Upon sample receipt, laboratory personnel will be responsible for sample custody. The original field chain-of-custody form will accompany samples requiring laboratory analysis. The laboratory will use chain-of-custody guidelines described in the USEPA guidance documents listed in Worksheet #2. Samples will be kept secured in the laboratory until the analysis and reporting are complete. Laboratory personnel having samples in their custody will be responsible for documenting and maintaining sample integrity.

Upon sample receipt, the laboratory sample custodian will verify the cooler seal, open the cooler, check and document the cooler temperature, and compare the contents against the field chain-of-custody form. If a sample container is missing, a sample container is broken upon receipt, the sample is in an inappropriate container, or the sample has not been preserved by appropriate means, the contact designated on the chain-of-custody form will be notified. The laboratory sample custodian will be responsible for logging in the samples, assigning each sample a unique laboratory ID number, labeling the sample bottle with the laboratory ID number, and moving the sample to an appropriate storage location to await analysis. The project name, field sample code, date sampled, date received, analysis required, storage location and date, and action for final disposal will be recorded in the laboratory tracking system. Relevant custody documentation will be placed in the project file.

Samples will be organized into sample delivery groups (SDGs) by the laboratory. An SDG may contain up to 20 field samples of the same matrix (field duplicates, trip blanks, and rinse blanks are considered field samples for the purposes of SDG assignment). Field samples received by the laboratory over a duration of no more than 14 calendar days can be assigned to a single SDG and processed through the laboratory (preparation, analysis, and reporting) as a group. Each SDG will include laboratory batch QC.

Once analysis is complete, the sample volume and extracts remaining will be maintained by the laboratory for at least 1 month after the final report is delivered. The laboratory will be responsible

for the eventual and appropriate disposal of the samples. The analytical laboratory will inform the project prior to disposal of samples or extracts. Unused portions of samples, sample extracts, and associated wastes will be disposed of by the laboratory in accordance with applicable rules and regulations.

UFP-QAPP Worksheet #18: Sampling Locations and Methods/SOP Requirements Table

Sampling Media	Sample ID[1]	Matrix	Depth	Analytical Group[2]	Number of Samples Per Sampling Event (Field Quality Control)	Sampling Standard Operating Procedure (SOP) Reference	Rationale for Sampling Location
Groundwater	To be determined (TBD)	Groundwater	TBD	• Polychlorinated biphenyl (PCB) Aroclors • PCB homologs • Total organic carbon (TOC) and dissolved organic carbon (DOC) • Cations (calcium, magnesium, sodium, potassium, manganese, iron; total and dissolved) • Alkalinity • Anions (sulfate, chloride, nitrate; total and dissolved) • Hardness • Total suspended solids • Total dissolved solids • Toxicity characteristic leaching procedure (TCLP) (full suite)[3]	TBD	A-7	See Predesign Investigation Work Plan (PDIWP) and Worksheets #10 and #11

Sampling Media	Sample ID[1]	Matrix	Depth	Analytical Group[2]	Number of Samples Per Sampling Event (Field Quality Control)	Sampling Standard Operating Procedure (SOP) Reference	Rationale for Sampling Location
Soil Samples	TBD	Soil (Composite)	Dredge Spoil Pile SC-2: 0 to 1 foot below grade surface Low-Activity Special Use Areas: 1-foot intervals between 1 and 4 feet bgs	• PCB Aroclors • PCB homologs • TCLP (full suite)[3]	TBD	A-5	See PDIWP and Worksheets #10 and #11
	TBD	Soil (Geotechnical)	Up to 40 feet bgs	• Moisture content • Particle size • Atterberg limits • Engineering Classification • Unconsolidated undrained (UU) triaxial • Consolidated undrained (CU) triaxial	TBD	A-5	
	TBD	Soil (Composite and Incremental Sampling Methodology [ISM])	Interim Measure and Nonresidential Areas: 1-foot intervals between 0 and 4 feet bgs depending on area	• PCB Aroclors • PCB homologs	TBD	A-12	

Sampling Media	Sample ID[1]	Matrix	Depth	Analytical Group[2]	Number of Samples Per Sampling Event (Field Quality Control)	Sampling Standard Operating Procedure (SOP) Reference	Rationale for Sampling Location
Sediment	TBD	Sediment	TBD	• PCB Aroclors • Metals (barium, chromium [total], cobalt, lead, manganese, nickel, and vanadium only) • Mercury • TCLP (full suite)[3] • TOC • Specific gravity • Moisture content • Particle size • Atterberg Limits • Dry density • Organic content • Unconfined compressive strength • Paint filter test • Total suspended solids (filtrate) • Oil and Grease (filtrate)	TBD	A-3	See PDIWP and Worksheets #10 and #11

Sampling Media	Sample ID[1]	Matrix	Depth	Analytical Group[2]	Number of Samples Per Sampling Event (Field Quality Control)	Sampling Standard Operating Procedure (SOP) Reference	Rationale for Sampling Location
Surface Water	TBD	Surface Water (Passive Sampler)	Mid-depth under base flow conditions and a minimum of 3 inches above sediment surface	• PCB homologs (dissolved) • PCB congeners (dissolved)	2 (2 events per year)	A-13	See PDIWP and Worksheets #10 and #11
	TBD	Surface Water (Grab Sample)	Mid-depth under base flow conditions[4] and a minimum of 3 inches above sediment surface	• PCB homologs (total and dissolved) • Metals (lead and total chromium only; total and dissolved) • Hexavalent chromium (total and dissolved)	2 (4 events per year)	A-13	

1. Sample naming conventions are described in Worksheet #17.
2. Analytical methods for each media are listed in PDIWP.
3. Toxicity characteristic leaching procedure (TCLP) (full suite) includes: TCLP metals (USEPA SW-846 6010C/7471B/7470A), TCLP volatile organic compounds (USEPA SW-846 8260C), TCLP semivolatile organic compounds (USEPA SW-846 8270D), corrosivity (USEPA SW-846 9045D), ignitability (USEPA SW-846 1030), and reactivity – cyanide/sulfide (Ch. 7 USEPA SW-846)
4. For surface water grab sampling, samples will not be collected if significant precipitation has occurred within 72 hours of the planned sampling date.

UFP-QAPP Worksheets #19 & #30: Sample Containers, Preservation, and Hold Times

Geotechnical Laboratory: Excel Geotechnical Laboratory, Nader Rad, 953 Forrest Street, Roswell, GA 30075, (770) 910-7537
Required accreditations/certifications: Not applicable
Sample Delivery Method: Courier or FedEx

Matrix	Analytical Group	Analytical Method/Standard Operating Procedure (SOP) Reference	Containers (Number, Size, and Type)	Minimum Sample Volume	Preservation	Analytical Holding Time
Solids (Sediment)	Moisture Content	ASTM International (ASTM) D2216	Sealed plastic bag	300 grams	None	N/A
	Specific Gravity	ASTM D854	Sealed plastic bag	300 grams	None	N/A
	Dry Density	ASTM D2937	Sealed plastic bag	300 grams	None	N/A
	Organic Content	ASTM D2974	Sealed plastic bag	300 grams	None	N/A
	Atterberg (Liquid and Plastic) Limits	ASTM D4318	Sealed plastic bag	300 grams	None	N/A
	Unconfined Compression Strength	ASTM D2166	one 3-inch-diameter Shelby tube	N/A	None	N/A
	Particle Size	ASTM D6913/ASTM D7928	Sealed plastic bag	300 grams	None	N/A
	Unconsolidated Undrained (UU) Triaxial	ASTM D2166	one 3-inch-diameter Shelby tube	N/A	None	N/A
Solids (Soils)	Moisture Content	ASTM D2216	Sealed plastic bag	300 grams	None	N/A
	Particle Size	ASTM D6913/ASTM D7928	Sealed plastic bag	300 grams	None	N/A
	Atterberg (Liquid and Plastic) Limits	ASTM D4318	Sealed plastic bag	300 grams	None	N/A

Matrix	Analytical Group	Analytical Method/Standard Operating Procedure (SOP) Reference	Containers (Number, Size, and Type)	Minimum Sample Volume	Preservation	Analytical Holding Time
Solids (Soils) cont.	Unconfined Compression Strength	ASTM D2166	one 3-inch-diameter Shelby tube	N/A	None	N/A
	Consolidated Undrained (CU) triaxial	ASTM D4767	one 3-inch-diameter Shelby tube	N/A	None	N/A

Analytical Laboratory: Eurofins TestAmerica Knoxville, John Andros, 5815 Middlebrook Pike, TN 37921, (912) 354-7858
Required accreditations/certifications: See Appendix 2 (List of Laboratory Certifications, Accreditations, Validations) of the Eurofins-TestAmerica (Knoxville) Quality Assurance Manual (included in Appendix B of this UFP-QAPP) for accreditations and certifications maintained by laboratory.
Sample Delivery Method: Courier or FedEx

Matrix	Analytical Group	Analytical Method/Standard Operating Procedure (SOP) Reference	Containers (Number, Size, and Type)	Minimum Sample Volume	Preservation	Analytical Holding Time
Aqueous (groundwater, surface water, and sediment leachates or filtrates)	PCB Congeners	USEPA Method 1668A/ KNOX-ID-0013	two 1-L amber glass	1 L	Temperature 0-6 degrees Celsius (°C)	40 days (analysis after extraction) 365 days (extraction)

Analytical Laboratory: Eurofins-TestAmerica Pittsburgh, John Andros, 301 Alpha Drive RIDC Park, Pittsburgh, PA 15238, (912) 354-7858
Required accreditations/certifications: See Appendix 2 (List of Laboratory Certifications, Accreditations, Validations) of the Eurofins-TestAmerica (Pittsburgh) Quality Assurance Manual (included in Appendix B of this UFP-QAPP) for accreditations and certifications maintained by laboratory.
Sample Delivery Method: Courier or FedEx

Matrix	Analytical Group	Analytical Method/Standard Operating Procedure (SOP) Reference	Containers (Number, Size, and Type)	Minimum Sample Volume	Preservation	Analytical Holding Time
Aqueous (groundwater, surface water, and sediment leachates or filtrates)	Polychlorinated Biphenyl (PCB) Aroclors	United States Environmental Protection Agency (USEPA) Method SW-846 8082A/PT-GC-005	two 1-Liter (L) amber glass	1 L	Temperature 0-6°C	40 days (analysis after extraction) 365 days (extraction)
	Total Dissolved Solids (TDS)	SM 2540C/PT-WC-045	1 x 500-milliliter (ml) plastic or glass	100 ml	Cool to 0-6°C but not frozen	7 days
	Total Suspended Solids (TSS)	SM 2540D/PT-WC-043	1 x 1000-ml plastic or glass	1000 ml	Cool to 0-6°C but not frozen	7 days
	Hardness	USEPA Method SM 2340B/PT-MT-WI-011	1 x 250-ml plastic	100 ml	Nitric acid (HNO_3) to pH<2; Cool to 0-6°C but not frozen	180 days
	Anions	USEPA Method 300.0/ PT-GC-009	1 x 125-ml plastic	20 ml	Cool to 0-6°C but not frozen	Fluoride, Chloride, Bromide, and Sulfate: 28 days Nitrite and Nitrate: 48 hours Ortho-phosphate: 48 hours; filter sample within 15 minutes of collection
	Total Organic Carbon (TOC) and Dissolved	USEPA Method 9060A/ PT-WC-017	2 x 40-ml volatile organic analysis (VOA) vials	40 ml	Sulfuric acid (H_2SO_4) to pH<2; Cool to 0-6°C but not frozen	28 days

Matrix	Analytical Group	Analytical Method/Standard Operating Procedure (SOP) Reference	Containers (Number, Size, and Type)	Minimum Sample Volume	Preservation	Analytical Holding Time
	Organic Carbon (DOC)					
	Oil and Grease	USEPA Method 1664B/PT-WC-028	2 x 1000-ml amber glass	1000 ml	Hydrochloric acid (HCL) to pH < 2, Cool to 0-6°C but not frozen	28 days
Solids (Sediment)	PCB Aroclors	USEPA Method SW-846 8082A/PT-GC-005	one 8-oz, amber glass with Teflon-lined lid	30 grams	Temperature 0-6°C	40 days (analysis after extraction) 365 days (extraction)
	Paint Filter Test	USEPA Method 9095B/ PT-WC-034	one plastic or glass jar	100 grams or 100 ml	Temperature 0-6°C	180 days
Solid (Soil)	PCB Aroclors	USEPA Method SW-846 8082A/PT-GC-005	one 8-oz, amber glass with Teflon-lined lid	30 grams	Temperature 0-6°C	40 days (analysis after extraction) 365 days (extraction)
Toxicity Characteristic Leaching Procedure (TCLP) Leachate (Groundwater and Soil/Sediment) USEPA Method SW-846/PT-OP-004	PCB Aroclors	USEPA Method SW-846 8082A/PT-GC-005	two 16-oz Teflon-lined amber glass jars (soils and sediment)	450 grams (soils and sediment) 700 ml (groundwater)	Temperature 0-6°C	Collection to Start of TCLP Leach: 14 days (VOCs and SVOCs) 28 days (Mercury) 180 days (Other Metals) End of TCLP Tumble to SVOC Extraction: 7 days End of TCLP Leach or SVOC Extraction to Analysis: 14 days (VOCs) 40 days (SVOCs) 28 days (Mercury) 180 days (Other Metals)
	Corrosivity (pH)	USEPA SW-846 9045D/ PT-WC-026	two 8-oz glass jars (metals, SVOCs, corrosivity, reactivity [cyanide and sulfide]; groundwater) one 4-oz glass jars (VOCs; groundwater) one 4-oz glass jars (ignitability; groundwater)			
	Ignitability	USEPA Chapter 7, Section 7.1/PT-WC-022				
	Reactivity (Cyanide)	USEPA SW-846 9010C/ PT-WC-018				
	Reactivity (Sulfide)	USEPA SW-846 9030B/9034/PT-WC-010				

Analytical Laboratory: Eurofins-TestAmerica Burlington, John Andros, 30 Community Drive, Suite 11, South Burlington, VT 05403, (912) 354-7858
Required accreditations/certifications: See Appendix 2 (List of Laboratory Certifications, Accreditations, Validations) of the Eurofins-TestAmerica (Burlington) Quality Assurance Manual (included in Appendix B of this UFP-QAPP) for accreditations and certifications maintained by laboratory.
Sample Delivery Method: Courier or FedEx

Matrix	Analytical Group	Analytical Method/Standard Operating Procedure (SOP) Reference	Containers (Number, Size, and Type)	Minimum Sample Volume	Preservation	Analytical Holding Time
Aqueous (groundwater, surface water, and sediment leachates or filtrates)	Polychlorinated Biphenyl (PCB) Aroclors	United States Environmental Protection Agency (USEPA) Method SW-846 8082A/BR-GC-005	two 1-Liter (L) amber glass	1 L	Temperature 0-6°C	40 days (analysis after extraction) 365 days (extraction)
	PCB Homologs	USEPA Method 680/ BR-MS-010	two 1-L amber glass	1 L	Temperature 0-6°C	
Solids (Sediment)	PCB Aroclors	USEPA Method SW-846 8082A/BR-GC-005	one 8-oz, amber glass with Teflon-lined lid	30 grams	Temperature 0-6°C	40 days (analysis after extraction) 365 days (extraction)
	TOC	Lloyd Khan/BR-WC-008	one 4-oz, amber glass	10 grams	Temperature 0-6°C	14 days
Solids (Soil)	PCB Aroclors	USEPA Method SW-846 8082A/BR-GC-005	one 8-oz, amber glass with Teflon-lined lid	30 grams	Temperature 0-6°C	40 days (analysis after extraction) 365 days (extraction)
	PCB Homologs	USEPA Method 680/ BR-MS-010	one 4-oz, amber glass	50 grams	Temperature 0-6°C	40 days (analysis after extraction) 365 days (extraction)
Toxicity Characteristic Leaching Procedure (TCLP) Leachate (Groundwater and Soil/Sediment)	PCB Aroclors	USEPA Method SW-846 8082A/BR-GC-005	two 16-oz Teflon-lined amber glass jars (soils and sediment) two 8-oz glass jars (SVOCs; groundwater)	450 grams (soils and sediment) 700 ml (groundwater)	Temperature 0-6°C	Collection to Start of TCLP Leach: 14 days End of TCLP Tumble to SVOC Extraction: 7 days End of SVOC Extraction to Analysis:

Matrix	Analytical Group	Analytical Method/Standard Operating Procedure (SOP) Reference	Containers (Number, Size, and Type)	Minimum Sample Volume	Preservation	Analytical Holding Time
USEPA Method SW-846/PT-OP-004						40 days (SVOCs)

Analytical Laboratory: Eurofins-TestAmerica Savannah, John Andros, 5102 LaRoche Avenue, Savannah, GA 31404, (912) 354-7858
Required accreditations/certifications: See Appendix 3 (List of Laboratory Certifications, Accreditations, Validations) of the Eurofins-TestAmerica (Savannah) Quality Assurance Manual (included in Appendix B of this UFP-QAPP) for accreditations and certifications maintained by laboratory.
Sample Delivery Method: Courier or FedEx

Matrix	Analytical Group	Analytical Method/Standard Operating Procedure (SOP) Reference	Containers (Number, Size, and Type)	Minimum Sample Volume	Preservation	Analytical Holding Time
Aqueous (groundwater, surface water, and sediment leachates or filtrates)	Alkalinity	SM 2320B/SA-GE-193	1 x 250-ml plastic	100 mL	Cool to 0-6°C but not frozen	7 days
	Metals/Cations	USEPA Method SW-846 6010C/SA-ME-070	1 x 250-ml plastic	100 ml	HNO_3 to pH<2; no HNO_3 preservation for samples to be filtered at the laboratory. If not preserved, temperature 0-6°C	180 days
	Hexavalent Chromium	USEPA Method SW-846 7196A/SA-GE-001	1 x 125-ml plastic	10 ml	Temperature 0-6°C	24 hours
Solids (Sediment)	Metals	USEPA Method SW-846 6010C/SA-ME-070	one 8-oz, amber glass	2 grams	Temperature 0-6°C	180 days
	Mercury	USEPA Method SW-846 7471B/SA-ME-028	one 4-oz, amber glass	5 grams	Temperature 0-6°C	28 days
Toxicity Characteristic Leaching	Metals	USEPA Method SW-846 6010C/SA-ME-070	two 16-oz Teflon-lined amber glass jars (soils and sediment)	450 grams (soils and sediment)	Temperature 0-6°C	Collection to Start of TCLP Leach:

Matrix	Analytical Group	Analytical Method/Standard Operating Procedure (SOP) Reference	Containers (Number, Size, and Type)	Minimum Sample Volume	Preservation	Analytical Holding Time
Procedure (TCLP) Leachate (Groundwater and Soil/Sediment) USEPA Method SW-846/PT-OP-004	Mercury	USEPA Method SW-846 7471B/7470A/SA-ME-028	---------- two 8-oz glass jars (metals, SVOCs, corrosivity, reactivity [cyanide and sulfide]; groundwater) one 4-oz glass jars (VOCs; groundwater) one 4-oz glass jars (ignitability; groundwater)	700 ml (groundwater		14 days (VOCs and SVOCs) 28 days (Mercury) 180 days (Other Metals) End of TCLP Tumble to SVOC Extraction: 7 days End of TCLP Leach or SVOC Extraction to Analysis: 14 days (VOCs) 40 days (SVOCs) 28 days (Mercury) 180 days (Other Metals)
	Volatile Organic Compounds	USEPA SW-846 8260C/ SA-VO-004				
	Semivolatile Organic Compounds	USEPA SW-846 8270D/ SA-SM-003				

UFP-QAPP Worksheet #20: Field Quality Control Sample Summary Table

Matrix	Analytical Group	Anticipated Concentration Level	Analytical Standard Operation Procedure Reference	No. of Sampling Locations or Areas (see Notes 1, 2, and 3)	No. of Field Duplicate Pairs	No. of MS/MSD Samples	No. of Equip. Blanks	No of Source Blanks	No. of Trip Blanks
Aqueous (groundwater, surface water, and sediment leachates or filtrates)	Polychlorinated biphenyl (PCB) Aroclors	Medium to High	USEPA Method SW-846 8082A/BR-GC-005	Groundwater: 3 Surface water: 2 Sediment leachates/filtrates: See Note 2	1 per 20 samples	1 per 20 samples	1 per 20 or 1 per day, whichever is less frequent, per sampling area	1 per lot of source water	N/A
	PCB homologs	Medium to High	USEPA Method 680/ BR-MS-010	Groundwater: 3 Surface water: 2	1 per 20 samples	1 per 20 samples	1 per 20 or 1 per day, whichever is less frequent, per sampling area	1 per lot of source water	N/A
	PCB Congeners	Medium to High	USEPA Method 1668A / KNOX-ID-0013	Surface water: 2	1 per 20 samples	1 per 20 samples	1 per 20 or 1 per day, whichever is less frequent, per sampling area	1 per lot of source water	N/A
	Total organic carbon (TOC)	Medium	USEPA 9060A/SA-GE-204	Groundwater: 3	1 per 20 samples	1 per 10 samples	1 per 20 or 1 per day, whichever is less frequent, per sampling area	1 per lot of source water	N/A
	Metals/Cations	Medium	USEPA Method 6010C/SA-ME-070	Groundwater: 3 Surface water: 2	1 per 20 samples	1 per 20 samples	1 per 20 or 1 per day, whichever is less frequent, per sampling area	1 per lot of source water	N/A
	Hexavalent Chromium	Low to medium	USEPA Method 7196A / SA-GE-001	Surface water: 2	1 per 20 samples	1 per 20 samples	1 per 20 or 1 per day, whichever is less frequent, per sampling area	1 per lot of source water	N/A
	Anions	Medium	Method 300.0/ PT-GC-009	Groundwater: 3	1 per 20 samples	1 per 10 samples	1 per 20 or 1 per day, whichever is less frequent, per sampling area	1 per lot of source water	N/A
	Hardness	Medium	USEPA Method 130.2 & SM2340B/SA-GE-201	Groundwater: 3	1 per 20 samples	NA	1 per 20 or 1 per day, whichever is less frequent, per sampling area	1 per lot of source water	NA

Matrix	Analytical Group	Anticipated Concentration Level	Analytical Standard Operation Procedure Reference	No. of Sampling Locations or Areas (see Notes 1, 2, and 3)	No. of Field Duplicate Pairs	No. of MS/MSD Samples	No. of Equip. Blanks	No of Source Blanks	No. of Trip Blanks
Aqueous (groundwater and sediment leachates or filtrates) cont.	Total Suspended Solids (TSS)	Medium	SM2540D/PT-WC-045	Groundwater: 3 Sediment leachates/filtrates: See Note 2	1 per 20 samples	NA	NA	NA	NA
	/ Total Dissolved Solids (TDS)	Medium	SM2540C/PT-WC-043	Groundwater: 3	1 per 20 samples	NA	NA	NA	NA
	Alkalinity	Medium	SM 2320B/SA-GE-193	Groundwater: 3	1 per 20 samples	NA	1 per 20 or 1 per day, whichever is less frequent, per sampling area	1 per lot of source water	N/A
	Oil and Grease	Low to Medium	USEPA Method 1664B/PT-WC-028	Sediment leachates/filtrates: See Note 2	N/A	1 per 20 samples	N/A	N/A	N/A
Solids (Sediment)	PCB Aroclors	Medium to High	USEPA Method SW-846 8082A/BR-GC-005	To be determined (see Note 2)	1 per 20 samples	1 per 20 samples	1 per 20 or 1 per day, whichever is less frequent, per sampling area	1 per lot of source water	N/A
	Metals	Medium	USEPA Method SW-846 6010C/SA-ME-070	To be determined (see Note 2)	1 per 20 samples	1 per 20 samples	1 per 20 or 1 per day, whichever is less frequent, per sampling area	1 per lot of source water	N/A
	Mercury	Medium	USEPA Method SW-846 7471B/SA-ME-028	To be determined (see Note 2)	1 per 20 samples	1 per 20 samples	1 per 20 or 1 per day, whichever is less frequent, per sampling area	1 per lot of source water	N/A

Matrix	Analytical Group	Anticipated Concentration Level	Analytical Standard Operation Procedure Reference	No. of Sampling Locations or Areas (see Notes 1, 2, and 3)	No. of Field Duplicate Pairs	No. of MS/MSD Samples	No. of Equip. Blanks	No of Source Blanks	No. of Trip Blanks
Solids (Sediment) cont.	TOC	Medium	Lloyd Khan/BR-WC-008	To be determined (see Note 2)	1 per 20 samples	1 per 20 samples	N/A	N/A	N/A
	Moisture Content	N/A	ASTM D2216	To be determined (see Note 2)	N/A	N/A	N/A	N/A	N/A
	Specific Gravity	N/A	ASTM D854	To be determined (see Note 2)	N/A	N/A	N/A	N/A	N/A
	Dry Density	N/A	ASTM D2937	To be determined (see Note 2)	N/A	N/A	N/A	N/A	N/A
	Organic Content	N/A	ASTM D2974	To be determined (see Note 2)	N/A	N/A	N/A	N/A	N/A
	Atterberg (Liquid and Plastic) Limits	N/A	ASTM D4318	To be determined (see Note 2)	N/A	N/A	N/A	N/A	N/A
	Unconfined Compression Strength	N/A	ASTM D2166	To be determined (see Note 2)	N/A	N/A	N/A	N/A	N/A
	Paint Filter Test	N/A	USEPA Method 9095B	To be determined (see Note 2)	N/A	N/A	N/A	N/A	N/A
	Particle Size	N/A	ASTM D6913/ASTM D7928	To be determined (see Note 2)	N/A	N/A	N/A	N/A	N/A
	Unconsolidated undrained (UU) triaxial	N/A	ASTM D2166	To be determined (see Note 2)	N/A	N/A	N/A	N/A	N/A

Matrix	Analytical Group	Anticipated Concentration Level	Analytical Standard Operation Procedure Reference	No. of Sampling Locations or Areas (see Notes 1, 2, and 3)	No. of Field Duplicate Pairs	No. of MS/MSD Samples	No. of Equip. Blanks	No of Source Blanks	No. of Trip Blanks
Solids (Soils)	PCB Aroclors	Medium to High	USEPA Method SW-846 8082A/BR-GC-005	Discrete borings: 3 Composite areas: 54 ISM areas: 3	1 per 20 samples	1 per 20 samples	1 per 20 or 1 per day, whichever is less frequent, per sampling area	1 per lot of source water	N/A
	PCB homologs	Medium to High	USEPA Method 680/ BR-MS-010	Discrete boring: 1 Composite areas: 1	1 per 20 samples	1 per 20 samples	1 per 20 or 1 per day, whichever is less frequent, per sampling area	1 per lot of source water	N/A
	Moisture Content	N/A	ASTM D2216	Discrete borings: 4	N/A	N/A	N/A	N/A	N/A
	Particle Size	N/A	ASTM D6913/ASTM D7928	Discrete borings: 4	N/A	N/A	N/A	N/A	N/A
	Atterberg (Liquid and Plastic) Limits	N/A	ASTM D4318	Discrete borings: 4	N/A	N/A	N/A	N/A	N/A
	Consolidated undrained (CU) triaxial	N/A	ASTM D4767	Discrete borings: 4	N/A	N/A	N/A	N/A	N/A
	Unconfined Compression Strength	N/A	ASTM D2166	Discrete borings: 4	N/A	N/A	N/A	N/A	N/A

Matrix	Analytical Group	Anticipated Concentration Level	Analytical Standard Operation Procedure Reference	No. of Sampling Locations or Areas (see Notes 1, 2, and 3)	No. of Field Duplicate Pairs	No. of MS/MSD Samples	No. of Equip. Blanks	No of Source Blanks	No. of Trip Blanks
TCLP Leachate (Groundwater and Soil/Sediment IDW)	TCLP	Medium	USEPA Method SW-846 1311/PT-OP-004	Not applicable (see Note 3)	N/A	N/A	N/A	N/A	N/A
	PCB Aroclors	Medium to High	USEPA Method SW-846 8082A/BR-GC-005 or PT-GC-005	Not applicable (see Note 3)	N/A	N/A	N/A	N/A	N/A
	Metals	Medium	USEPA Method SW-846 6010C/SA-ME-070	Not applicable (see Note 3)	N/A	N/A	N/A	N/A	N/A
	Mercury	Medium	USEPA Method SW-846 7471B/SA-ME-028	Not applicable (see Note 3)	N/A	N/A	N/A	N/A	N/A
	Volatile Organic Compounds	Low to Medium	USEPA SW-846 8260C/ SA-VO-004	Not applicable (see Note 3)	N/A	N/A	N/A	N/A	1 per cooler
	Semivolatile Organic Compounds	Low to Medium	USEPA SW-846 8270D/ SA-SM-003	Not applicable (see Note 3)	N/A	N/A	N/A	N/A	N/A
	Corrosivity (pH)	Medium	USEPA SW-846 9045D/ PT-WC-026	Not applicable (see Note 3)	N/A	N/A	N/A	N/A	N/A
	Ignitability	Low	USEPA Chapter 7, Section 7.1/PT-WC-022	Not applicable (see Note 3)	N/A	N/A	N/A	N/A	N/A
	Reactivity (Cyanide)	Low	USEPA SW-846 9010C/ PT-WC-018	Not applicable (see Note 3)	N/A	N/A	N/A	N/A	N/A
	Reactivity (Sulfide)	Low	USEPA SW-846 9030B/9034/PT-WC-010	Not applicable (see Note 3)	N/A	N/A	N/A	N/A	N/A

Notes:

1. The number of soil samples to be provided for analytical or geotechnical analyses cannot be provided as several sampling scopes of work are dependent on field observations; however the anticipated number of sample locations or areas has been provided where known.

2. The number of sediment sampling locations is dependent upon completion of Phase 1 of the sediment pre-design investigation (i.e., sediment probing and reconnaissance) as described in Section 3.3.2 of the OU-1/OU-2 Pre-Design Investigation Work Plan (P/S 2022).
3. The number of sampling locations or areas is not applicable to define for groundwater and soil/sediment IDW. Excess sample material, purge water, and spent decontamination liquids will be containerized in drums and tested to determine proper disposal methods.

IDW: investigation-derived waste

ISM: incremental sampling methodology

MS/MSD: matrix spike/matrix spike duplicate

UFP-QAPP Worksheet #21: Project Sampling Standard Operation Procedure (SOP) References Table

Reference Number	Title, Revision Date and/or Number	Originating Organization	Equipment Type	Modified for Project Work?	Comments
A-1	Field Sample Packaging, Handling, Shipping, and Chain-of-Custody Procedures	GSI, Anchor QEA, and Geosyntec	Refer to SOP in Attachment A	N	N/A
A-2	Field Cleaning and Decontamination Procedures	GSI, Anchor QEA, and Geosyntec	Refer to SOP in Attachment A	N	N/A
A-3	Sediment Probing and Sampling Procedures	GSI, Anchor QEA, and Geosyntec	Refer to SOP in Attachment A	N	N/A
A-4	Freezing Procedures for the Preservation of PCBs in Soil, Sediment, and Biota Samples	GSI, Anchor QEA, and Geosyntec	Refer to SOP in Attachment A	N	N/A
A-5	Surface and Subsurface Soil Sampling	GSI, Anchor QEA, and Geosyntec	Refer to SOP in Attachment A	N	N/A
A-6	Subsurface Drilling and Monitoring Well Installation Procedures	GSI, Anchor QEA, and Geosyntec	Refer to SOP in Attachment A	N	N/A
A-7	Groundwater Sampling Procedure	GSI, Anchor QEA, and Geosyntec	Refer to SOP in Attachment A	N	N/A
A-8	Monitoring Well/Borehole Abandonment Procedures	GSI, Anchor QEA, and Geosyntec	Refer to SOP in Attachment A	N	N/A
A-9	Procedures for Compositing or Homogenizing Soil or Sediment Samples	GSI, Anchor QEA, and Geosyntec	Refer to SOP in Attachment A	N	N/A
A-10	Slug Testing Procedure	GSI, Anchor QEA, and Geosyntec	Refer to SOP in Attachment A	N	N/A
A-11	Specific- Capacity and Pumping Test Procedures	GSI, Anchor QEA, and Geosyntec	Refer to SOP in Attachment A	N	N/A
A-12	Incremental Sampling Methodology Procedures	GSI, Anchor QEA, and Geosyntec	Refer to SOP in Attachment A	N	N/A
A-13	Surface Water Sampling, Filtering, and Measuring Procedures	GSI, Anchor QEA, and Geosyntec	Refer to SOP in Attachment A	N	N/A

UFP-QAPP Worksheet #22: Field Equipment Calibration, Maintenance, Testing, and Inspection Table

The following field instruments will be used in support of field sampling and field-testing tasks:

- Trimble differential global positioning system (DGPS) or equivalent (soil, groundwater, surface water and sediment)
- YSI Model Pro Plus multimeter or equivalent (groundwater and surface water)
- Hach Model 2100Q turbidimeter or equivalent (groundwater and surface water)

Instruments will be inspected and calibrated (if applicable) daily prior to use and as needed during use in accordance with the manufacturer's instructions and the Predesign Investigation Work Plan (PDIWP). The Predesign/Field Investigation Coordinator and Field Team Leads (or a qualified delegate) identified in UFP-QAPP Worksheet #3 & #5 (Project Organization and UFP-QAPP Distribution) will manage the field equipment calibration, maintenance, testing, and inspection in accordance with manufacturer's instructions and the PDIWP. Additionally, an inspection and calibration check (if applicable) will be completed at the end of each day in accordance with the manufacturer's instructions and the PDIWP. Pertinent data will be recorded on the appropriate field forms.

Geosyntec consultants

UFP-QAPP Worksheet #23: Analytical SOP References

Standard Operating Procedure (SOP)	Title, Revision Date, and/or Number	Revision	Definitive or Screening Data	Analytical Group/ Matrix	Instrument	Organization Performing Analysis	Modified for Project Work (Y/N)
Eurofins-TestAmerica, Burlington, Vermont							
BR-GC-005	Polychlorinated Biphenyls (PCBs) GC/ECD (SW846 8082A, USEPA TO4A/TO10A), 06/30/21	15	Definitive	PCB Aroclors/ Soil, Groundwater, Surface Water, and Sediment	Gas Chromatography (GC)/Electron Capture Detectors (ECD)	Eurofins-TestAmerica Burlington	N
BR-GT-006	Particle Size Analysis (ASTM International [ASTM] D 2217 and D422-63), 06/05/20	9	Definitive	Particle Size/ Soil and Sediment	Sieve/hydrometer	Eurofins-TestAmerica Burlington	N
BR-MS-010	PCBs Homologs by GC/MS (USEPA 680), 12/31/19	1	Definitive	PCB Homologs/ Groundwater, Surface Water, and Soil	GC/mass spectrometry (MS)	Eurofins-TestAmerica Burlington	N
BR-WC-008	Total Organic Carbon by Lloyd Kahn Method, 04/30/20	17	Definitive	TOC/Soil and Sediment	Drying Oven/Carlo Erba Elemental Analyzer/Costech Elemental Analyzer	Eurofins-TestAmerica Burlington	N
BR-EX-005	Separatory Funnel Extraction (SW-846 3510C), 06/25/2020	14	Definitive	PCBs Aroclors and Homologs/ Groundwater and Surface Water	--	Eurofins-TestAmerica Burlington	N
BR-EX-027	Automated Soxhlet Extraction (SW-846 3541), 06/30/21	5	Definitive	PCBs Aroclors and Homologs/ Soil and Sediment	Automated Soxhlet Extraction System	Eurofins-TestAmerica Burlington	N

Standard Operating Procedure (SOP)	Title, Revision Date, and/or Number	Revision	Definitive or Screening Data	Analytical Group/ Matrix	Instrument	Organization Performing Analysis	Modified for Project Work (Y/N)
Eurofins-TestAmerica, Pittsburgh, Pennsylvania							
PT-GC-005	Polychlorinated Biphenyls (PCBs) and PCBs as Congeners by GC/ECD, 5/19/20	12	Definitive	PCBs Aroclors and Congeners/ Soil, Groundwater, Surface Water, and Sediment	GC/ECD	Eurofins-TestAmerica Pittsburgh	N
PT-GC-009	Determination of Inorganic Anions by Ion Chromatography, 11/30/20	21	Definitive	Anions/Groundwater	Ion chromatograph	Eurofins-TestAmerica Pittsburgh	N
PT-MT-WI-011	2340B Hardness by Calculation, using Ca and Mg from ICP or ICPMS analysis	0	Definitive	Hardness/Groundwater	Inductively Coupled Plasma (ICP) atomic emission spectroscopy or ICP MS	Eurofins-TestAmerica Pittsburgh	N
PT-OP-001	Extraction of Organic Compounds from Waters, 11/5/20	21	Definitive	Semivolatile Organic Carbons (SVOCs)/ TCLP Leachate	ICP MS	Eurofins-TestAmerica Pittsburgh	N
PT-OP-004	Toxicity Characteristic Leaching Procedure (TCLP) and Synthetic Precipitation Leaching Procedure (SPLP). 4/10/20	10	Definitive	SVOCs, Sulfide, Cyanide, pH/ TCLP Leachate	--	Eurofins-TestAmerica Pittsburgh	N
PT-WC-010	Analysis for Sulfide, 4/28/20	19	Definitive	Sulfide/TCLP Leachate	--	Eurofins-TestAmerica Pittsburgh	N

Standard Operating Procedure (SOP)	Title, Revision Date, and/or Number	Revision	Definitive or Screening Data	Analytical Group/ Matrix	Instrument	Organization Performing Analysis	Modified for Project Work (Y/N)
PT-WC-018	Total Cyanide Distillation and Analysis, Semi-Automated Colorimetry, 4/13/20	21	Definitive	Cyanide/ TCLP Leachate	Spectrophotometer	Eurofins-TestAmerica Pittsburgh	N
PT-WC-022	Ignitability of Solids for Waste Characterization, 12/31/19	7	Definitive	Ignitability/ TCLP Leachate	--	Eurofins-TestAmerica Pittsburgh	N
PT-WC-026	Electrometric pH, 10/28/20	15	Definitive	pH/Soil, Groundwater, Sediment, TCLP Leachate	pH meter	Eurofins-TestAmerica Pittsburgh	N
PT-WC-028	Hexane Extractable Material (HEM; Oil and Grease) and Silica Gel Treated Hexane Extractable Material (SGT-HEM; TPH) Method 1664A/1664B and SW-846 Method 9070A, 5/25/20	16	Definitive	Oil and Grease/Sediment leachate	Oil and grease cartridge extraction station	Eurofins-TestAmerica Pittsburgh	N
PT-WC-034	Paint Filter – Free Liquids Test, 11/30/20	5	Definitive	Free Liquids/ Soil and Sediment	Paint filter	Eurofins-TestAmerica Pittsburgh	N
PT-WC-036	Flash Point by Setaflash (Small Scale) Closed-Cup Apparatus, 1/21/21	7	Definitive	Ignitability/ TCLP Leachate	Setaflash closed tester	Eurofins-TestAmerica Pittsburgh	N
PT-WC-043	Determination of Total and Volatile Suspended Solids in Waters and Wastes, 03/20/20	4	Definitive	TSS/Groundwater	--	Eurofins-TestAmerica Pittsburgh	N
PT-WC-045	Determination of Total Dissolved and Volatile Dissolved Solids in Waters and Wastes, 04/22/20	4	Definitive	TDS/Groundwater	--	Eurofins-TestAmerica Pittsburgh	N

Standard Operating Procedure (SOP)	Title, Revision Date, and/or Number	Revision	Definitive or Screening Data	Analytical Group/ Matrix	Instrument	Organization Performing Analysis	Modified for Project Work (Y/N)
Eurofins-TestAmerica, North Canton, Ohio							
NC-GC-045	Gas Chromatographic Analysis of PCBs Based on Methods 8082 and 8082A, 08/31/20	3	Definitive	PCBs/Ground water, Soil, Sediment	GC/ECD	Eurofins-TestAmerica North Canton	N
NC-OP-040	Soxhlet (Traditional) Extraction of Organic Compounds from Soils Based on Method SW846 3540C, 6/9/20	7	Definitive	SVOCs and PCBs/Soil and Sediment	GC/MS	Eurofins-TestAmerica North Canton	N
NC-OP-044	Soil Processing, 1/13/21	4	Definitive	All Analytes/ Soil	--	Eurofins-TestAmerica North Canton	N
NC-OP-046	Subsampling, 6/24/21	4	Definitive	All Analytes/ Soil	--	Eurofins-TestAmerica North Canton	N
Eurofins-TestAmerica, Savannah, Georgia							
SA-GE-190	Solid/Residue Determinations, 11/25/20	12B	Definitive	Residue Analytes/Soil, Groundwater, and Sediment	Drying oven	Eurofins-TestAmerica Savannah	N
SA-ME-028	Mercury Preparation and Analysis, 12/08/20	15A	Definitive	Mercury/Soil, Groundwater, and Sediment	Cold Vapor Atomic Absorption (CVAA)	Eurofins-TestAmerica Savannah	N
SA-ME-050	Liquid Preparation Procedures for ICP and ICP/MS, 9/14/21	22A	Definitive	Metals/ Groundwater and Leachate	Digestion Vessel	Eurofins-TestAmerica Savannah	N
SA-ME-051	Soil Preparation Procedures for ICP and ICP/MS, 06/03/21	15A	Definitive	Metals/Soil and Sediment	Digestion Vessel	Eurofins-TestAmerica Savannah	N

Standard Operating Procedure (SOP)	Title, Revision Date, and/or Number	Revision	Definitive or Screening Data	Analytical Group/ Matrix	Instrument	Organization Performing Analysis	Modified for Project Work (Y/N)
SA-ME-070	Elements by ICP, 12/09/20	19D	Definitive	Metals/Soil, Groundwater, and Sediment	ICP atomic emission spectroscopy	Eurofins-TestAmerica Savannah	N
SA-ME-074	Elements by ICP-MS, 12/09/20	13A	Definitive	Metals/Soil, Groundwater, and Sediment	ICP MS	Eurofins-TestAmerica Savannah	N
SA-SG-045	Organochlorine Pesticides and Polychlorinated Biphenyls (PCBs) by GC/ECD, 12/07/21	24	Definitive	PCBs and Pesticides/ Soil, Groundwater, and Sediment	GC/ECD	Eurofins-TestAmerica Savannah	N
SA-SM-033	Semi-Volatile Compounds by GC/MS, 11/20/21	11	Definitive	SSVOCs/Soil, Groundwater, and Sediment	GC/MS	Eurofins-TestAmerica Savannah	N
SA-GE-001	Measurement of Analytes using the Konelab Analyzer	9	Definitive	Hexavalent Chromium/Surface Water	Konelab Model 20 Autoanalyzer (or equivalent)	Eurofins-TestAmerica Savannah	N
Eurofins-TestAmerica, Savannah, Georgia							
SA-VO-004	Volatile Compounds by GC/MS, 10/19/21	11	Definitive	VOCs/Soil, Groundwater, and Sediment	GC/MS	Eurofins-TestAmerica Savannah	N
Eurofins-TestAmerica, Knoxville, Tennessee							
KNOX-ID-0013	Analysis of Polychlorinated Biphenyl (PCB) Isomers by Isotope Dilution HRGC/HRMS, 8/31/21	19	Definitive	PCBs/Surface Water	High resolution GC (HRGC)/High resolution MS (HRMS)	Eurofins-TestAmerica Knoxville	N

Standard Operating Procedure (SOP)	Title, Revision Date, and/or Number	Revision	Definitive or Screening Data	Analytical Group/ Matrix	Instrument	Organization Performing Analysis	Modified for Project Work (Y/N)
Excel Geotechnical Testing (EGT), Roswell, Georgia							
Not applicable	ASTM D854 – Standard Test Methods for Specific Gravity of Soil Solids by Water Pycnometer, 5/1/14	--	Definitive	Geotechnical/ Soil	--	EGT	N
Not applicable	ASTM D2216 - Standard Test Methods for Laboratory Determination of Water (Moisture) Content of Soil and Rock by Mass, 3/19/19	--	Definitive	Geotechnical/ Soil	--	EGT	N
Not applicable	ASTM D6913 – Standard Test Methods for Particle-Size Distribution (Gradation) of Soils Using Sieve Analysis, 2017 ASTM D7928 – Standard Test Method for Particle-Size Distribution (Gradation) of Fine-Grained Soils Using the Sedimentation (Hydrometer) Analysis, 6/10/21	--	Definitive	Geotechnical/ Soil	--	EGT	N
Not applicable	ASTM D4318 – Standard Test Methods for Liquid Limit, Plastic Limit, and Plasticity Index of Soils, 4/17/18	--	Definitive	Geotechnical/ Soil	--	EGT	N
Not applicable	ASTM D2937 - Standard Test Method for Density of Soil in Place by the Drive-Cylinder Method, 4/17/18	--	Definitive	Geotechnical/ Soil	--	EGT	N

Standard Operating Procedure (SOP)	Title, Revision Date, and/or Number	Revision	Definitive or Screening Data	Analytical Group/ Matrix	Instrument	Organization Performing Analysis	Modified for Project Work (Y/N)
Not applicable	ASTM D2974 – Standard Test Methods for Moisture, Ash, and Organic Matter of Peat and Other Organic Soils, 6/26/20	--	Definitive	Geotechnical/ Soil	--	EGT	N
Not applicable	ASTM D2166 – Standard Test Methods for Laboratory Determination of Water (Moisture) Content of Soil and Rock by Mass, 7/1/16	--	Definitive	Geotechnical/ Soil	--	EGT	N
Not applicable	ASTM D2850 – Standard Test Method for Unconsolidated-Undrained Triaxial Compression Test on Cohesive Soils, 11/15/15	--	Definitive	Geotechnical/ Soil	--	EGT	N
Not applicable	ASTM D4767 – Standard Test Method for Consolidated Undrained Triaxial Compression Test for Cohesive Soils, 4/10/20	--	Definitive	Geotechnical/ Soil	--	EGT	N

UFP-QAPP Worksheet #24: Analytical Instrument Calibration Table

For information regarding analytical laboratory instruments, calibration procedures and standards, and quality control, see Attachment B (Laboratory SOPs) for Standard Operating Procedures (SOPs) referenced in the following table.

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action (CA)	Person Responsible for CA	SOP Reference
GC/MS	See SOPs provided in Attachment B				Lab Manager/ Analyst	BR-MS-010, Rev. 1 SA-SM-033, Rev. 11 SA-VO-004, Rev. 11
HRGC/HRMS	See SOPs provided in Attachment B				Lab Manager/Analyst	KNOX-ID-0013, Rev. 19
GC/ECD	See SOPs provided in Attachment B				Lab Manager/ Analyst	BR-GC-005, Rev. 15 SA-SG-045, Rev. 24 NC-GC-045, Rev. 3 PT-GC-005, Rev. 12
pH Meter	See SOP provided in Attachment B				Lab Manager/ Analyst	PT-WC-026, Rev. 15
Ion Chromatograph	See SOP provided in Attachment B				Lab Manager/ Analyst	PT-GC-009, Rev. 21
ICP	See SOP provided in Attachment B				Lab Manager/ Analyst	SA-ME-070, Rev. 19D
ICP MS	See SOP provided in Attachment B				Lab Manager/ Analyst	SA-ME-074, Rev. 13A
CVAA	See SOP provided in Attachment B				Lab Manager/ Analyst	SA-ME-028, Rev. 15A

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action (CA)	Person Responsible for CA	SOP Reference
Carbon Analyzer	See SOP provided in Attachment B				Lab Manager/ Analyst	BR-WC-008, Rev. 17
Spectrophotometer	See SOP provided in Attachment B				Lab Manager/ Analyst	PT-WC-018, Rev. 21
Balance	See SOPs provided in Attachment B				Lab Manager/ Analyst	BR-GT-006, Rev. 9 PT-WC-028, Rev. 16 PT-WC-034, Rev. 5
Konelab Model 20 Autoanalyzer	See SOPs provided in Attachment B				Lab Manager/ Analyst	SA-GE-001, Rev. 9

CVAA: cold vapor atomic absorption
GC/ECD: gas chromatography/electron capture detector
GC/MS: gas chromatography/mass spectrometry
HRGC/HRMS: high resolution gas chromatography/high resolution mass spectrometry
ICP: inductively coupled plasma
ICP MS: inductively coupled plasma mass spectrometry

UFP-QAPP Worksheet #25: Analytical Instrument and Equipment Maintenance, Testing, and Inspection Table

For information regarding analytical laboratory instruments, testing and inspection procedures, and quality control, see Attachment B (Laboratory SOPs).

UFP-QAPP Worksheets #26 & #27: Sample Handling, Custody, and Disposal

Sample Collection (Labeling and Chain-of-Custody Form Completion), Packaging, and Shipment
Sample Collection (Personnel/Organization): Field Team Lead/Geosyntec, GSI, and Anchor QEA
Sample Packaging (Personnel/Organization): Field Team Lead/Geosyntec, GSI, and Anchor QEA
Coordination of Shipment (Personnel/Organization): Field Team Lead/Geosyntec, GSI, and Anchor QEA
Type of Shipment/Carrier: Overnight Shipping/Commercial Courier or Laboratory Courier
Sample Receipt, Inspection, and Analysis
Sample Receipt (Personnel/Organization): Sample Receiving Personnel/Eurofins-TestAmerica and EGT
Sample Custody and Storage (Personnel/Organization): Sample Receiving Personnel/ Eurofins-TestAmerica and EGT
Sample Preparation (Personnel/Organization): Sample Extraction/Preparation Personnel/ Eurofins-TestAmerica and EGT
Sample Determinative Analysis (Personnel/Organization): Analytical Testing Department Personnel/ Eurofins-TestAmerica and EGT
Sample Archiving
Field Sample Storage (No. of days from sample collection): Field Samples are stored for 30 days after submittal of the completed data package.
Sample Extract/Digestate Storage (No. of days from extraction/digestion): Sample extracts (as applicable) will be stored in the laboratory for 30 days after submittal of the completed data package unless notified by the client to archive for a longer period of time.
Sample Disposal
Personnel/Organization: Sample Receiving Personnel/ Eurofins-TestAmerica and EGT
Number of Days from Analysis: Field samples are stored for 30 days after submittal of the completed data package. Client will be notified prior to sample disposal by the laboratory.

Sample Custody

Sample collection and sample custody procedures are designed so that field custody of samples is maintained and documented. These procedures provide identification and documentation of the sampling event and the sample chain of custody from shipment of sample bottleware, through sample collection, to receipt of the sample by the subcontracted laboratory. When used in conjunction with the laboratory's custody procedures and the sample bottleware documentation, these data establish full legal custody and allow complete tracking of a sample from preparation and receipt of sample bottleware to sample collection, preservation, and shipping through laboratory receipt, sample analysis, and data validation. The chain of custody is defined as the sequence of persons who have the item in custody.

The persons responsible for sample custody and a brief description of their duties, are as follows:

- Laboratory Sample Custodian or Commercial Supplier: Verifies that the bottleware is certified clean and arranges for bottleware shipment to field sampling personnel or the contractor's equipment shop. Receives samples from shipping courier or field staff, inspects samples, compares received sample containers to Chain-of-Custody Record, logs samples into laboratory system for designated analyses, and secures sample containers until ready for analysis.
- Field Staff: Receives sample bottleware from laboratory, inspects bottleware for physical integrity, collects and preserves samples, retains bottleware and samples under custody until sample shipment, and relinquishes samples to shipping courier or to lab representative.
    - Laboratory Project Manager: Verifies reported laboratory analyses to the sample Chain-of-Custody Record and ensures that chain-of-custody documentation is incorporated into the project file.

A sample or other physical evidence is in custody if it is or was one of the following:

- In the field investigator's, transferee's, or lab technician's actual possession
- In the field investigator's, transferee's, or lab technician's view, after being in his/her physical possession
- In the field investigator's, transferee's, or lab technician's physical possession and then he/she secured it to prevent tampering
- Placed in a designated secure area

Chain-of-Custody Record

The field Chain-of-Custody Record is used to record the custody of all samples or other physical evidence collected and maintained. This form shall not be used to document the collection of split or duplicate samples. The Chain-of-Custody Record also serves as a mechanism to log samples for the analytical laboratories' sample custodian.

The following information must be supplied in the indicated spaces in detail to complete the field Chain-of-Custody Record:

- Project-specific information, including the project number and project name, must be added in the indicated space.
- The signatures of the sampling team leader must be included in the designated signature block.
- The sampling station number, date, and time of sample collection; grab or composite sample designation; and sample preservation type must be included on each line (each line shall contain only those samples collected at a specific location).
- The total number of sample containers must be listed in the indicated space for each sample, and the total number of individual containers must also be listed for each type of analysis under the indicated media or miscellaneous columns (it is impossible to have more than one media type per sample).
    - The field investigator and subsequent transferee(s) must document the transfer of the samples listed on the Chain-of-Custody Record in the spaces provided at the bottom of the form. Both the person relinquishing the samples and the person receiving them must sign the form and provide the date and time that this occurred in the proper space on the form. Usually, the last person receiving the samples or evidence should be a laboratory sample custodian.

Once the Chain-of Custody Record is completed, it becomes an accountable document and must be maintained in the project file. The suitability of any other form for chain of custody should be evaluated upon its inclusion of all of the above information in a legible format.

Laboratory Sample Custody Procedures

Once the samples reach the laboratory, they will be checked against information on the chain-of-custody form for anomalies. The condition, temperature, and appropriate preservation of samples will be checked and documented on the chain-of-custody form. Checking an aliquot of the sample using pH paper is an acceptable procedure. The occurrence of any anomalies in the received samples and their resolution will be documented in laboratory records. All sample information will then be entered into a tracking system, and unique analytical sample identifiers will be assigned. A copy of this information will be reviewed by the laboratory project manager for accuracy. Sample holding time will be tracked beginning with the collection of samples and will continue until the analysis is complete. Holding times will be observed as specified in Worksheet #19. All analyses will be documented with the chain-of-custody form. Procedures ensuring internal laboratory chain of custody will also be implemented and documented by the laboratory. Specific instructions concerning the analysis specified for each sample will be communicated to the analysts. Analytical batches will be created, and laboratory quality assurance/quality control (QA/QC) samples will be introduced into each batch.

While in the laboratory, samples will be stored in limited-access, temperature-controlled areas. Refrigerators, coolers, and freezers will be monitored for temperature 7 days a week. Acceptance criterion for the temperatures of the refrigerators and coolers will be 0 to 6 degrees Celsius (°C). The sample temperature will be noted on the chain of custody at the time of receipt.

Acceptance criterion for the temperatures of the freezers will be less than -10°C. All of the cold storage areas will be monitored by thermometers or temperature monitoring devices calibrated against National Institute of Standards and Technology (NIST)-traceable certified thermometers. Correction factors will be applied to each thermometer as indicated by the findings of the calibration. Records that include acceptance criteria will be maintained. Samples will be stored after analysis until disposed of in accordance with applicable local, state, and federal regulations. Disposal records will be maintained by the laboratory.

Standard operating procedures (SOPs) describing sample control and custody will be maintained by the laboratory.

UFP-QAPP Worksheet #28a: QC Samples Table for USEPA Method 8260C – Volatiles

Matrix	Aqueous (toxicity characteristic leaching procedure leachate, aqueous water quality control [QC] samples, equipment/source/trip blanks)
Analytical Group	Volatile Organic Compounds
Anticipated Concentration Level	Low to Moderate
Analytical Method/SOP Reference[1]	United States Environmental Protection Agency (USEPA) SW-846 Method 8260C/SA-VO-004, Rev. 11
Field Sampling Organization	Geosyntec Consultants, GSI, and Anchor QEA
Analytical Organization	Eurofins-TestAmerica, Savannah, Georgia
No. of Sample Locations	See Worksheet #18

QC Sample	Frequency/ Number	Method/Standard Operating Procedure (SOP) QC Acceptance Limits[2]	Corrective Action	Person(s) Responsible for Corrective Action	Measurement Performance Criteria[2]
Tune Standard (BFB)	At the beginning of each 12-hour clock	Refer to Attachment 7 of SOP	Troubleshoot; perform instrument maintenance; retune	Analyst	Refer to Attachment 7 of SOP
Initial Calibration (ICAL)	Upon instrument set-up and after failing continuing calibration verification (CCV)	Minimum 5-point response factor criteria (RRF) per Attachment 6 of SOP. % relative standard deviation (RSD) $\leq$20%. If %RSD >20%, use curve fit with $r^2 \geq 0.990$; Read back criteria for all levels of +/- 30% of true value	Troubleshoot; reanalyze standard(s), prepare new standard(s) and reanalyze; perform injector port maintenance and reanalyze standards; retune and reanalyze standards; replace column and reanalyze standards; clean source and reanalyze standards	Analyst	Minimum 5-point RRF per Attachment 6 of SOP. %RSD $\leq$20%. If %RSD >20%, use curve fit with $r^2 \geq 0.990$; Read back criteria for all levels of +/- 30% of true value
Initial Calibration Verification (ICV)	After each ICAL	%RSD <30% Poor Performer 30% < %RSD < 50% as per Attachment 11 of SOP	Troubleshoot; reanalyze standard, prepare new standard and reanalyze; recalibrate	Analyst	%RSD <30% Poor Performer 30% < %RSD < 50% as per Attachment 11 of SOP

Matrix	Aqueous (toxicity characteristic leaching procedure leachate, aqueous water quality control [QC] samples, equipment/source/trip blanks)
Analytical Group	Volatile Organic Compounds
Anticipated Concentration Level	Low to Moderate
Analytical Method/SOP Reference[1]	United States Environmental Protection Agency (USEPA) SW-846 Method 8260C/SA-VO-004, Rev. 11
Field Sampling Organization	Geosyntec Consultants, GSI, and Anchor QEA
Analytical Organization	Eurofins-TestAmerica, Savannah, Georgia
No. of Sample Locations	See Worksheet #18

QC Sample	Frequency/ Number	Method/Standard Operating Procedure (SOP) QC Acceptance Limits[2]	Corrective Action	Person(s) Responsible for Corrective Action	Measurement Performance Criteria[2]
Continuing Calibration Verification (CCV)	1 per 12-hour clock after BFB	Meet minimum response factor (RF) criteria in Attachment 6 of SOP. All analytes must be ≤20%RSD; no analyte >50%RSD. Poor performer %RSD may be >50% as per Attachment 11 of SOP. Reporting limit (RL) CCV is required to report nondetects (NDs) for analytes outside 20%RSD or RRF criteria. A nonconformance memo (NCM) is required to denote analytes outside criteria.	Troubleshoot; if CCV %RSD is above control limits, and there are no detections in the samples, the laboratory will report the ND results with an NCM; reanalyze standard; prepare new standard and reanalyze; recalibrate	Analyst	Meet minimum RF criteria in Attachment 6 of SOP. All analytes must be ≤20%RSD; no analyte >50%RSD. Poor performer %RSD may be >50% as per Attachment 11 of SOP. RL CCV is required to report NDs for analytes outside 20%RSD or RRF criteria. An NCM is required to denote analytes outside criteria.

Matrix	Aqueous (toxicity characteristic leaching procedure leachate, aqueous water quality control [QC] samples, equipment/source/trip blanks)
Analytical Group	Volatile Organic Compounds
Anticipated Concentration Level	Low to Moderate
Analytical Method/SOP Reference[1]	United States Environmental Protection Agency (USEPA) SW-846 Method 8260C/SA-VO-004, Rev. 11
Field Sampling Organization	Geosyntec Consultants, GSI, and Anchor QEA
Analytical Organization	Eurofins-TestAmerica, Savannah, Georgia
No. of Sample Locations	See Worksheet #18

QC Sample	Frequency/ Number	Method/Standard Operating Procedure (SOP) QC Acceptance Limits[2]	Corrective Action	Person(s) Responsible for Corrective Action	Measurement Performance Criteria[2]
Internal Standards (ISTD)	Spike in all continuing calibration verification internal standard (CCVIS), samples, and batch QC items	CCVIS: Area -50% to 100% CCV in ICAL; RT +/- 10 seconds from ICAL. Samples and batch QC items: Area -50% to 100% of previous CCVIS	Troubleshoot; evaluate chromatogram, spectra, and integrations; reanalyze extract; perform instrument maintenance and reanalyze extract; reextract and reanalyze if sufficient sample is available	Analyst	CCVIS: Area -50% to 100% CCV in ICAL; RT +/- 10 seconds from ICAL. Samples and batch QC items: Area -50% to 100% of previous CCVIS
Surrogate Compounds	Spiked in all samples and batch QC items	Within laboratory generated limits	Troubleshoot; evaluate chromatogram, spectra, and integrations; reanalyze sample if sufficient sample is available	Analyst	Within laboratory generated limits
Method Blank	1 per batch of up to 20 samples	No analytes detected ≥ ½ RL or > 1/10th the amount measured in a sample	Troubleshoot; reextract and reanalyze if sufficient sample is available; if second failure flag data as appropriate–refer to SOP SA-QA-017	Analyst	No analytes detected ≥ ½ RL or > 1/10th the amount measured in a sample

Matrix	Aqueous (toxicity characteristic leaching procedure leachate, aqueous water quality control [QC] samples, equipment/source/trip blanks)
Analytical Group	Volatile Organic Compounds
Anticipated Concentration Level	Low to Moderate
Analytical Method/SOP Reference[1]	United States Environmental Protection Agency (USEPA) SW-846 Method 8260C/SA-VO-004, Rev. 11
Field Sampling Organization	Geosyntec Consultants, GSI, and Anchor QEA
Analytical Organization	Eurofins-TestAmerica, Savannah, Georgia
No. of Sample Locations	See Worksheet #18

QC Sample	Frequency/ Number	Method/Standard Operating Procedure (SOP) QC Acceptance Limits[2]	Corrective Action	Person(s) Responsible for Corrective Action	Measurement Performance Criteria[2]
Laboratory Control Sample (LCS)	1 per batch of up to 20 samples	Within laboratory generated limits	Troubleshoot; reextract and reanalyze if sufficient sample is available; if second failure flag data as appropriate–refer to SOP SA-QA-017	Analyst	Within laboratory generated limits
Laboratory Control Sample Duplicate (LCSD)	1 per batch of up to 20 samples, when insufficient sample is provided for MS/MSD	Within laboratory generated limits	Troubleshoot; reextract and reanalyze if sufficient sample is available; if second failure flag data as appropriate–refer to SOP SA-QA-017	Analyst	Within laboratory generated limits
Matrix Spike/ Matrix Spike Duplicate (MS/MSD)	1 per batch of up to 20 samples	Within laboratory generated limits	Troubleshoot; reanalyze MS/MSD if second failure flag data as appropriate–refer to SOP SA-QA-017	Analyst	Within laboratory generated limits
Initial Demonstration of Capability (IDOC)	Initially, per analyst, per analyte/method/ matrix combination	Refer to SOP SA-QA-006	Refer to SOP SA-QA-006. Note unsupervised work must not begin until acceptable IDOC is obtained.	Analyst	Refer to SOP SA-QA-006

Matrix	Aqueous (toxicity characteristic leaching procedure leachate, aqueous water quality control [QC] samples, equipment/source/trip blanks)
Analytical Group	Volatile Organic Compounds
Anticipated Concentration Level	Low to Moderate
Analytical Method/SOP Reference[1]	United States Environmental Protection Agency (USEPA) SW-846 Method 8260C/SA-VO-004, Rev. 11
Field Sampling Organization	Geosyntec Consultants, GSI, and Anchor QEA
Analytical Organization	Eurofins-TestAmerica, Savannah, Georgia
No. of Sample Locations	See Worksheet #18

QC Sample	Frequency/ Number	Method/Standard Operating Procedure (SOP) QC Acceptance Limits[2]	Corrective Action	Person(s) Responsible for Corrective Action	Measurement Performance Criteria[2]
Continuing Demonstration of Capability (CDOC)	Annually, per analyst, per analyte/ method/matrix combination	Refer to SOP SA-QA-006	Refer to SOP SA-QA-006.	Analyst	Refer to SOP SA-QA-006
Refer to SOP SA-QA-006	Upon method/instrument setup, per analyte/ method/matrix combination. Then annually thereafter.	Refer to SOP SA-QA-007	Refer to SOP SA-QA-007	Analyst	Refer to SOP SA-QA-007
Refer to SOP SA-QA-007	Upon method/instrument setup, per analyte/ method/matrix combination.	Refer to SOP SA-QA-007	Refer to SOP SA-QA-007	Analyst	Refer to SOP SA-QA-007

Matrix	Aqueous (toxicity characteristic leaching procedure leachate, aqueous water quality control [QC] samples, equipment/source/trip blanks)
Analytical Group	Volatile Organic Compounds
Anticipated Concentration Level	Low to Moderate
Analytical Method/SOP Reference[1]	United States Environmental Protection Agency (USEPA) SW-846 Method 8260C/SA-VO-004, Rev. 11
Field Sampling Organization	Geosyntec Consultants, GSI, and Anchor QEA
Analytical Organization	Eurofins-TestAmerica, Savannah, Georgia
No. of Sample Locations	See Worksheet #18

QC Sample	Frequency/ Number	Method/Standard Operating Procedure (SOP) QC Acceptance Limits[2]	Corrective Action	Person(s) Responsible for Corrective Action	Measurement Performance Criteria[2]
Refer to SOP SA-QA-007	Upon method/instrument setup, per analyte/ method/matrix combination. Then annually thereafter.	Refer to SOP SA-QA-007	Refer to SOP-QA-007	Analyst	Refer to SOP SA-QA-007

1. SOPs are reviewed/revised on an annual schedule by the laboratory. The current version of the SOP will be followed at the time of sample receipt.
2. This is a summary of the acceptance limits and measurement performance criteria; refer to the method SOP included in Attachment B of this UFP-QAPP for specific or more information. The acceptance limits and measurement performance criteria are in accordance with the government-required limits or criteria (i.e., National Functional Guidelines and/or USEPA Region 4 *Laboratory Operations and Quality Assurance Manual* [USEPA 2022]).

UFP-QAPP Worksheet #28b: QC Samples Table for USEPA Method 8270D – Semivolatiles

Matrix	Aqueous (toxicity characteristic leaching procedure [TCLP] leachate, aqueous water quality control [QC] samples, equipment/source blanks)
Analytical Group	Semivolatile Organic Compounds
Anticipated Concentration Level	Low to Moderate
Analytical Method/SOP Reference[1]	United States Environmental Protection Agency (USEPA) SW-846 Method 8270D/SA-SM-003, Rev. 11
Field Sampling Organization	Geosyntec Consultants, GSI, and Anchor QEA
Analytical Organization	Eurofins-TestAmerica Savannah, Georgia
No. of Sample Locations	See Worksheet #18

QC Sample		Frequency/ Number	Method/Standard Operating Procedure (SOP) QC Acceptance Limits[2]	Corrective Action	Person(s) Responsible for Corrective Action	Measurement Performance Criteria[2]
Tune/Column Evaluation Standard	Spectrum Criteria	At the beginning of each 12-hour clock	Method criteria per Attachment 5 of SOP	Perform instrument maintenance; retune	Analyst	Method criteria per Attachment 5 of SOP
	Tailing Factor Criteria		Pentachlorophenol <2 Benzidine <2		Analyst	Pentachlorophenol <2 Benzidine <2
	Breakdown Criteria		<20%		Analyst	<20%

Matrix	Aqueous (toxicity characteristic leaching procedure [TCLP] leachate, aqueous water quality control [QC] samples, equipment/source blanks)
Analytical Group	Semivolatile Organic Compounds
Anticipated Concentration Level	Low to Moderate
Analytical Method/SOP Reference[1]	United States Environmental Protection Agency (USEPA) SW-846 Method 8270D/SA-SM-003, Rev. 11
Field Sampling Organization	Geosyntec Consultants, GSI, and Anchor QEA
Analytical Organization	Eurofins-TestAmerica Savannah, Georgia
No. of Sample Locations	See Worksheet #18

QC Sample	Frequency/ Number	Method/Standard Operating Procedure (SOP) QC Acceptance Limits[2]	Corrective Action	Person(s) Responsible for Corrective Action	Measurement Performance Criteria[2]
Initial Calibration (ICAL)	Upon instrument set-up and after failing continuing calibration verification (CCV)	5-point minimum response factor criteria (RRF) per Attachment 11 of SOP. %RSD ≤20%. If %RSD >20%, use curve fit w/ $r^2 \geq 0.990$; If linear fit, reporting limit (RL) Level: 30% of true; If r^2 <0.990, use %RSD (allowed for <10% of total # analytes; no analyte >50% RSD). % relative standard error (RSE) <50% for low point; <30% for all other points.	Reanalyze standard(s); prepare new standard(s) and reanalyze; perform injector port maintenance and reanalyze standards; replace column and/or clean source, and reanalyze standards	Analyst	5-point Minimum RRF per Attachment 11 of SOP. %RSD ≤ 20%. If %RSD > 20%, use curve fit w/ $r^2 \geq 0.990$; If linear fit, RL Level: 30% of true; If r^2 <0.990, use %RSD (allowed for <10% of total # analytes; no analyte >50% RSD). %RSE <50% for low point; <30% for all other points.

Matrix	Aqueous (toxicity characteristic leaching procedure [TCLP] leachate, aqueous water quality control [QC] samples, equipment/source blanks)
Analytical Group	Semivolatile Organic Compounds
Anticipated Concentration Level	Low to Moderate
Analytical Method/SOP Reference[1]	United States Environmental Protection Agency (USEPA) SW-846 Method 8270D/SA-SM-003, Rev. 11
Field Sampling Organization	Geosyntec Consultants, GSI, and Anchor QEA
Analytical Organization	Eurofins-TestAmerica Savannah, Georgia
No. of Sample Locations	See Worksheet #18

QC Sample	Frequency/ Number	Method/Standard Operating Procedure (SOP) QC Acceptance Limits[2]	Corrective Action	Person(s) Responsible for Corrective Action	Measurement Performance Criteria[2]
Initial Calibration Verification (ICV)	After each ICAL	% relative standard deviation (RSD) <30% Poor performers %RSD <50% as per Attachment 10 of SOP. A nonconformance memo (NCM) is required to denote analytes outside criteria.	Reanalyze standard; prepare new standard and reanalyze; recalibrate	Analyst	%RSD <30% Poor performers %RSD <50% as per Attachment 10 of SOP. An NCM is required to denote analytes outside criteria.

Matrix	Aqueous (toxicity characteristic leaching procedure [TCLP] leachate, aqueous water quality control [QC] samples, equipment/source blanks)
Analytical Group	Semivolatile Organic Compounds
Anticipated Concentration Level	Low to Moderate
Analytical Method/SOP Reference[1]	United States Environmental Protection Agency (USEPA) SW-846 Method 8270D/SA-SM-003, Rev. 11
Field Sampling Organization	Geosyntec Consultants, GSI, and Anchor QEA
Analytical Organization	Eurofins-TestAmerica Savannah, Georgia
No. of Sample Locations	See Worksheet #18

QC Sample	Frequency/ Number	Method/Standard Operating Procedure (SOP) QC Acceptance Limits[2]	Corrective Action	Person(s) Responsible for Corrective Action	Measurement Performance Criteria[2]
Continuing Calibration Verification (CCV)	1 per 12-hour clock after Tune/Column Evaluation Standard	RRF per Attachment 11 of SOP. %RSD ≤ 20%. Poor performers per Attachment 10 of SOP. See Note 1 in Corrective Action.	Reanalyze standard; prepare new standard and reanalyze; recalibrate Note 1: If <20% total # analytes >20%RSD, evaluate reporting limit (RL) CCV. RL CCV must be qualitatively identified. If client sample is nondetect for affected analyte, report unqualified result. If client sample has detection for affected analyte, report result as estimated.	Analyst	RRF per Attachment 11 of SOP. %RSD ≤ 20%. Poor performers per Attachment 10 of SOP. See Note 1 in Corrective Action.

Matrix	Aqueous (toxicity characteristic leaching procedure [TCLP] leachate, aqueous water quality control [QC] samples, equipment/source blanks)
Analytical Group	Semivolatile Organic Compounds
Anticipated Concentration Level	Low to Moderate
Analytical Method/SOP Reference[1]	United States Environmental Protection Agency (USEPA) SW-846 Method 8270D/SA-SM-003, Rev. 11
Field Sampling Organization	Geosyntec Consultants, GSI, and Anchor QEA
Analytical Organization	Eurofins-TestAmerica Savannah, Georgia
No. of Sample Locations	See Worksheet #18

QC Sample	Frequency/ Number	Method/Standard Operating Procedure (SOP) QC Acceptance Limits[2]	Corrective Action	Person(s) Responsible for Corrective Action	Measurement Performance Criteria[2]
Reporting Limit CCV	1 per clock, if needed. Analyze after CCV. When RRF criteria is not met in ICAL, or CCV RRF or CCV %RSD criteria is not met.	Affected analytes must be qualitatively identified.	Perform instrument maintenance; Reanalyze affected samples; Recalibrate	Analyst	Affected analytes must be qualitatively identified.
Method Blank	1 per batch of up to 20 samples	Less than ½ RL	Refer to SOP SA-QA-017	Analyst	Less than ½ RL

Matrix	Aqueous (toxicity characteristic leaching procedure [TCLP] leachate, aqueous water quality control [QC] samples, equipment/source blanks)
Analytical Group	Semivolatile Organic Compounds
Anticipated Concentration Level	Low to Moderate
Analytical Method/SOP Reference[1]	United States Environmental Protection Agency (USEPA) SW-846 Method 8270D/SA-SM-003, Rev. 11
Field Sampling Organization	Geosyntec Consultants, GSI, and Anchor QEA
Analytical Organization	Eurofins-TestAmerica Savannah, Georgia
No. of Sample Locations	See Worksheet #18

QC Sample	Frequency/ Number	Method/Standard Operating Procedure (SOP) QC Acceptance Limits[2]	Corrective Action	Person(s) Responsible for Corrective Action	Measurement Performance Criteria[2]
Laboratory Control Sample/ Laboratory Control Sample Duplicate (LCS/LCSD)	LCS: 1 per batch of up to 20 samples LCSD: 1 per batch of up to 20 samples when insufficient sample is provided for MS/MSD/sample duplicate (SD)	Within laboratory generated limits	Refer to SOP SA-QA-017	Analyst	Within laboratory generated limits
Matrix Spike/ Matrix Spike Duplicate (MS/MSD)	1 per batch of up to 20 samples	Within laboratory generated limits	Refer to SOP SA-QA-017	Analyst	Within laboratory generated limits
Initial Demonstration of Capability (IDOC)	Initially, per analyst, per analyte/method/ matrix combination	Refer to SOP SA-QA-006	Refer to SOP SA-QA-006. Note unsupervised work must not begin until acceptable IDOC is obtained.	Analyst	Refer to SOP SA-QA-006

Matrix	Aqueous (toxicity characteristic leaching procedure [TCLP] leachate, aqueous water quality control [QC] samples, equipment/source blanks)
Analytical Group	Semivolatile Organic Compounds
Anticipated Concentration Level	Low to Moderate
Analytical Method/SOP Reference[1]	United States Environmental Protection Agency (USEPA) SW-846 Method 8270D/SA-SM-003, Rev. 11
Field Sampling Organization	Geosyntec Consultants, GSI, and Anchor QEA
Analytical Organization	Eurofins-TestAmerica Savannah, Georgia
No. of Sample Locations	See Worksheet #18

QC Sample	Frequency/ Number	Method/Standard Operating Procedure (SOP) QC Acceptance Limits[2]	Corrective Action	Person(s) Responsible for Corrective Action	Measurement Performance Criteria[2]
Continued Demonstration of Capability (CDOC)	Annually, per analyst, per analyte/method/ matrix combination	Refer to SOP SA-QA-006	Refer to SOP SA-QA-006.	Analyst	Refer to SOP SA-QA-006
Reporting Limit Verification (RLV)	Upon method/ instrument set-up, per analyte/ method/matrix/ combination Perform RLV study annually thereafter.	Refer to SOP SA-QA-007	Refer to SOP SA-QA-007	Analyst	Refer to SOP SA-QA-007

Matrix	Aqueous (toxicity characteristic leaching procedure [TCLP] leachate, aqueous water quality control [QC] samples, equipment/source blanks)
Analytical Group	Semivolatile Organic Compounds
Anticipated Concentration Level	Low to Moderate
Analytical Method/SOP Reference[1]	United States Environmental Protection Agency (USEPA) SW-846 Method 8270D/SA-SM-003, Rev. 11
Field Sampling Organization	Geosyntec Consultants, GSI, and Anchor QEA
Analytical Organization	Eurofins-TestAmerica Savannah, Georgia
No. of Sample Locations	See Worksheet #18

QC Sample	Frequency/ Number	Method/Standard Operating Procedure (SOP) QC Acceptance Limits[2]	Corrective Action	Person(s) Responsible for Corrective Action	Measurement Performance Criteria[2]
Method Detection Limit (MDL) Study MDL Verification (MDLV)	Upon method, instrument set-up, per analyte method matrix combination Perform MDLV after MDL study annually thereafter.	Refer to SOP SA-QA-007	Refer to SOP SA-QA-007	Analyst	Refer to SOP SA-QA-007

1. SOPs are reviewed/revised on an annual schedule by the laboratory. The current version of the SOP will be followed at the time of sample receipt.
2. This is a summary of the acceptance limits and measurement performance criteria; refer to the method SOP included in Attachment B of this UFP-QAPP for specific or more information. The acceptance limits and measurement performance criteria are in accordance with the government-required limits or criteria (i.e., National Functional Guidelines and/or USEPA Region 4 Laboratory Operations and Quality Assurance Manual [USEPA 2022]).

UFP-QAPP Worksheet #28c: QC Samples Table for USEPA Method 8082A – Polychlorinated Biphenyls Aroclors and Congeners

Matrix	Aqueous (groundwater, porewater performance reference compounds, aqueous water quality control [QC] samples, equipment/source blanks)/Solid (soil and sediment)
Analytical Group	Polychlorinated Biphenyls Aroclors
Anticipated Concentration Level	Low to High
Analytical Method/SOP Reference[1]	United States Environmental Protection Agency (USEPA) SW-846 Method 8082A/ Primary: PT-GC-005, Secondary: BR-GC-005
Field Sampling Organization	Geosyntec Consultants, GSI, and Anchor QEA
Analytical Organization	Eurofins-TestAmerica Burlington, Vermont, or Pittsburgh, Pennsylvania
No. of Sample Locations	See Worksheet #18

QC Sample	Frequency/ Number	Method/Standard Operating Procedure (SOP) QC Acceptance Limits[2]	Corrective Action	Person(s) Responsible for Corrective Action	Measurement Performance Criteria[2]
Initial Calibration (ICAL)	Every 6 months, or more often as needed	Average response factor (RF): < 20% relative standard deviation (RSD) Linear Regression: Linear Correlation Coefficient >0.990 and intercept <1/2 of the reporting limit (RL)	Reanalyze standard(s), Prepare new standard(s) and reanalyze, Perform injector port maintenance and reanalyze standards, Replace column and/or clean source, and reanalyze standards	Analyst	Average RF: <20% RSD Linear Regression: Linear Correlation Coefficient >0.990 and intercept <1/2 RL
Initial Calibration Verification (ICV)	After each ICAL	≤20% RSD (80%–120% recovery)	If the ICV response does not meet this limit, repeat the ICV analysis once with a freshly made standard. If failure repeats, the initial calibration and ICV standards must be prepared fresh and calibration repeated.	Analyst	≤ 20% RSD (80%–120% recovery)

Matrix	Aqueous (groundwater, porewater performance reference compounds, aqueous water quality control [QC] samples, equipment/source blanks)/Solid (soil and sediment)
Analytical Group	Polychlorinated Biphenyls Aroclors
Anticipated Concentration Level	Low to High
Analytical Method/SOP Reference[1]	United States Environmental Protection Agency (USEPA) SW-846 Method 8082A/ Primary: PT-GC-005, Secondary: BR-GC-005
Field Sampling Organization	Geosyntec Consultants, GSI, and Anchor QEA
Analytical Organization	Eurofins-TestAmerica Burlington, Vermont, or Pittsburgh, Pennsylvania
No. of Sample Locations	See Worksheet #18

QC Sample	Frequency/ Number	Method/Standard Operating Procedure (SOP) QC Acceptance Limits[2]	Corrective Action	Person(s) Responsible for Corrective Action	Measurement Performance Criteria[2]
Continuing Calibration Verification (CCV)[3]	Beginning of run and every 20 samples	% RSD ≤20% (80%-120% recovery)	Reanalyze sample batch unless criteria listed in Section 9.10.4 of the SOP are met.	Analyst	%RSD < 20%
RT Windows	With each ICAL (mid-point updated daily with continuing calibration verification internal standard [CCVIS])	+ 3X Standard Deviation/ + 0.01 min	Reprocess samples; Evaluate instrument and perform maintenance as needed	Analyst	+ 3X SD / + 0.01 min
Internal Standards (IS)	All standards, samples, and QC	Response of IS must be within a range of 50% to 150% of the ICAL midlevel standard	Inspect equipment for malfunctions and corrected. Reanalysis of samples analyzed while the system was malfunctioning is required.	Analyst	Response of IS must be within a range of 50% to 150% of the ICAL midlevel standard
Surrogate Compounds	All samples and QC	Statistical control limits are maintained by laboratory	Reprepare and reanalyze	Analyst	Statistical control limits are maintained by laboratory
Method Blank	One per batch of up to 20 samples	Less than RL	Reextract and reanalyze sample batch unless criteria listed in Section 9.5.2 of the SOP are met.	Analyst	Less than RL

Matrix	Aqueous (groundwater, porewater performance reference compounds, aqueous water quality control [QC] samples, equipment/source blanks)/Solid (soil and sediment)
Analytical Group	Polychlorinated Biphenyls Aroclors
Anticipated Concentration Level	Low to High
Analytical Method/SOP Reference[1]	United States Environmental Protection Agency (USEPA) SW-846 Method 8082A/ Primary: PT-GC-005, Secondary: BR-GC-005
Field Sampling Organization	Geosyntec Consultants, GSI, and Anchor QEA
Analytical Organization	Eurofins-TestAmerica Burlington, Vermont, or Pittsburgh, Pennsylvania
No. of Sample Locations	See Worksheet #18

QC Sample	Frequency/ Number	Method/Standard Operating Procedure (SOP) QC Acceptance Limits[2]	Corrective Action	Person(s) Responsible for Corrective Action	Measurement Performance Criteria[2]
Laboratory Control Sample (LCS)	One per batch of up to 20 samples	Within laboratory generated limits	Reextract and reanalyze sample batch unless criteria listed in Section 9.6.5 of the SOP are met.	Analyst	Within laboratory generated limits
Matrix Spike/ Matrix Spike Duplicate (MS/MSD)	One per batch of up to 20 samples	Within laboratory generated limits; MS/MSD is not used for batch control	Reextract and reanalyze sample batch unless criteria listed in Section 9.6.5 of the SOP are met. If an MS/MSD is not possible due to limited sample, then an LCS duplicate may be analyzed to monitor batch precision.	Analyst	Within laboratory generated limits; MS/MSD is not used for batch control
Dual column agreement	All positive results	40% RPD		Analyst	40% RPD

1. SOPs are reviewed/revised on an annual schedule by the laboratory. The current version of the SOP will be followed at the time of sample receipt.
2. This is a summary of the acceptance limits and measurement performance criteria; refer to the method SOP included in Attachment B of this UFP-QAPP for specific or more information. The acceptance limits and measurement performance criteria are in accordance with the government-required limits or criteria (i.e., National Functional Guidelines and/or USEPA Region 4 Laboratory Operations and Quality Assurance Manual [USEPA 2022]).
3. If the analysis includes the use of internal standards, a closing CCV is not required at the end of the analytical run.

UFP-QAPP Worksheet #28d: QC Samples Table for USEPA Method 680 – Polychlorinated Biphenyls Homologs

Matrix	Aqueous (groundwater, surface water, aqueous water quality control [QC] samples, equipment/source blanks)/Solid (soil and sediment)
Analytical Group	Polychlorinated Biphenyls (PCB) Homologs
Anticipated Concentration Level	Low to High
Analytical Method/SOP Reference[1]	United States Environmental Protection Agency (USEPA) Method 680/BR-MS-010
Field Sampling Organization	Geosyntec Consultants, GSI, and Anchor QEA
Analytical Organization	Eurofins-TestAmerica Burlington, Vermont
No. of Sample Locations	See Worksheet #18

QC Sample	Frequency/ Number	Method/Standard Operating Procedure (SOP) QC Acceptance Limits[2]	Corrective Action	Person(s) Responsible for Corrective Action	Measurement Performance Criteria[2]
Tune (DFTPP)	Every 12 hours	See Table 4 of SOP regarding DFTPP key ions and abundance criteria	Reanalyze, retune mass spectrometer	Analyst	See Table 4 of SOP regarding DFTPP key ions and abundance criteria
Initial Calibration (ICAL)	Upon instrument set-up, and after unsuccessful CCV	Minimum of 5 calibration points. Meets Midlevel Standard Performance Criteria. %RSD < 20%.	Correct problem and repeat calibration	Analyst	Minimum of 5 calibration points. Meets Midlevel Standard Performance Criteria. %RSD < 20%.

Matrix	Aqueous (groundwater, surface water, aqueous water quality control [QC] samples, equipment/source blanks)/Solid (soil and sediment)
Analytical Group	Polychlorinated Biphenyls (PCB) Homologs
Anticipated Concentration Level	Low to High
Analytical Method/SOP Reference[1]	United States Environmental Protection Agency (USEPA) Method 680/BR-MS-010
Field Sampling Organization	Geosyntec Consultants, GSI, and Anchor QEA
Analytical Organization	Eurofins-TestAmerica Burlington, Vermont
No. of Sample Locations	See Worksheet #18

QC Sample	Frequency/ Number	Method/Standard Operating Procedure (SOP) QC Acceptance Limits[2]	Corrective Action	Person(s) Responsible for Corrective Action	Measurement Performance Criteria[2]
Performance Criteria	Each midlevel standard	Mass abundance ratios of all calibration congeners within acceptance range (see SOP). Baseline separation of PCB congener #87 from congeners #154 and #77. Signal-to-noise ratio of ≥5 for decachlorobiphenyl ion 499 and chrysene-d12 ion 241. Decachlorobiphenyl mass abundance: mass 500 ≥70% but ≤95% of mass 498	Recalibrate, performing maintenance as necessary.	Analyst	Mass abundance ratios of all calibration congeners within acceptance range (see SOP). Baseline separation of PCB congener #87 from congeners #154 and #77. Signal-to-noise ratio of ≥5 for decachlorobiphenyl ion 499 and chrysene-d12 ion 241. Decachlorobiphenyl mass abundance: mass 500 ≥70% but ≤95% of mass 498
Initial Calibration Verification (ICV)	After each ICAL	%RSD <20%	Correct problem and verify second source standard. If that fails, repeat initial calibration.	Analyst	%RSD < 20%

Matrix	Aqueous (groundwater, surface water, aqueous water quality control [QC] samples, equipment/source blanks)/Solid (soil and sediment)
Analytical Group	Polychlorinated Biphenyls (PCB) Homologs
Anticipated Concentration Level	Low to High
Analytical Method/SOP Reference[1]	United States Environmental Protection Agency (USEPA) Method 680/BR-MS-010
Field Sampling Organization	Geosyntec Consultants, GSI, and Anchor QEA
Analytical Organization	Eurofins-TestAmerica Burlington, Vermont
No. of Sample Locations	See Worksheet #18

QC Sample	Frequency/ Number	Method/Standard Operating Procedure (SOP) QC Acceptance Limits[2]	Corrective Action	Person(s) Responsible for Corrective Action	Measurement Performance Criteria[2]
Continuing Calibration Verification (CCV)	12 hours, following instrument tune and Window Defining Mix	%RSD <20%	Reprepare standard and reanalyze once, if still outside criteria perform corrective action, sequence can be restarted if two successive CCVs at different concentrations pass, otherwise repeat ICAL and all associated samples since last successful CCV, unless CCV is high and samples are nondetects.	Analyst	%RSD < 20%
Instrument Blank (ICB/CCB)	After ICV and every CCV	< ½ reporting limit (RL)	Terminate the analysis, correct problem, and reanalyze the previous 10 samples.	Analyst	< ½ RL
Internal Standards (ISTD)	Spiked in all standards, and batch QC items	Area ± 50% of ICAL or ± 30% of previous CCV	Evaluate chromatogram, spectra, and integrations. Reanalyze extract to confirm. Perform instrument maintenance as necessary. If sufficient sample is available, initiate a reextract request.	Analyst	Area ± 50% of ICAL or ± 30% of previous CCV
Surrogate Compounds	All samples and batch QC item.	See table 2: Sample QC Recovery Limits in BR-MS-010 for biphenyl specific limits.	Evaluate chromatogram, spectra, and integrations; Reanalyze extract(s); Reextract and reanalyze if sufficient sample is available.	Analyst	See table 2: Sample QC Recovery Limits in BR-MS-010 for biphenyl specific limits.

Matrix	Aqueous (groundwater, surface water, aqueous water quality control [QC] samples, equipment/source blanks)/Solid (soil and sediment)
Analytical Group	Polychlorinated Biphenyls (PCB) Homologs
Anticipated Concentration Level	Low to High
Analytical Method/SOP Reference[1]	United States Environmental Protection Agency (USEPA) Method 680/BR-MS-010
Field Sampling Organization	Geosyntec Consultants, GSI, and Anchor QEA
Analytical Organization	Eurofins-TestAmerica Burlington, Vermont
No. of Sample Locations	See Worksheet #18

QC Sample	Frequency/ Number	Method/Standard Operating Procedure (SOP) QC Acceptance Limits[2]	Corrective Action	Person(s) Responsible for Corrective Action	Measurement Performance Criteria[2]
Method Blank (MB)	1 per batch of up to 20 samples	< ½ RL	Examine project data quality objectives (DQOs) and take appropriate corrective action, which may include reanalysis of MB, reextraction of batch, and/or nonconformance report (NCR). Corrective action must be documented on NCR. If there are no detects in samples, or if all detects are >10X MB level, repreparation and reanalysis may not be required.	Analyst	< ½ RL
Laboratory Control Sample (LCS)	1 per batch of up to 20 samples	See Table 2 of SOP for recovery and relative percent difference (RPD) limits for LCS	Examine project DQOs and take appropriate corrective action, which may include reanalysis of LCS, reextraction of batch, and/or nonconformance memo (NCM). Corrective action must be documented on NCM. Flag all reported values outside of control limits.	Analyst	See Table 2 of SOP for recovery and RPD limits for LCS

Matrix	Aqueous (groundwater, surface water, aqueous water quality control [QC] samples, equipment/source blanks)/Solid (soil and sediment)
Analytical Group	Polychlorinated Biphenyls (PCB) Homologs
Anticipated Concentration Level	Low to High
Analytical Method/SOP Reference[1]	United States Environmental Protection Agency (USEPA) Method 680/BR-MS-010
Field Sampling Organization	Geosyntec Consultants, GSI, and Anchor QEA
Analytical Organization	Eurofins-TestAmerica Burlington, Vermont
No. of Sample Locations	See Worksheet #18

QC Sample	Frequency/ Number	Method/Standard Operating Procedure (SOP) QC Acceptance Limits[2]	Corrective Action	Person(s) Responsible for Corrective Action	Measurement Performance Criteria[2]
Laboratory Control Sample Duplicate (LCSD)	1 per batch of up to 20 samples, when insufficient sample is provided for MS/MSD/sample duplicate (SD).	See Table 2 of SOP for recovery and RPD limits for LCSD	Examine project DQOs and take appropriate corrective action, which may include re-analysis of LCSD, reextraction of batch, and/or NCR. Corrective action must be documented on NCR. Flag all reported values outside of control limits.	Analyst	See Table 2 of SOP for recovery and RPD limits for LCSD
Matrix Spike/ Matrix Spike Duplicate (MS/MSD)	1 per batch of up to 20 samples	See Table 2 of SOP for recovery and RPD limits for MS/MSD	Evaluate data and determine if a matrix effect or analytical error is indicated. If analytical error, reanalyze and/or reextract. Flag all reported values outside of control limits.	Analyst	See Table 2 of SOP for recovery and RPD limits for MS/MSD

1. SOPs are reviewed/revised on an annual schedule by the laboratory. The current version of the SOP will be followed at the time of sample receipt.
2. This is a summary of the acceptance limits and measurement performance criteria; refer to the method SOP included in Attachment B of this UFP-QAPP for specific or more information. The acceptance limits and measurement performance criteria are in accordance with the government-required limits or criteria (i.e., National Functional Guidelines and/or USEPA Region 4 Laboratory Operations and Quality Assurance Manual [USEPA 2022]) if appliable.

UFP-QAPP Worksheet #28e: QC Samples Table for USEPA Method 6010C – Cations/Metals

Matrix	Aqueous (groundwater, surface water, toxicity characteristic leaching procedure [TCLP] leachate, aqueous water quality control [QC] samples, equipment/source blanks)/Solid (soil and sediment)
Analytical Group	Cations/Metals
Anticipated Concentration Level	Low to Moderate
Analytical Method/SOP Reference[1]	United States Environmental Protection Agency (USEPA) Method SW-846 6010C / SA-ME-070
Field Sampling Organization	Geosyntec Consultants, GSI, and Anchor QEA
Analytical Organization	Eurofins-TestAmerica Savannah, Georgia
No. of Sample Locations	See Worksheet #18

QC Sample	Frequency/ Number	Method/Standard Operating Procedure (SOP) QC Acceptance Limits[2]	Corrective Action	Person(s) Responsible for Corrective Action	Measurement Performance Criteria[2]
Initial Calibration (ICAL)	Daily	Single Point: 1 standard and 1 blank Multipoint: Minimum 3 standards and 1 blank; Correlation coefficient ≥0.998	Recalibrate	Analyst	Single Point: 1 standard and 1 blank Multipoint: Minimum 3 standards and 1 blank; Correlation coefficient ≥0.998
High Standard	Immediately after every calibration	Recoveries within ±5% of expected values	New initial calibration	Analyst	Recoveries within ±5% of expected values
Initial Calibration Verification (ICV)	At the beginning of the analysis	Within ±10% of the true value	Recalibrate	Analyst	Within ±10% of the true value
Continuing Calibration Verification (CCV)	At the beginning and end of analysis, and after every 10 samples	Within ±10% of the true value; ≤5% relative standard deviation (RSD)	Terminate the analysis, troubleshoot and reanalyze the previous 10 samples	Analyst	Within ±10% of the true value; ≤5%RSD

Matrix	Aqueous (groundwater, surface water, toxicity characteristic leaching procedure [TCLP] leachate, aqueous water quality control [QC] samples, equipment/source blanks)/Solid (soil and sediment)
Analytical Group	Cations/Metals
Anticipated Concentration Level	Low to Moderate
Analytical Method/SOP Reference[1]	United States Environmental Protection Agency (USEPA) Method SW-846 6010C / SA-ME-070
Field Sampling Organization	Geosyntec Consultants, GSI, and Anchor QEA
Analytical Organization	Eurofins-TestAmerica Savannah, Georgia
No. of Sample Locations	See Worksheet #18

QC Sample	Frequency/ Number	Method/Standard Operating Procedure (SOP) QC Acceptance Limits[2]	Corrective Action	Person(s) Responsible for Corrective Action	Measurement Performance Criteria[2]
Calibration Blank (ICB/CCB)	After ICV and every CCV	Absolute value < ½ the reporting limit (RL)	Terminate the analysis, troubleshoot and reanalyze the previous 10 samples	Analyst	Absolute value < 1/2 RL
Interference Check Standards (ICSA/ICSAB)	At the beginning of the analysis	±20% of the true values Pay attention to false positives and false negatives for elements not present in solutions.	Terminate the analysis, correct problem, recalibrate, and reanalyze all samples since the last ICS that was in control	Analyst	±20% of the true values Pay attention to false positives and false negatives for elements not present in solutions.
Method Blank (MB)	1 per batch of up to 20 samples or less	Absolute value of result less than 1/2 RL or result <10% of the analyte level in the sample	Refer to SOP SA-QA-017	Analyst	Absolute value of result less than 1/2 RL or result <10% of the analyte level in the sample

Matrix	Aqueous (groundwater, surface water, toxicity characteristic leaching procedure [TCLP] leachate, aqueous water quality control [QC] samples, equipment/source blanks)/Solid (soil and sediment)
Analytical Group	Cations/Metals
Anticipated Concentration Level	Low to Moderate
Analytical Method/SOP Reference[1]	United States Environmental Protection Agency (USEPA) Method SW-846 6010C / SA-ME-070
Field Sampling Organization	Geosyntec Consultants, GSI, and Anchor QEA
Analytical Organization	Eurofins-TestAmerica Savannah, Georgia
No. of Sample Locations	See Worksheet #18

QC Sample	Frequency/ Number	Method/Standard Operating Procedure (SOP) QC Acceptance Limits[2]	Corrective Action	Person(s) Responsible for Corrective Action	Measurement Performance Criteria[2]
Laboratory Control Sample/ Laboratory Control Sample Duplicate (LCS/LCSD)	LCS: 1 per batch of up to 20 samples LCSD: 1 per batch, if insufficient sample provided for MS/MSD	Within laboratory generated limits	Refer to SOP SA-QA-017	Analyst	Within laboratory generated limits
Matrix Spike/ Matrix Spike Duplicate (MS/MSD)	1 per batch of up to 20 samples	Within laboratory generated limits	Refer to SOP SA-QA-017	Analyst	Within laboratory generated limits
Serial Dilution (1/5 Dilution)	1 per batch of up to 20 samples	If sample concentration greater than 50x method detection limit (MDL): percent difference ≤ 10%	Troubleshoot; redigest and reanalyze if sufficient sample available; otherwise, flag data as appropriate	Analyst	If sample concentration greater than 50x MDL: percent difference ≤ 10%

Matrix	Aqueous (groundwater, surface water, toxicity characteristic leaching procedure [TCLP] leachate, aqueous water quality control [QC] samples, equipment/source blanks)/Solid (soil and sediment)
Analytical Group	Cations/Metals
Anticipated Concentration Level	Low to Moderate
Analytical Method/SOP Reference[1]	United States Environmental Protection Agency (USEPA) Method SW-846 6010C / SA-ME-070
Field Sampling Organization	Geosyntec Consultants, GSI, and Anchor QEA
Analytical Organization	Eurofins-TestAmerica Savannah, Georgia
No. of Sample Locations	See Worksheet #18

QC Sample	Frequency/ Number	Method/Standard Operating Procedure (SOP) QC Acceptance Limits[2]	Corrective Action	Person(s) Responsible for Corrective Action	Measurement Performance Criteria[2]
Post-digestion Spike	1 per batch of up to 20 samples	75%–125% recovery	Troubleshoot; redigest and reanalyze if sufficient sample available; otherwise, flag data as appropriate	Analyst	75%–125% recovery
Reporting Limit Check Solution	At the beginning and end of analysis run when using both single point and multipoint calibrations.	+/- 30% of true concentration	Opening: Stop analysis, troubleshoot and reanalyze affected samples Capping: Qualify affected samples; reanalyze if required by project plan	Analyst	+/- 30% of true concentration

Matrix	Aqueous (groundwater, surface water, toxicity characteristic leaching procedure [TCLP] leachate, aqueous water quality control [QC] samples, equipment/source blanks)/Solid (soil and sediment)
Analytical Group	Cations/Metals
Anticipated Concentration Level	Low to Moderate
Analytical Method/SOP Reference[1]	United States Environmental Protection Agency (USEPA) Method SW-846 6010C / SA-ME-070
Field Sampling Organization	Geosyntec Consultants, GSI, and Anchor QEA
Analytical Organization	Eurofins-TestAmerica Savannah, Georgia
No. of Sample Locations	See Worksheet #18

QC Sample	Frequency/ Number	Method/Standard Operating Procedure (SOP) QC Acceptance Limits[2]	Corrective Action	Person(s) Responsible for Corrective Action	Measurement Performance Criteria[2]
%RSD (CV) of Multiple Exposures	Evaluate for all Calibration, QC, and samples	Conc. ≥ RL - Warning: ≤20% - Acceptance: ≤30%	If CV>20 but ≤30, review data for possible interferences; If interference present, reanalyze digest; If no interference present, report average; If CV≥30%, reanalyze digest, report the result that has the lower precision value or dilute the digestion and reanalyze	Analyst	Conc. ≥ RL - Warning: ≤20% - Acceptance: ≤30%
		Conc. < RL Use professional judgment	Use professional judgment		Conc. < RL Use professional judgment
Linear Range	Determined at least every 6 months in accordance with Attachment 7 of SOP.	% difference ≤10%	Reanalyze at a lower concentration	Analyst	% difference ≤10%

Matrix	Aqueous (groundwater, surface water, toxicity characteristic leaching procedure [TCLP] leachate, aqueous water quality control [QC] samples, equipment/source blanks)/Solid (soil and sediment)
Analytical Group	Cations/Metals
Anticipated Concentration Level	Low to Moderate
Analytical Method/SOP Reference[1]	United States Environmental Protection Agency (USEPA) Method SW-846 6010C / SA-ME-070
Field Sampling Organization	Geosyntec Consultants, GSI, and Anchor QEA
Analytical Organization	Eurofins-TestAmerica Savannah, Georgia
No. of Sample Locations	See Worksheet #18

QC Sample	Frequency/ Number	Method/Standard Operating Procedure (SOP) QC Acceptance Limits[2]	Corrective Action	Person(s) Responsible for Corrective Action	Measurement Performance Criteria[2]
Interelement Correction Factors (IEC)	Determined every 6 months. Verified at the beginning of an analysis run	Refer to ICSA, ICSAB criteria	See ICSA, ICSAB corrective action	Analyst	Refer to ICSA, ICSAB criteria
Lower Limit of Quantitation Check Sample	Annually	70%–130%	Reanalyze at a higher concentration, elevate RL accordingly	Analyst	70%–130%
Initial Demonstration of Capability (IDOC)	Initially, per analyst, per analyte/method/ matrix combination	Refer to SOP SA-QA-006	Refer to SOP SA-QA-006. Note unsupervised work must not begin until acceptable IDOC is obtained.	Analyst	Refer to SOP SA-QA-006
Continued Demonstration of Capability (CDOC)	Annually, per analyst, per analyte/ method/matrix combination	Refer to SOP SA-QA-006	Refer to SOP SA-QA-006	Analyst	Refer to SOP SA-QA-006

Matrix	Aqueous (groundwater, surface water, toxicity characteristic leaching procedure [TCLP] leachate, aqueous water quality control [QC] samples, equipment/source blanks)/Solid (soil and sediment)
Analytical Group	Cations/Metals
Anticipated Concentration Level	Low to Moderate
Analytical Method/SOP Reference[1]	United States Environmental Protection Agency (USEPA) Method SW-846 6010C / SA-ME-070
Field Sampling Organization	Geosyntec Consultants, GSI, and Anchor QEA
Analytical Organization	Eurofins-TestAmerica Savannah, Georgia
No. of Sample Locations	See Worksheet #18

QC Sample	Frequency/ Number	Method/Standard Operating Procedure (SOP) QC Acceptance Limits[2]	Corrective Action	Person(s) Responsible for Corrective Action	Measurement Performance Criteria[2]
Reporting Limit Verification (RLV)	Upon method/ instrument setup, per analyte/method/ matrix combination. Then annually thereafter	Refer to SOP SA-QA-007	Refer to SOP-QA-007	Analyst	Refer to SOP SA-QA-007
Method Detection Limit (MDL) Study	Upon method/ instrument setup, per analyte/method/ matrix combination	Refer to SOP SA-QA-007	Refer to SOP-QA-007	Analyst	Refer to SOP SA-QA-007
MDL Verification (MDLV)	Upon method/ instrument setup, per analyte/method/ matrix combination. Then annually thereafter	Refer to SOP SA-QA-007	Refer to SOP-QA-007	Analyst	Refer to SOP SA-QA-007

Matrix	Aqueous (groundwater, surface water, toxicity characteristic leaching procedure [TCLP] leachate, aqueous water quality control [QC] samples, equipment/source blanks)/Solid (soil and sediment)
Analytical Group	Cations/Metals
Anticipated Concentration Level	Low to Moderate
Analytical Method/SOP Reference[1]	United States Environmental Protection Agency (USEPA) Method SW-846 6010C / SA-ME-070
Field Sampling Organization	Geosyntec Consultants, GSI, and Anchor QEA
Analytical Organization	Eurofins-TestAmerica Savannah, Georgia
No. of Sample Locations	See Worksheet #18

QC Sample	Frequency/ Number	Method/Standard Operating Procedure (SOP) QC Acceptance Limits[2]	Corrective Action	Person(s) Responsible for Corrective Action	Measurement Performance Criteria[2]
Instrument Detection Limit (IDL)	Upon method/instrument set-up. Then quarterly thereafter	Refer to SOP SA-QA-007	Refer to SOP-QA-007	Analyst	Refer to SOP SA-QA-007

1. SOPs are reviewed/revised on an annual schedule by the laboratory. The current version of the SOP will be followed at the time of sample receipt.
2. This is a summary of the acceptance limits and measurement performance criteria; refer to the method SOP included in Attachment B of this UFP-QAPP for specific or more information. The acceptance limits and measurement performance criteria are in accordance with the government-required limits or criteria (i.e., National Functional Guidelines and/or USEPA Region 4 Laboratory Operations and Quality Assurance Manual [USEPA 2022]) if appliable.

UFP-QAPP Worksheet #28f: QC Samples Table for USEPA Methods 7470A and 7471B – Mercury

Matrix	Aqueous (sediment filtrate, toxicity characteristic leaching procedure [TCLP] leachate. quality control [QC] samples, equipment/ source blanks)/Solid (soil and sediment)
Analytical Group	Mercury
Anticipated Concentration Level	Low to Moderate
Analytical Method/SOP Reference[1]	United States Environmental Protection Agency (USEPA) Methods 7470A and 7471B/SA-ME-028
Field Sampling Organization	Geosyntec Consultants, GSI, and Anchor QEA
Analytical Organization	Eurofins-TestAmerica Savannah, Georgia
No. of Sample Locations	See Worksheet #18

QC Sample	Frequency/ Number	Method/Standard Operating Procedure (SOP) QC Acceptance Limits[2]	Corrective Action	Person(s) Responsible for Corrective Action	Measurement Performance Criteria[2]
Initial Calibration (ICAL)	Daily	Correlation ≥ 0.995 (1 blank and 5 standards)	Troubleshoot; recalibrate	Analyst	Correlation ≥ 0.995 (1 blank and 5 standards)
Initial Calibration Verification (ICV)	At the beginning of the analysis	Within ±10% of the true value	Troubleshoot; recalibrate	Analyst	Within ±10% of the true value
Continuing Calibration Verification (CCV)	At the beginning and end of analysis, and after every 10 samples	Within ±10% of the true value	Terminate the analysis, correct problem, recalibrate, and reanalyze all samples since the last compliant CCV	Analyst	Within ±10% of the true value
Calibration Blank (ICB/CCB)	After ICV and every CCV	Absolute value of the calibration blank must be less than ½ the reporting limit (RL)	Terminate the analysis, correct problem, recalibrate, and reanalyze all samples since the last compliant calibration blank	Analyst	Absolute value of the calibration blank must be less than ½ RL

Matrix	Aqueous (sediment filtrate, toxicity characteristic leaching procedure [TCLP] leachate. quality control [QC] samples, equipment/ source blanks)/Solid (soil and sediment)
Analytical Group	Mercury
Anticipated Concentration Level	Low to Moderate
Analytical Method/SOP Reference[1]	United States Environmental Protection Agency (USEPA) Methods 7470A and 7471B/SA-ME-028
Field Sampling Organization	Geosyntec Consultants, GSI, and Anchor QEA
Analytical Organization	Eurofins-TestAmerica Savannah, Georgia
No. of Sample Locations	See Worksheet #18

QC Sample	Frequency/ Number	Method/Standard Operating Procedure (SOP) QC Acceptance Limits[2]	Corrective Action	Person(s) Responsible for Corrective Action	Measurement Performance Criteria[2]
RL Standard	After every calibration but not before the ICV. This standard is not a requirement of SW846, if a multipoint calibration encompassing the RL level is used	50%–150% of true value	Troubleshoot; recalibrate	Analyst	50%–150% of true value
Method Blank	1 per batch of up to 20 samples	Less than ½ RL	Refer to SOP SA-QA-017	Analyst	Less than ½ RL
Laboratory Control Sample (LCS)	1 per batch of up to 20 samples	Within laboratory generated limits	Refer to SOP SA-QA-017	Analyst	Within laboratory generated limits
Matrix Spike/ Matrix Spike Duplicate (MS/MSD)	1 per batch of up to 20 samples	Within laboratory generated limits	Refer to SOP SA-QA-017	Analyst	Within laboratory generated limits

Matrix	Aqueous (sediment filtrate, toxicity characteristic leaching procedure [TCLP] leachate. quality control [QC] samples, equipment/ source blanks)/Solid (soil and sediment)
Analytical Group	Mercury
Anticipated Concentration Level	Low to Moderate
Analytical Method/SOP Reference[1]	United States Environmental Protection Agency (USEPA) Methods 7470A and 7471B/SA-ME-028
Field Sampling Organization	Geosyntec Consultants, GSI, and Anchor QEA
Analytical Organization	Eurofins-TestAmerica Savannah, Georgia
No. of Sample Locations	See Worksheet #18

QC Sample	Frequency/ Number	Method/Standard Operating Procedure (SOP) QC Acceptance Limits[2]	Corrective Action	Person(s) Responsible for Corrective Action	Measurement Performance Criteria[2]
Post Digestion Spikes (PDS)	When MS/MSD is unacceptable	Within laboratory generated limits	Refer to SOP SA-QA-017	Analyst	Within laboratory generated limits
Initial Demonstration of Capability (IDOC)	Initially, per analyst, per analyte/method/ matrix combination	Refer to SOP SA-QA-006	Refer to SOP SA-QA-006. Note unsupervised work must not begin until acceptable IDOC is obtained.	Analyst	Refer to SOP SA-QA-006
Continued Demonstration of Capability (CDOC)	Annually, per analyst, per analyte/method/ matrix combination	Refer to SOP SA-QA-006	Refer to SOP SA-QA-006	Analyst	Refer to SOP SA-QA-006
Reporting Limit Verification (RLV)	Upon method/ instrument setup, per analyte/ method/matrix combination. Then annually thereafter	Refer to SOP SA-QA-007	Refer to SOP-QA-007	Analyst	Refer to SOP SA-QA-007

Matrix	Aqueous (sediment filtrate, toxicity characteristic leaching procedure [TCLP] leachate. quality control [QC] samples, equipment/ source blanks)/Solid (soil and sediment)
Analytical Group	Mercury
Anticipated Concentration Level	Low to Moderate
Analytical Method/SOP Reference[1]	United States Environmental Protection Agency (USEPA) Methods 7470A and 7471B/SA-ME-028
Field Sampling Organization	Geosyntec Consultants, GSI, and Anchor QEA
Analytical Organization	Eurofins-TestAmerica Savannah, Georgia
No. of Sample Locations	See Worksheet #18

QC Sample	Frequency/ Number	Method/Standard Operating Procedure (SOP) QC Acceptance Limits[2]	Corrective Action	Person(s) Responsible for Corrective Action	Measurement Performance Criteria[2]
Method Detection Limit (MDL) Study	Upon method/ instrument setup, per analyte/ method/matrix combination	Refer to SOP SA-QA-007	Refer to SOP-QA-007	Analyst	Refer to SOP SA-QA-007
MDL Verification (MDLV)	Upon method/ instrument setup, per analyte/ method/matrix combination. Then annually thereafter	Refer to SOP SA-QA-007	Refer to SOP-QA-007	Analyst	Refer to SOP SA-QA-007
Instrument Detection Limit (IDL)	Upon method/instrument set-up. Then quarterly thereafter	Refer to SOP SA-QA-007	Refer to SOP-QA-007	Analyst	Refer to SOP SA-QA-007

1. SOPs are reviewed/revised on an annual schedule by the laboratory. The current version of the SOP will be followed at the time of sample receipt.
2. This is a summary of the acceptance limits and measurement performance criteria; refer to the method SOP included in Attachment B of this UFP-QAPP for specific or more information. The acceptance limits and measurement performance criteria are in accordance with the government-required limits or criteria (i.e., National Functional Guidelines and/or USEPA Region 4 Laboratory Operations and Quality Assurance Manual [USEPA 2022]) if appliable.

UFP-QAPP Worksheet #28g: QC Samples Table for USEPA Method 300.0 – Anions

Matrix	Aqueous (groundwater, aqueous water quality control [QC] samples, equipment/source blanks)
Analytical Group	Anions
Anticipated Concentration Level	Low to Moderate
Analytical Method/SOP Reference	United States Environmental Protection Agency (USEPA) Method 300.0/PT-GC-009
Field Sampling Organization	Geosyntec Consultants, GSI, and Anchor QEA
Analytical Organization	Eurofins-TestAmerica Pittsburgh, Pennsylvania
No. of Sample Locations	See Worksheet #18

QC Sample	Frequency/ Number	Method/Standard Operating Procedure (SOP) QC Acceptance Limits[2]	Corrective Action	Person(s) Responsible for Corrective Action	Measurement Performance Criteria[2]
Initial Calibration (ICAL)	Prior to sample analysis; when CCV fails; at lease every 6 months	$r^2 \geq 0.995$ (Minimum three standards and one blank)	Recalibrate	Analyst	$r^2 \geq 0.995$ (Minimum three standards and one blank)
Low Level of Quantitation (LLOQ)	After analysis of ICAL	50%–150% of true value	Troubleshoot, reanalyze, if second failure perform ICAL	Analyst	50%–150% of true value
Initial Calibration Verification (ICV)	After each ICAL	Within ±10% of the true value	Troubleshoot; reanalyze ICV if second failure perform ICAL	Analyst	Within ±10% of the true value

Matrix	Aqueous (groundwater, aqueous water quality control [QC] samples, equipment/source blanks)
Analytical Group	Anions
Anticipated Concentration Level	Low to Moderate
Analytical Method/SOP Reference	United States Environmental Protection Agency (USEPA) Method 300.0/PT-GC-009
Field Sampling Organization	Geosyntec Consultants, GSI, and Anchor QEA
Analytical Organization	Eurofins-TestAmerica Pittsburgh, Pennsylvania
No. of Sample Locations	See Worksheet #18

QC Sample	Frequency/ Number	Method/Standard Operating Procedure (SOP) QC Acceptance Limits[2]	Corrective Action	Person(s) Responsible for Corrective Action	Measurement Performance Criteria[2]
Continuing Calibration Verification (CCV)	At the beginning and end of analysis, and after every 10 samples	Within ±10% of the true value	Terminate the analysis, correct problem, recalibrate, and reanalyze previous 10 samples; if results are associated with a failed CCV, they must be reported with qualifier in all cases	Analyst	Within ±10% of the true value
Continuing Calibration Blank (CCB)	After every CCV	Result < method detection limit (MDL)	Source of contamination should be investigated and corrected and all associated samples reanalyzed, unless one of the situations described in Section 9.1.1 of the SOP exists	Analyst	Result < MDL

Matrix	Aqueous (groundwater, aqueous water quality control [QC] samples, equipment/source blanks)
Analytical Group	Anions
Anticipated Concentration Level	Low to Moderate
Analytical Method/SOP Reference	United States Environmental Protection Agency (USEPA) Method 300.0/PT-GC-009
Field Sampling Organization	Geosyntec Consultants, GSI, and Anchor QEA
Analytical Organization	Eurofins-TestAmerica Pittsburgh, Pennsylvania
No. of Sample Locations	See Worksheet #18

QC Sample	Frequency/ Number	Method/Standard Operating Procedure (SOP) QC Acceptance Limits[2]	Corrective Action	Person(s) Responsible for Corrective Action	Measurement Performance Criteria[2]
Method Blank	1 per batch of up to 20 samples	Result < MDL	Source of contamination should be investigated and corrected and all associated samples reanalyzed, unless one of the situations described in Section 9.1.1 of the SOP exists	Analyst	Result < MDL
Laboratory Control Sample/ Laboratory Control Sample Duplicate (LCS/LCSD)	1 per batch of up to 20 samples	90%–110% of true value	Reanalyze all samples associated with unacceptable LCS	Analyst	90%–110% of true value

Matrix	Aqueous (groundwater, aqueous water quality control [QC] samples, equipment/source blanks)
Analytical Group	Anions
Anticipated Concentration Level	Low to Moderate
Analytical Method/SOP Reference	United States Environmental Protection Agency (USEPA) Method 300.0/PT-GC-009
Field Sampling Organization	Geosyntec Consultants, GSI, and Anchor QEA
Analytical Organization	Eurofins-TestAmerica Pittsburgh, Pennsylvania
No. of Sample Locations	See Worksheet #18

QC Sample	Frequency/ Number	Method/Standard Operating Procedure (SOP) QC Acceptance Limits[2]	Corrective Action	Person(s) Responsible for Corrective Action	Measurement Performance Criteria[2]
Matrix Spike/ Matrix Spike Duplicate (MS/MSD)	1 per batch of up to 10 samples	Within laboratory generated limits; 90%–110% ±20% RPD	Troubleshoot; reprepare and reanalyze if sufficient sample available; if an MS/MSD is not possible due to limited sample volume then an LCS duplicate may be analyzed.	Analyst	Within laboratory generated limits; 90%–110% ±20% RPD

1. SOPs are reviewed/revised on an annual schedule by the laboratory. The current version of the SOP will be followed at the time of sample receipt.
2. This is a summary of the acceptance limits and measurement performance criteria; refer to the method SOP included in Attachment B of this UFP-QAPP for specific or more information. The acceptance limits and measurement performance criteria are in accordance with the government-required limits or criteria (i.e., National Functional Guidelines and/or USEPA Region 4 Laboratory Operations and Quality Assurance Manual [USEPA 2022]) if applicable.

UFP-QAPP Worksheet #28h: QC Samples Table for SM 2340B– Hardness by Calculation

Matrix	Aqueous (toxicity characteristic leaching procedure [TCLP] leachate)
Analytical Group	Hardness
Anticipated Concentration Level	Low to Moderate
Analytical Method/SOP Reference[1]	Standard Methods 2340B/PT-MT-WI-011
Field Sampling Organization	Geosyntec Consultants, GSI, and Anchor QEA
Analytical Organization	Eurofins-TestAmerica Pittsburgh, Pennsylvania
No. of Sample Locations	See Worksheet #18

Quality Control (QC) Sample	Frequency/ Number	Method/Standard Operating Procedure (SOP) QC Acceptance Limits[2]	Corrective Action	Person(s) Responsible for Corrective Action	Measurement Performance Criteria[2]
Hardness is completed by calculation using calcium and magnesium concentrations from ICP or ICPMS analysis (United States Environmental Protection Agency [USEPA] Method 6010C). See UFP-QAPP Worksheet #28e for QC samples associated with USEPA Method 6010C.					

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2. This is a summary of the acceptance limits and measurement performance criteria; refer to the method SOP included in Attachment B of this UFP-QAPP for specific or more information. The acceptance limits and measurement performance criteria are in accordance with the government-required limits or criteria (i.e., National Functional Guidelines and/or USEPA Region 4 Laboratory Operations and Quality Assurance Manual [USEPA 2022]) if applicable.

UFP-QAPP Worksheet #28i: QC Samples Table for SM 2540D– Total Dissolved Solids and Total Suspended Solids

Matrix	Aqueous (groundwater, aqueous water quality control [QC] samples, equipment/source blanks)
Analytical Group	Total Dissolved Solids and Total Suspended Solids
Anticipated Concentration Level	Low to Moderate
Analytical Method / SOP Reference[1]	Standard Methods 2540C/D/PT-WC-043 and PT-WC-045
Field Sampling Organization	Geosyntec Consultants, GSI, and Anchor QEA
Analytical Organization	Eurofins-TestAmerica Pittsburgh, Pennsylvania
No. of Sample Locations	See Worksheet #18

QC Sample	Frequency/ Number	Method/Standard Operating Procedure (SOP) QC Acceptance Limits[2]	Corrective Action	Person(s) Responsible for Corrective Action	Measurement Performance Criteria[2]
Method Blank	1 per batch of up to 20 samples	Result < reporting limit (RL)	Reanalyze batch	Analyst	Result < RL
Laboratory Control Sample (LCS)	1 per batch of up to 20 samples	Within laboratory generated limits; 80% to 120% of true or certified value	Reanalyze all associated samples with an acceptable LCS, unless the original LCS recovery is high and sample results are <RL	Analyst	Within laboratory generated limits; 80% to 120% of true or certified value
Sample Duplicate (SD)	1 per batch of up to 10 samples	Relative percent difference (RPD) within laboratory generated limits; 10%	Reanalyze; if second failure flag data as appropriate and address in case narrative	Analyst	RPD within laboratory generated limits; 10%

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2. This is a summary of the acceptance limits and measurement performance criteria; refer to the method SOP included in Attachment B of this UFP-QAPP for specific or more information. The acceptance limits and measurement performance criteria are in accordance with the government-required limits or criteria (i.e., National Functional Guidelines and/or USEPA Region 4 Laboratory Operations and Quality Assurance Manual [USEPA 2022]) if applicable.

UFP-QAPP Worksheet #28j: QC Samples Table for Lloyd Khan – Total Organic Carbon

Matrix	Solid (soil and sediment)
Analytical Group	Organic Carbon
Anticipated Concentration Level	Low to Moderate
Analytical Method/SOP Reference[1]	Lloyd Khan/BR-WC-008
Field Sampling Organization	Geosyntec Consultants, GSI, and Anchor QEA
Analytical Organization	Eurofins-TestAmerica, Burlington, Vermont
No. of Sample Locations	See Worksheet #18

Quality Control (QC) Sample	Frequency/ Number	Method/Standard Operating Procedure (SOP) QC Acceptance Limits[2]	Corrective Action	Person(s) Responsible for Corrective Action	Measurement Performance Criteria[2]
Method Blank	1 per batch of up to 20 samples	Result < reporting limit (RL)	Troubleshoot; reanalyze if sufficient sample available; otherwise, flag data as appropriate	Analyst	Result < RL
Laboratory Control Sample/ Laboratory Control Sample Duplicate (LCS/LCSD)	1 per batch of up to 20 samples	Within laboratory generated limits; % recovery (75–125)	Troubleshoot; reanalyze if sufficient sample available; otherwise, flag data as appropriate	Analyst	Within laboratory generated limits; % recovery (75–125)
Matrix Spike/ Matrix Spike Duplicate (MS/MSD)	1 per batch of up to 20 samples	Within laboratory generated limits	Troubleshoot; reanalyze if sufficient sample available; otherwise, flag data as appropriate	Analyst	Within laboratory generated limits
Initial Calibration (ICAL)	Initial method set-up, after combustion chamber is changed (approx. every 200 drops)	Correlation coefficient must be >0.995	Troubleshoot; standards check, recalibrate	Analyst	Correlation coefficient must be >0.995
Initial Calibration Verification (ICV)	Following calibration	% recovery (75–125)	Troubleshoot; reanalyze ICV if second failure perform ICAL	Analyst	% recovery (75–125)

Matrix	Solid (soil and sediment)
Analytical Group	Organic Carbon
Anticipated Concentration Level	Low to Moderate
Analytical Method/SOP Reference[1]	Lloyd Khan/BR-WC-008
Field Sampling Organization	Geosyntec Consultants, GSI, and Anchor QEA
Analytical Organization	Eurofins-TestAmerica, Burlington, Vermont
No. of Sample Locations	See Worksheet #18

Quality Control (QC) Sample	Frequency/ Number	Method/Standard Operating Procedure (SOP) QC Acceptance Limits[2]	Corrective Action	Person(s) Responsible for Corrective Action	Measurement Performance Criteria[2]
Calibration Verification (CCV)	Every 20 drops and at the end of the analytical sequence	% recovery (75–125)	Terminate the analysis, correct problem, recalibrate, and reanalyze previous samples. If results are associated with a failed CCV, they must be reported with qualifier in all cases.	Analyst	% recovery (75–125)
Calibration Blank (CCB)	After every CCV	<RL	Source of contamination should be investigated and corrected and all associated samples reanalyzed.	Analyst	<RL

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2. This is a summary of the acceptance limits and measurement performance criteria; refer to the method SOP included in Attachment B of this UFP-QAPP for specific or more information. The acceptance limits and measurement performance criteria are in accordance with the government-required limits or criteria (i.e., National Functional Guidelines and/or USEPA Region 4 *Laboratory Operations and Quality Assurance Manual* [USEPA 2022]) if applicable.

UFP-QAPP Worksheet #28k: QC Samples Table for 9060A – Total and Dissolved Organic Carbon

Matrix	Aqueous (groundwater, aqueous water quality control [QC] samples, equipment/source blanks)
Analytical Group	Organic Carbon
Anticipated Concentration Level	Low to Moderate
Analytical Method/SOP Reference[1]	9060A/PT-WC-017
Field Sampling Organization	Geosyntec Consultants, GSI, and Anchor QEA
Analytical Organization	Eurofins-TestAmerica Pittsburgh, Pennsylvania
No. of Sample Locations	See Worksheet #18

QC Sample	Frequency/ Number	Method/Standard Operating Procedure (SOP) QC Acceptance Limits[2]	Corrective Action	Person(s) Responsible for Corrective Action	Measurement Performance Criteria[2]
Method Blank	1 per batch of up to 20 samples	Result < reporting limit (RL)	Troubleshoot; reanalyze if sufficient sample available; otherwise, flag data as appropriate	Analyst	Result < RL Note: If individual results and average are reported, each individual injection must meet the MB criteria.
Laboratory Control Sample/ Laboratory Control Sample Duplicate (LCS/LCSD)	LCS: 1 per batch of up to 20 samples LCSD: if insufficient sample provided for an MS/MSD	Within laboratory generated limits; % recovery (85–115); 20% relative standard deviation (RSD) for LCSD	Troubleshoot; reinject the standard once. If failure repeats, all associated samples must be reanalyzed unless one of the situations listed in section 9.3.1 of the SOP exists.	Analyst	Within laboratory generated limits; % recovery (85–115); 20% RSD for LCSD
Matrix Spike/ Matrix Spike Duplicate (MS/MSD)	1 per batch of up to 10 samples	Within laboratory generated limits; % recovery (75–125); 20% RSD	Troubleshoot; reanalyze if sufficient sample available; otherwise flag data as appropriate	Analyst	Within laboratory generated limits; % recovery (75–125); 20% RSD

Matrix	Aqueous (groundwater, aqueous water quality control [QC] samples, equipment/source blanks)
Analytical Group	Organic Carbon
Anticipated Concentration Level	Low to Moderate
Analytical Method/SOP Reference[1]	9060A/PT-WC-017
Field Sampling Organization	Geosyntec Consultants, GSI, and Anchor QEA
Analytical Organization	Eurofins-TestAmerica Pittsburgh, Pennsylvania
No. of Sample Locations	See Worksheet #18

QC Sample	Frequency/ Number	Method/Standard Operating Procedure (SOP) QC Acceptance Limits[2]	Corrective Action	Person(s) Responsible for Corrective Action	Measurement Performance Criteria[2]
Initial Calibration (ICAL)	At least every 6 months or more often as needed when CCVs or CCBs fail to meet acceptance criteria	Correlation coefficient must be >0.995	Troubleshoot; prepare fresh standards and repeat calibration.	Analyst	Correlation coefficient must be >0.995
Initial Calibration Verification (ICV)	Following calibration	% recovery (90–110)	Troubleshoot; prepare fresh standards and repeat calibration. reanalyze ICV if second failure perform ICAL	Analyst	% recovery (90–110)
Initial Calibration Blank (ICB)	Following ICV	< RL	Source of contamination should be investigated and corrected and repeat calibration if needed.	Analyst	< RL
Calibration Verification (CCV)	Every 10 samples and at end of run using midrange CCV	% recovery (90–110)	Terminate the analysis, correct problem, recalibrate, and reanalyze previous samples. If results are associated with a failed CCV, they must be reported with qualifier in all cases.	Analyst	% recovery (90–110)
Calibration Blank (CCB)	Every 10 samples	< RL	Source of contamination should be investigated and corrected and all associated samples reanalyzed.	Analyst	< RL

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2. This is a summary of the acceptance limits and measurement performance criteria; refer to the method SOP included in Attachment B of this UFP-QAPP for specific or more information. The acceptance limits and measurement performance criteria are in accordance with the government-required limits or criteria (i.e., National Functional Guidelines and/or USEPA Region 4 Laboratory Operations and Quality Assurance Manual [USEPA 2022]) if applicable.

UFP-QAPP Worksheet #28l: QC Samples Table for 9045D - Corrosivity (pH)

Matrix	Aqueous (toxicity characteristic leaching procedure [TCLP] leachate)
Analytical Group	Corrosivity (pH)
Anticipated Concentration Level	Low to Moderate
Analytical Method/SOP Reference[1]	United States Environmental Protection Agency (USEPA) SW-846 9045D/PT-WC-026
Field Sampling Organization	Geosyntec Consultants, GSI, and Anchor QEA
Analytical Organization	Eurofins-TestAmerica Pittsburgh, PA
No. of Sample Locations	See Worksheet #18

Quality Control (QC) Sample:	Frequency/ Number	Method/SOP QC Acceptance Limits[2]	Corrective Action	Person(s) Responsible for Corrective Action	Measurement Performance Criteria[2]
Initial Calibration	Daily	The slope of the calibration must meet > 95%, or the manufacturer's requirements if tighter. The pH readings must be within 0.05 pH units of each buffer solution value.	If the manufacturer's slope requirement is not met, the meter will display an error message. Pour fresh buffer solutions into the cups and repeat the calibration. If this does not resolve the problem, change the KCL solution in the electrode. If still having a problem the probe may need to be changed. Note any needed maintenance in the meter maintenance log, including electrode maintenance. If pH readings are not within 0.05 pH units of each buffer solution value, repeat adjustments on successive portions of the two buffer solutions until the readings are within 0.05 pH units (s.u.).	Analyst	The slope of the calibration must meet > 95%, or the manufacturer's requirements if tighter. The pH readings must be within 0.05 pH units of each buffer solution value.

Matrix	Aqueous (toxicity characteristic leaching procedure [TCLP] leachate)
Analytical Group	Corrosivity (pH)
Anticipated Concentration Level	Low to Moderate
Analytical Method/SOP Reference[1]	United States Environmental Protection Agency (USEPA) SW-846 9045D/PT-WC-026
Field Sampling Organization	Geosyntec Consultants, GSI, and Anchor QEA
Analytical Organization	Eurofins-TestAmerica Pittsburgh, PA
No. of Sample Locations	See Worksheet #18

Quality Control (QC) Sample:	Frequency/ Number	Method/SOP QC Acceptance Limits[2]	Corrective Action	Person(s) Responsible for Corrective Action	Measurement Performance Criteria[2]
Initial Calibration Verification/ Laboratory Control Sample (ICV/LCS)	Second source pH buffer 7 is analyzed after initial calibration	Reading between 6.95 to 7.05 pH units	If the ICV reading is outside the range, repeat once with a fresh portion of second source buffer. If the failure repeats, the initial meter calibration must be repeated with fresh buffers. Sample analysis must not proceed until the meter calibration is verified successfully.	Analyst	Reading between 6.95 to 7.05 pH units
Continued Calibration Verification (CCV)	pH buffer 7 is analyzed after every 10 samples and at the end of the sequence	Reading between 6.95 to 7.05 pH units	If a CCV falls outside the required range, repeat once with a fresh portion of buffer. If the failure repeats, the initial meter calibration must be repeated with fresh buffers. All samples analyzed after the last passing CCV must be reanalyzed.	Analyst	Reading between 6.95 to 7.05 pH units
Sample Duplicate (SD)	1 per batch of up to 10 samples	Relative percent difference (RPD) within laboratory generated limits; ≤ 2%	Reanalyze; two fresh portions of sample and duplicate must be analyzed until the readings stabilize within the required limit.	Analyst	RPD within laboratory generated limits; ≤ 2%

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UFP-QAPP Worksheet #28m: QC Samples Table for USEPA SW-846 Chapter 7, Section 7.1 - Ignitability

Matrix	Aqueous (toxicity characteristic leaching procedure [TCLP] leachate)
Analytical Group	Ignitability
Anticipated Concentration Level	Low to Moderate
Analytical Method/SOP Reference[1]	United States Environmental Protection Agency (USEPA) SW-846 Chapter 7, Section 7.1/PT-WC-022
Field Sampling Organization	Geosyntec Consultants, GSI, and Anchor QEA
Analytical Organization	Eurofins-TestAmerica Pittsburgh, Pennsylvania
No. of Sample Locations	See Worksheet #18

Quality Control (QC) Sample	Frequency/ Number	Method/Standard Operating Procedure (SOP) QC Acceptance Limits[2]	Corrective Action	Person(s) Responsible for Corrective Action	Measurement Performance Criteria[2]
Sample/Sample Duplicate (DUP)	1 per batch of up to 20 samples	The sample and sample duplicate pair is evaluated qualitatively. If both the sample and its duplicate are found to be either ignitable (YES/YES) or both not ignitable (NO/NO), the QC is considered in control.	If the sample and sample duplicate are out of control (YES/NO), the data must be qualified appropriately (e.g., sample heterogeneity) and an explanation provided in the report narrative.	Analyst	The sample and sample duplicate pair is evaluated qualitatively. If both the sample and its duplicate are found to be either ignitable (YES/YES) or both not ignitable (NO/NO), the QC is considered in control.

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2. This is a summary of the acceptance limits and measurement performance criteria; refer to the method SOP included in Attachment B of this UFP-QAPP for specific or more information. The acceptance limits and measurement performance criteria are in accordance with the government-required limits or criteria (i.e., National Functional Guidelines and/or USEPA Region 4 Laboratory Operations and Quality Assurance Manual [USEPA 2022]) if applicable.

UFP-QAPP Worksheet #28n: QC Samples Table for SW-846 9010C – Reactivity (Total Cyanide)

Matrix	Aqueous (groundwater, aqueous water quality control [QC] samples, equipment/source blanks)
Analytical Group	Reactivity (Cyanide)
Anticipated Concentration Level	Low to Moderate
Analytical Method/SOP Reference[1]	United States Environmental Protection Agency (USEPA) SW-846 9014 / PT-WC-018
Field Sampling Organization	Geosyntec Consultants, GSI, and Anchor QEA
Analytical Organization	Eurofins-TestAmerica, Pittsburgh, Pennsylvania
No. of Sample Locations	See Worksheet #18

QC Sample	Frequency/ Number	Method/Standard Operating Procedure (SOP) QC Acceptance Limits[2]	Corrective Action	Person(s) Responsible for Corrective Action	Measurement Performance Criteria[2]
Method Blank	After initial calibration verification (ICV) and continued calibration verification (CCVs)	Result < reporting limit (RL)	Troubleshoot; reanalyze if sufficient sample available; otherwise, flag data as appropriate	Analyst	Result < RL
Laboratory Control Sample/ Laboratory Control Sample Duplicate (LCS/LCSD)	LCS: 1 per batch of up to 20 samples LCSD: if insufficient sample provided for an MS/MSD	Within laboratory generated limits; % recovery (85–115); 20% relative standard deviation (RSD) for LCSD	If LCS recovery is above control limits and all associated samples are nondetect, results may be reported with the nonconformance memo (NCM). Samples associated with low LCS recovery, or samples with detected cyanide associated with high LCS recovery must be redistilled and reanalyzed.	Analyst	Within laboratory generated limits; % recovery (85–115); 20% RSD for LCSD
Low and High Distilled Standard Check	1 set per prep batch	Within laboratory generated limits; 90%–110%	Corrective actions are the same as for the LCS.	Analyst	Within laboratory generated limits; 90%–110%

Matrix	Aqueous (groundwater, aqueous water quality control [QC] samples, equipment/source blanks)
Analytical Group	Reactivity (Cyanide)
Anticipated Concentration Level	Low to Moderate
Analytical Method/SOP Reference[1]	United States Environmental Protection Agency (USEPA) SW-846 9014 / PT-WC-018
Field Sampling Organization	Geosyntec Consultants, GSI, and Anchor QEA
Analytical Organization	Eurofins-TestAmerica, Pittsburgh, Pennsylvania
No. of Sample Locations	See Worksheet #18

QC Sample	Frequency/ Number	Method/Standard Operating Procedure (SOP) QC Acceptance Limits[2]	Corrective Action	Person(s) Responsible for Corrective Action	Measurement Performance Criteria[2]
Matrix Spike/ Matrix Spike Duplicate (MS/MSD)	1 per batch of up to 20 samples	Within laboratory generated limits; % recovery (75–125); ≤ 20% RSD for MSD	Troubleshoot; reanalyze if sufficient sample available; otherwise, flag data as appropriate	Analyst	Within laboratory generated limits; % recovery (75–125); ≤ 20% RSD for MSD
Initial Calibration (ICAL)	Daily	Correlation coefficient must be ≥ 0.995, y-intercept ≤ +/- ½ RL	Troubleshoot; recalibrate	Analyst	Correlation coefficient must be ≥ 0.995, y-intercept ≤ +/- ½ RL
Initial Calibration Verification (ICV)	Following calibration	% recovery (90–110)	If the ICV does not verify the calibration curve, check the instrument conditions and if necessary, perform instrument maintenance, then analyze a new calibration curve followed by a fresh ICV.	Analyst	% recovery (90–110)
Continuing Calibration Verification (CCV)	Every 10 samples	% recovery (90–110)	Terminate the analysis, correct problem, recalibrate, and reanalyze previous samples. If results are associated with a failed CCV, they must be reported with qualifier in all cases.	Analyst	% recovery (90–110)

Matrix	Aqueous (groundwater, aqueous water quality control [QC] samples, equipment/source blanks)
Analytical Group	Reactivity (Cyanide)
Anticipated Concentration Level	Low to Moderate
Analytical Method/SOP Reference[1]	United States Environmental Protection Agency (USEPA) SW-846 9014 / PT-WC-018
Field Sampling Organization	Geosyntec Consultants, GSI, and Anchor QEA
Analytical Organization	Eurofins-TestAmerica, Pittsburgh, Pennsylvania
No. of Sample Locations	See Worksheet #18

QC Sample	Frequency/ Number	Method/Standard Operating Procedure (SOP) QC Acceptance Limits[2]	Corrective Action	Person(s) Responsible for Corrective Action	Measurement Performance Criteria[2]
Initial Calibration Blank (ICB) and Continuing Calibration Blank (CCB)	After ICV and CCVs	< RL	Source of contamination should be investigated and corrected and all associated samples reanalyzed.	Analyst	< RL
Low Level of Quantitation (LLOQ) Verification	Quarterly to validate quantitation capability at low analyte concentration levels	±35% of true value with ≤20% RSD between MDL verifications (MDLVs)	If recovery or RPD is outside the acceptance range, an MDL study must be performed and the RL (LLOQ) must be reestablished.	Analyst	±35% of true value with ≤20% RSD between MDLVs

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2. This is a summary of the acceptance limits and measurement performance criteria; refer to the method SOP included in Attachment B of this UFP-QAPP for specific or more information. The acceptance limits and measurement performance criteria are in accordance with the government-required limits or criteria (i.e., National Functional Guidelines and/or USEPA Region 4 Laboratory Operations and Quality Assurance Manual [USEPA 2022]) if applicable.

UFP-QAPP Worksheet #28o: QC Samples Table for SW-846 9030B/9034 – Reactivity (Sulfide)

Matrix	Aqueous (toxicity characteristic leaching procedure [TCLP] leachate)
Analytical Group	Reactivity (Sulfide)
Anticipated Concentration Level	Low to Moderate
Analytical Method / SOP Reference[1]	United States Environmental Protection Agency (USEPA) SW-846 9030B/9034 /PT-WC-010
Field Sampling Organization	Geosyntec Consultants, GSI, and Anchor QEA
Analytical Organization	Eurofins-TestAmerica Pittsburgh, Pennsylvania
No. of Sample Locations	See Worksheet #18

Quality Control (QC) Sample	Frequency/ Number	Method/Standard Operating Procedure (SOP) QC Acceptance Limits[2]	Corrective Action	Person(s) Responsible for Corrective Action	Measurement Performance Criteria[2]
Method Blank	1 per batch of up to 20 samples	Result < reporting limit (RL)	Reanalyze batch	Analyst	Result < RL
Initial Calibration Verification/ Continued Calibration Verification (ICV/CCV)	Prior to sample analysis, after every 10 titrations and at the end of the run	85%–115%	If the ICV does not meet recovery criteria, check calculations, re-standardize the iodine solution and titrant and repeat. If a CCV does not meet criteria, reanalyze once. If the failure repeats, re-standardize the iodine solution and titrant and repeat the analysis of all samples run after the last passing CCV.	Analyst	85%–115%
Initial Calibration Blank/Continued Calibration Blank (ICB/CCB)	Prior to sample analysis, after every 10 titrations and at the end of the run	Result < RL	Reprepare and reanalyze samples unless all associated samples are nondetect, sample quantity is insufficient to reanalyze, or outside of holding time	Analyst	Result < RL
Laboratory Control Sample (LCS)	1 per batch of up to 20 samples	Within laboratory generated limits; % recovery (85–115)	Troubleshoot; redistill and reanalyze if sufficient sample available; otherwise, flag data as appropriate	Analyst	Within laboratory generated limits; % recovery (85–115)

Matrix	Aqueous (toxicity characteristic leaching procedure [TCLP] leachate)
Analytical Group	Reactivity (Sulfide)
Anticipated Concentration Level	Low to Moderate
Analytical Method / SOP Reference[1]	United States Environmental Protection Agency (USEPA) SW-846 9030B/9034 /PT-WC-010
Field Sampling Organization	Geosyntec Consultants, GSI, and Anchor QEA
Analytical Organization	Eurofins-TestAmerica Pittsburgh, Pennsylvania
No. of Sample Locations	See Worksheet #18

Quality Control (QC) Sample	Frequency/ Number	Method/Standard Operating Procedure (SOP) QC Acceptance Limits[2]	Corrective Action	Person(s) Responsible for Corrective Action	Measurement Performance Criteria[2]
Matrix Spike/ Matrix Spike Duplicate (MS/MSD)	1per batch of up to 20 samples	Within laboratory generated limits; % recovery (75–125); 20% relative percent difference (RPD)	Troubleshoot; reanalyze if sufficient sample available; otherwise, flag data as appropriate	Analyst	Within laboratory generated limits; % recovery (75–125); 20% RPD

1. SOPs are reviewed/revised on an annual schedule by the laboratory. The current version of the SOP will be followed at the time of sample receipt.
2. This is a summary of the acceptance limits and measurement performance criteria; refer to the method SOP included in Attachment B of this UFP-QAPP for specific or more information. The acceptance limits and measurement performance criteria are in accordance with the government-required limits or criteria (i.e., National Functional Guidelines and/or USEPA Region 4 Laboratory Operations and Quality Assurance Manual [USEPA 2022]) if applicable.

UFP-QAPP Worksheet #28p: QC Samples Table for USEPA Method 9095B - Paint Filter Test

Matrix	Solids (Sediment)
Analytical Group	Paint Filter Test
Anticipated Concentration Level	Not applicable
Analytical Method/SOP Reference[1]	United States Environmental Protection Agency (USEPA) Method 9095B/PT-WC-034
Field Sampling Organization	Geosyntec Consultants, GSI, and Anchor QEA
Analytical Organization	Eurofins-TestAmerica Pittsburgh, Pennsylvania
No. of Sample Locations	See Worksheet #18

Quality Control (QC) Sample	Frequency/ Number	Method/Standard Operating Procedure (SOP) QC Acceptance Limits[2]	Corrective Action	Person(s) Responsible for Corrective Action	Measurement Performance Criteria[2]
Sample Duplicate (SD)	1 per batch of up to 20 samples	The sample and sample duplicate pair is evaluated qualitatively. Because this procedure does not report numeric results, no relative percent difference (RPD) can be applied to the duplicate. The sample and duplicate must agree on the presence or absence of free liquid.	If liquid is collected from one and not the other, 2 more portions of the sample must be analyzed.	Analyst	The sample and sample duplicate pair is evaluated qualitatively. Because this procedure does not report numeric results, no %RPD can be applied to the duplicate. The sample and duplicate must agree on the presence or absence of free liquid.

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2. This is a summary of the acceptance limits and measurement performance criteria; refer to the method SOP included in Attachment B of this UFP-QAPP for specific or more information. The acceptance limits and measurement performance criteria are in accordance with the government-required limits or criteria (i.e., National Functional Guidelines and/or USEPA Region 4 Laboratory Operations and Quality Assurance Manual [USEPA 2022]) if applicable.

UFP-QAPP Worksheet #28q: QC Samples Table for SW-846 1664B – Oil and Grease

Matrix	Aqueous (Sediment leachate)
Analytical Group	Oil and Grease
Anticipated Concentration Level	Low to Moderate
Analytical Method/SOP Reference[1]	United States Environmental Protection Agency (USEPA) SW-846 1664B/PT-WC-028
Field Sampling Organization	Geosyntec Consultants, GSI, and Anchor QEA
Analytical Organization	Eurofins-TestAmerica Pittsburgh, Pennsylvania
No. of Sample Locations	See Worksheet #18

Quality Control (QC) Sample	Frequency/ Number	Method/Standard Operating Procedure (SOP) QC Acceptance Limits[2]	Corrective Action	Person(s) Responsible for Corrective Action	Measurement Performance Criteria[2]
Method Blank	1 per batch of up to 20 samples	Result < reporting limit (RL)	Reanalyze batch	Analyst	Result < RL
Laboratory Control Sample (LCS)	1 per batch of up to 20 samples	Within laboratory generated limits; % recover (78–114)	Troubleshoot; redistill and reanalyze if sufficient sample available; otherwise, flag data as appropriate	Analyst	Within laboratory generated limits; % recovery (78–114)
Matrix Spike/ Matrix Spike Duplicate (MS/MSD)	1per batch of up to 20 samples	Within laboratory generated limits; %recovery (78–114); and 18% relative percent difference (RPD) for MSD	Troubleshoot; reanalyze if sufficient sample available; otherwise, flag data as appropriate	Analyst	Within laboratory generated limits; % recovery (78–114); 18% RPD
Balance Check	1 per batch of up to 20 samples	Criteria for 2 milligrams (mg) weight check is +/- 10% Criteria for 1 gram (g) weight check is +/- 0.5%	If the balance check is outside of acceptance criteria, do not begin weighing pans. Clean the balance pan, recalibrate the balance and check the required weights again.	Analyst	Criteria for 2 mg weight check is +/- 10% Criteria for 1 g weight check is +/- 0.5%

1. SOPs are reviewed/revised on an annual schedule by the laboratory. The current version of the SOP will be followed at the time of sample receipt.
2. This is a summary of the acceptance limits and measurement performance criteria; refer to the method SOP included in Attachment B of this UFP-QAPP for specific or more information. The acceptance limits and measurement performance criteria are in accordance with the government-required limits or criteria (i.e., National Functional Guidelines and/or USEPA Region 4 *Laboratory Operations and Quality Assurance Manual* [USEPA 2022]) if applicable.

UFP-QAPP Worksheet #28r: QC Samples Table for SW-846 7196A – Hexavalent Chromium

Matrix	Aqueous (Surface Water)
Analytical Group	Hexavalent Chromium
Anticipated Concentration Level	Low to Moderate
Analytical Method/SOP Reference[1]	United States Environmental Protection Agency (USEPA) SW-846 7196A/SA-GE-001
Field Sampling Organization	Geosyntec Consultants, GSI, and Anchor QEA
Analytical Organization	Eurofins-TestAmerica Savannah, Georgia
No. of Sample Locations	See Worksheet #18

Quality Control (QC) Sample	Frequency/ Number	Method/Standard Operating Procedure (SOP) QC Acceptance Limits[2]	Corrective Action	Person(s) Responsible for Corrective Action	Measurement Performance Criteria[2]
Method Blank	1 per batch of up to 20 samples	Result < 1/2 reporting limit (RL)	Evaluate according to SOP SA-QA-017	Analyst	Result < ½ RL
Laboratory Control Sample (LCS)	1 per batch of up to 20 samples	Within laboratory generated limits	Evaluate according to SOP SA-QA-017	Analyst	Within laboratory generated limits
LCS Duplicate	1 per batch of up to 20 samples (if insufficient sample provided for Matrix Spike/ Matrix Spike Duplicate [MS/MSD] or Sample Duplicate [SD])	Within laboratory generated limits	Evaluate according to SOP SA-QA-017	Analyst	Within laboratory generated limits
MS/MSD	1 per batch of up to 20 samples	Within laboratory generated limits	Evaluate according to SOP SA-QA-017	Analyst	Within laboratory generated limits
Initial Calibration (ICAL)	Daily	Correlation ≥ 0.995 and each point on curve must be within ± 10% of true value	Recalibrate	Analyst	Correlation ≥ 0.995 and each point on curve must be within ± 10% of true value

Matrix	Aqueous (Surface Water)
Analytical Group	Hexavalent Chromium
Anticipated Concentration Level	Low to Moderate
Analytical Method/SOP Reference[1]	United States Environmental Protection Agency (USEPA) SW-846 7196A/SA-GE-001
Field Sampling Organization	Geosyntec Consultants, GSI, and Anchor QEA
Analytical Organization	Eurofins-TestAmerica Savannah, Georgia
No. of Sample Locations	See Worksheet #18

Quality Control (QC) Sample	Frequency/ Number	Method/Standard Operating Procedure (SOP) QC Acceptance Limits[2]	Corrective Action	Person(s) Responsible for Corrective Action	Measurement Performance Criteria[2]
Second Source Initial Calibration Verification (ICV)	Immediately following the initial calibration; at a minimum, quarterly	within ± 10% of true value	Recalibrate	Analyst	within ± 10% of true value
Continuing Calibration Verification (CCV)	At the beginning and end of the analysis, and after every 10 samples	within ± 10% of true value	Terminate the analysis, fix the problem and reanalyze the previous samples.	Analyst	within ± 10% of true value
Initial Calibration Blank (ICB)	After ICV	Result < ½ RL	Terminate the analysis, correct problem and reanalyze the previous 10 samples.	Analyst	Result < ½ RL
Continuing Calibration Blank (CCB)	After every CCV	Result < ½ RL	Terminate the analysis, correct problem and reanalyze the previous 10 samples.	Analyst	Result < ½ RL
Batch Frequency	≤ 20 field samples or 24 hours	Not applicable	Not applicable	Not applicable	Not applicable
Linear Calibration Range (LCR) (also called Linear Dynamic Range, LDR)	Upon method/instrument set-up, and when LCR verification fails acceptance criteria.	within ± 10% of true value	This determines the range at which the instrument is considered linear. The highest concentration of the calibration curve cannot exceed the highest concentration of the LCR study.	Analyst	within ± 10% of true value

Matrix	Aqueous (Surface Water)
Analytical Group	Hexavalent Chromium
Anticipated Concentration Level	Low to Moderate
Analytical Method/SOP Reference[1]	United States Environmental Protection Agency (USEPA) SW-846 7196A/SA-GE-001
Field Sampling Organization	Geosyntec Consultants, GSI, and Anchor QEA
Analytical Organization	Eurofins-TestAmerica Savannah, Georgia
No. of Sample Locations	See Worksheet #18

Quality Control (QC) Sample	Frequency/ Number	Method/Standard Operating Procedure (SOP) QC Acceptance Limits[2]	Corrective Action	Person(s) Responsible for Corrective Action	Measurement Performance Criteria[2]
LCR Verification (LCRV) (also called LDR Verification, LDRV)	Every 6 months	within ± 10% of true value	Re-perform the LCR study	Analyst	within ± 10% of true value
Initial Demonstration of Capability (IDOC)	Initially, per analyst, per analyte/method combination	Refer to SOP SA-QA-006	Refer to SOP SA-QA-006 (Unsupervised work cannot begin until successful IDOC is obtained.)	Analyst	Refer to SOP SA-QA-006
Continuing Demonstration of Capability (CDOC)	Annually, per analyst, per analyte/method combination	Refer to SOP SA-QA-006	Refer to SOP SA-QA-006	Analyst	Refer to SOP SA-QA-006
Reporting Limit Verification (RLV)	Upon method/instrument set-up, per analyte/method/matrix combination.	Refer to SOP SA-QA-007	Refer to SOP SA-QA-007	Analyst	Refer to SOP SA-QA-007
Method Detection Limit Study (MDL)	Upon method/instrument set-up, per analyte/method/matrix combination and annually thereafter	Refer to SOP SA-QA-007	Refer to SOP SA-QA-007	Analyst	Refer to SOP SA-QA-007

Matrix	Aqueous (Surface Water)
Analytical Group	Hexavalent Chromium
Anticipated Concentration Level	Low to Moderate
Analytical Method/SOP Reference[1]	United States Environmental Protection Agency (USEPA) SW-846 7196A/SA-GE-001
Field Sampling Organization	Geosyntec Consultants, GSI, and Anchor QEA
Analytical Organization	Eurofins-TestAmerica Savannah, Georgia
No. of Sample Locations	See Worksheet #18

Quality Control (QC) Sample	Frequency/ Number	Method/Standard Operating Procedure (SOP) QC Acceptance Limits[2]	Corrective Action	Person(s) Responsible for Corrective Action	Measurement Performance Criteria[2]
MDL Verification (MDLV)	Upon method/instrument set-up, per analyte/method/matrix combination.	Refer to SOP SA-QA-007	Refer to SOP SA-QA-007	Analyst	Refer to SOP SA-QA-007

1. SOPs are reviewed/revised on an annual schedule by the laboratory. The current version of the SOP will be followed at the time of sample receipt.
2. This is a summary of the acceptance limits and measurement performance criteria; refer to the method SOP included in Attachment B of this UFP-QAPP for specific or more information. The acceptance limits and measurement performance criteria are in accordance with the government-required limits or criteria (i.e., National Functional Guidelines and/or USEPA Region 4 *Laboratory Operations and Quality Assurance Manual* [USEPA 2022]) if applicable.

UFP-QAPP Worksheet #29: Project Documents and Records Table

Sample Collection Documents and Records	On-Site Analysis Documents and Records	Off-Site Analysis Documents and Records	Data Assessment Documents and Records	Other
• Field notes and field forms • Boring logs • Well diagrams • Identification of quality control (QC) samples • Chain-of-custody forms • Air bills • Custody seals	• Equipment calibration logs • Field parameter data • Water level and water depth measurements • Soil lithology information • Waste labels and manifests	• Sample receipt, custody and tracking records • Equipment calibration logs • Sample preparation logs • Sample run logs • Equipment maintenance, testing, and inspection logs • Instrument printouts • Standards results • Corrective action forms • Sample disposal records • Raw data (electronic) • Analytical results: portable document format (PDF) files, and electronic data deliverables • Level II and Level IV standard data deliverables	• Field sampling assessment checklists • Laboratory assessment checklists • Data validation/ usability assessments • Corrective Action Plan • Final Report	• Corrective action communication • Approval authority responses/concurrence

UFP-QAPP Worksheets #31, #32 & #33: Assessments and Corrective Action

Assessments:

Assessment Type	Responsible Party & Organization	Frequency	Estimated Dates	Assessment Deliverable	Deliverable due date
Field Safety and Sampling Technical Systems Audit	Field Team Lead and Project Manager (Geosyntec)	At the beginning of each sampling event	TBD	Checklist including examinations of field sampling and measurement records, field instrument operating and calibration records, sample collection, handling and packaging in compliance with the established procedures, maintenance of quality control (QC) procedures, chain of custody, field and sample documentation, safety procedures	2 business days after assessment
Laboratory Technical Systems Assessment	Laboratory and Operations Managers (Eurofins-TestAmerica)	Annually	TBD	Per requirements of QA Manager	Not applicable
Data Quality Assessment	Field Team Lead and Quality Assurance (QA) Manager (Geosyntec) Laboratory Manager (Eurofins-TestAmerica)	Per analytical data package	TBD	Communication in the form of an email requesting additional laboratory forms, backup data that may be missing and/ or clarification of the analytical report.	2 weeks after receipt of data

TBD: to be determined

Assessment Response and Corrective Action:

Assessment Type	Responsibility for Responding to Assessment Findings	Assessment Response Documentation	Timeframe of Response	Responsibility for Implementing Corrective Action	Responsible for Monitoring Corrective Action Implementation
Field Safety and Sampling Technical Systems Audit	Project Manager/ Field Team Lead (Geosyntec)	Letter report or email	2 business days after assessment	Field Team Lead (Geosyntec)	Project Manager (Geosyntec)
Laboratory Technical Systems Assessment	Laboratory Manager/ Operational Manager (Eurofins TestAmerica)	Letter report or email	2 business days after assessment	Laboratory Manager/ Operational Manager (Eurofins TestAmerica)	Laboratory Manager/ Operational Manager (Eurofins TestAmerica)
Data Quality Assessment	Laboratory Manager/ Operational Manager (Eurofins TestAmerica)	Per requirements of Quality Assurance (QA) Manager	Per requirements of QA Manager	Laboratory Manager/ Operational Manager (Eurofins TestAmerica)	Laboratory Manager/ Operational Manager (Eurofins TestAmerica)

QA Management Reports Table:

Type of Report	Frequency (daily, weekly monthly, quarterly, annually, etc.)	Projected Delivery Date(s)	Person(s) Responsible for Report Preparation (Title and Organizational Affiliation)	Report Recipient(s) (Title and Organizational Affiliation)
Field Progress Reports	Daily during sampling events; email or oral communication	Daily during sampling events	Field Team Lead (Geosyntec)	Project Manager (Geosyntec)
Monthly Project Progress Report	Monthly for duration of the project	Monthly	Project Manager/Field Team Lead (Geosyntec)	Project Manager (Geosyntec)/On-Scene Coordinator (EPA)
Data Validation and Usability Assessment	Per analytical data package	Within 3 weeks of receiving analytical package	Data Validation Team/Geosyntec	Project Manager/Field Team Lead (Geosyntec)

UFP-QAPP Worksheet #34: Validation Process Table (Data Review Step I)

Verification Input	Description	Internal/ External	Responsible for Verification (Name, Organization)
Chain of Custody	Chain-of-custody forms and shipping documentation will be reviewed internally upon completion and compared with the packed sample shipment.	Internal	Validation Team/ Geosyntec
Field Notes	Field notes documenting all field activities will be maintained, scanned, and placed in the appropriate project directories.	Internal	Field Team Lead/ Geosyntec
Field Logs	Boring logs, well-installation logs, well-development records, field-sampling data sheets, and other field forms will be scanned and stored in project directories.	Internal	Field Team Lead/ Geosyntec
Laboratory Data	All data packages including electronic data deliverables (EDDs) will be verified by the lab for completeness and technical accuracy prior to submittal.	External	Project Manager/ Eurofins-TestAmerica and EGT
	All data packages including EDDs will be verified internally in accordance with the stated data validation procedures (100% Stage 2A).	Internal	Validation Team/ Geosyntec
Deliverables Review	All deliverables will be reviewed internally, and comments will be verified. Following internal verification, each deliverable will be distributed for external review by the United States Environmental Protection Agency (USEPA).	Internal	Validation Team/ Geosyntec

UFP-QAPP Worksheet #35: Validation Process Table
(Data Review Steps IIa and IIb)

The data validation strategy will be consistent with the applicable United States Environmental Protection Agency (USEPA) guidelines. Data validation will consist of 100% Stage 2A data validation, per analytical test and matrix.

Step IIa/IIb	Validation Input	Description	Responsible for Validation (Name, Organization)
IIa	Methods/SOPs	Ensure that all sampling and analytical SOPs are followed.	Field Team Lead, Validation Team/ Geosyntec
IIb	Sampling Schedule	Confirm that all sampling locations listed in Worksheet #18 are sampled and analyzed with the appropriate analytical methods.	Field Team Lead/ Geosyntec
IIa	Field Sheets	Review and edit field-sampling sheets for consistency and proper information.	Field Team Lead/ Geosyntec
IIa	Equipment Calibration	Perform proper calibration of groundwater gauging and sampling equipment, and document results.	Field Staff/Geosyntec
IIa/IIb	Performance Requirements	Check that the performance requirements specified by this UFP-QAPP are met.	Validation Team/ Geosyntec
IIa/IIb	All Required Elements of the Data Package	Check that all of the required reporting elements are present in the laboratory data package applicable to the requirements for Stage 2A validation.	Laboratory Personnel/ Eurofins TestAmerica and Validation Team, QA Manager/Geosyntec
IIb	Analytical QC Results	Verify that all method QC samples were run and met the referenced measurement performance criteria listed in Worksheets #12a -12i	Validation Team/ Geosyntec
IIb	Project Quantitation Limits	Compare sample results to project quantitation limits (PQLs) listed in Worksheets #15.	Validation Team/ Geosyntec
IIb	Deliverables	Keep deliverable review documentation during internal and external review process. Distribute deliverables on time as listed in Worksheets #14 and #16.	Validation Team/ Geosyntec

QC: quality control
SOP: standard operating procedure

Data Validation

The evaluation of the data will extend beyond method, procedural, or contractual compliance (verification) to check the analytical quality of the specific data set. The data will be evaluated in regard to compliance to the data quality objectives (DQOs) and measurement quality objectives. During data validation, data qualifiers will be assigned to provide the basis of describing data quality. Should nonconformance issues be generated from the laboratory, the validation procedure evaluates the effects of the nonconformance(s) on the quality and usability of the data set.

Step IIa denotes a list of validation activities, which include the following and are associated with methods, procedures, and contracts:

- Data Deliverables: Check that all required information on sampling and analysis are provided.
- Analytes: Check that all analytes were reported as required.
- Chain of Custody: Evaluate traceability of data and examine against procedural requirements.
- Holding Times: Check holding times for analysis.
- Sample Handling: Check that sample preservation, handling, and storage procedures were met.
- Sampling Methods and Procedures: Check that field measurement and performance criteria were met and, if field measurements and performance criteria did not meet specifications, be sure that is documented. Check that required sampling methods were used.
- Field Transcription: Check transcription accuracy of sampling data where applicable.
- Analytical Methods and Procedures: Evaluate whether the required methods and procedures were performed.
- Data Qualifiers: Check that the laboratory qualifiers were used correctly.
- Laboratory Transcription: Check accuracy of transcription where applicable.
- Proficiency Testing: Evaluate acceptance of proficiency testing sample results against performance requirements as specified by the project.
- Standards: Check that standards are traceable and meet project and contract requirements.
    - Communication: Check that required communication procedures were followed by field and laboratory personnel.

Step IIb denotes a list of validation activities, which include the following and are associated with comparison with measurement performance Criteria in the Predesign Investigation Work Plan (PDIWP):

- Data Deliverables and PDIWP: Check that data report from Step IIa was provided.
- Sampling Plan: Check whether the sampling plan was executed as specified.
- Sampling Procedures: Evaluate whether sampling procedures were followed with respect to equipment and proper sampling support.
- Collocated Field Duplicates: Compare results of collocated field duplicates with criteria established in the PDIWP.
- Project Quantitation Limits: Check that quantitation limits were achieved as outlined in the PDIWP and that the laboratory successfully analyzed a standard at the quantitation limit.
- Confirmatory Analysis: Evaluate the agreement of the laboratory results.
- Performance Criteria: Evaluate QC data against project-specific performance criteria in the PDIWP (i.e., evaluate quality parameters beyond those outlined in the methods).
- Data Qualifiers: Check that the lab qualifiers and the third-party data validator qualifiers applied in Step IIa were those specified in this UFP-QAPP and that any deviations were specified.
- Step IIb Validation Report: Summarize outcome of comparison of data to methods, procedures, and contracts, in this UFP-QAPP and include qualified data and explanation of all data qualifiers.

UFP-QAPP Worksheet #36: Validation Process Table (Data Review Steps IIa and IIb)

Step IIa/IIb	Matrix	Analytical Group	Validation Criteria	Data Validator (title and organizational affiliation)
IIa/IIb	Floodplain and Creek Bank Soil	All Analytical Parameters	Method and SOP criteria, EPA QA/G8, USEPA Region 4 Data Validation SOPs QAS-SOP-0025, QAS-SOP-12, QAS-SOP-13, QAS-SOP-14); and National Functional Guidelines for Data Review	Geosyntec Data Validation Team/ Geosyntec
IIa/IIb	Groundwater and Surface Water	All Analytical Parameters	Method and SOP criteria, EPA QA/G8, USEPA Region 4 Data Validation SOPs (QAS-SOP-0025, QAS-SOP-12, QAS-SOP-13, QAS-SOP-14); and National Functional Guidelines for Data Review	Geosyntec Data Validation Team/ Geosyntec
IIa/IIb	Snow Creek Sediment	All Analytical Parameters	Method and SOP criteria, EPA QA/G8, USEPA Region 4 Data Validation SOPs (QAS-SOP-0025, QAS-SOP-12, QAS-SOP-13, QAS-SOP-14); and National Functional Guidelines for Data Review	Geosyntec Data Validation Team/ Geosyntec

QAS: quality assurance section
SOP: standard operating procedure
USEPA: United States Environmental Protection Agency

Data Validation

The Geosyntec data validation team will validate all laboratory data in accordance with current United States Environmental Protection Agency (USEPA) National Functional Guidelines (USEPA 2020) and *Guidance on Environmental Data Verification and Data Validation* (USEPA 2002a) as well as USEPA Region 4 Data Validation SOPs (USEPA 2011, 2016a). The data validation strategy will be consistent with the applicable USEPA guidelines. Data validation will consist of 100% Stage 2A data validation, per analytical test and matrix. Field data will also be validated against the standard operating procedures (SOPs) and the acceptance criteria contained in the Predesign Investigation Work Plan (PDIWP).

The data reviewer is required request any missing information needed from the laboratory. Elimination of the data from the review process is not allowed. All data will be qualified, as necessary, in accordance with established criteria. Data summary packages will consist of sample results, quality control (QC) summaries, and all raw data associated with the sample results and QC summaries. During the data validation process, the qualifiers shown below will be applied to the data as applicable.

Data Qualifier Definitions

- U: The analyte was analyzed for but was not detected at or above the reported sample quantitation limit.

- J: The analyte was positively identified; the associated numerical value is the approximate concentration of the analyte in the sample.

- J+: The analyte was positively identified; however, the associated numerical value is likely to be higher than the concentration of the analyte in the sample due to positive bias of associated QC or calibration data or attributable to matrix interference.

- J-:The analyte was positively identified; however, the associated numerical value is likely to be lower than the concentration of the analyte in the sample due to negative bias of associated QC or calibration data or attributable to matrix interference.

- UJ: The analyte was not detected above the reported sample quantitation limit. However, the reported quantitation limit is approximate and may or may not represent the actual limit of quantitation necessary to accurately and precisely measure the analyte in the sample.

- R: The sample results are rejected due to serious deficiencies in the ability to analyze the sample and meet quality control criteria. The presence or absence of the analyte cannot be verified.

UFP-QAPP Worksheet #37: Usability Assessment (Data Review Step III)

To the extent possible, the validation team will follow the United States Environmental Protection Agency's (USEPA's) data quality assessment (DQA) process to verify that the type, quality, and quantity of data collected are appropriate for their intended use. DQA methods and procedures are outlined in USEPA's *Guidance, Data Quality Assessment: A Reviewer's Guide* (EPA QA/G-9R). The DQA process included five steps:

1. Review the data quality objectives (DQOs) and sampling design.
2. Conduct a preliminary data review.
3. Select a statistical test.
4. Verify the assumptions of the statistical test.
5. Draw conclusions from the data.

After the data are received from the field team or from the laboratory, validation of the data will occur as described in Worksheet #36. During validation, where necessary, validation qualifiers will be applied to the data indicating that it has limited use, should perhaps be examined more closely, or has dramatically failed one or more data quality indicator (DQI) criteria and has been rejected. This information will be supplied to the project team via a validation report and to the data management team through updates to the database. A DQA report will be prepared periodically summarizing the overall quality of the data, including field data, field quality control (QC) data, laboratory QC data, laboratory QC data trends, and laboratory data. This will further illustrate the limitations of any qualified data that may have resulted during data validation. It is incumbent on the project team to then use the data in an appropriate manner based on any limitations that have been identified.

Summarize the usability assessment process and all procedures (e.g., interim steps, and any statistics, equations, and computer algorithms that will be used)

Data usability is the process of evaluating the data validation results and determining the confidence with which any data point(s) may be used. Usability is determined by evaluating the data validation qualifier applied and the laboratory QC results. Concentration values may be considered to have a high degree of confidence because the associated method performance criteria were achieved. Estimated concentration results are evaluated with respect to the bias contributed to the value by the associated QC result. Bias direction can be estimated for data quality impacts due to surrogate recoveries, matrix spike recoveries, and laboratory control sample recoveries. Sample concentration results that are rejected during data validation are not used in the decision-making process and should not be reported.

Evaluative procedures used to assess overall measurement error associated with the project

Data usability is evaluated with respect to the DQOs presented in this UFP-QAPP to check that the opportunity for incorporating unacceptable and manageable error into the decision-making process is minimized to the extent possible. The acceptable error associated with the analytical data results and their use is contained in Worksheet #12.

The analytical data, data validation qualifiers, and QC results will be evaluated to determine the confidence with which the analytical data can be used in the project decision-making process. The criteria used in the data usability summary are presented as follows using the DQI criteria required for the predesign investigation (PDI) and measured as precision, accuracy, representativeness, completeness, comparability, and sensitivity (PARCCS).

PARCCS OVERVIEW

Introduction

This quality assurance (QA) program addresses both field and laboratory activities. QA objectives are formally measured by computing performance measures known as DQIs, which are compared to predefined measurement quality objectives (MQOs) specific to the project objectives. The DQIs for measurement data are expressed in terms of PARCCS. Evaluation of DQIs provides the mechanism for ongoing control and evaluation of data quality throughout the project and ultimately will be used to define the data quality achieved for the various measurement parameters. The field QA/QC program will be accomplished by collecting field duplicates, equipment blanks, source blanks, field blanks, and trip blanks. The analytical QA/QC program will be assessed through the internal laboratory QC performed, including method blanks, laboratory control sample (LCS) and laboratory control sample duplicates (LCSD) recoveries, surrogate recoveries, and matrix spike/matrix spike duplicate (MS/MSD) recoveries. The following sections describe the DQIs in greater detail, with a discussion of the associated MQOs.

Precision

Precision refers to the reproducibility or degree of agreement among duplicate measurements of a single analyte. The closer the numerical values of the measurements, the more precise the measurement. Poor precision stems from random errors (i.e., mechanisms that can cause both high and low measurement errors at random). Precision is usually stated in terms of standard deviation, but other estimates, such as the coefficient of variation (relative standard deviation), range (maximum value minus minimum values), and relative range, are common and may be used pending review of the data. Precision will be checked by collecting field duplicates and the analysis of MS/MSD and LCS/LCSD samples for the work performed at the site. The overall precision of measurement data is a mixture of sampling and analytical factors. Analytical precision is much easier to control and quantify than sampling precision; there are more historical data related to individual method performance, and the “universe” is not limited to the samples received in the laboratory. In contrast, sampling precision is unique to the project. Sampling precision will be measured through

the laboratory analysis of field duplicate samples. Laboratory precision will be measured through the analysis of MS/MSD and LCS/LCSD samples.

During the collection of data using field methods and/or instrumentation, precision is checked by reporting several measurements taken at one location and comparing the results. Precision will be determined from replicate samples and will be expressed as the relative percent difference (RPD) between replicate/duplicate sample results, computed as follows:

$$RPD = \frac{X_1 - X_2}{(X_1 + X_2)/2} \times 100$$

where X_1 and X_2 are reported concentrations for each replicate sample and subtracted differences represent absolute values. For field duplicates, the precision goals for this project are RPD = 35% for aqueous samples and RPD = 50% for solid samples. For laboratory duplicates, the RPD goals are dictated by the specific analytical and laboratory QC acceptance criteria.

Accuracy and Bias

Accuracy refers to the degree of difference between measured or calculated values and the true value. The closer the numerical value of the measurement comes to the true value or actual concentration, the more accurate the measurement. The converse of accuracy is bias, in which a systematic mechanism tends to consistently introduce errors in one direction or the other. Bias in environmental sampling can occur in one of three ways; these mechanisms and their associated diagnostic and management methods are as follows:

- High bias, which can stem from cross-contamination of sampling, packaging, or analytical equipment and materials. Cross-contamination is monitored through blank samples, such as equipment blanks, field blanks, trip blanks, filter blanks, and method blanks. These samples assess the potential for cross-contamination from, respectively, sampling equipment, ambient conditions, packaging and shipping procedures, field filters, and laboratory equipment. Data validation protocols described in Worksheet #36 present a structured approach for data qualification based on blank samples.
- Low bias, which can stem from the dispersion and degradation of target analytes (e.g., volatilization of chlorinated solvents during field sampling). The effects of these mechanisms are difficult to quantify. Sampling accuracy can be maximized by the adoption and adherence to a strict field QA program. Specifically, sampling will be conducted following standard protocols described in this UFP-QAPP (e.g., eliminating headspace in sampling vials for volatile organic compounds [VOCs] will reduce the potential for dispersion of VOCs during sampling). Through regular review of field procedures, deficiencies will be documented and corrected in a timely manner.
- High or low bias, due to poor recoveries, poor calibration, or other system control problems. The effects of these mechanisms on analytical accuracy may be expressed as the percent recovery of an analyte that has been added to the environmental sample at a

known concentration before analysis. Analytical accuracy in the laboratory will be determined through the analysis of LCSs and MS/MSDs. As with blank samples, data validation protocols provide a structured formula for data qualification based on erroneously high or low analyte recoveries.

Accuracy, when potentially affected by high or low recoveries as described in the third bullet above, is presented as percent recovery (%R) for MS/MSD recoveries, defined as:

$$\%\ R = \frac{Spiked\ Sample\ Concentration - Sample\ Concentration}{Spike\ Concentration} \times 100$$

Accuracy goals are presented as upper and lower control limits for percent recovery and are generated through the compilation of control charts and referenced in each laboratory method SOP attached to this UFP-QAPP. These limits in conjunction with validation-guidance-required percent recoveries will be used for assessing data accuracy.

Representativeness

Representativeness is defined by the degree to which the data accurately and precisely describe a characteristic of a population, parameter variations at a sampling point, a process condition, or an environmental condition. If the results are reproducible, the data obtained can be said to represent the environmental condition. Representativeness is evaluated by collecting a sufficient number of samples of an environmental medium, properly chosen with respect to place and time. The precision of a representative set of samples reflects the degree of variability of the sampled medium, as well as the effectiveness of the sampling techniques and laboratory analysis. Representativeness will be determined through consensus with Region 4 USEPA concerning appropriate sampling scope.

Completeness

Completeness is defined as the percentage of measurements made that are judged to be valid measurements. The completeness goal is essentially the same for all data uses in that a sufficient amount of valid data are to be generated. There are limited historical data on the completeness achieved by individual methods. However, the Contract Laboratory Program (CLP) data have been found to be 80% to 85% complete on a nationwide basis. The percent completeness for each set of samples will be calculated as follows:

$$\%\ Completeness = \frac{Valid\ Data}{Total\ Data\ Planned} \times 100$$

The QA objective for completeness for all parameters will be 90%.

Comparability

Comparability expresses the confidence with which one data set can be compared to another data set measuring the same property. Comparability is evaluated by using established and approved analytical methods, consistency in the basis of analysis (e.g., wet weight, volume), consistency in reporting units (micrograms per liter [µg/L], milligrams per liter [mg/L]), and analysis of standard reference materials. By using standard sampling and analytical procedures, data sets will be comparable.

Sensitivity

Sensitivity refers to the minimum magnitude at which analytical methods can resolve quantitative differences among sample concentrations. If the minimum magnitude for a particular analytical method is sufficiently below an action level or risk-screening criterion, then the method sensitivity is deemed sufficient to fully evaluate the data set with respect to the desired reference values. Frequently, risk-based screening levels fall below the sensitivity of even the most sensitive analytical methods. In such cases, it is necessary to review the qualifications of several laboratories, both from the standpoint of sensitivity as well as other DQIs, to select the best laboratory for the project. The method detection limit (MDL) is a theoretical limit determined through an MDL study, in which the concentration of a spiked solution is tested at least seven times. At least seven method blanks are also used to generate an MDL. The standard deviation of the recovered concentrations (σ_{rec}) is computed and multiplied by the t-distribution value to arrive at the MDL. The higher of the two MDLs is chosen as the MDL for the analyte and matrix. This practice helps alleviate the reporting of false positive results due to background contamination. The reporting limits (RLs) are the lowest quantitative value reported and are generally based on the lowest concentration standard on the calibration curve. Analytical sensitivity is readily evaluated by comparing method RLs to risk-based screening values. The results of this analysis are presented in Worksheet #15, which demonstrate the suitability of the selected methods to the project requirements.

Identify the personnel responsible for performing the usability assessment

Data usability is first evaluated by the laboratory performing the fixed-base analysis, the data validation team, and the QA Manager. Usability of data collected in the field is first determined by the field team, Field Team Lead, and Project Manager. Once the data are validated, the final usability of the data are determined by the project team, specifically the technical leaders for the project and the Project Manager.

Documentation that will be generated during usability assessment and how usability assessment results will be presented so that they identify trends, relationships (correlations), and anomalies

Data usability will be documented through validation reports as well as through the issuance of DQA reports, which will summarize how the data reflect the specific criteria for the DQIs assigned to the

project. A DQA report will be included in interim and final reports where applicable and will provide discussions of the DQI assessment.

ATTACHMENT A

Field Standard Operating Procedures

Attachment A-1
Field Sample Packing, Handling, Shipping, and Chain-of-Custody Procedures

1.0 HANDLING

1. Complete sample label with the following information:
   a. Sample type (sediment, soil, etc.)
   b. Site name and Project number
   c. Sample identification code and other sample identification information, if applicable
   d. Analysis required
   e. Date
   f. Time sampled
   g. Sample type (composite or grab)
   h. Preservative, if applicable (if used, ice should be listed as a preservative)
   i. Sampler's name or initials
2. Cover the label with clear packing tape to secure the label onto the container.
3. Complete the chain-of-custody (COC) forms (an example form is attached).
4. Store samples in a cooler on ice or in a refrigerator until shipped to the lab.

2.0 PACKING

1. Ensure that sample containers have the sample labels securely affixed to the container with clear tape.
2. Check caps on the sample containers to ensure that they are properly sealed.
3. Complete the COC form with sampling information and ensure that recorded information matches the sample labels.
4. Secure, using tape, the outside drain plug at the bottom of the cooler, line the inside of the cooler with a garbage bag or other plastic liner.
5. Wrap sample containers in individual plastic bags. For glass containers, use bubble wrap bags or wrap containers with a second layer of cushioning material.
6. Place 1 to 2 inches of cushioning material at the bottom of the cooler.
7. Place the sealed sample containers into the cooler.
8. Include a bottle of water clearly labeled as "temperature blank" in the cooler.

9. Place ice in plastic bags and seal. Place loosely in the cooler.
10. Fill the remaining space in the cooler with cushioning material.
11. If releasing to a laboratory custodian, both the releaser and receiver will sign and date the COC form indicating the transfer of custody.
12. Indicate on the COC form if shipping by overnight service, include the air bill number on the COC form, sign and date the COC form, place COC forms in a plastic bag and seal. Tape the forms to the inside of the cooler lid.
13. Close the lid of the cooler, and secure with packing tape.
14. Wrap strapping tape around both ends of the cooler at least twice.
15. Mark the cooler on the outside with the following information if shipping by express carrier: shipping address; return address; "Fragile" labels; and arrows indicating “this side up.” Cover the labels with clear plastic tape. Place a signed custody seal on at least two sides of the cooler/lid interface.

3.0 SHIPPING

Samples will be hand delivered or delivered by an express carrier (e.g., Federal Express) using same day or overnight service.

4.0 CHAIN OF CUSTODY (COC)

Field custody documentation consists of both field logbooks, field forms and COC forms. A sample is in a person’s custody if

- it is in that person’s possession;
- it is in that person’s view, after being in his or her possession;
- it was in that person’s possession and he or she locked it up to prevent tampering; or
- it is in a secure area, under the control of that person.

The following custody procedures will be followed in the field:

1. The sampling crew will be responsible for the care and custody of the samples collected until they are transferred to the laboratory or otherwise stored at a controlled location (e.g., in on-site locked storage).
2. When shipping to the laboratory, each sample shipped will be entered on the COC form. Samples sharing a single COC form will be packaged and shipped together. The COC form will contain the following information:
    a. Name and address of laboratory
    b. Method of shipment (courier or overnight service)
    c. Air bill number (if applicable)
    d. Project name

e. Project contact information

f. Signatures for release and acceptance of custody

g. Unique sample identification numbers (must be the same as the identification numbers of the sample labels)

h. Date and time of sample collection

i. Sample matrix

j. Preservation (if any, including ice)

k. Analyses required

3. Samples will be packaged and shipped or transported to the analytical laboratory for analysis.

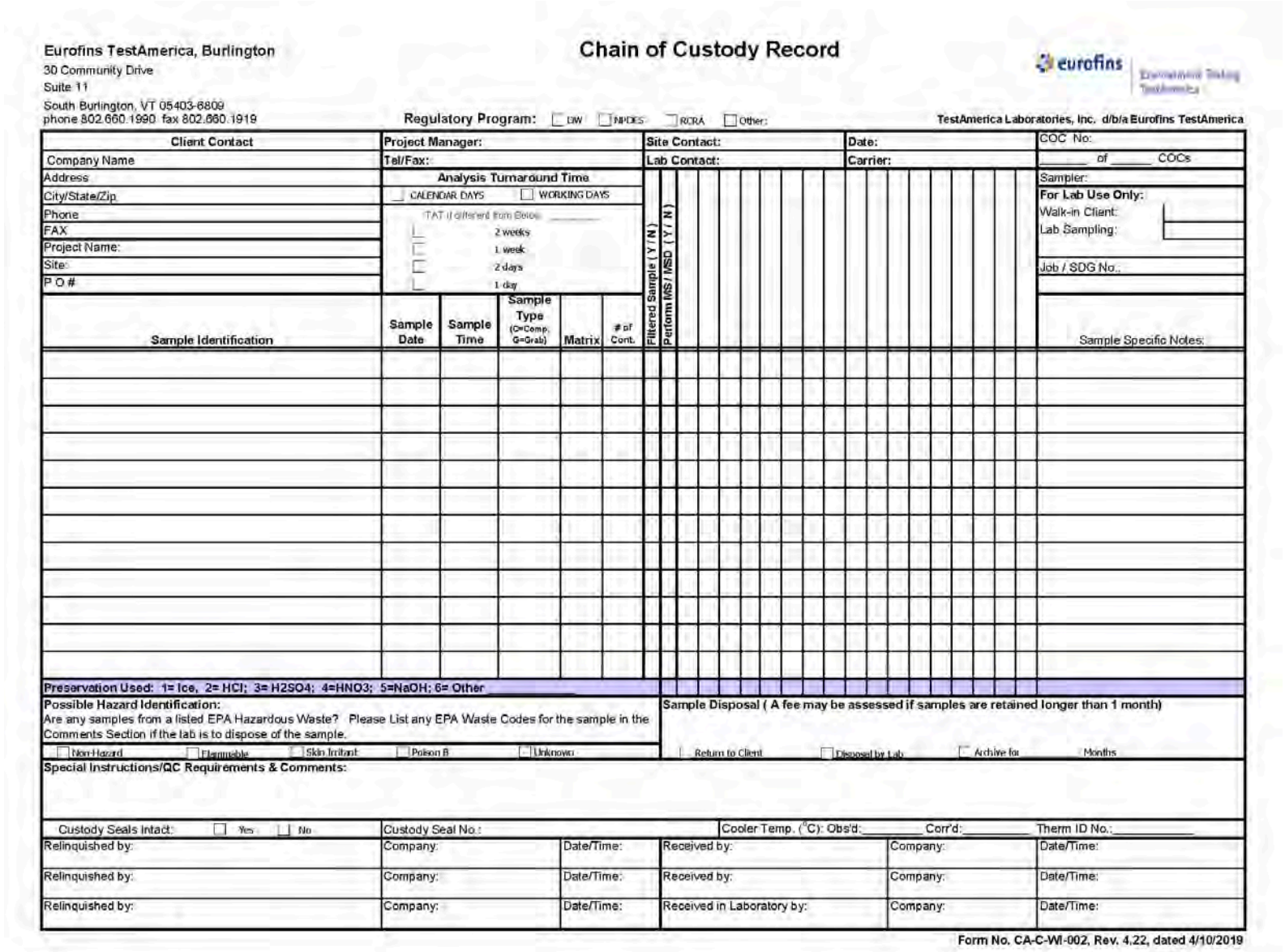
4. The original COC form will accompany the samples to the laboratory. Copies of the COC will be retained in the project files. The samples and signed COC forms will remain in the possession of the sampling crew until the samples are delivered to the express carrier, hand delivered to a mobile or permanent laboratory, or placed in secure storage.

If samples are shipped via express carrier, the carrier, air bill number and date shipped will be noted on the COC form, and the form will be placed inside the cooler and sealed with COC seals.

If samples are delivered directly to the laboratory or to a laboratory custodian or courier, the releaser and the receiver will sign and date the COC indicating the date and time of transfer.

If samples are split with a government agency or other party, a separate COC will be prepared for those samples and marked to indicate with whom the samples are being split.

Attachment 1: Example of Chain-of-Custody Form



Eurofins TestAmerica, Burlington
30 Community Drive
Suite 11
South Burlington, VT 05403-6809
phone 802.660.1990 fax 802.660.1919

Chain of Custody Record

eurofins | Environment Testing TestAmerica

Regulatory Program: ☐ DW ☐ NPDES ☐ RCRA ☐ Other:

TestAmerica Laboratories, Inc. d/b/a Eurofins TestAmerica

Client Contact	Project Manager:	Site Contact:	Date:	COC No:
Company Name	Tel/Fax:	Lab Contact:	Carrier:	____ of ____ COCs
Address	Analysis Turnaround Time			Sampler:
City/State/Zip	☐ CALENDAR DAYS ☐ WORKING DAYS			For Lab Use Only:
Phone	TAT if different from Below			Walk-in Client:
FAX	☐ 2 weeks			Lab Sampling:
Project Name:	☐ 1 week			
Site:	☐ 2 days			Job / SDG No.:
P O #	☐ 1 day			

Sample Identification	Sample Date	Sample Time	Sample Type (C=Comp, G=Grab)	Matrix	# of Cont.	Filtered Sample (Y / N)	Perform MS / MSD (Y / N)	Sample Specific Notes:

Preservation Used: 1= Ice, 2= HCl; 3= H2SO4; 4=HNO3; 5=NaOH; 6= Other _____

Possible Hazard Identification:
Are any samples from a listed EPA Hazardous Waste? Please List any EPA Waste Codes for the sample in the Comments Section if the lab is to dispose of the sample.

☐ Non-Hazard ☐ Flammable ☐ Skin Irritant ☐ Poison B ☐ Unknown

Sample Disposal (A fee may be assessed if samples are retained longer than 1 month)

☐ Return to Client ☐ Disposal by Lab ☐ Archive for _____ Months

Special Instructions/QC Requirements & Comments:

Custody Seals Intact: ☐ Yes ☐ No | Custody Seal No.: | Cooler Temp. (°C): Obs'd:_____ Corr'd:_____ | Therm ID No.:_____

Relinquished by:	Company:	Date/Time:	Received by:	Company:	Date/Time:
Relinquished by:	Company:	Date/Time:	Received by:	Company:	Date/Time:
Relinquished by:	Company:	Date/Time:	Received in Laboratory by:	Company:	Date/Time:

Form No. CA-C-WI-002, Rev. 4.22, dated 4/10/2019

Attachment A-2
Field Cleaning and Decontamination Procedures

1.0 INTRODUCTION

This procedure describes the cleaning and decontamination of equipment used for sampling. If disposable sampling equipment is not used, sampling equipment must be cleaned before it is used to collect the next sample.

Construction or other equipment not involved in sampling might require decontamination before being moved to another work location on the site or off-site. Dry decontamination, steam cleaning or power washing may be used to clean large equipment in accordance with applicable procedures not included in this Uniform Federal Policy Quality Assurance Project Plan (UFP-QAPP).

Decontamination fluids will be collected, contained, and disposed of in accordance with project requirements and applicable regulations.

2.0 MATERIALS

The following materials will be available, as required, during field cleaning and decontamination activities:

- Personal protective and safety equipment as specified by the task-specific Job Safety Analysis (JSA)
- Analyte-free water
- Non-phosphate detergent (Alconox® or equivalent)
- Appropriate cleaning solvent (e.g., n-propanol, hexane, 10% nitric acid)
- Rinse collection containers
- Knife
- Brushes
- Aluminum foil
- Heavy-duty trash bags or other plastic sheeting
- Spray bottles
- Resealable plastic bags
- Plastic sheeting

3.0 CLEANING PROCEDURE FOR SMALL EQUIPMENT AND SAMPLING DEVICES

Follow the health and safety procedures specified in the task-specific JSA. Cleaning of reusable sampling equipment (e.g., scoops, mixing bowls, spatulas) will follow the decontamination procedures presented below:

- Dry brush or otherwise remove visible material
- Nonphosphate detergent and water wash
- Water rinse
- For organic sampling, rinse equipment with organic solvent (e.g., n-propanol)
- For metals or inorganic sampling, rinse equipment with 10% nitric acid in water solution
- Analyte-free water rinse
- Allow to air dry and wrap in aluminum foil (do not use aluminum foil for metals samples).

Equipment used to collect samples for low reporting limits or specific parameters may be cleaned for those specific parameters using appropriate solvents (e.g., hexane or 10% nitric acid).

Cleaning and decontamination fluids will be collected in appropriate containers and treated at an on-site or off-site treatment facility or otherwise disposed of according to applicable regulations.

Attachment A-3
Sediment Probing and Sampling Procedures

1.0 SCOPE AND APPLICATION

Lexan® tubing will be the primary method used to collect sediment cores. The tubing may be replaced with a calibrated rod if only probing is needed. Coarse material may need to be collected to characterize biota and chemistry. Coarse substrates can range from pebbles and gravel to cobbles, boulders, or bedrock. If the creek bed cannot be penetrated by the Lexan tubing due to large cobbles, boulders, or bedrock, three attempts should be made using a standard split-spoon sampler, stainless-steel bucket auger, trowels or hand collecting, as appropriate. Depending on the sampling plan and objectives, the sampling point may be moved a few feet and the new location documented, in attempt to retrieve a sample. If no sample can be obtained, document the three attempts and proceed to the next location.

Additional methods of sediment sampling (e.g., handheld dredge sampling, Ekman grab sampling) are also described and will be conducted as appropriate to collect surficial sediment samples. Surficial sediment is defined as the upper 2 inches of sediment.

Handheld dredge methods are provided for collecting bulk sediment for benthic macroinvertebrate toxicity studies. Sediment toxicity benchmark (e.g., simultaneously extracted metals/acid-volatile sulfide, total organic carbon) and bulk sediment toxicity (e.g., freshwater amphipod bioassay) sampling studies may involve the use of these procedures for collection of the top 6 inches of sediment.

2.0 EQUIPMENT LIST

The following collection equipment and materials will be available, as required, during sediment sampling:

- Personal protective equipment (PPE) as required by the task-specific Job Safety Analysis (JSA)
- Global positioning system (GPS) unit
- Chest waders
- Cleaning supplies and equipment
- Boat and motor
- Aluminum or stainless-steel tray (do not use aluminum if sediment samples will be analyzed for metals)
- Surveyor’s rods
- Duct tape
- Indelible ink markers

- Lexan tubing with end caps, stainless-steel bucket auger, and/or stainless-steel sediment corer
- Stainless-steel core driver block
- Handheld dredge with rope
- Calibrated rod for sediment depth measurement
- Brass push rod
- Hacksaw, handsaw, or knife
- Vacuum pump
- Stainless-steel Ekman grab sampler (0.25 square feet [ft^2]) with handle (or equivalent)
- Teflon® or polyethylene siphon
- Stainless-steel or plastic ruler
- Stainless-steel spoons
- Stainless-steel mixing bowl
- Stainless-steel trowel
- Camera with date and time setting
- Transport container (cooler) with ice
- Appropriate sample containers and forms
- Field notebook

3.0 CAUTIONS

Cautions include hazards associated with working around water (e.g., drowning, falling on slippery surfaces). Cautions also include biological hazards such as venomous snakes, tick bites, etc. Physical, biological and other site-specific hazards are described in the task-specific Job Safety Analysis (JSA).

4.0 HEALTH AND SAFETY CONSIDERATIONS

Personnel will wear appropriate clothing for working conditions (e.g., light colored, high visibility, long-sleeved clothing) and will have access to warm or cold beverages to avoid dehydration, overheating, or cold stress. Personnel will wear personal flotation devices when working around water. Communication systems and the need for a float plan will be addressed in the task-specific JSA and will be discussed during a task kick-off meeting and during daily health and safety meetings. Physical, biological, and other site-specific hazards are described in the task-specific JSA.

5.0 PROCEDURES

The procedures for sediment probing and sampling using different collection methods are discussed in the following subsections.

5.1 Procedure for Sediment Probing

The calibration rod will be used to probe sediment depths. Measurements made of location, depth, time, and field samples as well as pertinent observations (e.g., staining, odors) will be documented in the field notebook or on task-specific sampling forms.

5.2 Procedure for Lexan Tube Sampling

1. Identify the proposed sample location in the field notebook or sample log along with other appropriate information collected during sediment sampling activities.
2. Don PPE as required by the task-specific JSA.
3. Position boat (if used) or sampling device over the sampling location and record station position.
4. At each sample location, lower a section of Lexan tube until it just reaches the top of sediment. Measure the depth of water. (Sections of Lexan tube might need to be spliced together in deep water locations).
5. Push the Lexan tube into the sediment by hand, then slowly drive the tube into the sediment until the targeted depth (e.g., 2 feet) is achieved, or until refusal. Measure the depth of sediment. If the procedure is being conducted to determine sediment depth (probing), a calibrated rod may be used in place of the Lexan tube. If the procedure is being performed to collect samples for laboratory analysis, continue with Step 6.
6. Drive the tube several more inches, using a stainless-steel core driver and measure the distance. This procedure is performed to obtain a “plug” at the bottom of the core and prevent the loose sediment from escaping.
7. Place a vacuum pump on the top end of the Lexan tube and create a vacuum to prevent the sediments/plug from escaping during retrieval.
8. Slowly pull the tube from the sediment, twisting it slightly as it is removed (if necessary).
9. Before the tube is fully removed from the water, place a cap on the bottom end of the tube while it is still submerged.
10. Keeping the tube upright, wipe the bottom end dry and seal the cap with duct tape. Measure and record the length of sediment recovered and evaluate the integrity of the core. If the core appears to have poor recovery or if the sampling plan requires, attempt a second core adjacent to the first location. The

sampler's judgement will determine which core should be used for the collection of sample increments.

11. Transport the core sample to the sample processing station.
12. While keeping the core upright, use a handsaw to make a horizontal cut in the tube approximately one inch above the sediment.
13. Re-cap the cut end of the tube, seal the cap with duct tape and mark this end as "TOP" with indelible ink marker.
14. Wipe the tube dry.
15. Place a completed sample label on the tube.
16. Record the following information on both the tube and on the cap:
    a. sample number
    b. sampling date
    c. sampling time
17. Place the core sample upright in a container with ice.
18. Repeat the above procedures until all core samples are collected.
19. Sediment cores will be extruded from the Lexan tubing onto an aluminum or stainless-steel tray.
20. If the core is to be photographed, the photograph log will be filled out and a calibrated measuring rod or ruler will be photographed along with the core.
21. Cores will be sectioned into the required depth increments as specified in the sampling plan. Depth intervals and whether and how they are adjusted for poor recovery will be noted in the field book or on the sample logs. Each increment for analysis will be individually packaged. Describe and record sample descriptions.
22. Record the following sediment characteristics in the field logbook or on the core log form:
    a. texture
    b. color
    c. presence of debris
    d. presence of an oily sheen
    e. biological structures
    f. odor (e.g., hydrogen sulfide)
23. Core sections may be frozen to facilitate sectioning when sediment is extremely loose.

24. For cores that require sectioning into thin intervals (e.g., 2 centimeters [cm]), extruding can be accomplished using a mechanical extruder that pushes the core material out through the top of the core tube. Sediment samples are cut into the desired section thickness. Sediment touching the inner sides of the tube will be discarded. Each section will be individually placed into sample containers.
25. The handsaw or knife used to section the core should be decontaminated between each cut.
26. Samples will be stored in coolers on ice until transfer to the laboratory. Label sample containers in accordance with the procedures in Attachment A-1.
27. Record appropriate information in the field notebook.
28. Fill out chain-of-custody form, and handle, pack and ship the samples in accordance with the procedures described in Attachment A-1 to the Uniform Federal Policy Quality Assurance Project Plan (UFP-QAPP) for Operable Unit 1/Operable Unit 2 (OU-1/OU-2) Anniston PCB Site.

5.3 Procedures for Handheld Dredge Sampling

1. Identify the proposed sample location and field conditions in the field notebook or on the sample log form.
2. Don PPE as required by the task-specific JSA.
3. At each sample location, drop the opened dredge from side of boat making sure that the end of the rope is kept inside the boat.
4. Once the dredge has been allowed to settle into the bottom sediments, a hard pull on the rope will close the dredge and capture the sediment inside.
5. Retrieve the dredge into the boat.
6. Open the dredge to allow the sediment to empty onto a stainless-steel tray.
7. Record the sediment description (e.g., appearance, color, grain size).
8. If sampling for chemical analysis, use a stainless-steel spoon or spatula to collect the sample and place it into appropriate sample containers.
9. Label sample containers in accordance with the procedures with Attachment A-1.
10. Fill out chain-of-custody form, and handle, pack and ship the samples in accordance with the procedures described in Attachment A-1.

For bulk sediment collection, repeat steps 3 through 6 until sufficient sediment volume is collected. If chemical laboratory analyses are being performed, rinse blanks will be obtained by pouring deionized water through a cleaned dredge onto a cleaned stainless-steel tray and then into appropriate sample containers. Rinse blanks will be collected at the start and finish of sampling activities.

5.4 Procedures for Ekman Grab Sampling

5.4.1 Grab Sampler Deployment

1. Identify the proposed sample location in the field notebook or sample log along with other field conditions.
2. Don PPE as specified in the task-specific JSA.
3. Before each station is sampled, decontaminate the inner surfaces of the grab sampler and sampling utensils.
4. If the water depth is less than 9 feet, attach the grab sampler to the metal handles.
5. Place the grab sampler on a decontaminated surface and open it.
6. Securely place the two release wires around the release pins.
7. Lower the sampler through the water column at a slow and steady speed.
8. Allow the grab sampler to contact the bottom gently, with only its weight being used to force it into the sediments. The sampler should not be allowed to "free fall" to the bottom because this can result in premature triggering, an excessive wake or improper orientation upon contact with the bottom.
9. Deploy trigger weight to release the doors on the bottom of the grab sampler.

5.4.2 Grab Retrieval

1. After the grab sampler has rested on the bottom for approximately 5 seconds, begin retrieving it at a slow and steady rate.
2. After the grab sampler breaks the water surface, gently lower it onto a decontaminated surface, while maintaining the grab sampler in an upright position.
3. Open the doors on the top of the grab sampler, and inspect the sample for the following acceptability criteria:
    a. The sampler is not overfilled with sample to the point that the sediment surface presses against the top of the sampler or is extruded through the top of the sampler.
    b. Overlying water is present (indicates minimal leakage).
    c. The overlying water is not excessively turbid (indicates minimal disturbance or winnowing).
    d. The desired penetration depth is achieved (i.e., 4 to 5 cm for a 2-inch-deep surficial sample).
4. Penetration depth will be determined by placing a ruler against the center of the inside edge of the opening on top of one side of the grab sampler and extending it into the grab sampler until it contacts the top of the sample. The penetration

depth is determined by the difference between that measurement and the total depth of the grab sampler.

5.5 Sample Removal and Processing

Samples will be handled appropriately for the designated physical, chemical and biological analyses according to the following procedures:

1. For acceptable samples, remove the overlying water by slowly siphoning it off near one or more sides of the grab sampler. Provide that the siphon does not contact the sediments or that fine-grained suspended sediment is not siphoned off. If sediment is suspended in the overlying water, allow it to settle before siphoning off the water.
2. After the overlying water is removed, characterize the sample as specified in the study design. Characteristics that are often recorded include:
    a. texture
    b. color
    c. biological structures (e.g., shells, tubes, macrophytes)
    d. presence of debris (e.g., twigs, leaves)
    e. presence of an oily sheen
    f. odor (e.g., hydrogen sulfide, oil)
3. After the sample is characterized, remove the desired interval (e.g., top 2 inches for a surface sample) using a stainless-steel spatula or equivalent alternative. Materials other than sediment (e.g., large shells, stones, leaves) should be removed and noted on the field log sheet.
4. Transfer the surface sediment to a stainless-steel mixing bowl for homogenization (see Attachment A-12). Additional grab samples may be required to collect the volume of sediment specified in the study design. The mixing bowl should be covered while additional samples are being collected to prevent sample contamination (e.g., from precipitation, splashing water).
5. After a sufficient volume of sediment is transferred to the mixing bowl, homogenize the contents of the bowl using stainless-steel spoons until the texture and color of the sediment appears to be uniform.
6. After the sample is homogenized, distribute subsamples to the various containers as required by the sampling plan.
7. Fill out chain-of-custody form, and handle, pack and ship the samples in accordance with the procedures described in Attachment A-1.

6.0 WASTE MANAGEMENT

Dispose of wastes in accordance with project requirements and applicable regulations.

7.0 DATA RECORDING

Field notes will be recorded during sampling activities, and at a minimum, will include the following:

- Names of field crew and oversight personnel
- Weather conditions
- Date, time and sample location (GPS coordinates if specified)
- Photograph number when pictures are taken (if necessary)

8.0 QUALITY ASSURANCE AND QUALITY CONTROL

Field quality control samples including rinsate or equipment blanks, field duplicates, and additional volume for laboratory spiked samples may be required as specified in the sampling plan. The sediment sampling will be conducted consistent with the procedures outlined in this SOP. Deviations from the SOP will be discussed with the Project Manager prior to changing any field procedures.

Attachment A-4
Freezing Procedures for the Preservation of PCBs in Soil, Sediment, and Biota Samples

1.0 INTRODUCTION

Soil, sediment, and biota samples will be frozen to preserve samples for future analyses. The time between sampling and freezing will be minimized to the extent possible.

2.0 PROCEDURE FOR FREEZING CORE SAMPLES

After samples are collected, they will be photographed and stored in a freezer located in a controlled, limited-access location. Cores will be stored until they are required for analysis or until it is deemed that no further analyses are needed. The holding time for polychlorinated biphenyls (PCBs) in frozen samples is one year. The end use of the data should be considered before choosing to analyze samples outside of technical holding times.

2.1 Materials

The following materials will be available, as required, during sample freezing activities:

- Personal protective equipment (PPE) as required by the task-specific Job Safety Analysis (JSA)
- Cleaning supplies and equipment
- Tape
- Lexan tubing with end caps
- Indelible ink markers
- Transport container with ice
- Freezer, capable of maintaining a temperature of -10°C
- Appropriate sample containers and forms
- Field notebook

2.2 Procedure

1. Collect samples according to the relevant procedure or field sampling plan.
2. Check the contents of the cooler against the chain of custody (COC). Sample labels should be checked for completeness and any necessary corrections made.
3. Place sediment cores in the freezer in an upright position.
4. Note the date and time of freezing on the COC.

5. Cores will remain in an upright position until they are thoroughly frozen, at which time they may be stored stacked horizontally on shelves in the freezer.
6. Cores selected for analysis will be thawed at room temperature in a vertical position prior to processing. Processing will consist of extrusion and sectioning of the core and placement into laboratory provided sample containers. Samples will then be shipped to the laboratory.

2.3 Quality Control

The freezer(s) containing frozen samples will be checked daily during active field work to confirm proper working order and to record the temperature. If a freezer malfunctions, it will be repaired or replaced immediately. After field work is complete, the freezer will be checked at least monthly to verify it continues to work properly. To control/record access to the freezer, a completed COC sticker should be placed across the freezer door opening at the end of each workday during the active sampling effort and after field work is complete. Information on the COC sticker will be recorded in the field notebook. The next person who opens the freezer should first verify the COC sticker is in place, and place a new COC sticker across the freezer door opening—recording pertinent details in the field notebook—when work is complete.

2.4 Disposal

Sample cores will be stored frozen. After the investigation is complete, remaining cores will be disposed of in accordance with project requirements and applicable regulations.

3.0 PROCEDURE FOR FREEZING BIOTA SAMPLES

After biota samples are collected, they will be wrapped, labeled, placed in a cooler, frozen or shipped to the analytical laboratory for analysis. The samples will be stored frozen at the laboratory until they are required for analysis, relinquished to others under COC, or until it is deemed that no further analyses are needed.

3.1 Materials

The following materials will be available, as required, during biota freezing activities:

- PPE as specified by the task-specific JSA
- Cleaning equipment
- Field notebook and forms
- Aluminum foil
- Freezer paper
- Transport container with ice
- Freezer capable of maintaining a temperature of -20 °C

3.2 Procedure

1. Collect biota samples as provided in the relevant field sampling plan.
2. Measure and weigh samples.
3. Field process samples, when necessary.
4. Wrap samples in aluminum foil and then freezer paper with both an inner and outer sample label.
5. Enter the sample information on a COC form.
6. Place the sample in an insulated cooler with ice for transportation.
7. Upon arrival at the laboratory, the contents of the cooler are checked against the COC. Sample labels should be checked for completeness and any deviations brought to the attention of the project manager.
8. Place the samples in the freezer. Note the date and time of freezing on the COC.

3.3 Quality Control

The temperature of the freezer(s) will be monitored continuously via a temperature probe attached to a computerized data system. The freezer(s) containing frozen samples will be manually checked daily during active field work to confirm proper working order and to record the temperature. If a freezer malfunctions, it will be repaired or replaced immediately. After field work is complete, the freezer will be checked at least monthly to verify it continues to work properly. To control/record access to the freezer, a completed COC sticker should be placed across the freezer door opening at the end of each workday during the active sampling effort. Information on the COC sticker will be recorded in the field notebook. The next person who opens the freezer should first verify the COC sticker is in place, and place a new COC sticker across the freezer door opening—recording pertinent details in the field notebook. Laboratory COC procedures are included in the laboratory Quality Assurance Plan.

3.4 Disposal

Samples will be stored frozen until analysis. After the investigation is complete, the samples will be disposed of in accordance with project requirements and applicable regulations.

Attachment A-5
Surface and Subsurface Soil Sampling

1.0 INTRODUCTION

Surface and subsurface soil sampling may be conducted using hand tools. Depth sampling will be performed using hand tools, conventional drill rigs, and/or direct-push technology (DPT) drilling methods. Manual sampling procedures are described below.

2.0 MATERIALS

Sample bottles and preservatives appropriate for the parameters to be analyzed will be used, as well as analyte-free water, coolers, and ice.

The following materials will be available, as needed, for soil sampling activities:

- Personal protective equipment (PPE) and safety equipment as required by the task-specific Job Safety Analysis (JSA)
- Disposable aluminum pie plates or stainless-steel bowls (do not use aluminum if samples are collected for metals analysis)
- Disposable wooden spatulas, spoons or stainless-steel spoons
- Stainless-steel spades
- Stainless-steel hand augers
- Conventional/DPT drill rig and associated equipment
- Global positioning system (GPS) unit
- Indelible ink pens
- Ruler or survey rod
- Zip-top plastic bags
- Transport container (cooler) with ice
- Appropriate sample containers and forms
- Field notebooks

Documentation methods may include sample log forms, chain-of-custody (COC) forms, sample labels and seals, field logbooks, and a camera.

3.0 SOIL SAMPLING PROCEDURE

Soil samples may be collected in intervals from the ground surface to a variety of depths. Sample locations will be identified using stakes, flagging, or other appropriate means, and will be noted

in field logbooks and/or soil sampling logs. Sample points will be located using a GPS, or alternatively using measurements from structures and/or permanent property.

1. Clean equipment that will come in contact with the soil sample (see Attachment A-2) for the procedure for field cleaning/decontamination or use clean, disposable equipment). Collect field and equipment blanks in accordance with the appropriate field sampling plan and Uniform Federal Policy Quality Assurance Project Plan (UFP-QAPP) to document possible contamination from sampling equipment.
2. Clear the ground surface of brush, root mat, grass, leaves, or other debris
3. Use a spade, spoon, scoop, or hand auger to collect a sample over the required depth interval.
4. Use a ruler to ensure the sample is collected to the correct depth and record the top and bottom depths from the ground surface.
5. Collect samples below the surface interval by removing the surface interval first. Clean equipment that will come in contact with the soil sample (see Attachment A-2 to review the procedure for field cleaning/decontamination or use clean, disposable equipment). Then collect the deeper interval. It may be necessary to prevent the hole from collapsing by removing a wider section from the surface or using cut polyvinyl chloride (PVC) tubing or pipe to maintain the opening.
6. Collect samples for volatile organic compounds (VOCs) as discrete samples using Encore® samplers or cut syringes and will be collected before other samples are collected and homogenized (see Attachment A-13 for the procedure for VOC solid sample collection).
7. Samples for other analyses will be homogenized across the required interval or mixed with other discrete grab samples a to form a composite sample (Attachment A-9 for the procedure for compositing samples).
8. Place sample in clean sample container; label with sample ID, date, and time of collection; and place on ice. Prepare for packaging and shipping to the laboratory in accordance with the SOP for sample handling, packaging, shipping and COC (Attachment A-1).
9. Backfill sample holes to grade with native material or with clean builder's sand or other suitable material.

Attachment A-6
Subsurface Drilling and Monitoring Well Installation Procedures

1.0 INTRODUCTION

The following procedures will be implemented during drilling, installing, and developing temporary or permanent monitoring wells. Equipment will be decontaminated as outlined in Attachment A-2 (Field Cleaning/Decontamination Procedures) to the Anniston PCB Site Operable Unit 1/Operable Unit 2 Uniform Federal Policy Quality Assurance Project Plan (UFP-QAPP). Subsurface drilling and monitoring well installation will comply with the Alabama Department of Environmental Management (ADEM) *Alabama Environmental Investigation and Remediation Guidance* (AEIRG) (ADEM 2017) and the United States Environmental Protection Agency (USEPA) *Design and Installation of Monitoring Wells Guidance* (USEPA 2013).

2.0 GENERAL DRILLING PROCEDURES

The drilling and well installation procedures will conform to state and local regulations and guidance. The drill rig will be cleaned and decontaminated according to the guidelines described in Attachment A-2 of the UFP-QAPP and will conform with the procedures detailed in Appendix E.4 of the AEIRG. Drill rigs will not be used if they are leaking fluids that could enter the borehole or contaminate equipment that is placed in the borehole. Use of rags or absorbent materials to absorb fluids leaking form the drill rig is not acceptable. Drilling lubricants will not be used within the borehole.

The boreholes will be advanced using hollow-stem augers, rotosonic drilling, or other site-appropriate drilling methods outlined in Appendix B.2 of the AEIRG, and Section 2.2 in USEPA 2013, with preferred methods being those that case the borehole during drilling. Continuous soil samples will be collected throughout the length of the borehole for lithologic description.

Soil samples will be inspected and logged by qualified field personnel. The soil description will follow the Unified Soil Classification System (USCS) general format and include the following:

- Consistency or density
- Color
- Structural characteristics
- Composition with major components in capital letters
- Minor characteristics
- Geologic description in capital letters

For environmental soil samples, additional descriptions (such as odor, staining, oily sheen) will be noted in field notebooks. A log of drilling activities will be kept in a bound field notebook. Information in the field notebook will include location, date and time, personnel and equipment present, down time, materials used, samples collected, blow count measurements (if performed), and other observations or information that is relevant to the data collection objectives.

3.0 MONITORING WELL INSTALLATION

Typically, monitoring wells will be double-cased (a Type-III well per AEIRG) to prevent cross-contamination of the borehole from the shallow soils during drilling. Initially, a large-diameter pilot borehole (nominally 12 inches) will be advanced to a depth of approximately 5 feet for shallow residuum wells and to a depth of approximately 40 feet for deep residuum wells. Per AEIRG stipulations (Appendix B.3.3) the pilot borehole should extend a minimum of 2 feet, if possible, into the confining layer underlying the respective saturated zones. If the pilot borehole is installed into bedrock, 1-foot penetration is adequate. An 8-inch or 10-inch diameter casing will be placed in the pilot borehole and the annular space grouted (see grout specification below in Section 9.0) by tremie method per AEIRG procedures contained in Appendix B.3.3 The grout will be allowed to cure for a minimum of 24 hours prior to continuing drilling. A 6-inch or 8-inch nominal diameter (depending on the diameter used for the surface casing) borehole will be advanced through the surface casing to the target depth of the well.

If the rotosonic drilling method is used, temporary override casings may be used in place of permanent surface casings as an acceptable alternative to prevent cross-contamination of the borehole from shallow soils during drilling. This approach is referred to as the "telescoping method" and is conducted as follows:

- A 4-inch-diameter core barrel is advanced to the depths specified above for surface isolation (5 feet for shallow residuum wells, 40 feet for deep residuum wells, at least 2 feet into confining layers underneath saturated zones, or 1 foot into a bedrock-confining layer underneath a saturated zone), collecting soil cores continuously.

- A 6-inch-diameter or 8-inch-diameter inner casing is then drilled to the same depth as the 4-inch core barrel. Water may be used, if necessary, to assist in removal of materials between the 4-inch core barrel and inner casing.

- An 8-inch-diameter or 10-inch-diameter (depending on diameter used for the inner casing) override casing is drilled to the same depth, again, using water to remove material between the casings.

- If deemed necessary, a second override casing of 10-inch-diameter or 12-inch-diameter (depending on diameter used for the first override casing) is drilled to the same depth, again, using water to remove material between the first and second override casings. If the second override casing (10-inch-diameter or 12-inch-diameter) was used, then the 4-inch core barrel, inner casing, and first override casing (8-inch-diameter or 10-inch-diameter) are then sequentially advanced from 5 to 10 feet below ground surface.

- The 4-inch core barrel and inner casing are then sequentially advanced to the target depth of the well. After the boring is drilled to depth, the core barrel is removed and the well installed within the inner casing.

- The inner casing is removed as the sand pack is set (so that the bottom of the inner casing is set to the top of the sand pack interval), raised again as the bentonite seal is set (so that the bottom of the inner casing is set to the top of the bentonite seal interval), and then the inner casing is completely removed from the borehole as grout is placed between the inner and override casings.

- After removal of the inner casing, the annular space between the well casing and the override casings is topped off with grout and then the override casings are removed from the borehole. Grout is topped off one last time and left to set before a pad is built.

Permanent or temporary surface casings may be eliminated for wells installed in areas with measured low concentrations of PCBs in the soil.

Monitoring wells will be constructed using a 2-inch-diameter, flush-threaded, Schedule 40 polyvinyl chloride (PVC) casing and a section of slotted, Schedule 40 PVC screen. Well screen lengths will depend on site-specific considerations and will not exceed the maximum length of 10 feet stipulated by the AEIRG (Appendix B.3.2) and will not be less than 5 feet in length (Section 2.5; USEPA 2013). ADEM approval will be required if longer screens are warranted, due to large fluctuations in water table levels for example. An appropriately sized quartz filter pack will be used to fill the annular space between the borehole and the well and will act as a filter to keep fine-grained materials from clogging the well screen. The filter pack and screen slot size will be based on the formation material and the procedures described in Region 4 guidance documents (USEPA 2013). Before the screen and casing are placed into the borehole, a minimum of 6 inches of filter pack material will be placed at the bottom of the borehole to serve as firm footing.

The filter pack will be placed by tremie pipe or by pouring the sand through the hollow stem augers (or casing) as the augers (or casing) are withdrawn, to a minimum of 2 feet above the top of the well screen. A bentonite seal (minimum thickness of 2 feet of dry/non-hydrated pellets) will be installed on top of the filter pack. The bentonite will be allowed to hydrate for a minimum of 8 hours or the manufacturer's recommended time, whichever is greater. If the water table is below the pellet seal, potable or higher quality water will be added repeatedly to hydrate the pellets prior to grouting. The total volume of water added will be recorded on the monitoring well installation log. The remainder of the annulus will be filled with a bentonite/cement grout by tremie method up to 2 feet below ground surface or below the frost line, whichever is greater. The grout will be allowed to set for a minimum of 24 hours before the well surface is completed. Permanent monitoring wells will be completed at the surface with a 3-foot by 3-foot (minimum 6-inch thickness) concrete pad with locking anodized aluminum protective cover. The pad should be slightly sloped to allow drainage from the protective casing and off the pad.

Procedures for completing the monitoring well will include the following:

1. Only the well screen and filter pack portion of the completed monitoring well should be adjacent to the single water-bearing zone to be monitored.
2. The liquid slurry grouts will be placed by pumping the grout through a tremie pipe to the appropriate locations, or, if bentonite pellets are used, the pellets will be properly hydrated and compacted in place using the following steps:
    a. The bentonite pellets will be poured slowly down the well annulus or tremied into place to mitigate bridging of the pellets within the borehole above the desired depth.
    b. The pellets will be compacted in place using a PVC tremie pipe with a cap.
    c. The pellets will be allowed to hydrate for a minimum of 8 hours or the manufacturer's recommended time (whichever is longer) prior to continuing with well construction activities. Hydration will be completed by existing groundwater in the borehole (if present) or by adding clean water as they are placed into the borehole.
3. The protective surface casing will be set in both the cement grout and the concrete pad.

4.0 CASING REQUIREMENTS

The well casing requirements are as follows:

- Casing will be new and unused.
- Casings will be joined only with water-tight flush-joint threads; glue or solvent will not be used to join casing.
- PVC will conform to the ASTM International (ASTM) Standard F480 or NSF International (NSF) Standard 14 (Plastic Pipe System).
- The casing will be straight and plumb within the borehole. The casing will be notched or marked at the top to provide a point of reference for measuring groundwater elevations.

5.0 WELL SCREEN REQUIREMENTS

Well screen requirements are as follows:

- Requirements that apply to casing, except for strength requirements, also apply to well screen.
- Monitoring wells will not be screened across more than one water-bearing unit.
- Screens will be factory slotted.
- Screen slots will be sized to prevent 90% of the filter pack from entering the well. In formations consisting primarily of fines (silts and clays), the procedures for well screen design might require filter packs and screen slot sizes that are not commercially available. USEPA guidelines state the selection of 0.010-inch screen

slots with a 20-40 sand filter pack or 0.005-inch screen slots with 100 sand filter pack for very fine formations will be acceptable practice (USEPA 2013).

- The bottom of the screen will be capped, and the cap will be joined to the screen by threads. For wells where production of very fine sediment could occur, a 1- to 3-foot section of blank PVC casing may be placed below the screen to serve as a sump.

6.0 ANNULAR SPACE REQUIREMENTS

The annular space requirements are as follows:

- The annular space will be filled with a filter pack, including a 6-inch layer at the bottom of the borehole, a bentonite seal, and casing grout between the well string and the borehole wall.
- As the annular space is being filled, the well string will be centered. Centralizers are recommended for monitoring wells deeper than 50 feet.

7.0 FILTER PACK REQUIREMENTS

The filter pack will consist of silica sand and will extend from the bottom of the borehole to at least 2 feet above the top of the well screen. The filter pack material will be clean, inert, and well-rounded and will contain less than 2% flat particles. The sand will be certified free of contaminants by the vendor or contractor.

The filter pack will have a uniform, well-sorted, grain size and uniformity coefficient compatible with the formation materials and the screen. In formations consisting primarily of fines (silts and clays), the procedures for well screen design might require filter packs and screen slot sizes that are not commercially available. USEPA guidelines state the selection of 0.010-inch screen slots with a 20–40 sand filter pack or 0.005-inch screen slots with 100 sand filter pack for very fine formations will be acceptable practice (USEPA 2013).

The filter pack will not extend across more than one water-bearing unit. The filter pack will be placed by pouring the sand through the drilling string as the drill string is withdrawn.

8.0 BENTONITE SEAL REQUIREMENTS

The bentonite seal requirements are as follows:

- The bentonite seal will consist of at least 2 feet of dry/non-hydrated bentonite between the filter pack and the casing grout.
- The bentonite will be hydrated before casing grout is placed.
- Only 100% sodium bentonite will be used.

9.0 CASING GROUT REQUIREMENTS

The casing grout requirements are as follows:

- The casing grout will extend from the top of the bentonite seal to 2 feet below ground surface or below the frost line, whichever is greater, per AEIRG Appendix B.3.
- The grout will be mixed in the following proportions: 5% bentonite powder and 95% Portland cement, consistent with Section 2.3.5 in USEPA (2018)
- Grout will be pump tremied using either a side-discharge or a bottom-discharge tremie pipe.

10.0 SURFACE COMPLETION REQUIREMENTS

The aboveground surface completion of the permanent monitoring wells will be completed by extending the well a minimum of 2.5 feet above the ground surface (Section 2.3.6; USEPA 2013). A protective casing will be provided for each well, which will be seated in a 3-foot by 3-foot by 6-inch thick concrete surface pad. Flush mounted surface completions will be completed using a commercially available well box, at least 12 inches in diameter, seated in a 3-foot by 3-foot by 6-inch thick concrete pad. To allow atmospheric equilibration of the headspace within the monitoring well, a small-diameter (e.g., 0.25 inches) vent hole will be placed in the well casing, or a ventilated well cap will be used. A weep hole will be installed in the protective casing to prevent the casing from filling with water. The diameter of the protective casing will be at least 2 inches greater than the diameter of the well casing. The concrete pad will be sloped away from the well. A lockable cap or lid will be installed on the protective casing. The identity of the well will be permanently marked on the casing cap, the protective cover, or the concrete pad.

11.0 MONITORING WELL COMPLETION DIAGRAMS

A completion diagram will be submitted for each monitoring well installed. The diagram, at a minimum, will include the following information:

- Well identification (this will be the same as the boring identification described)
- Drilling method
- Drilling company name and driller name(s)
- Field personnel name(s)
- Installation date(s)
- Elevations of ground surface and the top of casing
- Total boring depth
- Lengths and descriptions of the screen and casing
- Lengths and descriptions of the filter pack, bentonite seal, casing grout, and any back-filled material
- Elevation of water surface before and immediately after development

- USCS description of the material penetrated by the boring

12.0 MONITORING WELL DEVELOPMENT

Well development will be conducted in accordance with procedures in AEIRG Appendix B.4., and USEPA (2013) Section 2.7. Monitoring wells will not be developed until at least 24 hours after installing the surface pads or, if a surface pad is not installed, at least 24 hours after grouting the annular space. The monitoring wells will be developed using bailing or pumping to produce representative formation water. Wells will be developed using a standard size pump with a surge block and/or a stainless-steel or Teflon® bailer with nylon rope. Development will be continued until representative water that is free of cuttings or other materials introduced during well construction is obtained. Representative water is generally assumed to have been obtained when the water is free of visible sediment and the pH, temperature, and specific conductivity have stabilized to within ±0.1 standard units, ±0.5°C, and ±10%, respectively. Ideally, the measured turbidity readings should be less than 10 Nephelometric Turbidity Units (NTUs) or should have stabilized to within ±10% for values greater than 10 NTUs.

The minimum duration of well development will vary in accordance with the method used to develop the well. For example, surging and pumping the well may provide a stable, sediment-free sample within minutes; whereas, bailing the well may require several hours of continuous effort to obtain a clear sample. Field personnel will determine appropriate development methods based on site-specific conditions observed during well construction. Field personnel will also record the methods of well development, duration, pH, temperature, turbidity, and specific conductivity readings in the well-development log.

Well development water will be contained and disposed of in accordance with project requirements and applicable regulations.

13.0 REFERENCES

ADEM. 2017. *Alabama Environmental Investigation and Remediation Guidance (AEIRG)*. Revision 4.0. February.

USEPA. 2018. *Design and Installation of Monitoring Wells*. United States Environmental Protection Agency, Region 4, Science and Ecosystem Support Division, Athens, Georgia. SESDGUID-101-R2, January 16. Available at: https://www.epa.gov/sites/production/files/2016-01/documents/design_and_installation_of_monitoring_wells.pdf.

Attachment A-7
Groundwater Sampling Procedure

1.0 INTRODUCTION

The general procedures for groundwater sampling are outlined below and were developed based on the U.S. Environmental Protection Agency (USEPA) *Standard Operating Procedures (SOP) for Low-Stress (Low Flow)/Minimal Drawdown Ground-Water Sample Collection* (USEPA 2015), the USEPA *Region 4 Groundwater Sampling Operating Procedure* (USEPA 2017), and the Alabama Department of Environmental Management (ADEM) *Alabama Environmental Investigation and Remediation Guidance* (AEIRG) (ADEM 2017). Sampling activities will be documented in the field notebook. Additionally, sampling data will be recorded on a groundwater sampling log form specific to each well sampled. Before groundwater sampling begins, wells will be inspected for signs of tampering or other damage. If tampering is suspected, (e.g., casing is damaged, lock or cap is missing) field personnel will record that information in the field notebook and on the well sampling form.

Before starting sampling activities, field personnel will place plastic sheeting on the ground surrounding the well. The plastic sheeting will be used to provide a clean working area around the wellhead and prevent soil or other debris from contacting sampling equipment. Water will be removed from the protective casing or the vaults around the well casing prior to venting and purging. Purging and sampling will be performed using the low-flow method in a manner that minimizes aeration of the water column and the agitation of sediments in the well and formation. Equipment will not be allowed to free-fall into a well.

The following information will be recorded when a well is purged and sampled: (1) depth to water before and after purging and (2) the condition of each well. Additionally, the total depth of the monitoring well will be sounded annually and after sample collection.

2.0 MATERIALS

Positive-pressure (bladder) pumps will be used to evacuate wells and collect samples. Equipment to operate these dedicated pump systems include a pressure source (e.g., air compressor, compressed air or nitrogen cylinders) a power source (e.g., battery or electric generator), and a controller. In addition, nondedicated sampling apparatus, such as surface discharge tubing and valving, might be required.

If wells do not have dedicated pump systems, a disposable pump system with accessories small enough to enter the wells will be necessary. Equipment that could contact the sample will be made of acceptable materials and cleaned before use. Acceptable materials that may contact groundwater samples are stainless steel, fluorocarbon resin (Teflon®), low-density polyethylene (LDPE), and glass.

Sample bottles and preservatives appropriate for the parameters to be sampled will be necessary as well as organic-free deionized water, coolers, and ice. The following materials will be available, as required, during groundwater sampling activities:

- Personal protective equipment and safety equipment as required by the task-specific Job Safety Analysis (JSA)
- Multiparameter meter or individual meters capable of measuring pH, specific conductivity, dissolved oxygen (DO), temperature, oxidation/reduction potential (ORP), and turbidity
- Calibration standards for field measurements
- Filtration apparatus, if necessary
- Electric water-level indicator with 0.01-foot increments
- Indelible ink markers
- Transport container with ice
- 55-gallon steel drums fitted with bung holes or suitable tank(s) for containing purged groundwater
- Appropriate sample containers and chain-of-custody forms
- Field notebook and groundwater sampling forms

3.0 WATER LEVEL MEASUREMENT PROCEDURE

Water-level measurements will be taken after wells have been installed and developed and water levels have recovered. Static water levels will also be measured each time a well is sampled and before any equipment enters the well. Any substantial change in conditions (e.g., barometric pressure change after a weather change) that may affect water levels during the water level measurement event will be recorded in the field log. If the casing cap is not vented, allow time for pressure equilibration after the cap is removed prior to measurement, and collect several measurements to confirm the water level is stabilized.

The groundwater level will be measured to the nearest 0.0l foot using an electric water-level indicator. Water levels will be measured from the notch or permanent mark located at the top of the well casing and recorded on the groundwater sampling form. If well casings are not notched or marked, measurements will be taken from the north side of the top of the well casing.

Following water level measurement, the total depth of the well from the top of the casing will be determined using the monitoring well installation log (or an updated well depth table) and recorded on the groundwater sampling form. The water level depth will then be subtracted from the total depth of the well to determine the height of the water column present in the well casing. For wells that are part of the sampling program, the total depth of the well will be measured using a weighted tape or electric water-level indicator on an annual basis after sample collection. The total depth measurement will be recorded in an updated well depth table. If well condition indicates a need for well redevelopment to remove accumulated sediment, the total well depth will be measured again after redevelopment.

4.0 FIELD INSTRUMENT CALIBRATION

Field measurements of temperature, specific conductance, DO, pH, ORP, and turbidity will be made during purging and developing each well. Meters will be calibrated according to the guidance supplied in the equipment manual using appropriate calibration standards. Calibration standards for all equipment should be National Institute of Standards and Technology (NIST) traceable. The calibration of the field equipment will be checked at the beginning of each day and after every 10 hours of use or at the manufacturer's suggested interval, whichever is less. The field equipment will only be recalibrated when necessary and in accordance with manufacturer's requirements.

5.0 LOW-FLOW PURGING PRIOR TO SAMPLING

Monitoring wells are purged to evacuate water that has been stagnant in the well and may not be representative of the aquifer. Purging will be accomplished using the low-flow method described in USEPA (2015, 2017) and ADEM (2017) guidance. Other sampling methods, which may be applicable for certain situations at the site, are described in these guidance documents. Using the low-flow method, the pump intake is positioned within the screened interval and the well is purged at a rate that will cause no more than 0.33 feet of drawdown. If this is not practicable (i.e., the flow rate is less than 200 milliliters per minute [ml/min]), greater drawdown will be allowed for a purge rate of 100 to 200 ml/min. For wells completed with screen intervals across the water table, the pump intake will be placed near the top of the water column. If drawdown approaches the screened interval (for wells with screen intervals below the water table) or if drawdown approaches the bottom of the well (for wells with screen intervals across the water table), the pump will be turned off and allowed to recover (the well does not have to fully recover before resuming the purging effort).

During purging, the water level within the well and the water-quality stabilization parameters (i.e., temperature, pH, specific conductivity, turbidity, DO, and ORP) will be monitored and recorded on the groundwater sampling form. Water quality will be assumed stabilized when the water is free of visible sediment and the pH, temperature, specific conductivity, and ORP have stabilized to within ±0.1 standard units, ± 3°C, ±3%, and ±10 millivolts (mV) respectively. Also, the measured turbidity readings should be less than 10 Nephelometric Turbidity Units (NTUs) or should have stabilized to within ±10% for values greater than 10 NTUs. DO stabilization criteria are ±10% for values greater than 0.5 milligrams per liter (mg/L) or 3 consecutive readings below 0.5 mg/L. However, if parameters do not stabilize and all other protocol are followed, well purging will be considered complete after a minimum of three well volumes have been removed, provided the well does not purge dry. Wells that purge dry will be considered properly purged with one well volume. The well volume is defined as the volume of submerged casing and screen. One well volume can be calculated using the following equation:

$$V = \pi (D/2)^2 \times h \times 7.48 \text{ gallons per cubic foot (gal/ft}^3\text{)}$$

where:

V= well volume in gallons

h = height of water column in feet

π = pi or 3.14 (dimensionless)

D = the inside diameter of the well casing (feet)

Water removed from the well during purging will be collected and disposed of according to project requirements and applicable regulations.

6.0 SAMPLE COLLECTION

Field personnel will wait at least 24 hours after wells are developed before sampling. This will allow the aquifer to recover. Groundwater sampling data will be maintained in a hard copy and electronic format and will include specific field sampling data, inspection results, and well maintenance information.

7.0 SAMPLE CONTAINERS AND STORAGE

Groundwater samples will be labeled with indelible ink marker, sealed, and stored in coolers with securely closed lids for storage and transport. The samples will be packed with ice and maintained at a temperature below 6°C, but above freezing. Samples must be received by the analytical laboratory in sufficient time to conduct the requested analyses within the specified holding time.

8.0 SAMPLE ACQUISITION

8.1 Volatile Organic Compounds (VOCs)

Samples for VOCs will be obtained from the well using a dedicated positive pressure (bladder) pump or a disposable pump system and will be collected directly from the pump discharge tube into 40-ml, glass vials provided by the laboratory. Contact with air and sample agitation should be minimized. These samples will not be filtered. Samples for analysis by USEPA SW846 Method 8260 will be preserved with hydrochloric acid (HCl) to a pH less than 2. Immediately after the sample is collected, a Teflon-lined silicon septum cap will be tightened onto the vial. There should be no air bubbles remaining within the vial once the cap has been fastened.

8.2 Semivolatile Organic Compounds (SVOCs), Polychlorinated Biphenyls (PCBs), and Pesticides

Samples for extractable SVOCs, PCBs, or pesticide analyses will be collected directly from a positive-pressure (bladder) pump discharge port or bailer into appropriate sample bottles with Teflon-lined lids. Samples should not be allowed to overflow the sample bottle. No preservatives

will be added to the samples. The same procedures will be used to collect samples for PCB congeners and for dioxin analyses.

Filtered and unfiltered samples for PCB analysis will be collected as specified in the appropriate field sampling plan and according to program-specific requirements. For monitoring wells sampled, filtration using a 0.1-micron filter is specified.

Filtration is best accomplished by using an in-line filter system in which the sample is directly fed from the discharge port of positive-pressure (bladder) pump through the filter and into the appropriate sample bottle. A less preferred but acceptable method is collecting an adequate sample from a positive-pressure (bladder) pump or bailer into a properly cleaned and prepared glass bottle, then immediately filtering it using a peristaltic pump and collecting it directly into the appropriate sample bottle. In addition, aliquots of unfiltered samples will be collected directly from the pump discharge port into the appropriate sample bottle.

8.3 Metals

Unfiltered and filtered samples for metals analysis will be collected using the sampling method described above. Samples for metals will be filtered in the field, as above. Additionally, these groundwater samples (filtered and unfiltered) will be placed into containers with nitric acid to obtain a pH less than 2. In-line sample filters will be used to fill the preserved containers.

9.0 SAMPLE PRESERVATION

Sample containers will be provided by the laboratories with premeasured preservatives. Additional preservatives may also be provided by the laboratory. Samples requiring field preservation should be preserved immediately upon collection in the field or after filtration.

10.0 REFERENCES

ADEM. 2017. *Alabama Environmental Investigation and Remediation Guidance* (AEIRG). Revision 4.0. February.

USEPA. 2015. *Standard Operating Procedures (SOP) for Low-Stress (Low Flow) / Minimal Drawdown Ground-Water Sample Collection*. U.S. Environmental Protection Agency, June 17. Available at: https://www.epa.gov/quality/standard-operating-procedure-low-stress-low-flow-minimal-drawdown-ground-water-sample.

USEPA. 2017. *Groundwater Sampling Operating Procedure*. U.S. Environmental Protection Agency, Region 4, Science and Ecosystem Support Division, Athens, Georgia. SESDPROC-301-R4, April 26. Available at: https://www.epa.gov/sites/production/files/2016-01/documents/design_and_installation_of_monitoring_wells.pdf.

Attachment A-8
Monitoring Well/Borehole Abandonment Procedure

1.0 INTRODUCTION

This document outlines the general procedures for abandoning (or decommissioning) a permanent or temporary monitoring well or piezometer. Well abandonment will follow the guidelines provided by the Alabama Department of Environmental Management (ADEM) *Alabama Environmental Investigation and Remediation Guidance* (AEIRG) (ADEM 2017).

2.0 MATERIALS

The following materials will be available, as required:

- Personal protective equipment (PPE) and safety equipment as required by the task-specific Job Safety Analysis (JSA)
- Portland® cement
- Bentonite powder
- Field notebook

3.0 DECOMMISSIONING PROCEDURES

3.1 General

Well abandonment procedures include sealing the well to prevent vertical or horizontal migration of contaminants and decontaminating drilling rigs and other down-hole tools prior to and after working on a well. Decommissioning will be conducted by a drilling contractor registered with the state of Alabama. Field personnel under contract to Solutia will oversee the decommissioning of the wells and record the following in the field notebook:

- Well location
- Date
- Time on-site
- Equipment used
- The depth and material used for each layer of sealant
- Measurement of static water level and depth prior to decommissioning
- Materials used and quantity of each material used
- Whether additional materials were added to compensate for settlement

3.2 Standard Decommissioning Procedures

In accordance with ADEM guidance (ADEM 2017), the standard procedure for well abandonment includes complete removal of the well casing and screen from the borehole. The procedure presented below will be used:

- The monitoring well riser and screen will be over-drilled to the original boring depth with an auger or drill bit at least two inches larger than the outer diameter of the well casing. Well materials will be removed from the auger or drill casing during drilling. Well casing, backfill material, and well construction materials should be completely removed from the ground.
- The clean borehole will be backfilled with grout consisting of 5% bentonite and 95% Portland cement. The tremie method will be used to pressure grout the borehole from the bottom up to ground surface.
- A mixing system will be used to prepare the grout and a positive displacement pump will be employed to pump the grout into the borehole.
- Boreholes will be checked at least 24 hours but no more than 48 hours after grouting to ensure the grout has cured properly and settling has not occurred. If settling is observed, grout will be added to fill the hole to within 2 feet of ground surface.
- Well surface completions will be demolished and removed.
- The borehole will be finished with a concrete surface plug for the upper 2 feet of the borehole. A concrete surface pad will be installed only for wells in areas of heavy traffic.

3.3 Additional Decommissioning Procedures for Wells with Surface Casing

For the wells with a surface casing (i.e., double-cased wells), the following modifications will be made:

- The monitoring well casing and screen will be over-drilled as described above; however, the borehole will be grouted to the base of the surface casing instead of to ground surface.
- The surface casing will be over-drilled, removed from the ground, and pressure grouted using the methods described above.
- In the event that a surface casing cannot be removed due to size, alignment, or materials of construction, ADEM will be notified, and the casing will be grouted in place. Per ADEM (2017) guidelines, surface casings with known construction design and without casing integrity issues may be left in the ground and filled with concrete, grout, neat cement, or a bentonite-cement mixture (i.e., 5% bentonite and 95% Portland cement).

3.4 Contingent Decommissioning Procedures

In some cases, it may not be possible to successfully remove the well casing. For such wells, ADEM will be notified with a request for approval to abandon the well by perforating the casing and grouting in place.

3.5 Decontamination of Drilling Equipment

Equipment used for plugging and abandoning wells and piezometers will be decontaminated between well locations and prior to leaving the site. Decontamination will involve steam cleaning, high-pressure washing, or other appropriate method using water from a municipal water supply or from the Solutia facility. Decontamination rinsate will be contained in an isolated, bermed pad and collected in tanks or drums. The rinsate will be characterized either by sampling and laboratory analysis or by process knowledge based on groundwater sampling results for the wells to be decommissioned. The rinsate will be managed and disposed of in accordance with applicable regulations depending on the rinsate characterization and concentrations of potential constituents of concern.

3.6 Waste Management

Excess grout and other well-abandonment-associated waste (e.g., well casings, surface casings, well pad concrete, protective bollards) will be managed and disposed of in accordance with applicable regulations.

4.0 REPORTING

A final report describing the procedures of the well decommissioning program will be prepared for submittal to ADEM and USEPA Region 4. The report will include the following information:

- Site name
- Site address
- Well type
- Reason for abandonment
- Well ID
- Abandonment date
- Abandonment supervisor
- Coordinates of well location
- Well location on site map
- Description of abandonment methods including total depth, sealant used, etc.
- Waste disposal records
- Photographs documenting abandonment

5.0 REFERENCES

ADEM. 2017. *Alabama Environmental Investigation and Remediation Guidance (AEIRG)*. Alabama Department of Environmental Management, Revision 4.0, February.

Attachment A-9
Procedures for Compositing or Homogenizing Soil or Sediment Samples

1.0 INTRODUCTION

The general procedures to be used in composting/homogenizing samples are outlined below.

2.0 MATERIALS

The following materials will be available, as required, when composting or homogenizing samples:

- Personal protective equipment (PPE) and safety equipment as required by the task-specific Job Safety Analysis (JSA)
- Stainless-steel spoon (or the disposable equivalent)
- Stainless-steel bowl (or the disposable equivalent)
- Appropriate sample containers and forms
- Field notebook

3.0 PROCEDURE FOR COMPOSTING OR HOMOGENIZING SAMPLES

Samples might require homogenization across a given depth interval, or several discrete grabs (usually five) might be combined into a composite sample. Samples for volatile organic compound (VOC) analysis will not be homogenized or composited. The procedure for mixing samples is provided below.

1. Mix the materials in a stainless-steel bowl (or the disposable equivalent) using a stainless-steel spoon (or disposable equivalent.) Note: When preparing samples for metals analyses, do not use disposable aluminum (or metal tools or trays other than stainless steel) as they may influence analytical results.
2. Flatten the pile by pressing the top without further mixing.
3. Divide the circular pile into equal quarters by dividing out two diameters at right angles.
4. Mix each quarter thoroughly with a spoon.
5. Mix two quarters (as described above) to form halves. Then mix two halves to form a composite or homogenous sample.

6. Place composite or homogenized sample into specified containers. Remaining material will be disposed of in accordance with project requirements and applicable regulations.

Attachment A-10
Slug Testing Procedure

1.0 INTRODUCTION

The general procedures for aquifer slug testing at the Anniston Polychlorinated Biphenyl (PCB) Site are outlined below and were developed based on the United States Environmental Protection Agency (USEPA) *Compendium of ERT Groundwater Sampling Procedures* (USEPA 1999), the *Ohio EPA Technical Guidance Manual for Ground Water Investigation* (Ohio EPA 2006), and *The Design, Performance, and Analysis of Slug Tests* (Butler 1998). Slug testing activities will be documented in the field notebook. Additionally, key slug testing data and parameters will be recorded on a slug testing log form specific to each well tested. Before slug testing begins, wells will be inspected for signs of tampering or other damage. If tampering is suspected (e.g., casing is damaged, lock or cap is missing), field personnel will record that information in the field notebook and on the slug testing form and will initiate corrective action if necessary, in consultation with the Anniston Remediation Group.

Before slug testing begins, field personnel will place plastic sheeting on the ground surrounding the well. The plastic sheeting will provide a clean working area around the wellhead and prevent soil or other debris from contacting slug testing equipment. Water will be removed from the protective casing or the vaults around the well casing prior to venting and slug testing.

Slug testing will be performed in a manner that creates measurable water rises or water drawdowns within the well. Equipment will not be allowed to free fall into a well.

The following information will be recorded when a well is tested:

- Depth to water before, during and after each slug test, as well as the time of the measurement
- The size(s) (i.e., length and diameter and/or volume) of the slug used for the test
- The elevation of the pressure transducers inside the well.

Additionally, the total depth of the monitoring well will be sounded and recorded before the slug test(s) begins.

2.0 MATERIALS

Polyvinyl chloride (PVC) slugs of known volume will be used to displace the water level within the well. The slugs will be carefully lowered or removed from the well using string strong enough to support the weight of the slug without breaking. In addition, pressure transducers (e.g., an In-Situ LevelTROLL 500) will be used to continuously record the water levels during the slug testing activities. Vented pressure transducer cables should be long enough to place the

transducer 1 foot above the bottom of each well being tested or monitored. Desiccant packs with the proper connection to the wellhead end of the vented cables should be used to protect the transducer from moisture.

The following additional materials will be used during slug testing activities:

- Personal protective equipment (as required by the site-wide health and safety plan [Site-Wide HASP] for the Anniston PCB Site)
- Transducer Bluetooth reader (e.g., In-Situ Wireless TROLL Com) to download water level data
- Electric water-level indicator with 0.01-foot increments
- Stopwatch
- Indelible ink markers
- Field notebook and slug testing forms

3.0 WATER LEVEL MEASUREMENT PROCEDURE

The water level will be measured after wells have been installed and developed and water levels have recovered. Static water levels (SWLs) will also be measured each time a well is tested, and before any equipment enters the well. Any substantial change in conditions (e.g., barometric pressure change after a weather change) that may affect water levels during the slug test will be recorded in the field log. If the casing cap is not vented, allow time for the pressure to equilibrate after the cap is removed prior to measurement, and collect several measurements to confirm the water level is stable.

The groundwater level will be measured to the nearest 0.01 foot using an electric water-level indicator. Water levels will be measured from the notch or permanent mark located at the top of the well casing and recorded on the slug testing form. If well casings are not notched or marked, measurements will be taken from the north side of the top of the well casing.

Once the water level has been measured, the total depth of the well from the top of the casing will be measured using an electric water-level indicator recorded on the slug testing form. The water level depth will then be subtracted from the total depth of the well to determine the height of the water column present in the well casing.

4.0 PREPARATION PRIOR TO SLUG TESTING

A pressure transducer will be placed in each well before the slug test begins. Pressure transducers shall be placed 1 foot above the bottom of the well. The pressure transducer cable shall be properly secured at the wellhead (e.g., using a spring clamp or carabiner). The deployed pressure transducer shall be configured to record in units of “feet of head” at a rate of 1 reading per second with a linear sampling interval. The transducer will be set to start recording shortly

before the initial slug test begins. The desiccant pack must be properly attached to the wellhead end of the pressure transducer cable to prevent moisture from entering the cable after initializing the transducer. Avoid connecting/disconnecting the desiccant pack or Bluetooth reader to the vented cable during the tests to avoid affecting the data being collected by the transducer. Monitoring well caps should be closed when they are not being tested or monitored.

5.0 SLUG TESTING

The following steps shall be conducted to perform a slug test:

1. Determine the initial size of the slug to be used. When deployed, the slug should be fully submerged (the top of the slug should be within 1 foot of the SWL) but remain above the transducer (the bottom of the slug should remain 1 foot above the transducer to avoid any chance of interference between the slug and transducer). The longest possible length of slug to be used is therefore 2 feet less than the distance between the SWL and the top of the transducer. The expected water level rise in the well caused by the slug can be calculated as follows:

$$Expected\ water\ level\ rise = \frac{diameter\ of\ slug\ (in\ inches)^2 * length\ of\ slug\ (ft)}{diameter\ of\ well\ (in\ inches)^2}$$

   The expected water level rise must not be greater than the depth to the SWL in the well to avoid overflowing the well. Select an appropriate length and diameter of the slug based on the above criteria and record both on the slug test field form. The initial slug selected should be smaller than the largest possible slug to minimize the chance of a very long initial slug test if the hydraulic conductivity of the aquifer is very low.

2. Measure the required length of string so that when deployed, the slug is fully submerged and the top of the slug is within 1 foot of the SWL.
3. Slowly lower the PVC slug into the well until the bottom of the slug is approximately 1 foot above the SWL. Record the start and end times for the slug test on the field form and in the field notebook.
4. Quickly lower slug into the well in a fast but controlled manner to minimize splashing and rapid oscillations of the water table and so that top of the slug will be placed at the measured depth. This phase of the test is referred to as the slug-in portion.
5. Monitor the water level in the well with the electronic water-level meter following the slug deployment, recording the initial reading every 2 minutes during the first 10 minutes of the test, and at least every 10 minutes after that. Take care to measure the water level without submerging the water-level meter to avoid affecting the transducer data. Calculate the rate of water level drop to estimate total time it will take to return to the static level.

6. Once the water level has returned to within 5% of the initial head displacement (Butler 1998), the slug-out portion of the test may start. To begin the slug-out phase of the test, quickly remove the slug in a controlled manner to minimize splashing and rapid oscillations of the water table while also avoiding tangling the slug string with the pressure transducer cable.
7. Once slug is removed, monitor the water level in the well with the electronic water-level meter similarly as was done during the slug-in phase of the test. The time for the slug-out phase of the test should be comparable to the time required for the previous slug-in phase.
8. Evaluate the water level rise and fall after the first set of slug-in and slug-out tests to determine slug size for the remaining test(s), ensuring that subsequent tests have measurable initial head displacements and are not excessively long in duration. Preferably, three sets of slug-in and slug-out tests (six total tests) should be performed with at least two different slug volumes; however, if six tests are not feasible due to a very low hydraulic conductivity and long recovery times, a minimum of three tests with usable data must be conducted. The next slug test may commence when the water level in the well has returned to 5% or less of the initial head displacement; if the next test will use a different volume slug, the next test may begin when the water level in the well has returned to within 5% of the expected initial head displacement of the next slug to be used (Butler 1998).
9. At the end of the slug testing, download the groundwater level data from the pressure transducer. Decontaminate the PVC slugs, pressure transducer, and vented cable with approved site decontamination procedures. The electronic data should be quickly viewed in the field to ensure proper function of the transducer and general usability of the data.

6.0 REFERENCES

Butler, J.J. 1998. *The Design, Performance, and Analysis of Slug Tests*. Boca Raton, Fla: Lewis Publ.

Ohio EPA. 2006. *Technical Guidance Manual for Ground Water Investigations (Chapter 4).* Ohio Environmental Protection Agency. December. Available at https://www.epa.state.oh.us/portals/28/documents/TGM-04_final1206W.pdf.

USEPA. 1999. *Compendium of ERT Groundwater Sampling Procedures.* United States Environmental Protection Agency. January. Available at https://www.epa.gov/sites/default/files/2015-06/documents/fieldsamp-ertsops.pdf.

Attachment A-11
Specific-Capacity and Pumping Test Procedures

1.0 INTRODUCTION

The general procedures for aquifer specific-capacity (also known as a "step test") and pumping test (also known as a "constant rate test") at the Anniston Polychlorinated Biphenyl (PCB) Site are outlined below and were developed based on the United States Environmental Protection Agency (USEPA) *Compendium of ERT Groundwater Sampling Procedures* (USEPA 1999) and the *Ohio EPA Technical Guidance Manual for Ground Water Investigation* (Ohio EPA 2006). A specific-capacity test is conducted prior to a pumping test to estimate the largest pumping rate a pumping well could sustain without causing the well to go dry. The specific-capacity test and pumping test will be documented in the field notebook. Additionally, key pumping data and parameters will be recorded on a pumping test log form specific to the well being tested. Before conducting specific-capacity or pumping tests, wells will be inspected for signs of tampering or other damage. If tampering is suspected (e.g., casing is damaged, lock or cap is missing), field personnel will record that information in the field notebook and on the pumping test form and will initiate corrective action, if necessary, in consultation with the Anniston Remediation Group.

Before specific-capacity or pumping tests begin, field personnel will place plastic sheeting on the ground surrounding the well. The plastic sheeting will provide a clean working area around the wellhead and prevent soil or other debris from contacting pumping test equipment. Water will be removed from the protective casing or the vaults around the well casing prior to venting and pumping test activities. The specific-capacity and pumping tests will be performed in a manner that should produce measurable water drawdowns within both the pumping well and the observation well(s).

The following information will be recorded when a well is tested:

- Depth to water before, during, and after specific-capacity and pumping test activities, as well as the time of the measurement in both the pumping well and the observation well(s)
- The pumping rate
- The elevation of the pressure transducers inside the well

Additionally, the total depth of the pumping and observation wells will be sounded before testing activities.

2.0 MATERIALS

A pump with variable speed controller will be used to remove water from the pumping well to create drawdown that can be measured accurately in the pumping well and the observation wells. Ensure with the pump vendor and/or manufacturer that the pump(s) selected for specific-capacity and pumping test activities are designed to run continuously within a well of specified diameter for extended periods of time. Pressure transducers (e.g., an In-Situ LevelTROLL 500) will be used to continuously record the water levels during the specific-capacity and pumping test activities at the pump well and the observation well(s). Vented pressure transducer cables should be of sufficient length to place the transducer 1 foot above the bottom of each well being tested or monitored. Desiccant packs with the proper connection to the wellhead end of the vented cables should be used to protect the transducer from moisture.

Equipment used to conduct the pumping tests should be used according to the manufacturer's operating manual. An appropriate maintenance check will be performed on each piece of equipment prior to use. If damaged or failed parts are identified during the maintenance check and it is determined that the damage can impact the equipment performance, the equipment will be removed from service until the part or parts are replaced or repaired.

The following additional materials will be used, as necessary, during specific-capacity tests and pumping tests:

- Personal protective equipment (as required by the site-wide health and safety plan [Site-Wide HASP] for the Anniston PCB Site)
- A power supply, such as a generator or power outlet, that is protected by continuous ground-fault circuit interrupters (GFCI)
- GFCI electrical extension cord
- Large water container/tank(s) (approximately 2,000 gallons of storage)
- Transducer Bluetooth reader (e.g., In-Situ Wireless TROLL Com) to download water level data
- An inline flow meter device (for measuring high flow rates of > 1 gallon per minute [gpm])
- A graduated bucket (for measuring intermediate flow rates of > 0.5 gpm and < 1 gpm)
- A measuring cup (for measuring low flow rates of < 0.5 gpm)
- Sufficient length of appropriate-diameter tubing for the pump(s) being used
- Electric water-level indicator with 0.01-foot increments
- Watch or stopwatch
- Indelible ink markers
- Field notebook and slug testing forms

3.0 WATER LEVEL MEASUREMENT PROCEDURE

The water level will be measured after wells have been installed and developed and water levels have stabilized. Static water levels will also be measured each time a well is tested and before any equipment enters the well. Any substantial change in conditions (e.g., barometric pressure change after a weather change) that may affect water levels during the tests will be recorded in the field log. If the casing cap of the pump well or the observation well(s) is not vented, allow time for the pressure to equilibrate after the cap is removed prior to measurement, and collect several measurements to confirm the water level is stable.

The groundwater level will be measured to the nearest 0.01 foot using an electric water-level indicator. Water levels will be measured from the notch or permanent mark located at the top of the well casing and recorded on the pumping test form. If well casings are not notched or marked, measurements will be taken from the north side of the top of the well casing.

Once the water level has been measured, the total depth of the well will be measured using an electric water-level indicator before specific-capacity and pumping test activities. The water level depth will then be subtracted from the total depth of the well to determine the height of the water column present in the well casing.

4.0 PREPARATION PRIOR TO SPECIFIC-CAPACITY AND PUMPING TEST

Although pumping will only be conducted in one well at a time, place a pressure transducer in each observation well near the well being tested before testing begins. Pressure transducers shall be placed 1 foot above the bottom of the well. The pressure transducers shall be properly secured at the wellhead. Configure the deployed pressure transducers to record in units of "feet of head" at a 5-second linear sampling interval and start recording shortly before the specific-capacity test or pumping test. The desiccants packs must be properly attached to the wellhead end of the pressure transducer cables to prevent moisture from entering the cables after configuring the transducers.

Set up the pump near the pumping well by ensuring the following:

- The pump is connected to a continuous power supply.
- The pump intake within the well is at least 1 foot above the pressure transducer if possible and well below the water table inside the well.
- The pressure transducer is not disturbed while lowering the pump or intake tubing.
- The appropriate pumping rate measurement method is in place at the surface of the well (i.e., in-line flow meter for high flow rates, graduated bucket for intermediate flow rates, or a measuring cup for low flow rates).
- An appropriately sized water container or tank is located near the well to collect the discharged water. Using an unsealed container to collect purge water is necessary

during specific-capacity and pumping tests to avoid back pressures, which can limit the performance and consistency of the pump and its flow rate, respectively.

5.0 SPECIFIC-CAPACITY TESTING

The specific-capacity test (or step test) is used to estimate the largest sustainable pumping rate that be achieved in the pumping well and is therefore conducted prior to the pumping test. During the specific-capacity test, the pumping rate is progressively increased at specified intervals. The following steps describe the activities conducted during the specific-capacity test.

1. Begin the specific-capacity test by setting the first pumping rate at 0.1 gpm (375 milliliters per minute [ml/min]). If drawdown records for groundwater sampling for the well are available, they can be used to estimate an initial pumping rate and flow rate increments. Record the time, flow rate, and depth to water at the following intervals:
    - Every 30 seconds for the first 3 minutes after turning on the pump or adjusting the flow rate
    - Every 1 minute for 3 minutes
    - Every 2 minutes for 4 minutes
    - Every 5 minutes until the next step
2. Maintain the well pumping rate at a constant flow for at least 30 minutes before increasing the flow rate or ending the specific-capacity test (Kruseman 2000).
3. After pumping the well for at least 30 minutes and recording the time, depth to water, and flow rates, increase the flow rate and pump the wells for another 30 minutes at a constant flow rate while recording the depth to water, time, and flow rates. Continue increasing the flow rate every 30 minutes until the water level drops to within 1 foot of the pump intake (i.e., the well approaches going dry) or until the maximum flow rate of the pump is reached. The following are suggested flow rate steps for the specific-capacity test (these increments are only an example and may be adjusted in the field based on observed conditions and pump capability):
    - 0.1 gpm
    - 0.25 gpm
    - 0.5 gpm
    - 0.75 gpm
    - 1.0 gpm
    - 1.5 gpm
    - 2.0 gpm
    - 2.5 gpm

- 3.0 gpm

4. Allow groundwater levels in the pumping well and observation wells to fully recover to static water level conditions after the specific-capacity test (or at least 24 hours after the specific-capacity test) before performing the constant-discharge pumping test.
5. At the end of the specific-capacity test, download the groundwater level data from all the pressure transducers for analysis and leave the pressure transducers on and in place. Ensure the desiccant packs are properly attached to the wellhead end of the pressure transducer cables to prevent moisture from entering the cables.

6.0 PUMPING TEST

The pumping test is conducted using a constant pumping rate, as follows:

1. Set up the equipment to conduct the pumping test at the same well where the specific capacity test was conducted.
2. Select a constant pumping rate to use during the pumping test based on the results from the specific-capacity test.
3. Conduct the pumping test for at least 8 hours. While conducting the pumping test, record the depth to water using the electronic water level meter and flow rate at the pumping well at the following frequencies:
   - Every 15 seconds for the first 2 minutes after turning on the pump
   - Every 30 seconds for 2 minutes
   - Every 1 minute for 2 minutes
   - Every 2 minutes for 12 minutes
   - Every 5 minutes for 10 minutes
   - Every 10 minutes for 30 minutes
   - Every 30 minutes until the end of the pumping test
4. Record the depths to water at the observation wells (existing monitoring wells other than the pump well) every 30 minutes and record the time of the measurements.
5. Record the time the pumping test is stopped on the field form and field notebook.
6. Record the rising depth to water in the pumping well at the following frequencies for only 20 minutes:
   - Every 15 seconds for the first 2 minutes after turning on the pump
   - Every 30 seconds for 2 minutes

- Every 1 minute for 2 minutes
- Every 2 minutes for 12 minutes
- Every 5 minutes for 10 minutes

7. If the initial pumping well discharge rate causes the pumping well to go dry, then the pumping test will cease. Data collected to the time that the pumping well went dry will be analyzed to determine if it is sufficient to estimate the transmissivity and storage coefficient. If the data are not sufficient, then the aquifer will be allowed to recover to the initial depth to water measurements, and the test will be repeated at a lower pumping rate.
8. Wait to download the transducer data for at least 8 hours after the pump is turned off to collect sufficient water level recovery data.

7.0 REFERENCES

Kruseman, G.P., N. de Ridder, and J.M. Verweij. 2000. Analysis and Evaluation of Pumping Test Data, Second Edition. Wageningen, Netherlands: International Institute for Land Reclamation and Improvement. https://www.hydrology.nl/images/docs/dutch/key/Kruseman_and_De_Ridder_2000.pdf.

Ohio EPA. 2006. Technical Guidance Manual for Ground Water Investigations (Chapter 4). Ohio Environmental Protection Agency. December. Available at https://www.epa.state.oh.us/portals/28/documents/TGM-04_final1206W.pdf.

USEPA. 1999. Compendium of ERT Groundwater Sampling Procedures. United States Environmental Protection Agency. January. Available at https://www.epa.gov/sites/default/files/2015-06/documents/fieldsamp-ertsops.pdf.

Attachment A-12
Incremental Sampling Methodology Procedures

1.0 INTRODUCTION

The general procedures for using the Incremental Sampling Methodology (ISM) at the Anniston Polychlorinated Biphenyl (PCB) Site are outlined below and were developed based on the Interstate Technology and Regulatory Council's (ITRC's) *ISM-2 Technical/Regulatory Guidance: Incremental Sampling Methodology (ISM) Update* (ITRC 2020).

ISM has been developed to decrease the effect of sample variability and provides a reasonably unbiased estimate of mean contaminant concentrations in a volume of soil targeted for sampling. ISM provides representative samples of specific soil volumes defined as decision units (DUs). An incremental sample is created by collecting many (usually 30 to 100) equal-volume increments in an unbiased manner from throughout the entire DU. The combined increments (frequently totaling a kilogram or more) are typically processed at the laboratory and subsampled to provide an analytical aliquot of only a few grams that is used for analysis. The final analytical aliquot is the target sample. The number of increments to obtain the desired ISM sample mass must be calculated with this information included in the approved field sampling plan (FSP) (ITRC 2020).

2.0 MATERIALS

Sampling equipment should be used according to the manufacturer's operating manual. An appropriate maintenance check will be performed on each piece of equipment prior to use. If damaged or failed parts are identified during the maintenance check and it is determined that the damage can impact the equipment performance, the equipment will be removed from service until the part or parts are replaced or repaired. Typical equipment required for ISM soil sampling includes the following:

- Stainless steel multi-increment sampling tool or equivalent (e.g., Enterprise Ventures Corporation [EVC] SOIL STICK™ or sampling tool described in the United States Environmental Protection Agency [USEPA] Method 8330B)
- 0.750-inch-diameter x 2.50-inch-long coring bore cutter and plunger (diameters range from 0.75 to 1.625 inches)
- Shovel, pickax or equivalent hand tool
- Wooden stakes and/or steel flags
- Rope or twine
- Measuring tape or measuring wheel
- Nitrile gloves
- Sharpie

- Appropriate sample containers sufficient to contain the sample volume for shipment to the laboratory
- Chain-of-custody forms
- Field notebook
- Direct-push drill rig with drill rods and soil core liners (if sampling subsurface soil)

3.0 SOIL SAMPLING PROCEDURE

The following procedure is for sampling surface soil using a two-person field team:

1. Review the FSP and health and safety plan (HASP) and don the appropriate personal protective equipment.
2. Stake the four corners of the DU; use a GPS to locate the corners, if necessary, per the FSP.
3. Physically measure the grid distances for sampling cells (e.g., 2 rows/15 columns measuring 10 feet long and 10 feet wide for cells within a 20-foot x 150-foot DU).
4. Lay out twine or rope to create the grid pattern or mark the rows and columns.
5. From the first grid cell, randomly select the increment location for the initial increment sample.
6. Remove plant material or surface debris (trash, twigs, rocks, etc.) from the location being sampled with hand tools.
7. Collect the increment using the sampling tool and extract the soil into the appropriate storage container.
8. Collect increment samples from adjacent cells sequentially using the same relative location as the first grid cell until all grid cells have been sampled.
9. Label sample for shipment and store samples appropriately for the parameter to be analyzed (e.g., place samples on ice in a cooler if necessary).
10. If collecting replicate samples, repeat steps 5 through 9 for each increment replicate sample (triplicate ISM sampling is recommended per the ITRC 2020 guidance document [ITRC 2020]).
11. Record sampling activities in the field notebook.
12. Once all increments have been collected for the DU, decontaminate equipment and change gloves before moving onto the next DU.

The following procedure is for sampling subsurface soil using a two-person sampling team and a drilling crew:

1. Repeat steps 1 through 6 above.

2. Advance the direct-push rig's drill rod with soil core liner to the depth specified in the FSP.
3. To avoid excessive ISM sample volumes, collect a wedge subsample of the soil core from the depth interval specified in the FSP (e.g., between 4.75 feet and 5.0 feet below ground surface).
4. Repeat steps 8 through 11 above.
5. Once all increments have been collected for the DU, decontaminate equipment and change gloves before moving onto the next DU. The drill rig will be cleaned and decontaminated according to the guidelines described in Attachment A-2 to the site-wide quality assurance project plan for the Anniston PCB Site and will conform with the procedures detailed in Appendix E.4 of the *Alabama Environmental Investigation and Remediation Guidance* (ADEM 2017).

4.0 REFERENCES

ADEM. 2017. *Alabama Environmental Investigation and Remediation Guidance (AEIRG)*. Revision 4.0. February.

ITRC. 2020. *Technical/Regulatory Guidance: Incremental Sampling Methodology (ISM) Update*. The Interstate Technology and Regulatory Council. October.

Attachment A-13
Surface Water Sampling, Filtering, and Measuring Procedures

1.0 INTRODUCTION

The procedures to be used while collecting water column samples are outlined below. The general procedures for measuring water quality parameters (e.g., turbidity, specific conductivity, pH, and temperature) in situ with a combination water-quality meter are also described.

2.0 MATERIALS

The following materials will be available, as required, during water column sampling:

- Personal protective equipment (PPE) and safety equipment as required the task specific Job Safety Analysis (JSA)
- Cleaning equipment
- Boat and motor
- Surveyor's rod
- Duct tape
- Graduated beaker
- Electronic depth sounder
- Rope
- Flagging
- Large glass mixing container
- Teflon® stirring rod
- Combination water-quality meter (Horiba U-10 or equivalent) or individual meters for each parameter of interest
- Standard solutions for calibration
- Analyte-free water
- Extra batteries for the meter
- Appropriate transport containers and packing, labeling, and shipping materials (coolers) with ice
- Appropriate filters and filtering apparatus (if necessary)
- Forceps (stainless steel)
- Compressed nitrogen gas tank
- Appropriate sample containers and forms
- Field notebook

- Appropriate water sampler and supporting equipment from among the following:
  - Automated ISCO composite water sampler
  - Wildco Kemmerer stainless steel bottle sampler or equivalent
  - Peristaltic pump with short pieces of medical-grade silicone tubing and Teflon tubing
  - SP3^{TM} passive sampling device and support or equivalent

3.0 WATER COLUMN SAMPLING PROCEDURES USING AN AUTOMATED COMPOSITE SAMPLER

Water column samples will be collected using an ISCO sampler or equivalent. The ISCO will be set to collect water when the stream gauge reaches the desired water level or stage. The sampler will be set to automatically collect surface water during each event. If the sampler is triggered to collect samples and the water level drops before sufficient sample volume is collected, the sample containers will be emptied, decontaminated, and reset to collect sufficient sample during the next triggering event.

The automated sampler may be used for volatile organic compounds (VOCs) provided the sampler is used to directly fill the appropriate container. Alternatively, VOC samples may be collected manually using the procedures described in Section 4, as soon as practical after the automated sampler is triggered. Samples for VOCs will not be collected after manually mixing or homogenizing in a larger container.

Sampling equipment will be dedicated for each sampling event or will be decontaminated between each use. The ISCO will be calibrated and set to collect composite samples directly into the appropriate containers or into a single five-gallon (carboy) sample during a high-water event. If a carboy is used as an intermediate homogenization step, a Teflon stirring rod and stirring plate will be used to composite the sample inside the carboy and to keep the sediment suspended in the container. Sample aliquots will be transferred from the carboy to an appropriate container or to a filter and container using a peristaltic pump.

4.0 WATER COLUMN SAMPLING USING MANUAL PROCEDURES

4.1.1 Kemmerer Stainless-Steel Bottle Sampler

Water column samples may be collected from a boat using a stainless-steel Kemmerer water sampler or equivalent. If depth-integrated samples are required, a composite will be prepared by collecting along a transect at various depths. The number of horizontal locations and depths will depend on the width and depth of the river at the point of interest and should be specified in the sampling plan prior to field work. Composite samples should not be collected for VOC analysis. Where possible, single grab samples should be taken for VOCs by directly submerging the sampling container or by collecting single grab samples using the procedures described in this section or Section 4.1.2.

1. Identify sampling location in field notebook along with other appropriate information.
2. Don PPE and safety equipment as specified in the task-specific JSA
3. Decontaminate the sampling equipment before each use.
4. Position the boat at the sampling station in the river.
5. Measure the total depth of the water column with an electronic depth sounder or weighted line.
6. Lower the water sampler to the deepest depth interval and at least 1 foot above the sediment bed and release trigger. Take care not to disturb the bottom sediments during sampling.
7. Raise the water sampler from the water column with minimal disturbance.
8. Transfer the individual grab samples to a larger container to comprise the composite sample for the given location.
9. Additional casts to the shallower depths will be required to obtain the depth-integrated sample.
10. Move to the next location along the transect until the composite sample is completed.
11. Mix the entire sample volume with the stirring rod and transfer the appropriate volume into the laboratory sample jars. Sample containers may be pre-preserved by the laboratory or preservative may be added in the field, if required.
12. Prepare field duplicates by aliquoting the composite into two sets of containers.
13. Label sample containers and secure the sample jar caps tightly.
14. Repeat steps 4–13 at the remaining stations.
15. After sampling containers have been filled, collect and record representative meter readings for pH, temperature, conductivity, and turbidity for the transect.
16. Place filled sample containers on ice in a cooler.
17. Follow procedures for preserving samples and packing, handling, and shipping with associated chain-of-custody (COC) procedures of samples.
18. Record required information on the appropriate forms and field notebook.

4.1.2 Peristaltic Pump Sampler

At locations where a peristaltic pump will be used, the procedures described in this section will be followed. If composite samples are required, samples from appropriate intervals and depths along a transect will be collected as described in Section 4.1.1 above. Composite samples should not be collected for VOCs, and a peristaltic pump is not preferred for VOCs. If samples are

required for VOCs and use of a peristaltic pump is necessary, samples will be collected by filling the VOC container directly from the peristaltic pump as described in this section.

1. Identify sampling location in field notebook along with other appropriate information.
2. Don PPE and safety equipment as specified in the task-specific JSA.
3. Clean the sampling equipment before each use.
4. Install clean, medical-grade silicone tubing in the pump head, as per the manufacturer's instructions. Allow sufficient tubing on the discharge side to facilitate convenient dispensation of liquid into the sample container and only enough on the suction end for attachment to the intake line. This practice will minimize sample contact with the silicone pump tubing.
5. Select the length of Teflon® tubing necessary to reach the required sample depth and attach to intake side of pump tubing. Taping the Teflon tubing to a surveyor's rod will facilitate reaching the required depth.
6. Allow several liters of sample to pass through the system (if possible) before actual sample collection. Collect this purge volume and return the water to the creek after the sample aliquot has been withdrawn.
7. Use a graduated cylinder or beaker as a measuring device and collect equal volumes of water at the required transect locations and depths.
8. Combine the individual grab samples into one composite sample for the given location (except for VOC sampling, where samples will be collected directly in laboratory sample jars and not composited).
9. Mix the entire sample volume with the stir plate and rod and transfer the appropriate volume into the laboratory sample jars. Containers may be pre-preserved or preservative added in the field if required. Secure the sample jar caps tightly.
10. After sample is collected, follow steps 15–18 described in Section 4.1.1.
11. Discard tubing between sampling locations and not reused.
12. Label sample containers and place filled sample containers on ice in a cooler. Follow procedures for preserving samples, as well as packing, handling, and shipping with associated COC procedures of samples.

5.0 WATER COLUMN SAMPLING USING PASSIVE SAMPLING METHODS

Please refer to SiREM's *SP3™ Sampler Deployment and Retrieval* standard operating procedure that is appended to this document. The document contains the procedures to complete deployment and retrieval of SP3™ passive sampling devices.

6.0 MEASURING WATER QUALITY PARAMETERS IN SITU

Measurements will be taken using a combination water meter or individual meters for the parameters of interest (e.g., turbidity, pH, temperature) as specified in the field sampling plan. The general standard procedures of calibration, operation, and maintenance are described below.

Calibration: The calibration will be checked daily following the manufacturer's instructions. If a reading falls outside of 95% of the expected value, the meter will be recalibrated. Calibration information will be recorded in the field notebook or on a specific field form. Where possible, the meter will be calibrated with at least two concentrations that bracket the range of interest.

Operation: The meter will be operated following the manufacturer's instructions. Sequential readings will be taken to determine that the instrument is stabilized. The final reading will be recorded in the field notebook or on a specific form.

Maintenance: The meter will be maintained according to the manufacturer's instructions. Maintenance information will be recorded with calibration information. Replacement or alternate meter(s) and probes will be available on-site or ready for overnight shipment, as necessary.

SP3™ Sampler Deployment and Retrieval

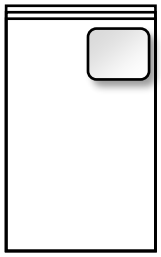
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SP3™ Sampler Deployment and Retrieval

Deployment

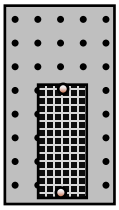
1. The SP3™ samplers are shipped in a cooler at 4°C or lower. Each SP3™ sampler is stored in an opaque Ziploc bag and the opaque bag is stored inside a large clear plastic Ziploc bag.



Clear Ziploc Bag

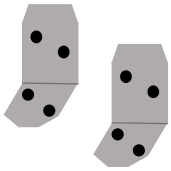


Opaque Ziploc Bag

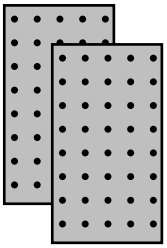


SP3™ Sampler

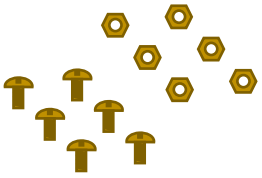
2. Also included in the cooler are bags of pre-cleaned wing brackets, landing wings and screws and nuts used to attach the landing wings to the SP3™.



Wing Brackets

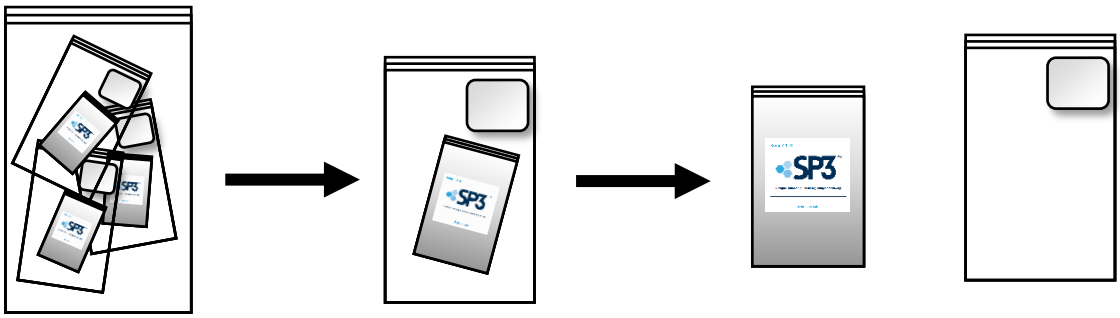


Landing Wings



Screws and Nuts

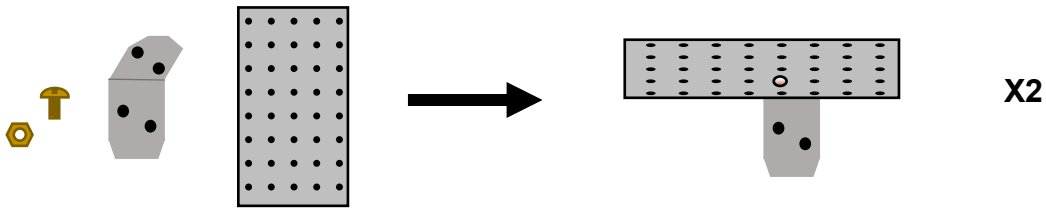
3. Keep the SP3™ samplers cool (4°C or lower) and in the dark prior to use.
4. No more than 30 minutes prior to deployment, remove a single bagged SP3™ sampler from the large bag of samplers or cooler. Then remove the labeled opaque Ziploc sampler bag from the clear plastic Ziploc bag (keep Ziploc bag for next step).



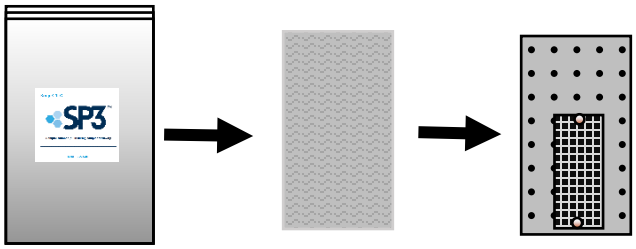
SP3

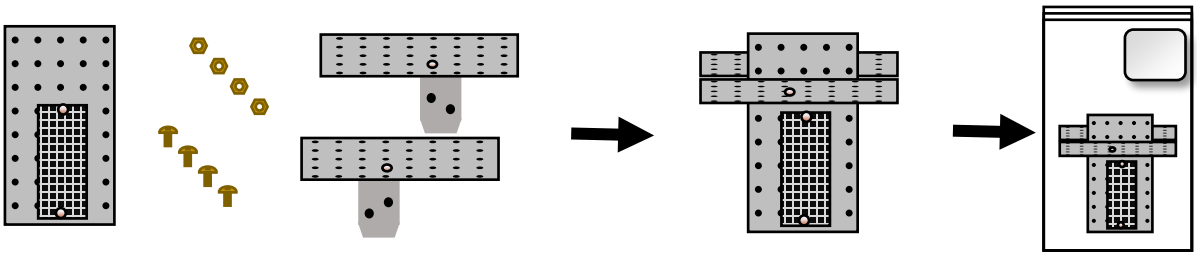
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5. Prepare the landing wing apparatus by screwing the wing bracket to the landing wings:

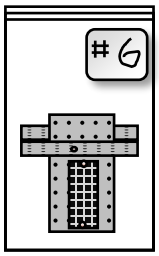


6. Remove the SP3™ sampler from the opaque Ziploc sample bag (keep bag for re-use upon retrieval). Unwrap the aluminum foil around the sample and attach the prepared wing assemblies to the top of the plate (the end of the plate that will remain above the sediment surface when deployed [see below]) using the two screws per wing apparatus. Place the entire SP3™ sampler in the original clear Ziploc bag, and seal bag.





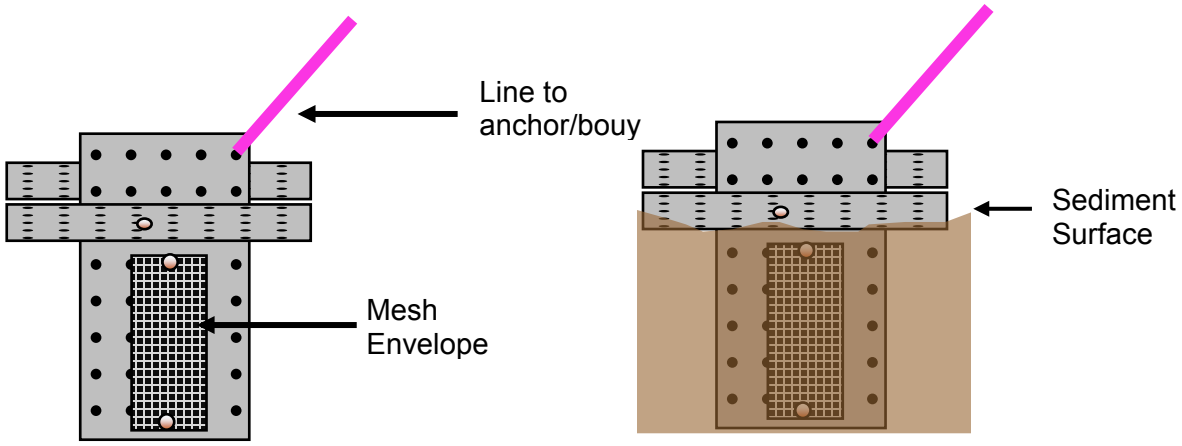
7. It is recommended to mark the sample station at which the sampler will be deployed on the outside label (using a permanent marker or grease pen) so that it can be easily read during deployment.



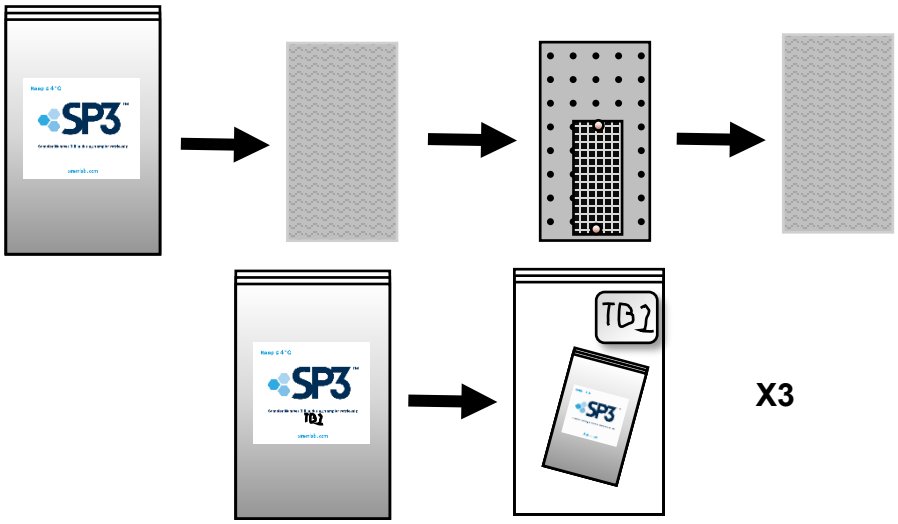
8. Keep the bag out of direct sunlight and heat, to the extent practicable, during the above preparation steps.

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9. Provide the bag for deployment to (i.e., push pole technician, SCUBA diver, wader or direct from small boat). Note, multiple bags can be prepared for deployment if multiple samplers will be deployed in a single mobilization. It is acceptable if water enters the bag. Minimize the time the sampler is underwater in the bag, to the extent practicable, up to a maximum time of approximately 30 minutes.
10. At the deployment location, remove the sampler from the plastic bag, attach the sampler to your anchor or bouy line, etc. Insert the sampler plate into the sediment to the desired depth and retain plastic bag, if possible, for re-use after retrieval.



11. If needed, plant a marker flag. Record the GPS co-ordinates of the deployed sampler and date of deployment.
12. Deploy all but three samplers (as needed). The remaining 3 samplers are for Field Blank (FB) use. For each FB sampler, remove the sampler from its labeled opaque Ziploc sampler bag and unwrap it from the aluminum foil. Expose the sampler to air/light/ambient conditions at sampling site for 5-10 minutes (or however long it took to prepare a sampler for deployment). Re-wrap the sampler in aluminum foil and place back in the labeled opaque Ziploc sampler bag, mark label with a unique sample code (e.g., “FB1”), and place in a clear ziploc bag.



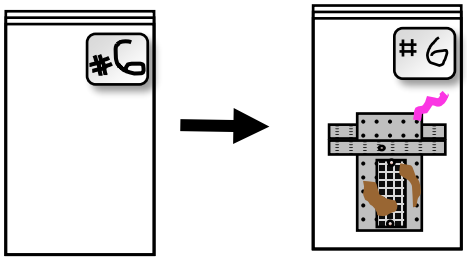
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13. For FB samplers, maintain at 4°C or less until they can be shipped to the analytical lab (ideally within 1 to 4 days of deployment).
14. Shipping of FB samplers:
    a. Include ice (double-ziploc bagged) or bagged blue ice packs.
    b. Samplers.
    c. Extra packaging (bubble wrap) as needed.
    d. Chain of custody
        i. Note EPA 1668A for PCB congeners, and/or modified EPA 8270 for PAHs (specify parent PAHs, alkylated PAHs, or both)
        ii. Test America contact is Ryan Henry
    e. Address to ship:
        Test America
        5815 Middlebrook Pike
        Knoxville, TN 37921
        Attn: Sample Receiving, Ryan Henry
        Phone: 865-291-3000
    f. Ship overnight for next morning delivery.

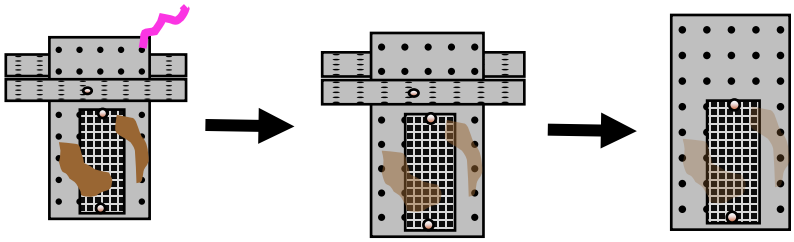
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Retrieval

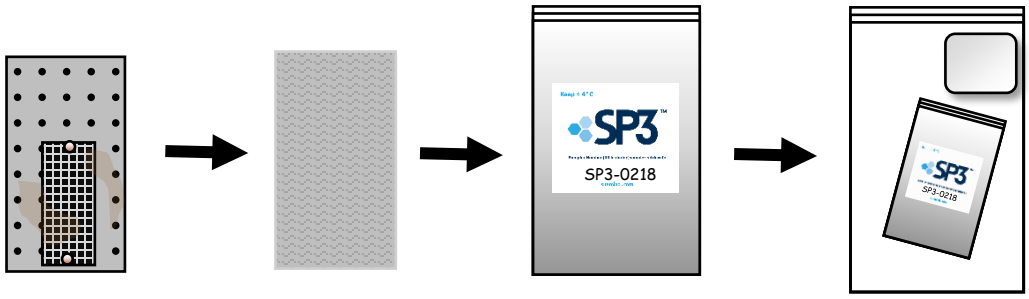
15. Use the original clear Ziploc bag for sample location, retrieve sampler from sediment, place in bag (seal if possible), and return bag to cooler within approximately 20 minutes.



16. Detach any remaining rope from the anchor, wipe off any excess sediment, and remove the wing assembly. Some sediment remaining on the sampler is acceptable.



17. Wrap the sampler in aluminum foil, place the wrapped SP3™ sampler in labeled opaque Ziploc sampler bag, mark label with the unique sample ID (e.g., “SP3-0218”), and place in a Ziploc bag.



18. Place in a cooler with ice/cold packs and maintain cold until shipping; samples can be held for several days as needed as long as they are kept cool (4°C or lower) and dark.
19. Ship the cooler as per the instructions in number 14 described above.

SP3™ Data Analysis

Surface Water Sampling, Filtering, and Measuring Procedures
February 2022

ATTACHMENT A:

DATA ANALYSIS METHODS

Attachment A:

Data Analysis Methods
Concentrations of Freely Dissolved Analytes
Measured via SP3™ Passive Samplers

The concentration of analyte PCBs (Table A1-a) and pesticides (Table A1-b) in PE obtained from the information provided in the analytical report (Attachment B) are used in a multi-step data process to calculate C_{free} analytes as described below.

Step 1:

The concentrations of the PRCs in PE [PE_t] were used to calculate the elimination rate (k_e) values for the PRCs in each deployed sampler using the following equation (Lohmann, 2012):

$$PRC\ k_e = \ln\left(\frac{[PE_{t=0}]}{[PE_{t=final}]}\right) \div t_{final}$$

where:

$PE_{t=0}$	= the average concentration of the PRC present in the PE at the beginning of the deployment (obtained from an average measurement of the PRC control blanks)
$PE_{t=final}$	= the concentration of the PRC in the PE after the deployment (obtained from each deployed PE sampler)
t_{final}	= the deployment time (in days)
k_e	= the elimination rate (in days^{-1})

PRC k_e values for the PRCs in each sampler are shown in Table A2. The values are also expressed as a percentage of steady state (concentration at equilibrium). Several PRC k_e values were not calculated and were treated as outliers because $PE_{t=final}$ values were equal to or greater than $PE_{t=0}$ values.

Step 2:

The second step was to estimate k_e values for the non-PRC primary analytes (non-PRC PCB) in each of the deployed samplers. This was accomplished by developing a linear regression model using PRC k_e values (dependent variable, from Table A2) and PE-water partition coefficients (K_{PE}) for each PRC PCB (independent variable, Smedes et al., 2009). Note that regression models were specific to each sampler (i.e. not global to the whole deployment) as local geologic and hydrodynamic conditions can vary greatly within a site.

Values were $\log_{10}$-transformed per Tomaszewski and Luthy (2008). By entering the analyte-specific K_{PE} into the linear regression model developed for each sampler, k_e values for each of the primary analytes for each sampler were calculated.

Step 3:

Concentrations of some non-PRC PCBs (Table A1-a and A1-b) in PE were corrected for trace levels of non-PRC PCBs present in the PRC control blanks (due to trace levels present in the PRC spiking solutions). Using the sample specific k_e values, the expected amount of these trace primary analyte PCBs present in the sample at the end of deployment (*Trace PCB* $_{t = final}$) was calculated via the following equation:

$$[Trace\ PCB_{t=final}] = \frac{[Trace\ PCB_{t=0}]}{e^{k_e \times t_{final}}}$$

where:

Trace PCB$_{t = final}$ = the concentration of trace PCBs remaining in the sample at the end of the deployment

Trace PCB$_{t = 0}$ = the average concentration of the trace PCB in the PE at the beginning of the deployment (obtained from an average measurement of the trace PCBs in the PRC control blanks)

k_e = the elimination rate value predicted by the sampler-specific regression model (in days^{-1})

t_{final} = the deployment time (in days)

Concentrations of *Trace PCB* $_{t = final}$ values were then subtracted from the measured concentrations of non-PRC PCBs and pesticides in PE (Table A1-a and A1-b).

Step 4:

This step describes the calculation of sampling rate correction factors (*CF*s) for each primary analyte in each sampler. The following equation is used, as adapted from Lohmann (2012):

$$CF = \frac{1}{1 - e^{-k_e \times t_{final}}}$$

where:

k_e = the elimination rate value predicted by the sampler-specific regression model (in days^{-1})

t_{final} = the deployment time (in days).

Step 5:

The concentration of primary analyte in the PE of each sampler (obtained from Table A1-a and A1-b) were multiplied by the *CF* values to calculate the steady-state concentration of primary analytes.

Step 6:

In the final step, the steady-state concentrations are divided by K_{PE} values (Smedes et al. 2009) to obtain the concentrations of C_{free} for the primary analytes. These are reported in Table 1. C_{free} Method Detection Limits (MDLs) were calculated in the approach described above using the estimated MDL concentration in PE, as reported the analytical laboratory and shown in Table A1.

For samples in which the percentage of steady state was indicated to be less than 10% for a primary analyte, C_{free} was calculated and given an "L" qualifier in Table 1. Estimates associated with L-qualified values should be evaluated with caution due to the higher level of uncertainty associated with high CF values (i.e., higher than 10).

References Cited

Lohmann, R. 2012. Critical review of low-density polyethylene's partitioning and diffusion coefficients for trace organic contaminants and implications for its use as a passive sampler. Environ. Sci. Technol.: 46:606-618.

Smedes, P., Geerstma, R. W., van der Zande, T., and Booij, K. 2009. Polymer-water partition coefficients of hydrophobic compounds for passive sampling: Application of cosolvent models for validation. Environ. Sci. Technol. 43:7047-7054.

Tomaszewski, J.E., and Luthy, R.G. 2008. Field deployment of polyethylene devices to measure PCB concentrations in pore water of contaminated sediment. Environ. Sci. Technol. 42:6086-6091.

United States Environmental Protection Agency (USEPA). 2003. Table of PCB Species by Congener Number.

ATTACHMENT B

Laboratory Standard Operating Procedures

Title: SEPARATORY FUNNEL EXTRACTION (SW-846 3510C)

Approvals (Signature):

Don Dawicki
Laboratory Director

Kristine Dusablon
Quality Assurance Manager

Mariah Witas
Department Supervisor

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1.0 Scope and Application

This SOP describes the laboratory procedure for the isolation of organic compounds from aqueous samples by separatory funnel liquid-liquid extraction.

1.1 Analytes, Matrix(s), and Reporting Limits

Refer to analytical methods for analyte lists and reporting limits.

On occasion clients may request modifications to this SOP. These modifications are handled following the procedures outlined in the Quality Assurance Manual.

2.0 Summary of Method

A measured volume of sample, usually 1 L, is serially extracted at a specified pH with methylene chloride using a separatory funnel extractor (SFE). The extract is dried with anhydrous sodium sulfate then concentrated, and if necessary, exchanged into a solvent compatible for extract cleanup or the determinative analysis method.

This SOP is based on the following reference method:

- SW-846 Method 3510C Separatory Funnel Liquid-Liquid Extraction, Revision 3, December, 1996.

3.0 Definitions

Refer to the Laboratory's Quality Assurance Manual (QAM) for the Glossary of Terms, Definitions and Acronyms except as follows.

A list of terms and definitions are provided in Appendix A.

4.0 Interferences

Method interference may be caused by contaminants in solvents, reagents, glassware and other sample processing equipment that can cause interference and/or elevated baselines in chromatography. All reagents and solvents used during this procedure should be reagent grade or high purity in order to minimize interference. All glassware must be cleaned in accordance with laboratory SOP BR-QA-023 *Glassware Cleaning Procedure*.

The decomposition of some analytes has been demonstrated under basic extraction conditions. Organochlorine pesticides may dechlorinate, phthalate esters may exchange, and phenols may react to form tannates. These reactions increase with increasing pH, and are decreased by the shorter reaction times available in Method 3510. Method 3510 is preferred over Method 3520 for the analysis of these classes of compounds. However, the recovery of phenols may be optimized by using Method 3520, and performing the initial extraction at the acid pH.

5.0 Safety

Employees must abide by the policies and procedures in the NDSC Eurofins TestAmerica Laboratories Environmental Health and Safety Manual (CW-E-M-001), Radiation Safety Manual and this document. This procedure may involve hazardous material, operations and equipment.

This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

5.1 Specific Safety Concerns or Requirements

Nitrile gloves should be used when performing this extraction. Latex and vinyl gloves provide no significant protection against the organic solvents used in this SOP and should not be used.

During Kuderna-Danish (K-D) concentration do not allow the extract to boil to dryness. The solvent vapors remaining in the K-D apparatus may superheat and create an explosion or fire hazard.

The use of separatory funnels to extract aqueous samples with methylene chloride creates excessive pressure very rapidly. Initial venting should be done immediately after the sample container has been sealed and inverted. Vent the funnel into the hood away from people and other samples.

The following analytes have been tentatively classified as known or suspected, human or mammalian carcinogens: Benzo(a)anthracene, Benzidine, 3,3'-Dichlorobenzindine, Benzo(a)pyrene, alpha-BHC, beta-BHC, gamma-BHC, delta-BHC, Dibenz(a,h)anthracene, n-Nitrosodimethylamine, 4,4'-DDT, and polychlorinated biphenyl compounds. Primary standards of these toxic compounds should be prepared in hood.

The K-D apparatus has ground glass joints which can become stuck. Technicians must use Kevlar or other cut/puncture resistant gloves when separating stuck joints.

Special care is required to prepare a solution of 6N NaOH because considerable heat is liberated by the exothermic reaction. The solution may splatter or boil during preparation. Stir the sodium hydroxide, a little at a time, into a volume of water. Use borosilicate glass; inspect the glassware prior to use to make sure it is free from any cracks, scratches, or chips that would indicate weakness in the glass. Wear safety goggles and gloves when preparing the mixture.

5.2 Primary Materials Used

Table 1 lists those materials used in this method, which have a serious or significant hazard rating. **Note: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the SDS for each of the materials listed in the table.** A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the SDS for each material before using it for the first time or when there are major changes to the SDS.

6.0 Equipment and Supplies

Catalog numbers listed in this SOP are subject to change at the discretion of the vendor. Analysts are cautioned to be sure equipment used meets the specification of this SOP.

6.1 Extraction Equipment

- Separatory Funnel, 2 Liter with PTFE stopcock and cap. Fisher Scientific or equivalent.

- Glass Funnel – 100 mm. Fisher Scientific or equivalent.
- Class A Volumetric: 1 L capacity. Fisher Scientific or equivalent.
- Fiberglass Wool: 8 um, Fisher Scientific or equivalent.
- pH paper: Narrow and Wide range
- Mechanical Shaker (Optional)

6.2 Miscellaneous

- pH Paper/Strips: Range 1-14.
- Pasteur Glass Pipettes: 1 mL, disposable. Fisher Scientific or equivalent.
- 0.5 mL – 2.0 mL Syringes: Hamilton Gastight® Syringes or equivalent.
- Vials and Caps: 1.8, 4, 8, 16, and 40 mL with Teflon lined septa and screw caps. Fisher Scientific.

7.0 Reagents and Standards

7.1 Reagents

- Sodium Sulfate (granular, anhydrous), Na_2SO_4. J.T. Baker or equivalent. Purify by heating at 400°C for at least 4 hours.
- Methylene Chloride (CH_2Cl_2): Pesticide Quality, J.T. Baker or equivalent.
- Hexane, (C_6H_{14}), Pesticide Quality, J.T. Baker or equivalent.
- Acetone, ($(CH_3)_2CO$), Pesticide Quality, J.T. Baker or equivalent.
- Reagent Water: RO water filtered through a Nanopure System or equivalent.
- Sodium Hydroxide (NaOH): Reagent Grade, J.T. Baker.
- Sulfuric Acid, (H_2SO_4): Reagent Grade, J.T. Baker.
- Nitrogen, Ultra High Purity, Linde or equivalent.

7.1.1 Prepared Reagents

- NaOH Solution (6N): In a 2.5 L clear glass bottle dissolve 240 g NaOH in reagent water and dilute to 1 Liter. Store the solution in reagent bottle at room temperature. Assign an expiration date of 6 months from date of preparation unless the parent material expires earlier, in which case, use the earliest expiration date.

- H_2SO_4 Solution (1:1 v/v): Add 500 mL of reagent water to a 1 L volumetric flask. Slowly add 500 mL of H_2SO_4 to the flask to dilute to volume. Store the solution in reagent bottle at room temperature. Assign an expiration date of 6 months from date of preparation unless the parent material expires earlier, in which case, use the earliest expiration date.

7.2 Standards

Purchase stock standards as certified solutions from commercial vendors. Prepare surrogate and spiking solutions in the laboratory by diluting a known volume of the stock standard solutions in an appropriate solvent using the formulation specified in the analytical SOP. Record the preparation of standard in the LIMS (TALS) module established for this purpose.

Store prepared standard solutions in glass containers at 4°C or below. Unless otherwise specified, assign an expiration date from the date of preparation per the formulation or in

accordance with the expiration date of the parent standard, whichever is sooner. The recommended formulation for each standard used in this procedure is provided in the analytical method along with the recommended source materials, expiration dates and storage conditions.

8.0 Sample Collection, Preservation, Shipment and Storage

The laboratory does not perform sample collection so these procedures are not included in this SOP. Sampling requirements may be found in the published reference method.

Sample container, preservation techniques and holding times may vary and are dependent on sample matrix, method of choice, regulatory compliance, and/or specific contract or client requests. Listed below are the holding times and the references that include preservation requirements.

Matrix	Sample Container	Minimum Sample Size	Preservation	Holding Time[1]	Reference
Water	1 L Amber Glass	2 x 1L	Cool to ≤ 6° without freezing	Extraction: 7 days	SW-846 Chapter 4

[1]Extraction holding time is determined from sampling date. Footnotes to this table specify this information is guidance and does not represent EPA requirements.

Unless otherwise specified by client or regulatory program, after analysis, samples and extracts are retained for a minimum of 30 days after provision of the project report and then disposed of in accordance with applicable regulations.

9.0 Quality Control

9.1 Sample QC

The laboratory prepares the following quality control samples with each batch of samples.

QC Item	Frequency	Acceptance Criteria
Method Blank (MB)	1 in 20 or fewer samples	See Analytical SOP
Laboratory Control Sample (LCS)	1 in 20 or fewer samples	See Analytical SOP
Matrix Spike(s) MS/MSD *If there is not sufficient volume for MS/MSD perform an LCSD*	1 in 20 or fewer samples when sufficient sample volume is provided by the client.	See Analytical SOP
Sample Duplicate (SD)	Client Request	See Analytical SOP

9.2 Instrument QC

Refer to the analytical SOP for the determinative test method.

10.0 Procedure

10.1 Instrument Calibration

Verify the calibration of any mechanical pipettes used is current and if it is not, notify QA. The QA department checks the calibration of pipettes quarterly in accordance with the procedures specified in laboratory SOP BR-QA-012.

10.2 Equipment Preparation

Reusable Glassware: Wash and prepare using the procedures described in laboratory SOP BR-QA-023. Use a Sharpie marker to label each piece of glassware with the last 3 digits of the job ID followed by a dash and the sample ID. Extraction solvent will erase “sharpie” ink, so use caution during the extraction to avoid splashing solvent on the marking.

Create a drying funnel by adding a plug of Pyrex glass wool to a 100mm glass funnel. Add a sufficient amount of baked sodium sulfate to each funnel such that the funnel is approximately ¾ full. Rinse the funnel with methylene chloride collecting the solvent in a waste beaker. Dispose the waste solvent into the designated satellite container. Assemble a Kuderna-Danish (K-D) concentrator by attaching a 10mL concentrator tube to a 500mL evaporation flask. Add 1-2 boiling chips to the concentrator tube. Place this setup under the drying funnel.

10.3 Extraction

Remove the samples from refrigerated storage and let them warm to ambient temperature.

To prepare the method blank (MB) and laboratory control sample (LCS): Measure 1 L of reagent water into a 1L volumetric container, then transfer the measured volume into individually labeled separatory funnel extractor(s) (SFE).

Mark the meniscus of each sample on the sample container with a Sharpie marker.

Quantitatively transfer the entire sample into the SFE.

Using a calibrated pipette or a syringe, add the proper type and amount of spike and surrogate solutions to each QC and field sample following the spike regimen specified in the Extraction Conditions Workbook. Ask a peer analyst familiar with the procedure or the department supervisor to witness the addition of the spike solutions to each sample.

Note: The spiking scheme for all methods is provided in the Extraction Conditions Workbook located in the Controlled SOP distribution folder in the Burlington Public directory. The analyst must always refer to this workbook for the most current spiking information each time extraction is performed.

Check the pH of each sample using wide range pH paper. Adjust the pH, if necessary, using 1:1 Sulfuric acid solution or 6N Sodium hydroxide solution. See the extractions conditions workbook to determine the extraction pH for each test method. If using pH paper, do not dip the paper into the sample, instead aliquot a drop of sample onto the pH paper using a Pasteur piptette.

Rinse the empty sample container with ~ 60 mL of methylene chloride and pour the rinsate into the SFE. Place the stopper on the SFE and twist the stopper to secure it tight.

Caution: Methylene chloride rapidly creates excessive pressure that must be released. Hold the SFE with the bottom facing away from you into and into the fume hood, shake the SFE once then open the stopcock to vent the pressure into the fume hood. Repeat the shake/vent step two more times.

Note: The laboratory uses a mechanical shaker to shake SFEs but use of the mechanical shaker is not required by the method. Samples may also be manually shaken if the mechanical shaker is out of service.

Place the SFE(s) on the mechanical shaker, set the timer for 2 minutes and press start. After 2 minutes, remove the SFE to the holding cart designed for this purpose. Let the sample sit for 10 minutes to allow the solvent layer to separate.

Decant the solvent layer from each SFE into the corresponding drying funnel collecting the solvent in the KD concentrator.

- *If an emulsion forms and the emulsion interface between layers is more than 1/3 the size of the solvent layer, use a mechanical technique to separate the phases. The technique used to break the emulsion depends on the sample but may include stirring, filtration through glass wool, centrifugation or other techniques. Consult with DS to determine the optimum technique for each situation encountered.*

Repeat the extraction/decant process two more times using 60mL of fresh methylene chloride each time.

To measure the sample volume, fill the sample container with tap water to the mark of the meniscus and pour the water into a graduated cylinder for volume measurement. Record the sample volume in the TALS worksheet.

10.4 Extract Concentration

Refer to SOP No. BR-EX-031, Concentration Techniques, for the correct procedures regarding the concentration of extracts.

11.0 Corrective Action

When an out-of-control situation occurs that is not delineated in this corrective action table or the corrective actions listed do not adequately address the circumstances, a Corrective Action Report (CAR) (NCM), etc., must be developed (see SOP BR-QA-016) and the analyst must use his/her best analytical judgment and available resources to determine the corrective action to be taken. The out-of-control situation may be caused by more than one variable. The analyst should seek the assistance of his/her immediate supervisor, QA manager or other experienced staff if they are uncertain of the cause of the out-of-control situation. The analysis must not be resumed until the source of the problem and an in-control status is re-established. All samples associated with the out-of-control situation must be reanalyzed after in-control status has been re-established or if authorization is received from the supervisor or QA Manager for release with data qualification.

12.0 Calculations / Data Reduction

12.1 Calculations

Calculations are provided in the analytical SOP for each method parameter.

12.2 Data Review

Refer to laboratory SOP BR-QA-019 for the required elements of each step of data review.

12.2.1 Primary Review

Review the batch worksheet for correctness and completeness. Record any problems encountered during the extraction process with a nonconformance memo (NCM).

Note: If the TALS log-in does not include cleanup methods in the method chain and there is reason to believe the extract may require cleanup (color, odor, viscosity, etc.) notify the Department Supervisor (DS) of the situation so he/she can determine if cleanup should be performed prior to analysis.

12.2.2 Secondary Review

Review the batch worksheet for correctness and completeness and to ensure the extraction performed is consistent the SOP and project specifications.

If errors are found or if the record is incomplete, initiate a 2nd Level Review NCM to record the observation.

For additional guidance regarding the laboratory's protocol and required elements for data review refer to laboratory SOP BR-QA-019.

13.0 Method Performance

13.1 Method Detection Limit Study (MDL)

The method detection limit (MDL) is the lowest concentration that can be detected for a given analytical method and sample matrix with 99% confidence that the analyte is present. MDLs reflect a calculated (statistical) value determined under ideal laboratory conditions in a clean matrix, and may not be achievable in all environmental matrices. An initial method detection limit study is performed in accordance with SOP BR-QA-005. The laboratory maintains MDL studies for analyses performed; these are verified at least annually unless method or program requirements require a greater frequency.

13.2 Demonstration of Capabilities (DOC)

13.2.1 Four aliquots of the QC check sample are analyzed using the same procedures used to analyze samples, including sample preparation. The concentration of the QC check sample may be equivalent to a mid- level calibration.

13.2.2 Calculate the average recovery and standard deviation of the recovery for each analyte of interest.

13.2.3 If any analyte does not meet the acceptance criteria, the test must be repeated. Only those analytes that did not meet criteria in the first test need to be evaluated. TNI 2016 requires consecutive passing results. Repeated failure for any analyte indicates the need for the laboratory to evaluate the analytical procedure and take corrective action.

13.2.4 Until the IDOC is approved by the QA Manager (or designee); the trainer and trainee must be identified in the batch record.

13.3 Training Requirements

The Group Leader is responsible for ensuring that this procedure is performed by an associate who has been properly trained in its use and has the required experience. A new analyst must be working under documented supervision prior to approval of the IDOC. Documentation that a new analyst is performing under supervision must be entered into the batch record (View Batch Information) until that analyst's IDOC has been approved by the QA Manager (or designee). See requirements for demonstration of analyst proficiency in SOP BR-QA-011.

14.0 Pollution Control

It is Eurofins TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage and reagent stability). Employees must abide by the policies in Section 13 of the NDSC Eurofins TestAmerica Laboratories Environmental Health and Safety Manual (CW-E-M-001) for "Waste Management and Pollution Prevention."

15.0 Waste Management

Waste management practices are conducted consistent with all applicable rules and regulations. Excess reagents, samples and method process wastes are disposed of in an accepted manner. Waste description rules and land disposal restrictions are followed. Waste disposal procedures are incorporated by reference to BR-EH-001 *Hazardous Waste*. The following waste streams are produced when this method is carried out.

- Organic Solvents - Satellite container: 55 Gallon Covered and Vented Drum.
- Extracted water samples - Satellite Container: 55 Gallon Covered and Vented Drum.
- Vials containing extracts - Satellite Container: 5 Gallon Covered Bucket (inside fume hood).
- Solid Waste - Satellite Container: Solid Waste 5 Gallon Plastic Bucket (inside fume hood).

16.0 References / Cross-References

- SW-846 Method 3510C Separatory Funnel Liquid-Liquid Extraction, Revision 3, December 1996.
- Laboratory SOP BR-EX-017
- Laboratory SOP BR-QA-019
- Laboratory SOP BR-QA-005
- Laboratory SOP BR-EH-001
- Laboratory SOP BR-EX-031
- Corporate Environmental Health and Safety Manual

17.0 Method Modifications

There are no modifications of the referenced method.

18.0 Attachments

- Table 1: Primary Materials Used
- Appendix A: Terms and Definitions

19.0 Revision History

Revision 14:

- Title Page: Updated approval signatures, Copyright info, Branding, and Dates.
- Throughout: Updated SOP references to match currently used ones
- Section 11.0: Added section as required by ETA corporate policy.
- Section 13.0: Updated language and guidance as required by ETA corporate policy.

Revision 13:

- Title Page: Updated approval signatures and copyright date.
- Section 6.0: removed equipment and supplies for extract concentration.
- Section 9.1: Instructions for LCSD added in Sample QC table
- Section 10.1: changed reference to pipettor calibration SOP.
- Section 10.2: Updated instructions on the labeling procedure of samples and minor re-wording of instructions.
- Section 10.3: Updated instructions to state sample transfer to SFE prior to sample spiking and to rinse the empty sample container with MeCl2 as a final step before the first extraction.
- Section 10.4: Removed concentration steps and added instructions to reference concentration SOP.
- Section 15.0: Added reference to concentration SOP
- Section 18: removed older revision history and added statement that previous revisions are retained by QA.

Previous revisions are retained by the QA department.

Table 1: Primary Materials Used

Material[1]	Hazards	Exposure Limit[2]	Signs and symptoms of exposure
Acetone	Flammable	1000 ppm-TWA	Inhalation of vapors irritates the respiratory tract. May cause coughing, dizziness, dullness, and headache.
Hexane	Flammable Irritant	500 ppm-TWA	Inhalation of vapors irritates the respiratory tract. Overexposure may cause lightheadedness, nausea, headache, and blurred vision. Vapors may cause irritation to the skin and eyes.
Sulfuric Acid[1]	Corrosive Oxidizer Dehydra-dator	1 mg/m^3	This material will cause burns if comes into contact with the skin or eyes. Inhalation of vapors will cause irritation of the nasal and respiratory system.
Sodium Hydroxide	Corrosive Poison	2 ppm, 5 mg/m^3	This material will cause burns if comes into contact with the skin or eyes. Inhalation of Sodium Hydroxide dust will cause irritation of the nasal and respiratory system.
Methylene Chloride	Carcinogen Irritant	25 ppm-TWA 125 ppm-STEL	Causes irritation to respiratory tract. Has a strong narcotic effect with symptoms of mental confusion, light-headedness, fatigue, nausea, vomiting and headache. Causes irritation, redness and pain to the skin and eyes. Prolonged contact can cause burns. Liquid degreases the skin. May be absorbed through skin.

[1] *Always add acid to water to prevent violent reactions.*
[2] *Exposure limit refers to the OSHA regulatory exposure limit.*

Appendix A: Terms and Definitions

Batch: environmental samples that are prepared and/or analyzed together with the same process, using the same lot(s) of reagents. A preparation/digestion batch is composed of one to 20 environmental samples of similar matrix, meeting the above criteria. An analytical batch is composed of prepared environmental samples (extracts, digestates and concentrates), which are analyzed together as a group.

Corrective Action: the action taken to eliminate the cause of an existing nonconformity, defect or other undesirable occurrence in order to prevent recurrence.

Holding Time: the maximum time that a sample may be held before preparation and/or analysis as promulgated by regulation or as specified in a test method.

Intermediate Standard: a solution made from one or more stock standards at a concentration between the stock and working standard. Intermediate standards may be certified stock standard solutions purchased from a vendor and are also known as secondary standards.

Laboratory Control Sample (LCS): a blank matrix spiked with a known amount of analyte(s) processed simultaneously with and under the same conditions as samples through all steps of the procedure.

Matrix Spike (MS): a field sample to which a known amount of target analyte(s) is added.

Matrix Spike Duplicate (MSD): a second replicate matrix spike

Method Blank (MB): a blank matrix processed simultaneously with and under the same conditions as samples through all steps of the procedure. Also known as the preparation blank (PB).

Non-conformance: an indication, judgment, or state of not having met the requirements of the relevant specification, contract or regulation.

Preservation: refrigeration and/or reagents added at the time of sample collection to maintain the chemical, physical, and/or biological integrity of the sample.

Quality Control Sample (QC): a sample used to assess the performance of all or a portion of the measurement system.

Stock Standard: a solution made with one or more neat standards usually with a high concentration. Also known as a primary standard. Stock standards may be certified solutions purchased from a vendor.

Surrogate: a substance with properties that mimic the analyte of interest but that are unlikely to be found in environmental samples.

Title: AUTOMATED SOXHLET EXTRACTION (SW-846 3541)

Approvals (Signature/Date):

06/30/2021	06/02/2021
Don Dawicki Date	Ryan Hammond Date
Laboratory Director	Operations Manager / EHS Coordinator
06/02/2021	06/17/2021
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1.0 Scope and Application

This SOP describes the laboratory procedure for automated Soxhlet extraction of organic analytes from soils, sediment and waste solids and the subsequent concentration of the extracts in preparation for analysis by GC or GC/MS.

1.1 Analytes, Matrices, and Reporting Limits

Refer to analytical methods for analyte lists and reporting limits.

2.0 Summary of Method

A measured amount of sample, usually 15 g, is mixed with anhydrous Sodium sulfate or a mixture of anhydrous Sodium sulfate and Magnesium sulfate to create a free-flowing mixture that is placed in an extraction thimble and extracted with an appropriate solvent for 3 hours using soxtherm extraction. Following extraction, the extract is dried, concentrated, and relinquished to the analytical department(s) or if necessary, exchanged into a solvent compatible for extract cleanup procedures.

This procedure is based on the following reference method:

SW-846 Method 3541, Automated Soxhlet Extraction, Revision 0, 1994. Test Methods for Evaluating Solid Waste Physical/Chemical Methods, Third Edition, September 1986.

3.0 Definitions

A list of terms and definitions are provided in Appendix A.

4.0 Interferences

Method interferences may be caused by contaminants in solvents, reagents, glassware and other sample processing equipment. These contaminants can lead to elevated baselines and discrete artifacts that appear in gas chromatograms. These materials are demonstrated to be free of interferences by the analysis of method blanks. Interferences from Phthalate esters can also pose a problem for the chlorinated pesticides analysis. To avoid this type of interference, the use of plastics in the laboratory must be minimized.

5.0 Safety

Employees must abide by the policies and procedures in the NDSC Eurofins TestAmerica Laboratories Environmental Health and Safety Manual (CW-E-M-001), and this document. This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

5.1 Specific Safety Concerns or Requirements

Nitrile gloves should be used when performing this extraction. Latex and vinyl gloves provide no significant protection against the organic solvents used in this SOP, and should not be used.

The following analytes have been tentatively classified as known or suspected human or mammalian carcinogens: Benzo(a)anthracene, Benzidine, 3,3'-dichlorobenzindine, Benzo(a)pyrene, alpha-BHC, beta-BHC, gamma-BHC, delta-BHC, Dibenz(a,h)anthracene, n-Nitrosodimethylamine, 4,4'-DDT, and polychlorinated biphenyl compounds. Primary standards of these toxic compounds should be prepared in hood.

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During Kuderna-Danish (K-D) concentration, do not allow the extract to boil to dryness. The solvent vapors remaining in the K-D apparatus may superheat and create an explosion or fire hazard.

The K-D apparatus has ground glass joints that can become stuck. Technicians must use Kevlar or other cut/puncture resistant gloves when separating stuck joints.

5.2 Primary Materials Used

Table 1 lists those materials used in this method, which have a serious or significant hazard rating. **Note: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the SDS for each of the materials listed in the table.** A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the SDS for each material before using it for the first time or when there are major changes to the SDS.

6.0 Equipment and Supplies

Catalog numbers listed in this SOP are subject to change at the discretion of the vendor. Analysts are cautioned to be sure equipment used meets the specification of this SOP.

6.1 Extraction Equipment

- Automated Soxhlet Extraction System: Gerhardt SX001-6 place extraction unit or equivalent.
- 54 mm Glass beaker, AMK Glass or equivalent.
- 45 mm Extraction Thimble with holder, AMK Glass or equivalent.
- Water Chiller, VWR or equivalent.

6.2 Miscellaneous

- Disposable glass Pasteur pipette and bulb.
- Top Loading balance: Capable of measuring to 0.01 gram accuracy. Mettler Model Number PM4800 or equivalent.
- Teflon and Stainless Steel Spatulas
- Adjustable pipette, capable of measuring 200 uL to 5 mL, Finnpipette or equivalent.
- 0.5 mL – 2.0 mL Hamilton Gastight® syringes or equivalent.
- 2, 4, 8, or 16 mL Teflon lined screw vial Fisher Scientific or equivalent

7.0 Reagents and Standards

7.1 Reagents

- Sodium sulfate (granular, anhydrous) (Na_2SO_4), J.T. Baker or equivalent. Purify by heating at 400°C for at least 4 hours
- Methylene chloride (CH_2CI_2), Pesticide quality, J.T Baker or equivalent.
- Hexane, (C_6H_{14}), Pesticide Quality. J.T. Baker or equivalent.
- Acetone, ($(CH_3)_2CO$), Pesticide quality. J.T. Baker or equivalent.
- Nitrogen, Ultra High Purity, Linde or equivalent.

7.2 Prepared Reagents

- Methylene chloride/Acetone (3:1): In a 4 L amber glass bottle, mix 3 L Methylene chloride with 1 L Acetone. Store the solution in a fume hood.
- Hexane /Acetone (1:1): In a 4L amber glass bottle mix 2L Hexane with 2L Acetone. Store the solution in a fume hood.

7.3 Standards

Purchase stock standards as certified solutions from commercial vendors. Prepare surrogate and spiking solutions in the laboratory by diluting a known volume of the stock standard solutions in an appropriate solvent. Record the preparation of standard in the LIMS (TALS) module established for this purpose.

Store prepared standard solutions in glass containers at 4°C or below. Unless otherwise specified, assign an expiration date of 6 months from the date of preparation or in accordance with the expiration date of the parent standard, whichever is sooner. The recommended formulation for each standard used in this procedure is provided in the analytical method along with the recommended source materials, expiration dates and storage conditions.

8.0 Sample Collection, Preservation, Shipment and Storage

The laboratory does not perform sample collection so these procedures are not included in this SOP. Sampling requirements may be found in the published reference method.

Matrix	Sample Container	Minimum Sample Size	Preservation	Holding Time[1]	Reference
Solid	Glass with PTFE lined lid	50 g	4°C	Extraction: 14 days	SW-846 Ch 4 (Table 4-1)

[1] *Extraction holding time is determined from sampling date.*

Unless otherwise specified by client or regulatory program, after analysis, samples and extracts are retained for a minimum of 30 days after provision of the project report and then disposed of in accordance with applicable regulations.

9.0 Quality Control

9.1 Sample QC

The laboratory prepares the following quality control samples with each batch of samples.

QC Item	Frequency	Acceptance Criteria
Method Blank (MB)	1 in 20 or fewer samples	See Analytical SOP
Laboratory Control Sample (LCS)	1 in 20 or fewer samples	See Analytical SOP
Matrix Spike(s) MS/MSD	1 in 20 or fewer samples	See Analytical SOP
Sample Duplicate (SD)	Client Request	See Analytical SOP

Surrogate spikes are added to all field and QC samples before preparation and/or analysis to assess the effect of the sample matrix on the accuracy of the method in the specific sample matrix. Matrix Spikes (MS) and Matrix Spike Duplicates (MSD) are performed upon request or when sufficient sample volume is received. When there is insufficient volume for MS/MSD, an LCS duplicate (LCSD) is analyzed to assess method precision and accuracy and an NCM is initiated. The situation documented with the “MS/MSD/DUP – Insufficient Volume” NCM.

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9.2 Instrument QC

For information regarding instrument QC refer to the analytical SOP for the determinative test method.

10.0 Procedure

10.1 Instrument Calibration

Check the calibration of the balance each day of use prior to use and record these checks in the logbook designated for this purpose.

Check the calibration of any mechanical pipettes quarterly in accordance with the procedures specified in laboratory SOP BR-QA-012.

10.2 Glassware & Equipment Set-Up

Prepare glassware using the procedures described in laboratory SOP BR-QA-023 and rinse with Acetone and Methylene chloride prior to use. Label the glassware for each field and QC sample clearly and unambiguously during each step of the extraction procedure. Solvent will erase grease pens and “sharpie ink”; so use caution to ensure labels are not obliterated during the procedure.

Turn on the water chiller to the soxtherm unit. Check to ensure there is sufficient 1:1 glycol/water in the unit and refill if necessary.

Initiate the cleaning of the system using program 1 and using 140 mL of the 1:1 Acetone/Methylene chloride solution. Discard the solution to the appropriate waste collection vessel after use. The cleaning should be performed once per month, or sooner if needed.

The recommended operating conditions for the soxtherm units are:

Operating Conditions (Program #1)

Temp Max: 150°C

Boiling Time: 55 Minute

Solvent Reduction A: 5 X 15 mL

Extraction Time: 55 Minute

Solvent Reduction B: 1X15 mL

Cool Time: 10 Minute

Total time: 2 hours 55 minutes

10.3 Sample Preparation

Mix samples thoroughly following the procedures specified in laboratory SOP BR-QA-020. Do not decant water from sediment samples if a large amount of water is present, contact the Project Manager for further guidance.

Initiate an extraction batch in TALS module ADII.

Place a labeled weighing dish on the balance and tare the balance. Measure 15 g (+/- 1 g) into the tared weighing dish and upload the sample mass into the TALS worksheet. Repeat for each sample and any designated MS/MSD or sample duplicates. Use Sodium sulfate for the method blank and LCS.

Mix each sample with a sufficient amount of drying agent to ensure a free-flowing mixture then

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transfer the sample into an extraction thimble with glass wool in the bottom.

Add ~6-12 silicon carborundum chips to each extraction beaker then place the extraction thimble into an extraction beaker.

Using a syringe add the proper type and volume of surrogate solution to the sample container and add the proper type and volume of spike solution to the sample container. Refer to the Extraction Workbook for proper spikes and volumes. Add 120 mL of extraction solvent to each extraction beaker.

For 8082A, methods use 1:1 Acetone/Hexane solution as the extraction solvent.

For 8270, use 3:1 Methylene Chloride:Acetone.

NOTE: If a sample is very wet and would require an excessive amount of Sodium sulfate to reach a free flowing state, resulting in the extraction thimble being overfilled, it may be permissible to reduce the amount of sample used, provided the project reporting limits can still be met. It may be necessary to reduce the final extract volume to meet the criteria; in this instance, all spiking levels should be adjusted accordingly so that spiking levels in the final extract is unaffected. Consult with the department manager and/or the technology manager prior to performing this modification.

10.4 Extraction

Place the extraction beakers into the extraction unit to ensure a tight seal between the extraction beaker and extraction unit. Check to ensure the chiller system is on and operating properly.

Turn on the soxtherm controller, press number “1” on controller pad to select program 1 and depress the enter key twice to begin the extraction process.

Periodically check the extraction units and do not allow the solvent in the extraction beaker to evaporate to dryness. If the solvent volume becomes low, lift the beakers off the boiling plate by switching the pneumatic control switch and add an additional amount of solvent to assure sufficient solvent volume until the extraction cycle is complete.

When the extraction cycle is finished, remove the beakers from the extraction unit and allow them to cool. Shut the soxtherm and Chiller “off” by depressing the “on” button. Concentrate the extracts to an appropriate final volume.

10.5 Extract Concentration

Refer to SOP BR-EX-031 for concentration techniques

10.6 Extract Preparation & Handling

For final extract volumes less than or equal to 1.0 mL adjust the extract volume using a calibrated graduated concentrator tube. For final extract volumes greater than 1.0 mL use a reference vial. Transfer the extract to labeled Teflon lined screw cap via and store refrigerated. Complete the batch worksheet and perform primary review.

11.0 Corrective Action

When an out-of-control situation occurs, a Corrective Action Report (CAR) (NCM), etc., must be developed (see SOP BR-QA-016) and the analyst must use his/her best analytical judgment and available resources to determine the corrective action to be taken. The out-of-control situation may be caused by more than one variable. The analyst should seek the assistance of his/her immediate supervisor, QA manager or other experienced staff if they are uncertain of the cause of the out-of-control situation. The analysis must not be resumed until the source of the problem and an in-control

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status is re-established. All samples associated with the out-of-control situation must be reanalyzed after in-control status has been re-established or if authorization is received from the supervisor or QA Manager for release with data qualification.

12.0 Calculations / Data Reduction

12.1 Calculations

Calculations are provided in the analytical SOP for each method parameter.

12.2 Data Review

Primary Review: Review the batch worksheet for correctness and completeness. Record any problems encountered during the extraction process with a non-conformance memo (NCM).

Secondary Review: Review the batch worksheet for correctness and completeness and to ensure the extraction performed is consistent the SOP and project specifications. Print the output worksheets and release extracts and output worksheet to the analytical department or to the next step in the method chain such as extract cleanup.

13.0 Method Performance

13.1 Method Detection Limit Study (MDL)

The method detection limit (MDL) is the lowest concentration that can be detected for a given analytical method and sample matrix with 99% confidence that the analyte is present. MDLs reflect a calculated (statistical) value determined under ideal laboratory conditions in a clean matrix, and may not be achievable in all environmental matrices. An initial method detection limit study is performed in accordance with SOP BR_QA-005. The laboratory maintains MDL studies for analyses performed; these are verified at least annually unless method or program requirements require a greater frequency.

13.2 Demonstration of Capabilities

All personnel are required to perform an initial demonstration of proficiency (IDOC) on the instrument they will be using for analysis prior to testing samples. On-going proficiency must be demonstrated annually. IDOCs and on-going proficiency demonstrations are conducted as follows.

13.2.1 Four aliquots of the QC check sample are analyzed using the same procedures used to analyze samples, including sample preparation. The concentration of the QC check sample can be equivalent to a mid- level calibration.

13.2.2 Calculate the average recovery and standard deviation of the recovery for each analyte of interest.

13.2.3 If any analyte does not meet the acceptance criteria, the test must be repeated. Only those analytes that did not meet criteria in the first test need to be evaluated. TNI 2016 requires consecutive passing results. Repeated failure for any analyte indicates the need for the laboratory to evaluate the analytical procedure and take corrective action.

13.2.4 Until the IDOC is approved by the QA Manager (or designee); the trainer and trainee must be identified in the batch record.

13.3 Training Requirements

The Group Leader is responsible for ensuring that this procedure is performed by an associate who has been properly trained in its use and has the required experience. A new analyst must be working under documented supervision prior to approval of the IDOC. Documentation that a new

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analyst is performing under supervision must be entered into the batch record (View Batch Information) until that analyst's IDOC has been approved by the QA Manager (or designee). See requirements for demonstration of analyst proficiency in SOP BR-QA-011.

14.0 Pollution Control

It is Eurofins TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage and reagent stability). Employees must abide by the policies in Section 13 of the NDSC Eurofins TestAmerica Laboratories Environmental Health and Safety Manual (CW-E-M-001) for "Waste Management and Pollution Prevention."

15.0 Waste Management

Waste management practices are conducted consistent with all applicable rules and regulations. Excess reagents, samples and method process wastes are disposed of in an accepted manner. Waste description rules and land disposal restrictions are followed. Waste disposal procedures are incorporated by reference to BR-EH-001 Hazardous Waste. The following waste streams are produced when this method is carried out.

- Solid Waste – Satellite Container: 5 Gallon Polyethylene Bucket
- Solvent Waste- Satellite Container: Steel 55 Gallon Drum

16.0 References / Cross-References

- SW-846 Method 3541, Automated Soxhlet Extraction, Revision 0, 1994. Test Methods for Evaluating Solid Waste Physical/Chemical Methods, Third Edition, September 1986.
- Corporate Environmental Health and Safety Manual (CW-E-M-001)
- Laboratory SOP BR-EH-001 *Hazardous Waste*
- Laboratory SOP BR-EX-002 *Extract Cleanup Procedures*
- Laboratory SOP BR-QA-023 *Glassware Cleaning*
- Laboratory SOP BR-EX-031 Concentration Techniques
- Laboratory SOP BR-QA-012 Support Equipment
- Laboratory SOP BR-QA-020 *Procedures for Sample Homogenization & Subsampling*
- Laboratory SOP BR-QA -019 *Data Review*
- Laboratory SOP BR-QA-005 *Determination of LOD, LOQ, & RL*
- Laboratory SOP BR-WC-006 *Percent Solids*

17.0 Method Modifications

#	Method Reference	Modification	Technical Justification
1	SW846 3541	The method calls for the use of 1:1 Methylene Chloride:Acetone for 8270 compounds. TA Burlington uses 3:1.	Used to alleviate aldol-condensation seen in semi samples.

18.0 Attachments

- Table 1: Primary Materials Used

- Appendix A: Terms and Definitions

19.0 Revision History

Revision 5.0, effective date 06/30/2021

- Revision incorporates TNI required updates from 2021 audit findings.
- Title Page: Updated headers, footers, signatories and copyright date
- Sections 5 and 14: Updated to reflect current NDSC required language.
- Section 9.1: Added the use of the NCM when insufficient sample volume to perform batch QC.
- Section 10.2: Updated to include length of automated extraction in response to TNI 2021 audit.
- Section 10.2: Update reference to Glassware Cleaning SOP.

Revision 4.0, effective date 04/13/2020

- Title Page: Updated approval signatures and Copyright date
- Section 10.2 &10.4: Updated to include that the chiller uses 1:1 glycol/water and that Nitrogen is not adjusted.
- Section 10.3: Minor changes to reflect bench practice.
- Throughout: Fixed minor typos/errors.

Previous revisions are retained by the QA department.

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Table 1: Primary Materials Used

Material[1]	Hazards	Exposure Limit[2]	Signs and symptoms of exposure
Acetone	Flammable	1000 ppm-TWA	Inhalation of vapors irritates the respiratory tract. May cause coughing, dizziness, dullness, and headache.
Hexane	Flammable Irritant	500 ppm-TWA	Inhalation of vapors irritates the respiratory tract. Overexposure may cause lightheadedness, nausea, headache, and blurred vision. Vapors may cause irritation to the skin and eyes.
Methylene Chloride	Carcinogen Irritant	25 ppm-TWA 125 ppm-STEL	Causes irritation to respiratory tract. Has a strong narcotic effect with symptoms of mental confusion, light-headedness, fatigue, nausea, vomiting and headache. Causes irritation, redness and pain to the skin and eyes. Prolonged contact can cause burns. Liquid degreases the skin. May be absorbed through skin.

[1] *Always add acid to water to prevent violent reactions.* [2] *Exposure limit refers to the OSHA regulatory exposure limit.*

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Appendix A: Terms and Definitions

Acceptance Criteria: specified limits placed on characteristics of an item, process or service defined in requirement documents.

Analyte: The specific chemicals or components for which a sample is analyzed. (EPA Risk Assessment Guide for Superfund, OSHA Glossary).

Batch: environmental samples that are prepared and/or analyzed together with the same process, using the same lot(s) of reagents. A preparation/digestion batch is composed of one to 20 environmental samples of similar matrix, meeting the above criteria. An analytical batch is composed of prepared environmental samples (extracts, digestates and concentrates), which are analyzed together as a group.

Corrective Action: the action taken to eliminate the cause of an existing nonconformity, defect or other undesirable occurrence in order to prevent recurrence.

Holding Time: the maximum time that a sample may be held before preparation and/or analysis as promulgated by regulation or as specified in a test method.

Laboratory Control Sample (LCS): a blank matrix spiked with a known amount of analyte(s) processed simultaneously with and under the same conditions as samples through all steps of the procedure.

Matrix Spike (MS): a field sample to which a known amount of target analyte(s) is added.

Matrix Spike Duplicate (MSD): a second replicate matrix spike

Method Blank (MB): a blank matrix processed simultaneously with and under the same conditions as samples through all steps of the procedure. Also known as the preparation blank (PB).

Non-conformance: an indication, judgment, or state of not having met the requirements of the relevant specification, contract or regulation.

Preservation: refrigeration and/or reagents added at the time of sample collection to maintain the chemical, physical, and/or biological integrity of the sample.

Quality Control Sample (QC): a sample used to assess the performance of all or a portion of the measurement system.

Reporting Limit (RL): the level to which data is reported for a specific test method and/or sample.

Stock Standard: a solution made with one or more neat standards usually with a high concentration. Also known as a primary standard. Stock standards may be certified solutions purchased from a vendor.

Surrogate: a substance with properties that mimic the analyte of interest but that are unlikely to be found in environmental samples.

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Title: Polychlorinated Biphenyls (PCBs) by GC/ECD (SW846 8082A, USEPA TO4A/TO10A)

Approvals (Signature/Date):

06/30/2021	06/02/2021
Don Dawicki Date	Ryan Hammond Date
Laboratory Director	Operations Manager / EHS Coordinator
06/02/2021	06/30/2021
Kristine Dusablon Date	Mark Fausel Date
Quality Assurance Manager / Technical Manager	Department Supervisor

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1.0 Scope and Application

This SOP describes the laboratory procedure used to determine the concentration of polychlorinated biphenyls (PCBs) as Aroclors using dual column gas chromatography with electron capture detectors (GC/ECD).

This SOP is applicable to instrument analysis only. Extraction and extract cleanup procedures are provided in separate SOPs.

1.1 Analytes, Matrices, and Reporting Limits

This procedure may be used for a variety of matrices including: air, sediment, soil, tissue, and water.

The list of target compounds that can be determined from this method along with the associated reporting limits (RL) is provided in Table 1.

2.0 Summary of Method

2.0uL of extract is injected into a dual capillary column gas chromatograph equipped with electron capture detectors (GC/ECD). The chromatographic data is used to determine the list of analytes provided in Table 1. NOTE: Injection volumes used must be validated and all samples are to be analyzed with the same injection volume used for standard solutions within the initial calibration. Quantitation is accomplished by averaging the concentration of three to five peaks for each Aroclor against a calibration curve. Internal standards are used to compensate for instrument variation.

This SOP is based on the following reference methods:

- SW-846 Method 8082A Polychlorinated Biphenyls (PCBs) by Gas Chromatography, Revision 0, February 2007.
- Compendium Method TO-4A, Determination of Pesticides and Polychlorinated Biphenyls in Ambient Air using High Volume Polyurethane Foam (PUF) Sampling Followed By Gas Chromatographic/Multi-Detector Detection (GC/MD), January 1999 (EPA/625/R-96/010b)
- Compendium Method TO-10A, Determination of Pesticides and Polychlorinated Biphenyls in Ambient Air using Low Volume Polyurethane Foam (PUF) Sampling Followed By Gas Chromatographic/Multi-Detector Detection (GC/MD), January 1999 (EPA/625/R-96/010b)

If the laboratory procedure is modified from the above reference method, a list of modifications will be provided in Section 16.0 of this SOP.

3.0 Definitions

Refer to the Laboratory's Quality Assurance Manual (QAM) for the Glossary of Terms, Definitions and Acronyms. Additionally, a list of terms and definitions are provided in Appendix A.

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4.0 Interferences

- Method interference may be caused by contaminants in the extraction solvent. Solvents should be stored away from organochlorine compounds to minimize contamination.

- Non-target compounds co-extracted from the sample matrix can also cause interference, the extent of which will vary depending on the nature of the samples. Elemental sulfur is often found in sediment samples and its presence will result in broad peaks. Samples are screened prior to analysis, and those samples that contain high levels of sulfur are subject to sulfur cleanup (SW-846 3660B). Cleanup procedures that may be used for this method include: GPC (SW-846-3640A), silica gel (SW-846 3630C), Florisil (SW-846 3620B), and Sulfuric acid Cleanup (SW-846 3665A).

- Phthalate esters introduced during sample preparation can pose a problem in the determination of target analytes. Common flexible plastics contain varying amounts of phthalate esters. These phthalate esters can be easily extracted or leached during extraction. To minimize this interference, avoid contact with any plastic materials.

5.0 Safety

Employees must abide by the policies and procedures in the NDSC Eurofins TestAmerica Laboratories Environmental Health and Safety Manual (CW-E-M-001), and this document. This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

5.1 Specific Safety Concerns or Requirements

The gas chromatograph contains zones that have elevated temperatures. The analyst must be aware of the locations of those zones and must cool them to room temperature prior to working on them.

There are areas of high voltage in the gas chromatograph. Depending on the type of work involved, either turn the power to the instrument off or disconnect it from its source of power.

5.2 Primary Materials Used

Table 2 lists materials used in this method which have a serious or significant hazard rating. **Note: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the SDS for each of the materials listed in the table.** A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the SDS for each material before using it for the first time or when there are major changes to the SDS.

6.0 Equipment and Supplies

Catalog numbers listed in this SOP are subject to change at the discretion of the vendor. Analysts are cautioned to be sure equipment used meets the specification of this SOP.

6.1 Miscellaneous

- Autosampler Vials, National Scientific or equivalent.
- Hydrogen Generator: Parker Hannafin H2-1200 or equivalent. Alternatively, UHP Hydrogen in cylinders may be used.
- Volumetric Glassware, Class “A” pipettes and flasks ranging in concentration from 0.5mLs to 500 mLs.
- Volumetric Syringes, Class “A” (10µl, 25µl, 50µl, 100µl, 250µl and 500µl), Hamilton or equivalent.

6.2 Analytical System

- Computer Hardware/Software: GC Acquisition Platform – ChemStation. Data Processing – TestAmerica Chrom, current version.
- GC/ECD: with dual columns, dual ECDs, and auto-sampler capable of injecting a 0.2-2.0 µL aliquot split onto two columns: Agilent Technologies 6890N with 7683 Series injector, or equivalent.
- Column Splitter: Siltek Angled Y Presstight Connector (Restek part# 20469) or equivalent. Used with the intent of splitting the injected volume equally into the two analytical columns
- GC Columns: A dual fused silica capillary column system that will provide simultaneous primary and confirmation analyses:
  - RTX-CLP, (30m x 0.32 mm ID x 0.5µm)
  - RTX-CLPII, (30m x 0.25 mm ID x 0.5µm)

Equivalent columns may be used provided the elution orders are documented and compound separations are maintained.

7.0 Reagents and Standards

7.1 Reagents

- Hexane, Ultra-Resi Analyzed, JT Baker or equivalent.

7.2 Standards

Purchase stock standard solutions from commercial vendors and from these prepare calibration and working standards by diluting a known volume of stock standard in an appropriate solvent to the final volume needed to achieve the desired concentration. The recommended formulation for

each standard used in this procedure is provided in Appendix B along with the recommended source materials, expiration dates and storage conditions.

8.0 Sample Collection, Preservation, Shipment and Storage

The laboratory does not perform sample collection, so these procedures are not included in this SOP. Sampling requirements may be found in the published reference method. Sample container, preservation techniques and holding times may vary and are dependent on sample matrix, method of choice, regulatory compliance, and/or specific contract or client requests. Listed below are the holding times and the references that include preservation requirements.

Matrix	Sample Container	Minimum Sample Size	Preservation	Extract Holding Time	Reference
Air	Polyurethane Foam Plug (PUF)	1 PUF	Chilled to 4°C	40 Days	TO-10A
Air	PUF	1 PUF	Chilled to 4°C	40 Days	TO-4A
Water	Glass	1 L	Chilled to 4°C	40 Days	SW-846 8082A
Solid	Glass	50 g	Chilled to 4°C	40 Days	SW-846 8082A

Analytical holding time is determined from date of initiation of extraction.

Unless otherwise specified by client or regulatory program, after analysis, samples and extracts are retained for a minimum of 30 days after provision of the project report and then disposed of in accordance with applicable regulations.

Note: EPA SW-846 Update IV Chapter 4 re-defined the holding times for PCBs as “NONE”. The EPA Methods Update Rule re-defined holding times for PCBs as one year from collection to extraction and one year from extraction to analysis. The laboratory incorporates the extended preparation holding times for PCBs on a project-specific basis, as allowed by that particular state agency and client.

9.0 Quality Control

9.1 Sample QC

The laboratory prepares the following quality control samples with each batch of samples.

QC Item	Frequency	Acceptance Criteria
Method Blank (MB)	1 in 20 or fewer field samples	See Table 3
Laboratory Control Sample (LCS)	1 in 20 or fewer field samples	See Table 3
Laboratory Control Sample Duplicate (LCSD)	1 in 20 or fewer field samples, if volume is not present for MS/MSD	See Table 3
Matrix Spike(s) MS/MSD	1 in 20 or fewer field samples	See Table 3

Sample Duplicate (SD)	Client Request	See Table 3

9.2 Instrument QC

The following instrument QC is performed:

QC Item	Frequency	Acceptance Criteria
Initial Calibration (ICAL)	Initially; when ICV or CCV fail	See Table 3
Second Source Calibration Verification (ICV)	Once, after each ICAL	See Table 3
Continuing Calibration Verification (CCV)	8082: Daily, every 20 field samples or 12 hours (whichever is sooner) TO4/TO10: Daily, every 10 field samples	See Table 3
Retention Time Windows	Once every 24 hours for all Aroclors reported (8082A). Once every 24 hours for Aroclor 1016/1260 (TO4/TO10)	Reference Section 10.2

10.0 Procedure

10.1 Instrument Operating Conditions

Install a five meter deactivated guard column into the injection port and connect the guard column to the separate analytical columns using a glass "Y". The analytical columns are installed into independent ECD detectors.

The recommended instrument operating conditions are as follows:

Initial Temperature	130ºC
Temperature Program	30ºC per minute to 210ºC; 10ºC per minute to 250ºC, hold 0.37 minutes; 20.0ºC per minute to 300°C. 30ºC per minute to 320 ºC. Hold for 3 minutes.
Detector Temperature	300ºC
Injector Temperature	220ºC
Injection port liner	Gooseneck splitless liner w/ deactivated glass wool
Injection volume	2.0µL Split pulse injection at 20psi actual
Carrier Gas	Hydrogen @ 10-15psi actual

Temperature and flow conditions can be moderately adjusted to satisfy chromatographic conditions that must produce Gaussian shaped peaks with defined separation of target analytes.

10.2 Retention Time Window Establishment

Whenever a new GC column is installed, establish RT windows for each analyte by analyzing three standards over a 72-hour period. Calculate the mean RT and Standard Deviation (SD).

The RT window is calculated as the mean RT ± 3SD. If the SD is <0.01 minutes, a default SD of 0.01 minutes may be used.

If this procedure results in RT windows that are too tight, favoring false negatives, the laboratory may opt to use an alternate method to determine the RT windows. An alternate method consists of using a RT window of ± 0.05 minutes. The center of the RT window is set at the midpoint calibration level in the initial calibration sequence. RT windows are then updated daily (minimum frequency), re-centering the windows on the retention times established in a CCV.

When determining retention time windows, analyze a standard of DDT, DDE and DDD. Ensure that the Aroclor peaks being used do not interfere with the major single-peak pesticides.

10.3 Instrument Calibration

10.3.1 Initial Calibration (ICAL)

Analyze a minimum of five calibration levels for each Aroclor. The following Aroclors may be combined in the standards: 1016/1260 (also known as 1660), 1221/1254, 1232/1262, 1242/1268. Aroclor 1248 may not be combined with other Aroclors.

Tetrachloro-m-xylene and Decachlorobiphenyl are calibrated from the Aroclor 1660 standards. Chrom settings for acquisition of other Aroclor calibrations must not have surrogate selected to calibrate.

Note: If the calibration will be used only for 8082A analysis, a single point calibration may be used except for Aroclors 1016 and 1260. The full set of Aroclors do not need to be calibrated if used for projects with a limited requested list. Every initial calibration block requires at least one standard with Aroclor 1016 to be analyzed for pattern recognition purposes, however.

Prepare the calibration standards using the formulations provided in Appendix B then transfer 100 µg/L to an autosampler vial insert. Add 4uL of the internal standard (IS) solution, then crimp the vial tightly.

Place the vials in the autosampler, and initiate the analytical sequence.

A minimum of five quantitation peaks must be chosen for each Aroclor with the exception of Aroclor 1221 which requires a minimum of 3 peaks. The peaks must be characteristic of the Aroclor in question.

The peaks used for quantitation in each Aroclor standard must be at least 25% of the height of the largest Aroclor peak with the exception of Aroclor 1268 where the requirement is 10%.

Each set of 5 peaks should include at least one peak that is unique to that Aroclor.

Calculate the Calibration Factor (CF), mean CF, and Percent Relative Standard Deviation (%RSD) for each analyte on both columns using the peak height, adjusted for internal standard response.

The %RSD for each target analyte must be less than or equal to 20% in order to use the mean CF for quantification. This evaluation is performed for each quantitation peak chosen for each

Aroclor. All peaks must individually pass the 20% evaluation.

Alternately, use linear regression with a correlation coefficient (r) ≥ 0.995. Linear regression requires a minimum of five calibration points.

Whichever calibration model is selected, it should be subjected to an additional check to establish the representativeness of the data that were used to produce it. This check is the refitting of each calibration point response back to the calibration model, or the comparison of the calculated amount of the standard against the expected amount. Calculate the concentration of each calibration standard from the final calibration curve. Calculate the %Error for each point. All points except the lowest point must have an absolute %Error of < 30%. The lowest standard (RL level) must have an absolute %Error < 50%. If these conditions are not met, perform corrective action and recalibrate the instrument.

The laboratory CHROM software provides an Initial Calibration %Drift report which shows the % Error for each calibration point. This report must be reviewed in addition to the %RSD / Linear Response Factor.

%Error is the absolute value of the difference between the calculated concentration and the true concentration divided by the true concentration x100%.

- For example, if the 0.20mg/L standard reads back as 0.17mg/L:

[(0.20 – 0.17)/0.20] x 100% = 15%

The mid-level AR1660 ICAL standard must meet a resolution criteria of <75% for the triplet near the end of the AR1260 pattern on at least one column. The resolution may be calculated per the equation in Appendix C.

10.3.2 Second Source Calibration Verification (ICV)

Immediately after each calibration and prior to the analysis of any QC or field samples, verify the accuracy of the initial calibration by analyzing a standard prepared from a second source of Aroclor 1660.

Prepare the ICV using the formulation provided in Appendix B. Aliquot 100uL of the working standard into a vial, and add 4uL of the IS solution. Crimp tightly. Inject the ICV standard onto the instrument in the same manner as performed for the initial calibration standards.

For 8082A, the percent difference must be within ± 20% of the expected value for the average of the quantitation peaks for each Aroclor. The retention time (RT) of each peak must be within the established RT window. Acceptance criteria must be met on both columns.

For TO-4A/TO-10A, the percent difference must be within ± 15% of the expected value for the average of the quantitation peaks for each Aroclor. The retention time (RT) of each peak must be within the established RT window. Acceptance criteria must be met on both columns.

If this criterion is not met, correct the problem and reanalyze the ICV. If reanalysis fails, remake the calibration standards and/or perform instrument maintenance and recalibrate. The acceptance criteria must be met on both columns. See Table 3.

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10.3.3 Continuing Calibration Verification (CCV)

Analyze a CCV (AR1660) at or below the mid-calibration range each day before sample analysis. This CCV is used to update the retention times of AR1660.

Analyze a CCV (AR1660) at least every 20 field samples for 8082A. If Aroclors other than 1660 are identified in a sample, a CCV of the identified Aroclor must be analyzed within 12 hours of the sample. Bracketing CCVs are not required.

Analyze a CCV (AR1660) at least every 10 field samples for TO4A/TO10A. The laboratory does not perform a CCV for the remaining Aroclors unless requested for the project or by regulatory requirement.

A CCV (AR1660) must be analyzed at least every 12 hours of operation.

Aliquot 100uL of the needed standard into a vial. Add 4uL of the IS solution, and crimp tightly. . Inject the CCV standard onto the instrument in the same manner as performed for the initial calibration standards.

The data system calculates an average calibration factor (CF) for each set of 5 quantitation peaks. The percent difference from the average calibration factor in the initial calibration is then calculated.

For 8082A, the percent difference or drift must be within ±20% for the average of the quantitation peaks and the retention time (RT) must be within the established RT window. Acceptance criteria must be met on both columns.

For TO4A/TO10A, the percent difference or drift must be within ±15% for the average of the quantitation peaks and the retention time (RT) must be within the established RT window. Acceptance criteria must be met on both columns.

The AR1660 CCV standard must meet a resolution criteria of <75% for the triplet near the end of the AR1260 pattern on at least one column. The resolution may be calculated per the equation in Appendix C.

If the CCV fails, it may be repeated once. If repeat analysis fails, corrective action must be taken. See Table 3.

10.3.4 Retention Time Updates

For 8082A analysis, all target analytes must have at least one CCV per 24 hours to update the retention time. If the samples have been screened and the Aroclors present in the samples are known, only those Aroclors must have the retention times updated daily.

For TO4A and TO10A analysis, only Aroclor 1660 is required to be analyzed every 24 hours. Update retention times of the other Aroclors as necessary.

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10.4 Troubleshooting

Check the following items in case of calibration failures:

- ICAL Failure – Perform injection port maintenance, install new guard column, check detector ends to see if detector jet has slipped. In extreme cases, install new columns, particularly if the chromatography has degraded as evidenced by peak shapes.
- CCV Failure – Perform Injection port maintenance; if injection port maintenance does not result in acceptable performance install a new guard column and remove one or more loops from each analytical column.
- Needle crushed during injection - Replace the needle and check the injection port for obstructions and check the autosampler for misalignment. Remove and clean the needle guide if necessary.
- Auto-sampler failure - Reset the auto-sampler and GC system. Replace the z-axis belt or clean the needle guide as necessary.
- Power failure - Reset run in ChemStation and re-acquire or re-initiate run sequence.

10.5 Sample Analysis

It is recommended that samples are screened to determine dilutions, Aroclor identifications, and the potential need for further sample cleanup prior to analysis.

Samples are typically screened at a 10x dilution against a single-point calibration curve. Screen data is not uploaded to TALS and is not reported.

10.6 Sample Analysis

Remove the extract from refrigerated storage and warm to room temperature.

Transfer 100 µL of extract to an autosampler vial. Add 4uL of IS solution, and crimp the vial tightly.

Place the vials in the autosampler in a sequence that begins with the calibration standards followed by the analysis of an ICV, QC samples, field samples and continuing calibration verification standards (CCVs).

Enter the sample ID's into the data acquisition program in the order that the samples were placed in the autosampler tray and initiate the analytical sequence.

Example analytical sequences follow:

Full Calibration Sequence	
Injection Number	**Lab Description**
1-2	Conditioning Blank
3-7	AR1221/1254 Calibration Block
8-12	AR1232/1262 Calibration Block
13-17	AR1242/1268 Calibration Block

18-22	AR1248 Calibration Block
23-27	AR1660 and Surrogate Calibration Block
28-32	Initial Calibration Verifications

Example Sample Analysis Sequence	
Injection Number	**Lab Description**
1-2	Conditioning Blank
4	AR1660 CCV
	CCVs for other Aroclors, as needed
5-24	Field Sample Injections*
	Repeat sample/CCV blocks as necessary*

* Injection sequence is dependent on method requirements (See Table 3).

Cleaning blanks (PIBLK) consisting of hexane may be analyzed after high-level samples at the discretion of the analyst for samples which contain large concentrations of late-eluting compounds.

11.0 Corrective Action

When an out-of-control situation occurs that is not delineated in this corrective action table or the corrective actions listed do not adequately address the circumstances, a Corrective Action Report (CAR) (NCM), etc., must be developed (see SOP BR-QA-016) and the analyst must use his/her best analytical judgment and available resources to determine the corrective action to be taken. The out-of-control situation may be caused by more than one variable. The analyst should seek the assistance of his/her immediate supervisor, QA manager or other experienced staff if they are uncertain of the cause of the out-of-control situation. The analysis must not be resumed until the source of the problem and an in-control status is re-established. All samples associated with the out-of-control situation must be reanalyzed after in-control status has been re-established or if authorization is received from the supervisor or QA Manager for release with data qualification.

12.0 Calculations / Data Reduction

12.1 Qualitative Identification

The data processing system identifies the target analytes by comparing the retention time of the peaks to the established retention time windows.

Review and accept or reject the qualitative identifications made by the data processing system using the following guidelines:

Compare the retention time of the peak to the established RT window, taking into account the shift of the surrogate peaks. If the surrogate peaks have shifted, open the retention time window in the direction of the shift. The processing system identifies the peak in the retention time window that is closest to the expected retention time set in the Chrom method, so the peak may need to be re-identified if a shift has occurred. The data system does not recognize Aroclor

patterns. The analyst manually identifies Aroclors by comparing the pattern in the samples to the patterns in the initial calibration standards. Weathering of PCB's in the environment may alter the PCB's to the point that the pattern no longer matches the pattern established for that Aroclor in the initial calibration. The laboratory takes the best pattern match approach to the identification and quantification of weathered PCB's.

Look for shoulders on the side of large peaks that may be peaks of interest. The processing system does not always automatically integrate shoulders from larger peaks, so manual integration (split) of the shoulder may be necessary.

Each target analyte must be detected on both columns for qualitative identification to be made.

12.2 Quantitative Identification

Using an average of the chosen quantification peaks per Aroclor the data system calculates the corrected concentration for each target analyte using the equations given in Appendix C. If sample interference is suspected, the laboratory may remove quantification peaks per column, keeping a minimum of 3 quantitation peaks per Aroclor on each column. The lower value between the two columns is reported as the primary result, unless the project specifies otherwise and the TALS formatter uses another formatting rule.

12.3 Calculations

See Appendix C.

12.4 Data Review

See laboratory SOP BR-QA-019 for detailed data review requirements.

12.4.1 Primary Review

Review project documents to ensure those project requirements were met. If project requirements were not met, immediately notify the project manager (PM) to determine an appropriate course of action.

Confirm qualitative and quantitative identification criteria using the criteria provided in Sections 11.1 and 11.2. If the data system does not properly integrate the peaks, perform manual integration in accordance with corporate SOP CA-Q-S-002.

Upload the data files from the data processing system (Chrom) to the laboratory information management system (TALS). Complete the batch information for standards and reagents and verify ICAL and QC sample associations. Review the results and set results to primary, secondary, acceptable or rejected as appropriate. Dilute and reanalyze samples whose average of peak results exceed the calibration range. The dilution analysis should result in a determination within the calibration range, preferably in the upper half of the calibration range. A more concentrated analysis is not necessary unless the project requires it. Dilution analyses may be performed to minimize matrix interference.

If a sample was analyzed immediately following a high concentration sample, review the results of the sample for any sign of carryover. If carryover is suspected, reanalyze the sample.

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Create a non-conformance memo (NCM) for any calibration, QC and sample data that is reported outside established acceptance criteria and/or schedule necessary corrective action. Set batch to 1st level review and complete the data review checklist.

12.4.2 Secondary Data Review

Verify quantitative and qualitative identification in the initial calibration standards and spot check such for ~15% of the remaining data in the batch.

If manual integrations were performed, review each integration to verify that the integration meets the requirements for manual integration as specified in corporate SOP CA-Q-S-002. If an error is suspected or found consult with the analyst that performed the integration and request correction or notify the Department Supervisor, Operations Manager, or QA Manager. Do not "fix" the integration. Reintegration by a secondary data reviewer must not be performed except in limited circumstances as approved by the department supervisor or other laboratory management. In those instances where the secondary reviewer performs the integration, this person is now considered the primary analyst and each integration performed by the secondary reviewer must be subsequently reviewed by a peer analyst or the department supervisor to verify the integration is consistent and compliant with the requirements specified CA-Q-S-002.

Review project documents to ensure those project requirements were met. If project requirements were not met, immediately notify the project manager (PM) to determine an appropriate course of action.

Verify that the acceptance criteria for the calibration and QC items listed in Table 1 were met. If the results do not fall within the established limits verify the recommended corrective actions were performed. If not, initiate corrective actions and/or verify an NCM was created to document the criteria exception. Verify analytical results are qualified accordingly. Set batch to 2nd level review and complete the data review checklists.

Run and review the deliverable. Fix any problems found. When complete set the method chain to lab complete and forward any paperwork to report/project management.

13.0 Method Performance

13.1 Method Detection Limit Study (MDL)

The method detection limit (MDL) is the lowest concentration that can be detected for a given analytical method and sample matrix with 99% confidence that the analyte is present. MDLs reflect a calculated (statistical) value determined under ideal laboratory conditions in a clean matrix, and may not be achievable in all environmental matrices. An initial method detection limit study is performed in accordance with BR-QA-005. The laboratory maintains MDL studies for analyses performed; these are verified at least annually unless method or program requirements require a greater frequency.

13.2 Demonstration of Capabilities

All personnel are required to perform an initial demonstration of proficiency (IDOC) on the instrument they will be using for analysis prior to testing samples. On-going proficiency must be

demonstrated annually. IDOCs and on-going proficiency demonstrations are conducted as follows.

13.2.1 Four aliquots of the QC check sample are analyzed using the same procedures used to analyze samples, including sample preparation. The concentration of the QC check sample can be equivalent to a mid- level calibration.

13.2.2 Calculate the average recovery and standard deviation of the recovery for each analyte of interest.

13.2.3 If any analyte does not meet the acceptance criteria, the test must be repeated. Only those analytes that did not meet criteria in the first test need to be evaluated. TNI 2016 requires consecutive passing results. Repeated failure for any analyte indicates the need for the laboratory to evaluate the analytical procedure and take corrective action.

13.2.4 Until the IDOC is approved by the QA Manager (or designee); the trainer and trainee must be identified in the batch record.

13.3 Training Requirements

The Group Leader is responsible for ensuring that this procedure is performed by an associate who has been properly trained in its use and has the required experience. A new analyst must be working under documented supervision prior to approval of the IDOC. Documentation that a new analyst is performing under supervision must be entered into the batch record (View Batch Information) until that analyst's IDOC has been approved by the QA Manager (or designee). See requirements for demonstration of analyst proficiency in BR-QA-011.

14.0 Pollution Control

It is Eurofins TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage and reagent stability). Employees must abide by the policies in Section 13 of the NDSC Eurofins TestAmerica Laboratories Environmental Health and Safety Manual (CW-E-M-001) for "Waste Management and Pollution Prevention."

15.0 Waste Management

Waste management practices are conducted consistent with all applicable rules and regulations. Excess reagents, samples and method process wastes are disposed of in an accepted manner. Waste description rules and land disposal restrictions are followed. Waste disposal procedures are incorporated by reference to BR-EH-001. The following waste streams are produced when this method is carried out.

- Vials containing sample extracts: Satellite container: 15 gallon bucket connected to a fume hood.
- Solvent Waste: Satellite container: 1 L glass bottle located in fume hood.

16.0 References / Cross-References

- SW-846 Method 8082A Polychlorinated Biphenyls (PCBs) by Gas Chromatography, Revision 0, February 2007.
- Compendium Method TO-4A, Determination of Pesticides and Polychlorinated Biphenyls in Ambient Air using High Volume Polyurethane Foam (PUF) Sampling Followed By Gas Chromatographic/Multi-Detector Detection (GC/MD), January 1999 (EPA/625/R-96/010b)
- · Compendium Method TO-10A, Determination of Pesticides and Polychlorinated Biphenyls in Ambient Air using Low Volume Polyurethane Foam (PUF) Sampling Followed By Gas Chromatographic/Multi-Detector Detection (GC/MD), January 1999 (EPA/625/R-96/010b)
- Corporate Environmental Health and Safety Manual (CW-E-M-001)
- Laboratory SOP BR-QA-002, Traceability, Handling, & Storage of Standards & Reagents
- Laboratory SOP BR-QA-011, Employee Training and Analyst Demonstration of Proficiency
- Laboratory SOP BR-EH-001, Tracking and Collection of Hazardous Waste
- Laboratory SOP BR-QA-014, Laboratory Records
- Laboratory SOP BR-QA-005, Procedures for Detection Limits, Limits of Detection, Limits of Quantitation
- Corporate SOP CA-Q-S-002, Manual Integrations
- Corporate SOP CA-Q-P-003, Calibration Curves and the Selection of Calibration Points
- Laboratory SOP BR-QA-005

17.0 Method Modifications

Method Reference	Modification
TO4A 13.1/TO10A 12.1	The laboratory uses five-point calibration curves for all Aroclors instead of the three-point curves required in the reference method
	The laboratory calibrates with all Aroclors, not only 1242 and 1254 or 1260
	The use of internal standards has been incorporated with this method based on guidance from method 8082A section 11.6.8. Per that guidance, bracketing CCVs are not required.
	Continuing calibration verification is performed using Aroclor 1016 and 1260, in place of Aroclor 1242 and 1260
	Due to the relative height of target peaks, only 3 peaks are used for the quantitation of Aroclor 1221

18.0 Attachments

- Table 1: Target Compound List and Reporting Limit
- Table 1A: Accuracy and Precision Limits
- Table 2: Primary Materials Used
- Table 3: QC Summary & Recommended Corrective Action (8082A)
- Table 3: QC Summary & Recommended Corrective Action (TO4A & TO10A)
- Appendix A: Terms and Definitions
- Appendix B: Standard Preparation Tables
- Appendix C: Equations

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19.0 Revision History

BR-GC-005, Rev 15 effective date 06/30/2021

- Revision incorporates finding from 2021 TNI audit.
- Title Page: Updated headers, footers, signatories and copyright date
- Section 10.3.1: Added language to better identify TNI Standard requirement for calibration standard read back evaluation.
- Section 10.3.3: Changed language to correct that CCV is analyzed ever 10 samples for TO4A and TO10A.
- Sections 5.0 and 14.0: Updated to include required NDSC reference and language.
- Section 17.0: Added method modification for CCV and three 1221 peaks.
- Section 19.0: Added effective dates
- Throughout: Added clarifying language, fixed minor typographical errors.

BR-GC-005, Rev 14 effective date 04/10/2020

- Title page: Updated Approval Signatures and Copyright Date.
- Section 6.1/6.2: Alternative equipment used listed.
- Section 10.1: Added minor clarifying statement pertaining to chromatographic conditions and added the type of injection port liner used.
- Section 10.3.2: Clarified the ICV requirements for TO-4A/TO-10A and 8082A.
- Section 10.6: Deleted “Retention Time Update Standard” line in Full Calibration Sequence Table.
- Throughout: Added clarifying language, fixed minor typographical errors.

Previous Revisions are retained by the QA department.

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Table 1: Routine Target Analyte List & Reporting Limits (RL)

ANALYTE	Routine Reporting Limit (RL)[1,2]				
	Water (ug/L)	Solid (ug/Kg)	Air (ug/m^3 TO10A)[3]	Air (ug/m^3 TO4A)[4]	Air (ug/PUF)
AR1016	0.50	17	0.10	0.00033	0.10
AR1221	0.50	17	0.10	0.00033	0.10
AR1232	0.50	17	0.10	0.00033	0.10
AR1242	0.50	17	0.10	0.00033	0.10
AR1248	0.50	17	0.10	0.00033	0.10
AR1254	0.50	17	0.10	0.00033	0.10
AR1260	0.50	17	0.10	0.00033	0.10
AR1262	0.50	17	0.10	0.00033	0.10
AR1268	0.50	17	0.10	0.00033	0.10

[1]The routine RL is the unadjusted value that can be achieved in a blank matrix.
[2]The RL for tissue matrix is project defined.
[3]RL based on a collection volume of 1m^3 or 1,000L
[4]RL based on a collection volume of 300m^3 or 300,000L

Table 1A: Routine Accuracy and Precision Limits[1]

Analyte	In-House Limits (%R)			Precision (RPD) (≤)
	Water	Solid	Air	
AR1016	55-120	55-120	65-125	30
AR1260	60-125	55-125	65-125	30
Surrogate: Decachlorobiphenyl (DCB)	30-150	45-125	60-120	NA
Surrogate:TCX (Advisory)[2]	55-120	30-130	60-120	NA

[1] The limits in this table are those used as of the effective date of this SOP. Current limits are stored in the LIMS database.
[2] The control limits for TCX (Tetrachloro-m-xylene) are advisory. Corrective action is not performed when recovery is outside limits.

Table 2: Primary Materials Used

Material[1]	Hazards	Exposure Limit[2]	Signs and symptoms of exposure
Hexane	Flammable Irritant	500 ppm-TWA	Inhalation of vapors irritates the respiratory tract. Overexposure may cause lightheadedness, nausea, headache, and blurred vision. Vapors may cause irritation to the skin and eyes.

[1] Always add acid to water to prevent violent reactions.
[2] Exposure limit refers to the OSHA regulatory exposure limit.

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Table 3: QC Summary, Frequency, Acceptance Criteria and Recommended Corrective Action (8082A)

QC Item	Frequency	Acceptance Criteria	Recommended Corrective Action[1]
ICAL	Before sample analysis, when CCVs indicate calibration is no longer valid; after major instrument maintenance	RSD for each analyte ≤ 20% -OR- Linear Regression: r ≥ 0.995	Correct problem, reanalyze, and repeat calibration.
ICV	After each initial calibration	(% R) ± 20% from expected value	Correct problem and verify second source standard. If that fails, repeat initial calibration.
CCV	8082:Daily before sample analysis, every 20 field samples or 12 hours (whichever is sooner). TO4/TO10:Daily before sample analysis, every 10 field samples.	% Difference or Drift ≥20%	See Section 10.3. Perform maintenance such as Injection Port maintenance, check for leaks, and change the lines / septa.
MB	One per extraction batch of 20 or fewer field samples	Target Analyte < RL	Examine project DQO's and take appropriate corrective action, which may include re-analysis of MB, re-extraction of batch, and/or non-conformance memo (NCM). If there are no detects in samples, or if all detects are > 10 X MB level, re-prep and reanalysis may not be required.
LCS	One per extraction batch of 20 or fewer field samples	See Table 1A	Examine project DQO's and take appropriate corrective action, which may include re-analysis of LCS, re-extraction of batch, and/or non-conformance memo (NCM). Flag all reported values outside of control limits
LCSD	One per extraction batch of 20 or field fewer samples, when MS/MSD is not present	See Table 1A	Examine project DQO's and take appropriate corrective action, which may include re-analysis of LCS, re-extraction of batch, and/or non-conformance memo (NCM). Flag all reported values outside of control limits.
MS/MSD	One per extraction batch of 20 or fewer field samples	See Table 1A	Evaluate data and determine if a matrix effect or analytical error is indicated. If analytical error, re-analyze and/or re-extract. Flag all reported values outside of control limits.
SD	Per client request	See Table 1A	Evaluate data and determine if a matrix effect or analytical error is indicated. If analytical error, re-analyze and/or re-extract. Flag all reported values outside of control limits.
Internal Standard	All field and QC samples	Area between 50-200% of area of daily calibration internal standard area	Evaluate data and determine if a matrix effect or analytical error is indicated. If analytical error, re-analyze and/or re-extract. Flag all reported values outside of control limits.
Surrogate	All field and QC samples	See Table 1A	Evaluate data and determine if a matrix effect or analytical error is indicated. If analytical error, re-analyze or re-extract. If matrix effect, review project DQOs to determine if a matrix effect must be confirmed by re-analysis. Flag all reported values outside of control limits.

[1]The recommended corrective action may include some or all of the items listed in this column. The corrective action taken may be dependent on project data quality objectives and/or analyst judgment but must be sufficient to ensure that results will be valid. If corrective action is not taken or is not successful, data must be flagged with appropriate qualifiers.

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Table 3A: QC Summary, Frequency, Acceptance Criteria and Recommended Corrective Action (EPA TO4 & TO10)

QC Item	Frequency	Acceptance Criteria	Recommended Corrective Action[1]
ICAL	Before sample analysis, when CCVs indicate calibration is no longer valid; after major instrument maintenance	Option 1: RSD for each analyte ≤ 20%- OR- Linear Regression: r ≥ 0.995	Correct problem, reanalyze, repeat calibration.
ICV	After each initial calibration	(% R) ± 15 % from expected value	Correct problem and verify second source standard. If that fails, repeat initial calibration.
CCV	Daily before sample analysis, every 10 field samples or 12 hours (whichever is sooner).	% Difference or Drift ±15%	Re-analyze once, if still outside criteria perform corrective action, sequence can be re-started if two successive CCVs pass, otherwise repeat ICAL and all associated samples since last successful CCV, unless CCV is high and bracketed samples are non-detects.
Method Blank	One per extraction batch of 20 or fewer field samples	Target Analyte < RL	Examine project DQO's and take appropriate corrective action, which may include re-analysis of MB, re-extraction of batch, and/or NCM. Corrective action must be documented in an NCM. If there are no detects in samples, or if all detects are > 10 X MB level, re-prep and reanalysis may not be required.
Field Blank	One per sampling event	Target Analyte < RL	Notify client.
LCS	One per extraction batch of 20 or fewer field samples	See Table 1A	Examine project DQO's and take appropriate corrective action, which may include re-analysis of LCS, re-extraction of batch, and/or non-conformance memo (NCM). Flag all reported values outside of control limits
LCSD	One per extraction batch of 20 or fewer field samples, when MS/MSD is not present	See Table 1A	Examine project DQO's and take appropriate corrective action, which may include re-analysis of LCS, re-extraction of batch, and/or non-conformance memo (NCM). Flag all reported values outside of control limits.
Field Spike	One per sampling event	%R (65-125)	Evaluate data and determine if a matrix effect or analytical error is indicated. If analytical error, re-analyze and/or re-extract.
Internal Standard	All field and QC samples	Area between 50-200% of area of daily calibration internal standard area	Evaluate data and determine if a matrix effect or analytical error is indicated. If analytical error, re-analyze and/or re-extract. Flag all reported values outside of control limits.
Surrogate	All field and QC samples	%R (60-120)	Evaluate data and determine if a matrix effect or analytical error is indicated. If analytical error, re-analyze or re-extract. If matrix effect, review project DQOs to determine if a matrix effect must be confirmed by re-analysis. Flag all reported values outside of control limits.

[1]*The recommended corrective action may include some or all of the items listed in this column. The corrective action taken may be dependent on project data quality objectives and/or analyst judgment but must be sufficient to ensure that results will be valid. If corrective action is not taken or is not successful, data must be flagged with appropriate qualifiers.*

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Appendix A: Terms and Definitions

Acceptance Criteria: specified limits placed on characteristics of an item, process or service defined in requirement documents.

Accuracy: the degree of agreement between an observed value and an accepted reference value. Accuracy includes a combination of random error (precision) and systematic error (bias) components which are due to sampling and analytical operations; a data quality indicator.

Analyte: The specific chemicals or components for which a sample is analyzed. (EPA Risk Assessment Guide for Superfund, OSHA Glossary).

Batch: environmental samples that are prepared and/or analyzed together with the same process, using the same lot(s) of reagents. A preparation/digestion batch is composed of one to 20 environmental samples of similar matrix, meeting the above criteria. An analytical batch is composed of prepared environmental samples (extracts, digestates and concentrates), which are analyzed together as a group.

Calibration: a set of operations that establish, under specified conditions, the relationship between values of quantities indicated by a measuring instrument or measuring system, or values represented by a material measure or a reference material and the corresponding values realized by the standards.

Calibration Curve: the graphical relationship between the known values or a series of calibration standards and their instrument response.

Calibration Standard: A substance or reference used to calibrate an instrument.

Continuing Calibration Verification (CCV): a single or multi-parameter calibration standard used to verify the stability of the method over time. Usually from the same source as the calibration curve.

Corrective Action: the action taken to eliminate the cause of an existing nonconformity, defect or other undesirable occurrence in order to prevent recurrence.

Data Qualifier: a letter designation or symbol appended to an analytical result used to convey information to the data user. (Laboratory)

Demonstration of Capability (DOC): procedure to establish the ability to generate acceptable accuracy and precision.

Holding Time: the maximum time that a sample may be held before preparation and/or analysis as promulgated by regulation or as specified in a test method.

Initial Calibration: Analysis of analytical standards for a series of different specified concentrations used to define the quantitative response, linearity and dynamic range of the instrument to target analytes.

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Intermediate Standard: a solution made from one or more stock standards at a concentration between the stock and working standard. Intermediate standards may be certified stock standard solutions purchased from a vendor and are also known as secondary standards.

Laboratory Control Sample (LCS): a blank matrix spiked with a known amount of analyte(s) processed simultaneously with and under the same conditions as samples through all steps of the procedure.

Matrix Spike (MS): a field sample to which a known amount of target analyte(s) is added.

Matrix Spike Duplicate (MSD): a second replicate matrix spike

Method Blank (MB): a blank matrix processed simultaneously with and under the same conditions as samples through all steps of the procedure. Also known as the preparation blank (PB).

Method Detection Limit (MDL): the minimum amount of a substance that can be measured with a specified degree of confidence that the amount is greater than zero using a specific measurement system. The MDL is a statistical estimation at a specified confidence interval of the concentration at which relative uncertainty is ±100%. The MDL represents a range where qualitative detection occurs. Quantitative results are only produced in this range and qualified with the proper data reporting flag when a project requires this type of data reporting.

Non-conformance: an indication, judgment, or state of not having met the requirements of the relevant specification, contract or regulation.

Precision: the degree to which a set of observations or measurements of the same property, obtained under similar conditions, conform to themselves.

Preservation: refrigeration and/or reagents added at the time of sample collection to maintain the chemical, physical, and/or biological integrity of the sample.

Quality Control Sample (QC): a sample used to assess the performance of all or a portion of the measurement system.

Reporting Limit (RL): the level to which data is reported for a specific test method and/or sample.

Stock Standard: a solution made with one or more neat standards usually with a high concentration. Also known as a primary standard. Stock standards may be certified solutions purchased from a vendor.

Surrogate: a substance with properties that mimic the analyte of interest but that are unlikely to be found in environmental samples.

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Appendix B: Standard Preparation Tables

The standard formulations contained in this Appendix are recommended and are subject to change. If the concentration of the stock standard is different than those noted in this table, adjust the standard preparation formulation accordingly. Unless otherwise specified, prepare the standard solutions in hexane using Class A volumetric glassware and Hamilton syringes. Unless otherwise specified for a standard solution, assign an expiration date of 1 year from date of preparation unless the parent standard expires sooner in which case use the earliest expiration date. Store the prepared solutions under refrigeration at a temperature of 4ºC (±2) and protect from light. See laboratory SOP BR-QA-002 *Traceability, Handling, & Storage of Standards & Reagents* for further guidance.

The standards presented below are prepared for the calibration of 8082A to satisfy DoD calibration requirements. For Non-DoD sample analysis pairs of Aroclors can be combined by spiking the defined volume into the defined Final Volume. The following Aroclors can be combined into a single calibration; 1221 & 1254, 1232 & 1260, 1242 & 1262, 1248 & 1268. Aroclor 1242 is prepared by itself.

Intermediate Calibration Standards (10 mg/L)

Parent Standard	Vendor	Component	Stock Standard Concentration (mg/L)	Volume Added (mL)	Final Volume (mL)	Final Concentration (mg/L)
AR1660[1]	Restek #32039	Aroclor 1016 Aroclor 1260	1000	0.40	40	10
AR1254	Restek #32011	Aroclor 1254	1000	0.40	40	10
AR1248	Restek #32010	Aroclor 1248	1000	0.40	40	10
AR1242	Restek #32009	Aroclor 1242	1000	0.40	40	10
AR1232	Restek #32008	Aroclor 1232	1000	0.40	40	10
AR1221	Restek #32007	Aroclor 1221	1000	0.40	40	10
AR1262	Restek #32409	Aroclor 1262	1000	0.40	40	10
AR1268	Restek #32410	Aroclor 1268	1000	0.40	40	10

[1] Standard is a mix of AR1016/AR1260. Concentration shown is the concentration of each Aroclor in the mixed standard.

Intermediate ICV Standard (10 mg/L)

Parent Standard	Vendor	Component	Stock Standard Concentration (mg/L)	Volume Added (mL)	Final Volume (mL)	Final Concentration (mg/L)
AR1660	Restek	Aroclor 1016 Aroclor 1260	1000	0.40	40	10

Surrogate Solution (10 mg/L)

Parent Standard	Vendor	Component	Stock Standard Concentration (mg/L)	Volume Added (mL)	Final Volume (mL)	Final Concentration (mg/L)
Pesticide Surrogate	Restek #3200	TCX DCB	1000	0.40	40	10

Working ICV Standard (200 ug/L)

Parent Standard	Vendor	Component	Parent Standard Concentration (mg/L)	Volume Added (mL)	Final Volume (mL)	Final Concentration (ug/L)
Intermediate ICV	Laboratory Prepared	Aroclor 1016 Aroclor 1260	10	0.80	40	200
Surrogate	Laboratory Prepared	TCX DCB	10	0.080		20

AR1660 Calibration Standard: CAL Level 5 (800 ug/L)[1]

Parent Standard	Vendor	Component	Parent Standard Concentration (mg/L)	Volume Added (mL)	Final Volume (mL)	Final Concentration (ug/L)
AR1660 Intermediate	Laboratory Prepared	Aroclor 1016 Aroclor 1260	10	8.0	100	800
Surrogate	Laboratory Prepared	TCX DCB	10	0.80		80

[1] This standard is the parent standard for each level of the AR1660 calibration standards

AR1660 Calibration Standard(s): CAL Levels 1- 4

Parent Standard	Calibration Standard	Parent Standard Concentration (ug/L)	Volume Added (mL)	Final Volume (mL)	Final Concentration (ug/L)
AR1660 Level 5	AR1660 CAL Level 4	800	20	40	400
AR1660 Level 5	AR1660 CAL Level 3	800	10	40	200
AR1660 Level 5	AR1660 CAL Level 2	800	5.0	40	100
AR1660 Level 5	AR1660 CAL Level 1	800	2.5	40	50

AR1221 Calibration Standard: CAL Level 5 (800 ug/L)[1]

Parent Standard	Vendor	Component	Parent Standard Concentration (mg/L)	Volume Added (mL)	Final Volume (mL)	Final Concentration (ug/L)
AR1221 Intermediate	Laboratory Prepared	Aroclor 1221	10	8.0	100	800
Surrogate	Laboratory Prepared	TCX DCB	10	0.80		80

[1] This standard is the parent standard for each level of the AR1221 calibration standards

AR1221 Calibration Standard(s): CAL Levels 1- 4

Parent Standard	Calibration Standard	Parent Standard Concentration (ug/L)	Volume Added (mL)	Final Volume (mL)	Final Concentration (ug/L)
AR1221 Level 5	AR1221 CAL Level 4	800	20	40	400
AR1221 Level 5	AR1221 CAL Level 3	800	10	40	200
AR1221 Level 5	AR1221 CAL Level 2	800	5.0	40	100
AR1221 Level 5	AR1221 CAL Level 1	800	2.5	40	50

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AR1232 Calibration Standard: CAL Level 5 (800 ug/L)[1]

Parent Standard	Vendor	Component	Parent Standard Concentration (mg/L)	Volume Added (mL)	Final Volume (mL)	Final Concentration (ug/L)
AR1232 Intermediate	Laboratory Prepared	Aroclor 1232	10	8.0	100	800
Surrogate	Laboratory Prepared	TCX DCB	10	0.80		80

[1] This standard is the parent standard for each level of the AR1232 calibration standards

AR1232 Calibration Standard(s): CAL Levels 1- 4

Parent Standard	Calibration Standard	Parent Standard Concentration (ug/L)	Volume Added (mL)	Final Volume (mL)	Final Concentration (ug/L)
AR1232 Level 5	AR1232 CAL Level 4	800	20	40	400
AR1232 Level 5	AR1232 CAL Level 3	800	10	40	200
AR1232 Level 5	AR1232 CAL Level 2	800	5.0	40	100
AR1232 Level 5	AR1232 CAL Level 1	800	2.5	40	50

AR1242 Calibration Standard: CAL Level 5 (800 ug/L)[1]

Parent Standard	Vendor	Component	Parent Standard Concentration (mg/L)	Volume Added (mL)	Final Volume (mL)	Final Concentration (ug/L)
AR1242 Intermediate	Laboratory Prepared	Aroclor 1242	10	8.0	100	800
Surrogate	Laboratory Prepared	TCX DCB	10	0.80		80

[1] This standard is the parent standard for each level of the AR1242 calibration standards

AR1242 Calibration Standard(s): CAL Levels 1- 4

Parent Standard	Calibration Standard	Parent Standard Concentration (ug/L)	Volume Added (mL)	Final Volume (mL)	Final Concentration (ug/L)
AR1242 Level 5	AR1242 CAL Level 4	800	20	40	400
AR1242 Level 5	AR1242 CAL Level 3	800	10	40	200
AR1242 Level 5	AR1242 CAL Level 2	800	5.0	40	100
AR1242 Level 5	AR1242 CAL Level 1	800	2.5	40	50

AR1248 Calibration Standard: CAL Level 5 (800 ug/L)[1]

Parent Standard	Vendor	Component	Parent Standard Concentration (mg/L)	Volume Added (mL)	Final Volume (mL)	Final Concentration (ug/L)
AR1248 Intermediate	Laboratory Prepared	Aroclor 1248	10	8.0	100	800
Surrogate	Laboratory Prepared	TCX DCB	10	0.80		80

[1] This standard is the parent standard for each level of the AR1248 calibration standards

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AR1248 Calibration Standard(s): CAL Levels 1- 4

Parent Standard	Calibration Standard	Parent Standard Concentration (ug/L)	Volume Added (mL)	Final Volume (mL)	Final Concentration (ug/L)
AR1248 Level 5	AR1248 CAL Level 4	800	20	40	400
AR1248 Level 5	AR1248 CAL Level 3	800	10	40	200
AR1248 Level 5	AR1248 CAL Level 2	800	5.0	40	100
AR1248 Level 5	AR1248 CAL Level 1	800	2.5	40	50

AR1254 Calibration Standard: CAL Level 5 (800 ug/L)[1]

Parent Standard	Vendor	Component	Parent Standard Concentration (mg/L)	Volume Added (mL)	Final Volume (mL)	Final Concentration (ug/L)
AR1254 Intermediate	Laboratory Prepared	Aroclor 1254	10	8.0	100	800
Surrogate	Laboratory Prepared	TCX DCB	10	0.80		80

[1] This standard is the parent standard for each level of the AR1254 calibration standards

AR1254 Calibration Standard(s): CAL Levels 1- 4

Parent Standard	Calibration Standard	Parent Standard Concentration (ug/L)	Volume Added (mL)	Final Volume (mL)	Final Concentration (ug/L)
AR1254 Level 5	AR1254 CAL Level 4	800	20	40	400
AR1254 Level 5	AR1254 CAL Level 3	800	10	40	200
AR1254 Level 5	AR1254 CAL Level 2	800	5.0	40	100
AR1254 Level 5	AR1254 CAL Level 1	800	2.5	40	50

AR1262 Calibration Standard: CAL Level 5 (800 ug/L)[1]

Parent Standard	Vendor	Component	Parent Standard Concentration (mg/L)	Volume Added (mL)	Final Volume (mL)	Final Concentration (ug/L)
AR1262 Intermediate	Laboratory Prepared	Aroclor 1262	10	8.0	100	800
Surrogate	Laboratory Prepared	TCX DCB	10	0.80		80

[1] This standard is the parent standard for each level of the AR1262 calibration standards

AR1262 Calibration Standard(s): CAL Levels 1- 4

Parent Standard	Calibration Standard	Parent Standard Concentration (ug/L)	Volume Added (mL)	Final Volume (mL)	Final Concentration (ug/L)
AR1262 Level 5	AR1262 CAL Level 4	800	20	40	400
AR1262 Level 5	AR1262 CAL Level 3	800	10	40	200
AR1262 Level 5	AR1262 CAL Level 2	800	5.0	40	100
AR1262 Level 5	AR1262 CAL Level 1	800	2.5	40	50

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AR1268 Calibration Standard: CAL Level 5 (800 ug/L)[1]

Parent Standard	Vendor	Component	Parent Standard Concentration (mg/L)	Volume Added (mL)	Final Volume (mL)	Final Concentration (ug/L)
AR1268 Intermediate	Laboratory Prepared	Aroclor 1268	10	8.0	100	800
Surrogate	Laboratory Prepared	TCX DCB	10	0.80		80

[1] *This standard is the parent standard fo each level of the AR1268 calibration standards*

AR1268 Calibration Standard(s): CAL Levels 1- 4

Parent Standard	Calibration Standard	Parent Standard Concentration (ug/L)	Volume Added (mL)	Final Volume (mL)	Final Concentration (ug/L)
AR1268 Level 5	AR1268 CAL Level 4	800	20	40	400
AR1268 Level 5	AR1268 CAL Level 3	800	10	40	200
AR1268 Level 5	AR1268 CAL Level 2	800	5.0	40	100
AR1268 Level 5	AR1268 CAL Level 1	800	2.5	40	50

Internal Standard Solution (500 ug/L)

Parent Standard	Vendor	Component	Parent Standard Concentration (mg/L)	Volume Added (uL)	Final Volume (mL)	Final Concentration (ug/L)
1-Bromo-2-nitrobenzene Stock	Restek p/n 32279	1-Bromo-2-nitrobenzene	1000	20	40	500

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Appendix C: Equations

Relative Response Factor Calculation

$$RRF = \frac{A_x}{A_{is}} \times \frac{C_{is}}{C_x}$$

Where:
AX =Peak height for the Aroclor peak to be measured.
Ais = Peak height for specified internal standard.
Cis = Amount of the internal standard injected (ng).
Cx = Amount of the target compound or DMC injected (ng).

Mean Relative Response Factor Calculation

$$\overline{RRF} = \frac{\sum_{i=1}^{n} RRF_i}{n}$$

Where:
n = The number of calibration standards

Percent Relative Standard Deviation Calculation

$$\%RSD = \frac{Standard\ Deviation}{Mean} \times 100$$

$$Standard\ Deviation = \sqrt{\frac{\sum_{i=1}^{n} \left(x_i - \overline{x}\right)^2}{n-1}}$$

Where:
xi = Each individual value used to calculate the mean
$\overline{x}$ = The mean of n values
n = The total number of values

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Relative Response Factor Percent Difference Calculation

$$\%Difference_{RRF} = \frac{RRF_C - \overline{RRF_i}}{\overline{RRFi}} x100$$

Where:

$\overline{RRF_i}$ = Mean Relative Response Factor from the most recent initial calibration meeting
technical acceptance criteria
RRFc = Relative Response Factor from CCV standard.

Percent Drift = Calculated Concentration – Theoretical Concentration X 100%
Theoretical Concentration

Percent Recovery

$$\%R = \frac{C_s}{C_n} \times 100\%$$

Cs = Concentration of the Spiked Field or QC Sample
Cn = Nominal Concentration of Spike Added

Percent Recovery (%R) for MS/MSD

$$\%R = \frac{C_s - C_u}{C_n} \times 100\%$$

Cs = Concentration of the Spiked Sample
Cu = Concentration of the Unspiked Sample
Cn = Nominal Concentration of Spike Added

Relative Percent Difference

$$RPD = \frac{|C_1 - C_2|}{\left(\frac{C_1 + C_2}{2}\right)} \times 100\%$$

C1 = Measured Concentration of First Sample
C2 = Measured Concentration of Second Sample

Sample Concentration
Note: The concentrations of the 3-5 peaks chosen for quantification, determined by peak height, are calculated and the average is then taken for final calculation.

Concentration of Water Samples

$$Concentration\ ug/L = \frac{(A_x)(I_s)(V_t)(DF)(GPC)}{(A_{is})(\overline{RRF})(V_o)(V_i)}$$

Where:
Ax = Peak height for the peak to be measured.
Ais = Peak height for the internal standard.
Is = Amount of internal standard injected in nanograms (ng).
Vo = Volume of water extracted in milliliters (mL).
Vi = Volume of extract injected in microliters (µL).
Vt = Volume of the concentrated extract in microliters (µL)
$\overline{RRF}$ = Mean Relative response factor determined from the initial calibration standard.
DF = Dilution Factor (If no dilution is performed, DF = 1.0)
GPC = GPC Factor (If no GPC cleanup is performed, GPC = 1.0)

Concentration of Soil and Sediment Samples

$$Concentration\ ug/Kg\ (Dry\ Weight\ Basis) = \frac{(A_x)(I_s)(V_t)(DF)(GPC)}{(A_{is})(\overline{RRF})(V_i)(W_s)(D)}$$

Where:
Ax = Peak height for the peak to be measured.
Ais = Peak height for the internal standard.
Is = Amount of internal standard injected in nanograms (ng).
Ws = Weight of sample extracted in grams (g)
Vi = Volume of extract injected in microliters (µL).
Vt = Volume of the concentrated extract in microliters (µL)
$\overline{RRF}$ = Mean Relative response factor determined from the initial calibration standard.
DF = Dilution Factor (If no dilution is performed, DF = 1.0)
D = $\frac{100 - \%Moisture}{100}$
GPC = GPC Factor (If no GPC cleanup is performed, GPC = 1.0)

Concentration of Air Samples (ug/PUF)

$$\text{Concentration ug/PUF} = \frac{(A_x)(I_s)(1PUF)(DF)}{(A_{is})(\overline{RRF})(V_o)(V_i)}$$

Where:
Ax = Peak height for the peak to be measured.
Ais = Peak height for the internal standard.
Is = Amount of internal standard injected in nanograms (ng).
Vo = Volume of water extracted in milliliters (mL).
Vi = Volume of extract injected in microliters (µL).
Vt = Volume of the concentrated extract in microliters (µL)
$\overline{RRF}$ = Mean Relative response factor determined from the initial calibration standard.
DF = Dilution Factor (If no dilution is performed, DF = 1.0)

Concentration of Air Samples (ug/m3)

$$\text{Concentration ug/m3} = \frac{(A_x)(I_s)(V_t)(DF)}{(A_{is})(\overline{RRF})(V_o)(V_i)}$$

Where:
Ax = Peak height for the peak to be measured.
Ais = Peak height for the internal standard.
Is = Amount of internal standard injected in nanograms (ng).
Vo = Volume of air sampled via PUF apparatus (m3).
Vi = Volume of extract injected in microliters (µL).
Vt = Volume of the concentrated extract in microliters (µL)
$\overline{RRF}$ = Mean Relative response factor determined from the initial calibration standard.
DF = Dilution Factor (If no dilution is performed, DF = 1.0)

Resolution

(Height of Valley / Average of the Two Peak Heights)*100%

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Title: Particle Size Analysis (ASTM D422-63, D6913-17, and D7928-17)

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1.0 Scope and Application

This SOP describes the laboratory procedure for the determination of particle size distribution in soils.

2.0 Summary of Method

A portion of sample is soaked in a dispersing agent then partitioned into separate portions: material retained on a #10 sieve and material passing the #10 sieve. The material retained on the #10 sieve is dried to constant weight then passed through a large size sieve stack; the material retained on each sieve is measured and recorded. Material passing the #10 sieve is subject to hydrometer analysis then passed through a small size sieve stack; the material retained on each sieve is measured and recorded. All measurements: large and small sieves and hydrometer readings, and the hygroscopic moisture are used to establish the particle size distribution of the sample.

This SOP is based on the following reference methods:

- ASTM Standard D2217-8585 (Re-approved 1998) "Standard Practice for Wet Preparation of Soil Samples for Particle-Size Analysis and Determination of Soil Constants", ASTM International, West Conshohocken, PA 2003, DOI: 10.1520/C0033-03, www.astm.org

- ASTM Standard D422-63 (Re-approved 2007) "Standard Test Method for Particle-Size Analysis of Soils", ASTM International, West Conshohocken, PA 2003, DOI: 10.1520/C0033-03, www.astm.org

- ASTM Standard D6913-17 "Standard Test Methods for Particle-Size Distribution (Gradation) of Soils Using SieveAnalysis"

- ASTM Standard D7928-17 "Standard Test Method for Particle-Size Distribution (Gradation) of Fine-Grained SoilsUsing the Sedimentation (Hydrometer) Analysis"

NOTE: ASTM D2217 was withdrawn without replacement by ASTM in 2007. A withdrawn standard is an ASTM standard that has been discontinued by the ASTM Sponsoring Committee responsible for the standard.

NOTE: ASTM D422-63 was withdrawn by ASTM in January 2016. Individual methods for gradation by sieve analysis (ASTM D6913-17) and gradation by sedimentation/hydrometer analysis (ASTM D7928-17) were promulgated as replacement approaches. At the time of this writing, ASTM has not yet developed a combined sieve/hydrometer approach. The laboratory's procedure will utilize elements of these two new methods into a combined approach, effectively equivalent to ASTM D422.

If the laboratory has modified the procedure from the reference method(s) a list of modifications will be provided in Section 16.0.

3.0 Definitions

Not Applicable

4.0 Interferences

Not Applicable

5.0 Safety

Employees must abide by the policies and procedures in the NDSC Eurofins TestAmerica Laboratories Environmental Health and Safety Manual (CW-E-M-001), and this document. This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

5.1 Specific Safety Concerns or Requirements

None

5.2 Primary Materials Used

There are no materials with a health rating of 3 or 4 used in this procedure. A complete list of materials used in the method can be found in Section 7.0. If a chemical material is listed, employees must review the information in the SDS for each material before using it for the first time or when there are major changes to the SDS.

6.0 Equipment and Supplies

Catalog numbers listed in this SOP are subject to change at the discretion of the vendor. Analysts are cautioned to be sure equipment used meets the specification of this SOP.

- Top-Loading Balance, capable of weight measurement to 0.01 g
- Mechanical Stirring Device and Dispersion Cup
- Thermometer: Accurate to 0.5°C
- Mortar and Rubber Tipped Pestle
- Sedimentation Cylinder(s) 1000 mL
- Hydrometer: ASTM 151H in specification E 100.
- Sieves, of the following size(s): Gilson Company, Inc. or equivalent
  3.0” (75.00 mm)
  2.0” (50.00 mm)
  1.5” (37.50 mm)
  1.0” (25.00 mm)
  3/4" (19.00 mm)
  3/8” (9.50 mm)
  # 4 (4.75 mm)
  #10 (2.00 mm)
  #20 (850.0 um)
  #40 (425 um)
  #60 (250.0 um)
  #80 (180.0 um)
  #100 (150.0 um)

#200 (75.0 um)
- Drying Oven with temperature range of 60-110°C
- Stainless Steel Spatulas & Spoons
- Metal & Bristle Brushes
- Ro-Tap Sieve Shaker, W. S. Tyler or equivalent.
- Timing Device with second hand and capable of counting up to 25 hours

7.0 Reagents and Standards

- Reverse Osmosis (RO) water: In-House System
- Sodium Hexametaphosphate: ELE International or equivalent.

Sodium Hexametaphosphate Solution: Add 120 g of sodium hexametaphosphate and 2940 g of reagent water to a 1-gallon bottle. Add a stir rod to the container and place on a stir plate. Mix the solution until it is homogeneous. Assign an expiration date of 30 days from the date made unless the parent reagent expires sooner, in which case use the earlier expiration date. Store the prepared solution at ambient temperature.

8.0 Sample Collection, Preservation, Shipment and Storage

The laboratory does not perform sample collection, so these procedures are not included in this SOP. Sampling requirements may be found in the published reference method.

Sample container, preservation techniques and holding times may vary and are dependent on sample matrix, method of choice, regulatory compliance, and/or specific contract or client requests. Listed below are the holding times and the references that include preservation requirements.

Matrix	Sample Container	Minimum Sample Size	Preservation	Holding Time	Reference
Solid	Glass Jar w/ Teflon Lid	500 g	None	None	ASTM D422-63

Unless otherwise specified by client or regulatory program, after analysis, samples are retained for a minimum of 30 days after provision of the project report and then disposed of in accordance with applicable regulations.

9.0 Quality Control

Not Applicable

10.0 Procedure

10.1 Equipment Calibration

Check the calibration of the balance prior to use, using at least two Class S weights that bracket the range of use. Record in the logbook designated for this purpose.

Check the temperature of the drying oven(s) each day of use, prior to use. Record in the logbook designated for this purpose.

NOTE: The QA Manager or their designee checks the calibration of liquid in glass thermometers annually against a NIST-traceable thermometer following the procedures given in laboratory SOP BR-QA-004. Electronic / digital thermometers that are battery-operated are checked quarterly using the same procedure.

Calibrate the hydrometers every two years following the procedure given in BR-QA-012.

Calibrate the sieves six months following the procedure given in BR-QA-012.

10.2 Hygroscopic Moisture Determination

Label an aluminum pan with the Lab ID for each sample. Tare the balance, weigh each pan, and record the weight measurement in spreadsheet FSL024.

Mix the sample with a stainless steel spatula. Measure at least 10-15 g of each sample into the labeled aluminum pan and record the weight of sample in the spreadsheet.

Place the pan + sample in an oven maintained at a temperature of 110ºC and dry the sample for at least 16 hours. Reweigh each pan and record the weight measurement in the spreadsheet.

Percent solids are calculated using the equation given in Section 12.0.

10.3 Sample Preparation

Use the calculated percent solids and the sample characteristics for each sample to determine the amount needed for analysis. The target mass for dry clay samples is ~60g of material, and for dry sandy/gravel material the target mass is ~125g. For very wet samples (lower %Solids), use a larger sample mass. See Table 2 for guidance on sample masses. If there is an insufficient amount of sample available, initiate a nonconformance memo (NCM) and contact the PM for further instruction.

Place a 1000 mL plastic beaker on the balance and enter the weight measurement in the worksheet as the tare weight. Weigh the amount of sample for analysis and record the weight in the bench sheet.

Add 125 mL of sodium hexametaphosphate solution to each beaker. Stir to mix and soak the sample in this solution for 16 hours

10.4 Sample Partition

Rinse the sample slurry into a dispersion vessel using reagent water. Fill the dispersion vessel ½ full with reagent water and disaggregate well using a combination of agitation and homogenization.

NOTE: If the sample is not amenable to disaggregation, initiate a NCM to notify the PM of the anomaly and proceed to the next step without dispersing the sample.

Place a #10 sieve in a funnel above a 1000 mL graduated cylinder. Pour the sample through the sieve. Rinse the dispersion vessel with reagent water and pour the rinse through the sieve. Repeat until transfer is complete. Bring the volume in the graduated cylinder to 1000 mL with

reagent water. Cover the cylinder with a rubber stopper and equilibrate the sample to ambient temperature in preparation for hydrometer analysis.

Label a medium size aluminum dish with the sample's LAB ID, then transfer the sample material that was retained on the #10 sieve to the dish. Place the aluminum dish in the drying oven set at 110 ± 5° C and dry the sample material for at least 16 hours or until constant weight. Set aside for sieve analysis.

10.5 Hydrometer Analysis

Prepare a hydrometer rinse bath by adding 1000 mL of reagent water to a 1000 mL graduated cylinder.

Record the hydrometer ID and start time on the worksheet. Set the timer for the elapsed time and perform each task as listed in Table 1: Hydrometer Reading Table.

To shake the cylinder, rotate the flask up and down for one minute approximating at least 60 turns. One turn down and one turn up equals two turns.

To take a hydrometer reading, gently insert the hydrometer into the graduated cylinder and wait ~ 20 seconds. Read the hydrometer from the top of the meniscus to the nearest 0.0005. Enter the reading on the worksheet. After each reading, clean the hydrometer by twisting and dropping the hydrometer into the hydrometer rinse bath.

Insert a temperature probe into the cylinder to the same depth as the hydrometer reading. Read the temperature to the nearest 0.5°C and enter the temperature measurement on the worksheet. Rinse the temperature probe in the hydrometer rinse bath.

Repeat the above process taking hydrometer readings every 2, 5, 15, 30, 60, 250 and 1440 minutes as per Table 1 then proceed to small sieve analysis.

10.6 Sieve Analysis

Inspect the sample material in the aluminum pan and record a description of the non-soil material (e.g.- sticks, grass, wood, plastic), hardness of material, and shape of material in the worksheet.

Hardness qualifiers include hard, soft, and brittle. Shape qualifiers include well rounded, rounded, subrounded, subangular, and angular.

<u>Large Sieves</u>

Weigh the 3/4", 3/8", #4 and #10 sieves and enter the weight measurements in the worksheet as the tare weight.

Stack the sieves and then transfer the sample material from the aluminum dish to the sieve stack. If the sample material is less than 30 g, manually shake the sieve stack for 2 minutes. If the sample material is greater than 30 g, place the sieve stack into the Ro-tap machine and shake the sieve stack for 10 minutes.

Weigh each sieve and record these measurements in the worksheet.

Small Sieves

Quantitatively transfer the sample from the graduated cylinder to a #200 wet wash sieve. Ensure the entire sample has been transferred to the #200 wet wash sieve by rinsing the graduated cylinder several time with RO water. Using RO water, wash the sample through the #200 sieve until the water runs clear, then transfer the material retained on the sieve into a 250 mL glass beaker labeled with the sample's LAB ID.

Place the beaker in the drying oven and dry at a temperature of 110ºC for at least 16 hours. After 16 hours, remove the beaker from the oven and allow it to cool.

Gently mix the dried contents of the beaker with a rubber-tipped pestle to break up any soil aggregates that may have formed during the drying stage.

Tare the balance and weigh the sieve stack sized between #20 and #200 and record the tare weights.

Transfer the sample to the sieve stack and ensure complete transfer. Use hair or wire brushes to clean the beaker. Place the sieve stack on the RoTap machine and shake for ten minutes.

Weigh each sieve and record these measurements in the worksheet.

11.0 Corrective Action

When an out-of-control situation occurs, a Corrective Action Report (CAR) (NCM), etc., must be developed (see SOP BR-QA-016) and the analyst must use his/her best analytical judgment and available resources to determine the corrective action to be taken. The out-of-control situation may be caused by more than one variable. The analyst should seek the assistance of his/her immediate supervisor, QA manager or other experienced staff if they are uncertain of the cause of the out-of-control situation. The analysis must not be resumed until the source of the problem and an in-control status is re-established. All samples associated with the out-of-control situation must be reanalyzed after in-control status has been re-established or if authorization is received from the supervisor or QA Manager for release with data qualification.

12.0 Calculations / Data Reduction

12.1 Calculations

Sample Used (SU): Dry Preparation

$$SU = (pan + dry\ sample - pan) - (pan + non - soil\ material - pan) \otimes HMCF$$

Where:
HMCF = Hygroscopic moisture correction factor.

Sieve Analysis (Percent Finer = PF)

Large Sieves:

3 inch: PF = 100-100 (Sieve and Sample (3 inch) - Sieve (3 inch))/SU*

2 inch: PF = PF (3 inch) - 100(Sieve and Sample (2 inch) - Sieve (2 inch))/SU and so on through the #10 Sieve.*

Small Sieves:

#20: PF = PF(#10) - 100(mass passing #10/sample mass (Hyd))*(sieve and sample (#20) - sieve(#20))/sample used*

#40: PF = PF (#20) - 100(mass passing #10/sample mass (Hyd))*(sieve and sample (#40) - sieve (#40))/sample used and so on up through #10 sieve.*

Hydrometer Analysis

Particle size, Micron

1000*sqrt [930*viscosity/980*(SG-1))*(effective depth/time)]

Viscosity at sample temperature, poises
Effective Depth, cm = 16.29-264.5*(actual Hydrometer reading - 1) above equation for effective depth based on equation found with table 2 in method, in which 16.29 = 0.5*(14.0-67.0/27.8)+10.5 and 264.5 = (10.5-2.3)/0.031
Time, minutes = Time of hydrometer reading from beginning of sedimentation
Sqrt - square root
SG - Specific Gravity of soil
Viscosity - is the resistance of a liquid to flow
Percent Finer (PF):

PF = Constant*(actual hydrometer reading - hydrometer correction factor - 1)

Where:
Constant = (100,000/W)*SG/(SG-1)
W = (Total sample used *sample used for hydrometer analysis*HMCF)/Amount of total sample passing #10 sieve
Hydrometer Correction = slope*sample temperature + Intercept
Slope = ((low temp. reading -1)-(high temp. reading -1)/(low temp. - high temp.))
Intercept = (low temp. reading -1) - (low temp. * slope)

12.2 Data Reduction

12.2.1 Primary Data Review

Review project documents such as the Project Plan (PP), Project Memo or any other document/process used to communicate project requirements to ensure those project requirements were met. If project requirements were not met, immediately notify the project manager (PM) to determine an appropriate course of action.

Upload the batch information into LIMS and complete the batch editor and worksheet. Initiate NCMs for any anomalies observed during the preparation process. Set the status of the batch to first level review.

12.2.2 Secondary Data Review

Review project documents such as the Project Plan (PP), Project Memo or any other document/process used to communicate project requirements and verify those project requirements were met. If project requirements were not met, immediately notify the project manager (PM) to determine an appropriate course of action.

Check the batch editor and worksheet to verify the batch is complete and any outages are documented with an NCM along with the results of any corrective actions taken. Set the status of the batch to second level review. Review the deliverable and set the job to lab complete.
Sample results are reported from the laboratory's LIMS system using the formatter specified by the Project Manager.

13.0 Method Performance

13.1 Method Detection Limit Study (MDL)

Not applicable

13.2 Demonstration of Capabilities

All personnel are required to perform an initial demonstration of proficiency (IDOC) on the method they will be using for analysis prior to testing samples. On-going proficiency must be demonstrated when changes are made to the method. IDOCs and on-going proficiency demonstrations are conducted by analyzing a sample in quadruplicate. Use FQA053 to document results and verify if DOC meets acceptance criteria.

13.3 Training Requirements

The Group Leader is responsible for ensuring that this procedure is performed by an associate who has been properly trained in its use and has the required experience. A new analyst must be working under documented supervision prior to approval of the IDOC. Documentation that a new analyst is performing under supervision must be entered into the batch record (View Batch Information) until that analyst's IDOC has been approved by the QA Manager (or designee). See requirements for demonstration of analyst proficiency in SOP No. BR-QA-011.

14.0 Pollution Control

It is Eurofins TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage and reagent stability). Employees must abide by the policies in Section 13 of the NDSC Eurofins TestAmerica Laboratories Environmental Health and Safety Manual (CW-E-M-001) for "Waste Management and Pollution Prevention."

15.0 Waste Management

Waste management practices are conducted consistent with all applicable rules and regulations. Excess reagents, samples and method process wastes are disposed of in an accepted manner.

Waste description rules and land disposal restrictions are followed. Waste disposal procedures are incorporated by reference to BR-EH-001. The following waste streams are produced when this method is carried out.

- Solid Waste-Satellite Container: Solid Waste 5 Gallon Plastic Bucket (inside fume hood)
- Liquid Waste- 55 gallon poly drum

16.0 References / Cross-References

- ASTM Standard D2217-8585 (Re-approved 1998) "Standard Practice for Wet Preparation of Soil Samples for Particle-Size Analysis and Determination of Soil Constants", ASTM International, West Conshohocken, PA 2003, DOI: 10.1520/C0033-03, www.astm.org

- ASTM Standard D422-63 (Re-approved 2007) "Standard Test Method for Particle-Size Analysis of Soils", ASTM International, West Conshohocken, PA 2003, DOI: 10.1520/C0033-03, www.astm.org

- ASTM Standard D6913-17 "Standard Test Methods for Particle-Size Distribution (Gradation) of Soils Using SieveAnalysis"

- ASTM Standard D7928-17 "Standard Test Method for Particle-Size Distribution (Gradation) of Fine-Grained SoilsUsing the Sedimentation (Hydrometer) Analysis"

- Eurofins TestAmerica Corporate Safety Manual, current version.

- Laboratory SOPs as referenced, current version.

Note: ASTM D2217 was withdrawn without replacement by ASTM in 2007. A withdrawn standard is an ASTM standard that has been discontinued by the ASTM Sponsoring Committee responsible for the standard. ASTM D422-63 was withdrawn by ASTM in January 2016

Note: ASTM D422-63 was withdrawn by ASTM in January 2016. Individual methods for gradation by sieve analysis (ASTM D6913-17) and gradation by sedimentation/hydrometer analysis (ASTM D7928-17) were promulgated as replacement approaches. At the time of this writing, ASTM has not yet developed a combined sieve/hydrometer approach. The laboratory's procedure will utilize elements of these two new methods into a combined approach, effectively equivalent to ASTM D422.

17.0 Method Modifications

- The laboratory prepares samples for ASTM D422 using ASTM method D2217 rather then the suggested method ASTM D421.
- The laboratory does not use Apparatus A or B defined in ASTM D422-63 for dispersion of samples due to the difficulty that many samples present using apparatus A and the unavailability of apparatus B. Instead the laboratory manually disaggregates the samples.
- The laboratory does not provide for the hydrometer readings at 1 minute and 8 minutes that are referenced in ASTM D7928-17.

18.0 Attachments

- Table 1: Hydrometer Reading Table (For up to 12 Sedimentation Cylinders)
- Table 2: Percent Solids Table for Weight Determination for D422.

19.0 Revision History

BR-GT-006, Revision 9.0:
- Title Page: Updated approval signatures, Copyright info, Branding, and Dates.
- Section 2.0: Added references to ASTM D6913-17 and D7928-17
- Section 2.0: Added Note that ASTM D 422-63 was withdrawn by ASTM in January 2016
- Section 5.2: Removed not applicable and added guidance language required by ETA corporate policy.
- Section 11.0: Added section as required by ETA corporate policy.
- Section 12.2: Updated to current practice and remove use of QC checker.
- Section 13.0: Updated language and guidance as required by ETA corporate policy.
- Section 16.0: Added references to ASTM D6913-17 and D7928-17
- Section 16.0: Added Note that ASTM D 422-63 was withdrawn by ASTM in January 2016
- Section 17.0: Added the modification that the laboratory does not perform the hydrometer readings at 1 minute and 8 minutes as referenced in ASTM D7928-17.

BR-GT-006, Revision 8.1:
- Title Page: Updated approval signatures, Copyright info, branding, and Dates.
- Section 10.3: added guidance for visual evaluation of sample for mass to use.

Previous revisions are retained by the QA department

Table 1: Hydrometer Reading Table (For up to 12 Sedimentation Cylinders)

Elapsed Time (hr:min)	Task	Cyl. No.	Actual Time (min)	Elapsed Time (hr:min)	Task	Cyl. No.	Actual Time (min)
0:00	Shake	1		1:01	Read	10	5
0:01	**Place**	**1**		**1:02**	Shake	11	
0:01	**Shake**	**2**		**1:03**	Place	11	
0:02	Place	2		1:04	Read	9	15
0:03	Read	1	2	1:05	Read	11	2
0:04	Read	2	2	1:06	Read	7	31
0:06	Read	1	5	1:07	Read	3	58
0:07	Read	2	5	1:08	Read	11	5
0:08	Shake	3		**1:09**	Shake	12	
0:09	**Place**	**3**		**1:10**	Place	12	
0:09	**Shake**	**4**		1:11	Read	10	15
0:10	Place	4		1:12	Read	12	2
0:11	Read	3	2	1:13	Read	4	63
0:12	Read	4	2	1:14	Read	8	32
0:14	Read	3	5	1:15	Read	12	5
0:15	Read	4	5	1:18	Read	11	15
0:16	Read	1	15	1:19	Read	9	30
0:17	Read	2	15	1:21	Read	5	60
0:20	Shake	5		1:25	Read	12	15
0:21	Place	5		1:26	Read	10	30
0:23	Read	5	2	1:27	Read	6	59
0:24	Read	3	15	1:33	Read	11	30
0:25	Read	4	15	1:34	Read	7	59
0:26	Read	5	5	1:41	Read	12	31
0:27	Shake	6		1:42	Read	8	60
0:28	Place	6		1:52	Read	9	63
0:30	Read	6	2	1:53	Read	10	57
0:31	Read	1	30	2:06	Read	11	63
0:32	Read	2	30	2:07	Read	12	57
0:33	Read	6	5	4:17	Read	1	256
0:34	Shake	7		4:18	Read	2	256
0:35	Place	7		4:19	Read	3	250
0:36	Read	5	15	4:20	Read	4	250
0:37	Read	7	2	4:21	Read	5	240
0:38	Read	3	29	4:22	Read	6	234
0:39	Read	4	29	5:00	Read	7	265
0:40	Read	7	5	5:01	Read	8	259
0:41	Shake	8		5:02	Read	9	253
0:42	Place	8		5:03	Read	10	247
0:43	Read	6	15	5:04	Read	11	241
0:44	Read	8	2	5:05	Read	12	235
0:47	Read	8	5	24:01	Read	1	1440
0:48	Shake	9		24:02	Read	2	1440
0:49	Place	9		24:03	Read	3	1434
0:50	Read	7	15	24:04	Read	4	1434
0:51	Read	9	2	24:05	Read	5	1424
0:52	Read	5	31	24:06	Read	6	1418
0:54	Read	9	5	24:07	Read	7	1412
0:55	Shake	10		24:08	Read	8	1406
0:56	Place	10		24:09	Read	9	1400
0:57	Read	8	15	24:10	Read	10	1394
0:58	Read	10	2	24:11	Read	11	1388
0:59	Read	6	31	24:12	Read	12	1382
1:00	**Read**	**1**	**59**				
1:00	**Read**	**2**	**58**				

Source: Laboratory Prepared Reference Document

Table 2: Percent Solids Table for Weight Determination for D422.

Percent Solid Table

Quantities of sample (in grams) to be utilized in Wet method version of ASTM D854 and D422

% Sol	Spec Grav	Hydrometer Slt/Cl	Slt/Snd	Snd	Snd/Gr
	25	**50**	**75**	**100**	**200**
1	2500	5000	7500	10000	20000
2	1250	2500	3750	5000	10000
3	833	1667	2500	3333	6667
4	625	1250	1875	2500	5000
5	500	1000	1500	2000	4000
6	417	833	1250	1667	3333
7	357	714	1071	1429	2857
8	313	625	938	1250	2500
9	278	556	833	1111	2222
10	250	500	750	1000	2000
11	227	455	682	909	1818
12	208	417	625	833	1667
13	192	385	577	769	1538
14	179	357	536	714	1429
15	167	333	500	667	1333
16	156	313	469	625	1250
17	147	294	441	588	1176
18	139	278	417	556	1111
19	132	263	395	526	1053
20	125	250	375	500	1000
21	119	238	357	476	952
22	114	227	341	455	909
23	109	217	326	435	870
24	104	208	313	417	833
25	100	200	300	400	800
26	96	192	288	385	769
27	93	185	278	370	741
28	89	179	268	357	714
29	86	172	259	345	690
30	83	167	250	333	667
31	81	161	242	323	645
32	78	156	234	313	625
33	76	152	227	303	606
34	74	147	221	294	588
35	71	143	214	286	571
36	69	139	208	278	556
37	68	135	203	270	541
38	66	132	197	263	526
39	64	128	192	256	513
40	63	125	188	250	500
41	61	122	183	244	488
42	60	119	179	238	476
43	58	116	174	233	465
44	57	114	170	227	455
45	56	111	167	222	444
46	54	109	163	217	435
47	53	106	160	213	426
48	52	104	156	208	417
49	51	102	153	204	408
50	50	100	150	200	400

% Sol	Spec Grav	Hydrometer Slt/Cl	Slt/Snd	Snd	Snd/Gr
	25	**50**	**75**	**100**	**200**
51	49	98	147	196	392
52	48	96	144	192	385
53	47	94	142	189	377
54	46	93	139	185	370
55	45	91	136	182	364
56	45	89	134	179	357
57	44	88	132	175	351
58	43	86	129	172	345
59	42	85	127	169	339
60	42	83	125	167	333
61	41	82	123	164	328
62	40	81	121	161	323
63	40	79	119	159	317
64	39	78	117	156	313
65	38	77	115	154	308
66	38	76	114	152	303
67	37	75	112	149	299
68	37	74	110	147	294
69	36	72	109	145	290
70	36	71	107	143	286
71	35	70	106	141	282
72	35	69	104	139	278
73	34	68	103	137	274
74	34	68	101	135	270
75	33	67	100	133	267
76	33	66	99	132	263
77	32	65	97	130	260
78	32	64	96	128	256
79	32	63	95	127	253
80	31	63	94	125	250
81	31	62	93	123	247
82	30	61	91	122	244
83	30	60	90	120	241
84	30	60	89	119	238
85	29	59	88	118	235
86	29	58	87	116	233
87	29	57	86	115	230
88	28	57	85	114	227
89	28	56	84	112	225
90	28	56	83	111	222
91	27	55	82	110	220
92	27	54	82	109	217
93	27	54	81	108	215
94	27	53	80	106	213
95	26	53	79	105	211
96	26	52	78	104	208
97	26	52	77	103	206
98	26	51	77	102	204
99	25	51	76	101	202
100	25	50	75	100	200

POLYCHLORINATED BIPHENYLS (PCBs) BY GC/MS

(Methods: EPA 680)

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1.0 Scope and Application

This SOP gives the procedures for the determination of polychlorinated biphenyls (PCBs) by gas chromatography/mass spectrometry (GC/MS). PCBs are reported as homologues by the level of chlorination: monochlorobiphenyls, dichlorobiphenyls, trichlorobiphenyls, etc., up to decachlorobiphenyl.

1.1 Analytes, Matrix(s), and Reporting Limits

The routine matrices for this procedure are waters and soils; however, this procedure may be adapted to accommodate other matrices.

A complete target analyte list, the reporting limits (RL), the method detection limits (MDL), and the accuracy and precision criteria associated with this procedure are provided in the TALS Method Limit Groups (MLGs).

On occasion clients may request modifications to this SOP. These modifications are handled following the procedures outlined in Section xx in the Quality Assurance Manual.

2.0 Summary of Method

A measured volume or weight of sample is spiked with surrogate and extracted using an appropriate extraction procedure. The extract is concentrated to a volume of 1.0mL, and analyzed by GC/MS operated in the Selected Ion Monitoring Mode (SIM), using windows to monitor for the characteristic masses of the various PCB homologues. Qualitative identification of the target compounds in the extract is based on the presence of the peak within the SIM window and the mass ratio between the primary and confirmation ions. Quantitative analysis is performed using the internal standard technique. Results are reported as homologue groups (e.g., monochlorobiphenyls).

This SOP is based on EPA method 680.

3.0 Definitions

Refer to the Glossary Section of the Quality Assurance Manual (QAM) for a complete listing of applicable definitions and acronyms.

Congener – a member of a family or class of compounds such as polychlorinated biphenyls (PCBs). There are 209 congeners of PCBs

Homologue – a PCB isomer with the same level of chlorination; e.g., the monochlorobiphenyl isomers, the dichlorobiphenyl isomers, etc.

4.0 Interferences

4.1 Procedural Interferences

Interferences may be caused by contaminants in solvents, reagents, glassware, and other sample processing apparatus and can make identification and/or quantification of the target analytes difficult.

All sample collection containers are single-use disposable containers which limits the potential for contamination. All non-disposable labware must be scrupulously cleaned in accordance with BR-EX-017 Glassware Cleaning to ensure it is free from contaminants and does not contribute artifacts.

High purity reagents and solvents are used to help minimize interference problems. Acetone, hexane, and methylene chloride must be verified prior to use in accordance with the TestAmerica Solvent Lot Testing Program.

Instrument and/or method blanks are routinely used to demonstrate all reagents and apparatus are free from interferences under the conditions of the analysis.

4.2 Matrix Interferences

The sample may require dilution prior to analysis to reduce or eliminate the interferences.

Interfering contamination may occur when a sample containing low concentrations of analytes is analyzed immediately following a sample containing relatively high concentrations of analytes. As such, samples known to be clean should be analyzed first. To prevent carryover into subsequent samples, analysis of reagent blanks may be needed after the analysis of a sample containing high concentrations of analytes. Sulfuric Acid clean-up may be utilized.

5.0 Safety

Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual (CW-E-M-001) and this document. This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats, and closed-toe, nonabsorbent shoes are a minimum.

5.1 Specific Safety Concerns or Requirements

The toxicity or carcinogenicity of chemicals used in this method has not been precisely defined; each chemical should be treated as a potential health hazard, and exposure to these chemicals should be minimized.

Acetone is a flammable solvent. It can cause irritation to the respiratory tract. Overexposure can cause fatigue, lightheadedness, headache, dizziness, and blurred vision.

Hexane is a flammable solvent. It can cause irritation to the respiratory tract. Overexposure can cause fatigue, lightheadedness, headache, dizziness, and blurred vision.

Methylene chloride is a carcinogen and an irritant. It causes irritation to the respiratory tract and has a strong narcotic effect with symptoms of mental confusion, light-headedness, fatigue, nausea, vomiting, and headache. May be absorbed through the skin and can cause irritation and pain to the skin and eyes.

The gas chromatograph and mass spectrometer contain zones that have elevated temperatures. The analyst needs to be aware of the locations of those zones, and must cool them to room temperature prior to working on them.

The mass spectrometer is under deep vacuum. The mass spectrometer must be brought to atmospheric pressure prior to working on the source.

There are areas of high voltage in both the gas chromatograph and the mass spectrometer. Depending on the type of work involved, either turn the power to the instrument off, or disconnect it from its source of power.

5.2 Primary Materials Used

Table 1 lists materials used in this method which have a serious or significant hazard rating. **Note: This list does not include all materials used in the method. The table contains a**

summary of the primary hazards listed in the SDS for each of the materials listed in the table. A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the SDS for each material before using it for the first time or when there are major changes to the SDS.

6.0 Equipment and Supplies

CHROM and Chemstation software are used to acquire, store, reduce, and output mass spectral data. The software allows integration of the ion abundance of any specific ion between specified time or scan number limits, calculation of response factors as or construction of a linear regression calibration curve, calculation of response factor statistics (mean and standard deviation), and calculation of concentrations of analytes using either the calibration curve or the response factors.

GC/MS System with compatible data system, autosampler, splitless injector, and direct capillary interface.

- Gas chromatograph – Agilent 5890, 6890, 7890, or equivalent with compatible autosampler, split/splitless injector, and direct capillary interface.
- Mass spectrometer – Agilent 5973, 5975, or equivalent
- Recommended Capillary Column – Restek Rxi-5MS, 30m x 0.25mm ID, 0.5um film thickness. Equivalent columns can be used.

7.0 Reagents and Standards

7.1 Reagents

Hexane – reagent grade

Acetone – reagent grade

Methylene Chloride- reagent grade

7.2 Standards

Purchase stock standard solutions from commercial vendors and from these prepare calibration and working standards by diluting a known volume of stock standard in an appropriate solvent to the final volume needed to achieve the desired concentration. The recommended formulation for each standard used in this procedure is provided in Appendix A along with the recommended source materials, expiration dates and storage conditions.

8.0 Sample Collection, Preservation, Shipment, and Storage

The laboratory does not perform sample collection, so these procedures are not included in this SOP. Sampling requirements may be found in the published reference method. Sample container, preservation techniques and holding times may vary and are dependent on sample matrix, method of choice, regulatory compliance, and/or specific contract or client requests. Listed below are the holding times and the references that include preservation requirements.

Matrix	Sample Container	Minimum Sample Size	Preservation	Extract Holding Time	Reference
Water	Amber Glass	1 L	Chilled to 4°C	40 Days	USEPA 680
Solid	Glass	50 g	Chilled to 4°C	40 Days	USEPA 680

[1]Analytical holding time is determined from date of initiation of extraction.

Unless otherwise specified by client or regulatory program, after analysis, samples and extracts are retained for a minimum of 30 days after provision of the project report and then disposed of in accordance with applicable regulations.

9.0 Quality Control

9.1 Sample QC

The laboratory prepares the following quality control samples with each batch of samples.

QC Item	Frequency	Acceptance Criteria
Method Blank (MB)	1 in 20 or fewer samples	See Table 3
Laboratory Control Sample (LCS)	1 in 20 or fewer samples	See Table 3
Laboratory Control Sample Duplicate (LCSD)	1 in 20 or fewer samples, when volume is not available for MS/MSD	See Table 3
Matrix Spike(s) MS/MSD	1 in 20 or fewer samples	See Table 3
Sample Duplicate (SD)	Client Request	See Table 3

9.2 Instrument QC

The following instrument QC is performed:

QC Item	Frequency	Acceptance Criteria
Initial Calibration (ICAL)	Initially, when CCV fails and after major instrument maintenance	See Table 3
GC/MS Mass Calibration (Tune) and Ion Abundance Check (DFTPP)	To begin a calibration sequence or to open a new 12 hour analytical window for sample analysis	See Table 3
Second Source Verification (ICV)	Immediately after ICAL	See Table 3
Window Defining Solution (WDM)	Daily, following DFTPP	
Continuing Calibration Verification (CCV)	Daily, every 12 hour window before the analysis of samples	See Table 3

10.0 Procedure

10.1 Instrument Operating Conditions

The instrument conditions listed in this SOP are provided for guidance purposes. The actual conditions used by the laboratory may be slightly different from those listed here and must be documented in the instrument maintenance log, data system, and/or run log.

Instrument maintenance must be performed in accordance with Attachment 4 of this SOP.

The goal is to have maximum separation between the target compounds in the shortest run time while maintaining sufficient sensitivity to detect the target compounds at the MDL.

Instrument conditions may vary according to the sensitivity of each instrument. The following conditions are provided for guidance. The laboratory must document the conditions used for the analysis of SVOC by GC/MS.

Recommended Column:

Rxi-5MS 30m x 0.25mm ID, 0.50um film thickness or equivalent

Column Flow:

Approximately 1mL/min helium

GC Oven Temperatures:

Initial column temperature: 60°C for 1 minute

Column temperature program 1: 20°C per minute to 150°C, hold 1 minute

Column temperature program 2: 10°C per minute to 310°C, hold until DCB and 13C12-DCB elute

GC Injector Parameters:

Injector temperature: 280°C

Injector: splitless

Inlet purge time: 0.5 minutes

Injector liner: 4mm ID quartz or 4mm glass, deactivated

Sample injection volume: 1μL

Mass Spectrometer and Interface Parameters:

Mass spectrometer interface: 300°C

Mass spectrometer source temperature: 230°C

Mass range: SIM (see Table 5for ions to monitor)

Mass range for DFTPP analysis: 35-500amu at >1 scan per second.

10.2 Internal Standard (ISTD)

Prior to analysis, internal standards must be added to all standards, samples, and QC items. The concentration of the internal standard must be the same in all calibration samples, field samples, and QC samples: 0.75 ug/mL.

Chrysene-d10 is used as the internal standard unless matrix interferences are encountered. Alternatively, phenanthrene-d10 may be used. If phenanthrene-d10 must be used, the calibration must be re-evaluated and verified using the second internal standard. An NCM must be generated to denote this situation.

The response of the ISTD in the ICV/CCV must be within 30% of the response in the most recent CCV or within 50% of the mean response of the ISTD in the ICAL. If the response is outside of this range, the analysis of the CCV must be repeated and any samples associated with the CCV must also be re-analyzed. Repeated failure of the ISTD response will require re-calibration.

The response of the ISTD in the samples and batch QC items must be within 30% of the response of the opening CCV or within 50% of the mean response of the ISTDs in the ICAL.

If these criteria are not met, corrective action must be taken and the associated sample extracts re-analyzed.

10.3 Tune Evaluation (DFTPP)

A solution containing DFTPP (difluorotriphenyl phosphate) must be analyzed at the beginning of each 12-hour acquisition period as a check on the "tune" of the mass spectrometer. The DFTPP analysis is performed using scan analysis. The same tune parameters are used for the SIM analysis of the calibration standards and samples.

Meeting the DFTPP tuning criteria demonstrates that the instrument is measuring the proper masses in the proper ratios.

Inject a 1uL aliquot of the 10ng/uL DFTPP/column evaluation solution.

The spectrum of the DFTPP must meet the criteria listed in Table 4. Background subtraction must be straightforward, that is, no scan within the elution window of DFTPP may be subtracted from another scan within the elution window, and designed only to eliminate column bleed or instrumental background. Scans $\pm$ 2 scans from the apex can be evaluated for the DFTPP criteria. Consecutive scans within this range may be averaged to meet the criteria.

Note: The DFTPP analysis should be evaluated as to the relative size of the DFTPP peak under the m/z 198 profile. A benchmark area window should be established for each instrument and data system. Area outside of this window suggests instrumental problems such as a bad injection, clogged autosampler syringe, leaking injector, reduced or elevated detector sensitivity, improper electron multiplier voltage selection, wrong tune method or tune file selected for this analysis, PFTBA valve left open, etc.

If the DFTPP fails to meet the criteria, the instrument may require tuning (manually or automatically with PFTBA). Depending on the nature of the results from the DFTPP analysis, other corrective measures may include remaking the DFTPP standard, cleaning the mass spectrometer source, etc.

10.4 Window Defining Solution (WDM)

Analyze a 1uL aliquot of the window-defining solution in the scan mode from 7.35 amu to 500amu at >1 scan per second or in SIM mode using the quant ions identifying each chlorobiphenyl group. Use the same temperature program that will be used for the SIM analysis of ICAL, CCV, QC, samples and DFTPP.

Determine the retention times of the first and last eluting congeners at each level of chlorination. The quantitation and confirmation masses are Attachment 12.

Set the SIM parameters in the Chemstation method used for the analysis of ICAL, CCV, QC and samples as follows. Refer to Attachment 7 for the ion sets.

- Begin data acquisition with ion set #1 before the elution of PCB congener #1, the first eluting CL-1-PCB
- Stop the acquisition of ion set #1 and begin acquisition of ion set #2 approximately 10 seconds before the elution of PCB congener #104, the first eluting CL5-PCB.
- Stop the acquisition of ion set #2 and begin the acquisition of ion set #3 approximately 10 seconds after the elution of PCB congener #77, the last eluting CL4-PCB.
- Stop the acquisition of ion set #3 and begin the acquisition of ion set #4 approximately 10 seconds after the elution of 4,4'-DDT. (The retention time of 4,4'-DDT is determined from the scan analysis of the DFTPP solution that is analyzed at the beginning of each 12-hour clock.)

- Stop the acquisition of ion set #4 and begin the acquisition of ion set #5 approximately 10 seconds before the elution of PCB congener #208, the first eluting CL9-PCB.

10.5 Initial and Continuing Calibration

Calibrate the instrument prior to analysis using a minimum of 5 standards. The lowest level calibration standard must be at or below the reporting limit, and the remaining standards will define the working range of the analytical system. Note that a single PCB congener of each chlorination level is used for calibration and quantitation using the standards and criteria given in Section 10.6. Once the calibration has been established and verified with an ICV in accordance with Section 10.7, sample analysis may proceed.

Verify the calibration curve with a Continuing Calibration Verification (CCV) at the beginning and end of each 12 hour clock.

The initial calibration standard concentrations currently in use in the laboratory are listed in Appendix B. Refer to Section 7.2 for the standard preparation instructions.

10.6 Initial Calibration Criteria

Each peak utilized in the Initial calibration must meet identification criteria as listed in Section 11.0.

The preferred method of quantitation is the average response factor. The relative standard deviation (%RSD) of the calibration standards must be <20%.

In addition to meeting the calibration criteria, the following performance criteria must also be met for the mid-level standard.

1. mass abundance ratios of all calibration congeners within acceptance range (Refer to Attachment 8.)
2. baseline separation of PCB congener #87 from congeners #154 and #77, which may coelute.
3. signal-to-noise ratio of >=5 for decachlorobiphenyl ion 499 and chrysene-d12 ion 241
4. decachlorobiphenyl mass abundance: mass 500 >=70% but <=95% of mass 498

10.7 Second Source Initial Calibration Verification (ICV)

The calibration curve must be verified after the initial calibration is established, prior to any sample analyses, with a standard obtained from a second source.

The initial calibration verification standard concentration currently in use in the laboratory is equivalent to Level 3 of the ICAL. Refer to Appendix B for the standard preparation instructions.

The ICV must be within ±20% of the true value.

10.8 Continuing Calibration Verification (CCV)

The calibration must be verified at the beginning and end of each 12-hour clock with a mid-level standard.

The CCV standard concentration currently in use in the laboratory is equivalent to level 4 of the ICAL. Refer to Appendix B for the standard preparation instructions.

10.9 Instrument Blanks (ICB/CCB)

The instrument must be shown to be free from contamination by the analysis of calibration blanks. Instrument blanks are analyzed after each ICV or CCV.

Initial and continuing calibration blanks must be <1/2RL.

10.10 Surrogates

This procedure uses surrogates to evaluate the extraction process.
Tetrachloro-m-xylene and 13C12-Decachlorobiphenyl are used as the surrogates. 13C6-gamma-BHC (lindane) may also be used.

Prior to preparation, the surrogates are added to all samples and QC items. The concentration of the surrogate is the same in all field samples and QC samples. A concentration of 2.5ug/mL is used.

Recovery limits are listed in Table 3. If the percent recovery is outside of this range, the analysis of the sample must be repeated. Repeated failure of the surrogate percent recovery may indicate re-extraction is necessary.

10.11 Sample Analysis

Remove the extracts from the refrigerator and allow them to come to room temperature. Prior to analysis spike the ISTD and set up the instrument sequence.

The sample extract must be injected using the same injection volume used for the calibration standards. Samples that are known to be relatively clean should be analyzed first. Samples suspected of containing high concentrations should be analyzed last. Instrument blanks may be analyzed after suspected high concentration samples to allow the detector response to stabilize.

10.12 Example Analytical Sequence

Description	Comments
DFTPP	12-hour clock begins
WDM	Window Defining Mix
Initial Calibration	
ICV	Second Source
Blank	
Samples & Batch QC Items	
CCV (Capping)	Level 3 Not to exceed12 hours.
DFTPP	12-hour clock begins
WDM	Window Defining Mix
CCV	Level three
Samples & Batch QC Items	
CCV (Capping)	Level 3 Not to exceed12 hours.

11.0 Corrective Action

When an out-of-control situation occurs that is not delineated in this corrective action table or the corrective actions listed do not adequately address the circumstances, a Corrective Action Report (CAR) (NCM), etc., must be developed (see SOP BR-QA-016) and the analyst must use his/her best analytical judgment and available resources to determine the corrective action to be taken. The out-of-control situation may be caused by more than one variable. The analyst should seek the assistance of his/her immediate supervisor, QA manager or other experienced staff if they are uncertain of the cause of the out-of-control situation. The analysis must not be resumed until the source of the problem and an in-control status is re-established. All samples associated with the out-of-control situation must be reanalyzed after in-control status has been re-established or if authorization is received from the supervisor or QA Manager for release with data qualification.

12.0 Calculations / Data Reduction

12.1 Qualitative Identification

The default procedure will be to use only the quantitation and confirmation ions for identification and quantification of PCB congeners. Interference check ions will not be used routinely to evaluate peaks as PCB congeners unless specified in a client QAPP or agency requirement or the sample concentration is near a critical quantitation limit.

Examine the Selected Ion Current Profiles (SICP) for the internal standards. Confirm that the RT and response of the internal standards are within the acceptance criteria specified in the QC Summary. If the internal standard retention times have changed significantly or the peaks cannot be located, stop and analysis and correct the problem. Reanalyze any associated samples.

Evaluate the peaks for candidates to be identified as PCBs. A peak is tentatively identified as a PCB if:

- The peak falls within the retention time range bordered by the first and last eluting isomer of that chlorination level
- The quantitation and confirmation ions are present and the area ratios fall within the acceptance criteria in Table 5 and the peaks maximize within one scan of each other. Examine the data for the presence of a coeluting PCB of higher chlorination if both ions and the M-70 ions are present and the ratio does not fall within the acceptance limits.
- The calculated on-column amount must be greater then the MDL.
- At least one ion in the M-70 cluster must be present

Evaluate the internal standard and the surrogates to check for shifts in retention times and to evaluate for ISTD response and the surrogate recovery. The recovery criteria for surrogates are given in the TALS MLG.

Evaluate each peak that corresponds to a target compound. Observe the general appearance of the chromatogram for possible dilutions, matrix interferences, and the overall shapes of the peaks.

12.2 Quantitative Identification

The data system quantifies the concentration of the target compound based on the integrated abundance of the characteristic ion from the EICP using the equations given in Appendix B. If there is matrix interference with the primary ion, a secondary ion may be used for quantification by calculating a mean RF factor for that ion and using that ion to quantify the analyte in the sample. When secondary ion calculations are performed, initiate a nonconformance memo (NCM) to ensure the quantitation approach is reported in the project narrative.

This method reports the homologue group. As such, there may be multiple peaks identified that meet criteria. The individual peaks are summed to report a total for the homologue group.

12.3 Dilutions

Unless otherwise specified by a client QAPP, results from a single analysis are reported as long as the largest target analyte (when multiple analytes are present) is in the upper half of the calibration range.

For clients who require we provide lower detection limits, a general guide would be to report the dilution detailed above and one additional run at a dilution factor 1/10 of the dilution with the highest target in the upper half of the calibration curve. For example, if samples analyzed at a 1/50 dilution resulted in a target in the upper half of the calibration curve, the sample would be analyzed at a dilution factor of 1/5 to provide lower reporting limits.

Note: If samples are analyzed at a dilution factor of 1:6 or greater, report the surrogate result as 0%D, to denote a dilution above the Dilution Threshold. Control limits will not apply to samples analyzed at dilutions of 1:6 or greater.

12.4 Calculations

See Appendix B.

12.5 Troubleshooting

Troubleshooting: the following items can be checked in case of calibration, QC or instrument failures:

- Loss of sensitivity for higher boiling compounds and internal standards may be indicative of a leak at the inlet. Replace septa and/or re-tighten lower inlet connection.
- Carryover contamination may indicate empty rinse vials.

12.6 Data Review

12.7 Primary Review

Review the chromatography and quantitation in the data processing system to confirm quantitative and qualitative identification of each target analyte.

Upload the data files to TALS. Enter batch editor information and add the standards and reagents to the TALS batch. Review the results against acceptance criteria. If acceptance criteria are not met, make arrangements to perform corrective action.

Check the results of samples analyzed immediately after high concentration samples for signs of carry-over. Reanalyze the sample if carry over is suspected.

Dilute and reanalyze samples whose results exceed the calibration range. The diluted analysis should result in a determination within the upper half of the calibration curve.

Set results to primary, secondary, acceptable or rejected as appropriate.

Verify corrective action was taken for all results not within acceptance criteria. If corrective action is not taken, or was unsuccessful, record all instances where criteria are not met with a nonconformance memo (NCM). Be sure to provide explanation of your decision making in the internal comment section of the NCM. The internal comment section should list the reason the

NCM is suspected, which action (if any) was taken and why and the outcome of the action taken.

Review project documents such as the Project Plan (PP), Project Memo or any other document/process used to communicate project requirements to ensure those project requirements were met. If project requirements were not met, immediately notify the project manager (PM) to determine an appropriate course of action.

Set the batch to 1st level review.

12.8 Secondary Review

Review the project documents such as the Project Plan (PP), Project Memo or any other document/process used to communicate project requirements and verify project requirements were met. If project requirements were not met, immediately notify the project manager (PM) to determine an appropriate course of action.

Review the TALS batch editor to verify information is complete. Review the batch to verify that the procedures in this SOP were followed. If discrepancy is found, resolve the discrepancy and verify any modifications to the SOP are documented and approved.

Spot-check 15% of samples minimally in the batch to verify quantitative and qualitative identification.

If manual integrations were performed:

- Review each manual integration to verify that the integration is consistent and compliant with the requirements specified in corporate SOP CA-Q-S-002.
- Check to ensure an appropriate technical reason code is provided for each manual integration. Acceptable technical reason codes are provided in corporate SOP CA-Q-S-002.
- Generate a “before” and “after” chromatogram for every manual integration performed on an instrument performance check standard (Tune, ICAL, ICV, CCV), QC sample (MB, LCS) and for any manual integration performed on any surrogate or internal standard in any field sample.
- Generate the Manual Integration Summary Report. Document your review of manual integrations on the summary report and obtain any review signatures of integrations performed during secondary review as required.

  If the reviewer disagrees with the integration performed by the primary analyst, the secondary data reviewer should not change the integration. Instead, he/she should consult with the primary analyst that performed the integration and both the reviewer and the primary analyst should agree the integration should be changed. If consensus between the primary analyst and the peer reviewer cannot be achieved; both should consult with the Technical Manager or department management for resolution. Any changes to the integration should be performed by the primary analyst. If it is necessary for the secondary reviewer to perform the manual integration because the primary analyst is out of the office; the integration made by the peer reviewer must be reviewed by another peer reviewer or by department management to verify the integration was performed and documented in compliance to corporate SOP CA-Q-S-002. If the original analyst that performed the integration is out of the office, the data reviewer may consult with the Department Manager (DM), Department Supervisor (DS) or the Technical Manager (TM) to verify the change he/she thinks is needed is warranted and should be made.

Verify that the performance criteria for the QC items listed in Table 1 were met. If the results do not fall within the established limits verify that corrective actions were performed. If corrective action was not performed; verify the reason is provided and that the situation is properly documented with an NCM. Set samples to 2nd level review.

Run the QC checker and fix any problems found. Run and review the deliverable. Fix any problems found. When complete set the method chain to lab complete and forward any paperwork to report/project management.

12.9 Data Reporting

The report format, application of data qualifiers and creation of the data deliverable is performed by the LIMS using the formatter set by the project manager during log-in.

Records of electronic and hardcopy data are maintained as described in laboratory SOP BR-QA-014.

13.0 Method Performance

13.1 Method Detection Limit Study (MDL)

The method detection limit (MDL) is the lowest concentration that can be detected for a given analytical method and sample matrix with 99% confidence that the analyte is present. MDLs reflect a calculated (statistical) value determined under ideal laboratory conditions in a clean matrix, and may not be achievable in all environmental matrices. An initial method detection limit study is performed in accordance with SOP No. BR-QA-005. The laboratory maintains MDL studies for analyses performed; these are verified at least annually unless method or program requirements require a greater frequency.

13.2 Demonstration of Capabilities

All personnel are required to perform an initial demonstration of proficiency (IDOC) on the instrument they will be using for analysis prior to testing samples. On-going proficiency must be demonstrated annually. IDOCs and on-going proficiency demonstrations are conducted as follows.

13.2.1 Four aliquots of the QC check sample are analyzed using the same procedures used to analyze samples, including sample preparation. The concentration of the QC check sample can be equivalent to a mid- level calibration.

13.2.2 Calculate the average recovery and standard deviation of the recovery for each analyte of interest.

13.2.3 If any analyte does not meet the acceptance criteria, the test must be repeated. Only those analytes that did not meet criteria in the first test need to be evaluated. TNI 2016 requires consecutive passing results. Repeated failure for any analyte indicates the need for the laboratory to evaluate the analytical procedure and take corrective action.

13.2.4 Until the IDOC is approved by the QA Manager (or designee); the trainer and trainee must be identified in the batch record.

13.3 Training Requirements

The Group Leader is responsible for ensuring that this procedure is performed by an associate who has been properly trained in its use and has the required experience. A new analyst must be working under documented supervision prior to approval of the IDOC. Documentation that a new analyst is performing under supervision must be entered into the batch record (View Batch Information) until that analyst's IDOC has been approved by the QA Manager (or designee). See requirements for demonstration of analyst proficiency in SOP No. BR-QA-011.

14.0 Pollution Control

It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage and reagent stability). Employees must abide by the policies in Section 13 of the Environmental Health and Safety Manual (corporate SOP CW-E-M-001) for "Waste Management and Pollution Prevention."

15.0 Waste Management

Waste management practices are conducted consistent with all applicable rules and regulations. Excess reagents, samples and method process wastes are disposed of in an accepted manner. Waste description rules and land disposal restrictions are followed. Waste disposal procedures are incorporated by reference to BR-EH-001. The following waste streams are produced when this method is carried out.

- Vials containing sample extracts: Satellite container: 15 gallon bucket connected to a fume hood.
- Solvent Waste: Satellite container: 1 L glass bottle located in fume hood.

16.0 References / Cross-References

- TestAmerica Environmental Health and Safety Manual (CW-E-M-001)
- TestAmerica Burlington Addendum to the Environmental Health and Safety Manual
- *Method 680: Determination of Pesticides and PCBs in Water and Soils/Sediment by Gas Chromatography/Mass Spectrometry*. November 1985. Physical and Chemical Methods Branch, Environmental Monitoring and Support Laboratory, Office of Research and Development, USEPA, Cincinnati, OH

17.0 Method Modifications and Clarifications

- The reference method was written specifically for drinking water and source water samples; however, the laboratory may perform other types of water samples using this procedure.
- This SOP contains preparation and sample evaluation procedures for soils and tissues, which are not included in EPA Method 680. The holding times in use for soil(tissue?) samples (i.e., 14 days from collection to preparation and 40 days from preparation to analysis) have been adopted from EPA 8270 and associated prep methods.
- EPA SW-846 Update IV Chapter 4 re-defined the holding times for PCBs as "NONE". Additionally, the EPA Methods Update Rule re-defined holding times for PCBs as one year from collection to extraction and one year from extraction to analysis. Some states/agencies allow a 1 year holding time from collection to extraction, with 40 days extraction to analysis. Where allowed by the project, this extended holding time is incorporated by the laboratory via the use of HT-specific TALS methods.
- This procedure may be modified to analyze other matrices (e.g., waste samples) based on the needs of the client. This will need to be arranged by the Project Manager at the initiation of the project.
- Waste matrices are non-routine, and the laboratory is not certified for these matrices. The laboratory uses its routine soil RLs (converted for initial and final volumes, etc.), and soil QC limits to evaluate waste samples. Soil DOCs can be used to satisfy analyst demonstrations of capability for these types of non-routine matrices. Ottawa sand is used as the blank matrix for waste samples. The holding times in use for waste samples (i.e., one year from

collection to preparation and one year from preparation to analysis) have been adopted from the EPA Methods Update Rule (MUR).

- The method has been modified to include the use of a carbon-13 labeled analogue of decachlorobiphenyl (13C12-DCB) and tetrachloro-m-xylene as the surrogate in place of the labeled BHC and DDT compounds. 13C12-DCB can be subjected to the optional acid cleanup (the unlabeled analogue, decachlorobiphenyl, is routinely used in SW-846 Method 8082). A window-defining mix containing the first and last eluting isomers of each level of chlorination is used as an aid to establish and verify that the SIM windows are properly set.
- EPA Method 680 requires the analysis of a reagent blank (method blank) for each new batch of solvents or reagents. The laboratory only performs this with each batch of samples. EPA method 680 requires the analysis of a solvent blank (method blank without surrogates or internals) for each new batch of solvents or reagents in order to verify the solvent does not contain any impurities that could lead to “false positives”.
- EPA Method 680 uses both chrysene-d10 and phenanthrene-d10 as internal standards (and provides identification criteria for both). The laboratory only uses chrysene-d10 unless there is matrix interference.
- The default identification and quantitation procedure will be to use only the quantitation and confirmation ions for the PCB homologues. Interference check ions, as described in Section 11.2.3, will not be used routinely to evaluate peaks as PCB homologues unless specified in a client QAPP or agency requirement or the sample concentration is near a critical quantitation limit. Samples evaluated according to the default quantitation procedures may be slightly high biased.
- EPA Method 680 states that if samples are not going to be prepared within 72 hours the pH must be adjusted with NaOH or H2SO4 to a range of 5-9 and the preservation recorded (including volume used). The laboratory verifies sample pH prior to preparation, and adjusts the sample as needed into the 5-9 pH range at that time.

18.0 Tables and Appendices

Table 1: Primary Materials Used

Table 2: Sample QC Recovery Limits

Table 3: Routine QC Summary, Frequency, Acceptance Criteria and Recommended Corrective Action

Table 4: DFTPP Key Ions and Abundance Criteria

Table 5: Ions for SIM Acquisition

Table 6: Approximate Retention Times for PCB Isomer Groups and Calibration Congeners

Table 7: Corrections for Interference of PCB Containing Two Additional Chlorines

Table 8: First and Last Eluting Isomers

Table 9: Quantitation and Interference Check Ions

Table 10: Corrections for Interference of PCB Containing One Additional Chlorine

Appendix A: Standard Preparation

Appendix B: Calculations

19.0 Revision History

Revision 1:

- Updated cover page signatures, dates, copyright info, and branding
- Throughout: minor typographical corrections and formatting updates
- Throughout: Updated headers and sections to include corporate formatting and language.

Revision 0:

- New SOP

Table 1: Primary Materials Used

Material	Hazards	Exposure Limit[1]	Signs and Symptoms of Exposure
Acetone	Flammable	1000ppm TWA	Inhalation of vapors irritates the respiratory tract. May cause coughing, dizziness, dullness, and headache.
Hexane	Flammable Irritant	500ppm TWA	Inhalation of vapors irritates the respiratory tract. Overexposure may cause lightheadedness, nausea, headache, and blurred vision. Vapors may cause irritation to the skin and eyes.
Methylene Chloride	Carcinogen Irritant	25ppm TWA 125ppm STEL	Causes irritation to respiratory tract. Has a strong narcotic effect with symptoms of mental confusion, light-headedness, fatigue, nausea, vomiting and headache. Causes irritation, redness and pain to the skin and eyes. Prolonged contact can cause burns. Liquid degreases the skin. May be absorbed through skin.

[1]Exposure limit refers to the OSHA regulatory exposure limit.

Table 2: Sample QC Recovery Limits

Sample QC Recovery Limits				
Surrogate Recovery				
	Tetrachloro-m-xylene		**Decachlorobiphenyl-13C12**	
	Lower Limit (%)	**Upper Limit (%)**	**Lower Limit (%)**	**Upper Limit (%)**
Soil/Tissue	30	130	10	134
Water	30	130	25	113
Laboratory Control Sample and Matrix Spike Recovery and RPD Limits				
	Soil/Tissue		**Water**	
	Lower Limit (%)	**Upper Limit (%)**	**Lower Limit (%)**	**Upper Limit (%)**
Monochlorobiphenyls	30	130	42	130
Dichlorobiphenyls	30	130	49	130
Trichlorobiphenyls	30	130	51	130
Tetrachlorobiphenyls	40	140	54	130
Pentachlorobiphenyls	40	140	63	130
Hexachlorobiphenyls	40	140	62	130
Heptachlorobiphenyls	40	140	62	130
Octachlorobiphenyls	40	140	64	130
Nonachlorobiphenyls	30	130	70	195
Decachlorobiphenyl	30	130	59	130
RPD Limits:	50		40	

Table 3: Routine QC Summary, Frequency, Acceptance Criteria and Recommended Corrective Action

QC Item	Frequency	Criteria	Corrective Action
Clock Time	12 hours	Clock time starts with the injection of the DFTPP. Analysis of samples and QC items must conclude within expiration of clock time. Subsequent analysis requires new DFTPP.	Not applicable
Tune (DFTPP)	At beginning of each clock	See Table 4	Re-analyze, retune mass spectrometer
Initial Calibration (ICAL)	Upon instrument set-up, and after unsuccessful CCV	Minimum of 5 calibration points. Meets mid-Level Standard Performance Criteria. %RSD < 20%.	Correct problem and repeat calibration.
Performance Criteria	Each mid-level standard	Mass abundance ratios of all calibration congeners within acceptance range (see attachment XXX). Baseline separation of PCB congener #87 from congeners #154 and #77. Signal-to-noise ratio of >=5 for decachlorobiphenyl ion 499 and chrysene-d12 ion 241. Decachlorobiphenyl mass abundance: mass 500 >=70% but <=95% of mass 498	Recalibrate, performing maintenance as necessary.
Initial Calibration Verification (ICV)	After each ICAL	%D<20	Correct problem and verify second source standard. If that fails, repeat initial calibration.
Continuing Calibration Verification (CCV)	12 hours, following instrument tune and Window Defining Mix	%D<20%	Re-prepare standard and re-analyze once, if still outside criteria perform corrective action, sequence can be re-started if two successive CCVs at different concentrations pass, otherwise repeat ICAL and all associated samples since last successful CCV, unless CCV is high and samples are non-detects.
Instrument Blank (ICB/CCB)	After ICV and every CCV	<1/2RL	Terminate the analysis, correct problem and reanalyze the previous 10 samples.
Internal Standards (ISTD)	Spiked in all standards, samples, and batch QC items	Area ± 50% of ICAL or ± 30% of previous CCV	Evaluate chromatogram, spectra, and integrations. Reanalyzed extract to confirm. Perform instrument maintenance as necessary. if sufficient sample is available, initiate a Re-extract request.

Surrogate Compounds	All samples and batch QC item.	See Table 2	Evaluate chromatogram, spectra, and integrations; Reanalyze extracts(s); Re-extract and reanalyze if sufficient sample available
Method Blank (MB)	One per extraction batch	<1/2RL	Examine project DQO's and take appropriate corrective action, which may include re-analysis of MB, re-extraction of batch, and/or non-conformance report (NCR). Corrective action must be documented on NCR. If there are no detects in samples, or if all detects are > 10 X MB level, re-prep and reanalysis may not be required.
Laboratory Control Sample (LCS)	One per extraction batch	See Table 2	Examine project DQO's and take appropriate corrective action, which may include re-analysis of LCS, re-extraction of batch, and/or non-conformance memo (NCM). Corrective action must be documented on NCM. Flag all reported values outside of control limits.
Laboratory Control Sample Duplicate (LCSD)	One per extraction batch, when insufficient sample is provided for MS/MSD/SD	See Table 2	Examine project DQO's and take appropriate corrective action, which may include re-analysis of LCSD, re-extraction of batch, and/or non-conformance report (NCR). Corrective action must be documented on NCR. Flag all reported values outside of control limits.
Matrix Spike (MS)/Matrix Spike Duplicate (MSD)	One per extraction batch	See Table 2	Evaluate data and determine if a matrix effect or analytical error is indicated. If analytical error, re-analyze and/or re-extract. Flag all reported values outside of control limits.

Table 4: DFTPP Key Ions and Abundance Criteria

m/z	Ion Abundance Criteria
127	40-60% of mass 198
197	<1.0% of mass 198
198	Base peak, 100% relative abundance
199	5-9% of mass 198
275	10-30% of mass 198
365	>1% of mass 198
441	Present but less than mass 443
442	>40% of mass 198
443	17-23% of mass 442

Table 5: Ions for SIM Acquisition

ION Set 1 (a)	ION Set 2 (b)	ION Set 3 (c)	ION Set 4 (d)	ION Set5 (e)
152	186	248	240	356
153	188	249	241	358
186	220	254	288	360
187	222	256	290	390
188	254	288	322	392
189	255	290	324	394
190	256	322	326	424
220	258	323	356	425
221	288	324	357	426
222	289	326	358	428
224	290	328	360	430
255	292	357	362	432
256	294	358	391	462
258	323	360	392	464
290	324	362	394	466
292	326	392	396	496
294	328	394	398	498
	358	396	428	499
	360	398	430	500
	362		432	502
(a) Cl-1 to Cl-4 and Phenthrene-d10				
(b) Cl-3 to-Cl-6				
(c) Cl5 to Cl-7				
(d) Cl-6 to Cl-8 and Chrysene-d12				
(e) Cl-8 to Cl-10 and $^{13}C_{12}$-DCB				

Table 6: Approximate Retention Times for PCB Isomer Groups and Calibration Congeners

PCB Isomer Group	Approximate RRT Range	Calibration Congener	Approximate Calibration Congener RRT
Cl-1	0.30-0.35	2-chlorobiphenyl (1)	0.30
Cl-2	0.38-0.50	2,3-dichlorobiphenyl (5)	0.43
Cl-3	0.46-0.64	2,4,5-trichlorobipheny l(29)	0.54
Cl-4	0.55-0.82	2,2',4,6-tetrachlorobiphenyl (50)	0.56
Cl-5	0.64-0.92	2,2',3,4,5'-pentachlorobiphenyl (87)	0.80
Cl-6	0.75-1.1	2,2',4,4',5,6'-hexachlorobipheny l(154)	0.82
Cl-7	0.88-1.2	2,2',3,4',5,6,6'-heptachlorobiphenyl (188)	0.88
Cl-8	0.99-1.21	2,2',3,3',4,5,,6,6'-octachlorobiphenyl (200)	1.03
Cl-9	0.16-1.28	Decachlorobiphenyl	1.3
Cl-10	1.3	Decachlorobiphenyl	1.3

RRT = retention time relative to Chrysene-d12

Table 7: Corrections for Interference of PCB Containing Two Additional Chlorines

PCB Isomer Group	Quant Ion	Confirmation Ion	Ion Measured to Determine Interference	Percent Ion area to be subtracted from quant ion area	Percent Ion area to be subtracted from confirmation ion area
Cl-3	256	258	254	99	33
Cl-4	292	290	288	65	131
Cl-5	326	324	322	108	164
Cl-6	360	362	356	161	71
Cl-7	394	396	390	225	123

Table 8: Corrections for Interference of PCB Containing One Additional Chlorine

PCB Isomer Group	Quant ION	Ion Measured to Determine Interference	Percent Ion area to be subtracted from QUNAT ION Area
Cl-2	222	221	13.5
Cl-3	256	255	13.5
Cl-4	292	289	17.4
Cl-5	326	323	22.0
Cl-6	360	357	26.5
Cl-7	394	391	30.9
Cl-8	430	425	40.0

Table 9: First and Last Eluting Isomers

Congener	First Eluting Isomer	Last Eluting Isomer
Cl-1	2-chlorobiphenyl	4-Chlorobiphenyl
Cl-2	2,6-dichlorobiphenyl	4,4'-dichlorobiphenyl
Cl-3	2,2',6-trichlorobiphenyl	3,4,4'-trichlorobiphenyl
Cl-4	2,2',6,6'-tetrachlorobiphenyl	3,3',4,4'-tetrachlorobiphenyl
Cl-5	2,2',4,6,6'-pentachlorobiphenyl	3,3',4,4',5-pentachlorobiphenyl
Cl-6	2,2',4,4',6,6'-hexachlorobiphenyl	3,3',4,4',5,5'-hexachlorobiphenyl
Cl-7	2,2',3,4',5,6,6'-heptachlorobiphenyl	2,3,3',4,4',5,5'-heptachlorobiphenyl
Cl-8	2,2',3,3',5,5',6,6'-octachlorobiphenyl	2,3,3',4,4',5,5',6-octachlorobiphenyl
Cl-9	2,2',3,3',4,5,5',6,6'-nonachlorobiphenyl	2,2',3,3',4,4',5,5',6-nonachlorobiphenyl

Table 10: Quantitation and Interference Check Ions

(a) ratio of quantitation ion to confirmation ion

PCB Isomer Group	Quant Ion	Confirmation Ion	Expected Ratio(a)	Acceptable Ratio(a)	M-70 Confirmation Ions	Interference Check Ions M+70	Interference Check Ion M+35
Cl-1	188	190	3.0	2.5-3.5	152, 153	256, 258	222, 224
Cl-2	222	224	1.5	1.3-1.7	152, 153, 186, 188	292, 290, 294	256, 258
Cl-3	256	258	1.0	0.8-1.2	186, 188	326, 324, 328	290, 292, 294
Cl-4	292	290	1.3	1.1-1.5	220, 222	360, 362	326, 324, 328
Cl-5	326	324	1.6	1.4-1.8	254, 256, 258	394, 392, 396, 398	360, 362
Cl-6	360	362	1.2	1.0-1.4	288, 290, 292	430, 426, 428, 432	394, 392, 396, 398
Cl-7	394	396	1.0	0.8-1.2	322, 324		430, 428, 432
Cl-8	430	428	1.1	0.9-1.3	356, 358, 360	498, 494, 496, 500	464, 462, 466
Cl-9	464	466	1.3	1.1-1.5	390, 392, 394		498, 496, 500
Cl-10	498	500	1.1	0.9-1.3	424, 426, 428, 430		
Chrysene-d12	240	241	5.1	4.3-5.9			
Phenathrene-d10	188	189	6.6	6.0-7.2			
$^{13}C_{12}$-DCB (surrogate)	510	512					

Appendix A: Standard Preparation Tables

The standard formulations contained in this Appendix are recommended and are subject to change. If the concentration of the stock standard is different than those noted in this table, adjust the standard preparation formulation accordingly. Depending on the extraction solvent, repare the standard solutions in methylene chloride or hexane using Class A volumetric glassware and Hamilton syringes. Unless otherwise specified for a standard solution, assign an expiration date of 1 year from date of preparation unless the parent standard expires sooner in which case use the earliest expiration date. See laboratory SOP BR-QA-002 *Reagents & Standards Handling* for further guidance.

Tune Stock					
Solution	Analytes	Stock Concentration (ug/mL)	Amount Added	Final Volume	Final Concentration (ug/mL)
Ultra Scientific GCM-150-1	4,4'-DDT	1000	400uL	16mL	25
	Benzidine				
	DFTPP				
	Pentachlorophenol				

680 Calibration Stock					
Solution	Analytes	Stock Concentration (ug/mL)	Amount Added	Final Volume	Final Concentration (ug/mL)
Ultra Scientific CB-681MN	Decachlorobiphenyl (BZ #209)	250	400uL	4mL	25
	Monochlorobiphenyls (BZ #1)	50			5
	Dichlorobiphenyls (BZ #5)	50			5
	Trichlorobiphenyls (BZ #29)	50			5
	Tetrachlorobiphenyls (BZ #50)	100			10
	Pentachlorobiphenyls (BZ #87)	100			10
	Hexachlorobiphenyls (BZ #154)	100			10
	Heptachlorobiphenyls (BZ #188)	150			15
	Octachlorobiphenyls (BZ #200)	150			15
Restek 32027	Tetrachloro-m-xylene	200	100uL		5
Cambridge Isotope Labs EC-1410-10	Decachlorobiphenyl-13C12	40	2500uL		25
Ultra Scientific CB682MN	Nonachlorobiphenyls (BZ #208)	200	400		20
	Tetrachlorobiphenyls (BZ #77)	100			10
	Pentachlorobiphenyls (BZ #104)	100			10

Internal Standard Stock					
Solution	Analytes	Stock Concentration (ug/mL)	Amount Added	Final Volume	Final Concentration (ug/mL)
Ultra Scientific ATS-120	Chrysene-d12	2000	37.5uL	4	
Ultra Scientific ITS-230	Phenanthrene-d10	1000	75uL		18.75

Window Defining Solution Stock (Use as prepared)					
Solution	Analytes	Stock Concentration (ug/mL)	Amount Added	Final Volume	Final Concentration (ug/mL)
Ultra Scientific RPCW-101	BZ #1	100	40uL	4mL	1
	BZ #3				
Ultra Scientific RPCW-102	BZ #10				
	BZ #15				
Ultra Scientific RPCW-103	BZ #19				
	BZ #37				
Ultra Scientific RPCW-104	BZ #54				
	BZ #77				
Ultra Scientific RPCW-105	BZ #104				
	BZ #1026				
Ultra Scientific RPCW-106	BZ #155				
	BZ #169				
Ultra Scientific RPCW-107	BZ #188				
	BZ #189				
Ultra Scientific RPCW-108	BZ #202				
	BZ #205				
Ultra Scientific RPCW-109	BZ #206				
	BZ #208				
Ultra Scientific RPCW-110	BZ #209 (DCB)				

CALIBRATION COMPONENTS	Cal Point (amount added to 100uL)					
CALIBRATION COMPONENTS	**CAL 1 (0.5uL)**	**CAL 2 (2uL)**	**CAL 3 (10uL)**	**CAL 4 (20uL)**	**CAL 5 (40uL)**	**CAL 6 (100uL)**
Calibration Congeners						
2-chlorobiphenyl (1)	0.025	0.1	0.5	1	2	5
2,3-dichlorobiphenyl (5)	0.025	0.1	0.5	1	2	5
2,4,5-trichlorobiphenyl (29)	0.025	0.1	0.5	1	2	5

2,2',4,6-tetrachlorobiphenyl (50)	0.05	0.2	1	2	4	10
2,2',3,4,5'-Pentachlorobiphenyl (87)	0.05	0.2	1	2	4	10
2,2',4,4',5,6'-hexachlorobiphenyl (154)	0.05	0.2	1	2	4	10
2,2',3,4',5,6,6'-heptachlorobiphenyl (188)	0.075	0.3	1.5	3	6	15
2,2',3,3',4,5,5',6,6'-octachlorobiphenyl (200)	0.075	0.3	1.5	3	6	15
Decachlorobiphenyl	0.125	0.5	2.5	5	10	25
Retention Time Congeners						
3,3',4,4-tetrachlorobiphenyl (77)				2		
2,2',4,6,6'-Pentachlorobiphenyl (104)				2		
2,2',3,3',4,5,5',6,6'-nonachlorobiphenyl (208)				2		
Surrogate						
$^{13}C_{12}$-Decachlorobiphenyl	0.2	0.5	2.5	5	10	25
Internal Standards						
Phenathrene-d10	0.75	0.75	0.75	0.75	0.75	0.75
Chrysene-d12	0.75	0.75	0.75	0.75	0.75	0.75

ICV Stock (used as prepared)					
Solution	Analytes	Stock Concentration (ug/mL)	Amount Added	Final Volume	Final Concentration (ug/mL)
AccuStandard M-680A	Monochlorobiphenyls (BZ #1)	50	80uL	4mL	1
	Dichlorobiphenyls (BZ #5)	50			1
	Trichlorobiphenyls (BZ #29)	50			1
	Tetrachlorobiphenyls (BZ #50)	100			4
	Pentachlorobiphenyls (BZ #87)	100			4
	Hexachlorobiphenyls (BZ #154)	100			2
	Heptachlorobiphenyls (BZ #188)	150			3
	Octachlorobiphenyls (BZ #200)	150			3
	Decachlorobiphenyl (BZ #209)	250			5
AccuStandard M-680-RT	Tetrachlorobiphenyls (BZ #77)	100	80uL		4
	Pentachlorobiphenyls (BZ #104)	100			4
	Nonabichlorophenyls (BZ #208)	200			8

Appendix B: Equations

Equation 1: Relative Response Factor Calculation

$$RRF = \frac{A_x}{A_{is}} \times \frac{C_{is}}{C_x}$$

Where:
A_X = Area of the characteristic ion for the compound to be measured (Table 6).
A_{is} = Area of the characteristic ion for specified internal standard (Table 5).
C_{is} = Amount of the internal standard injected (ng).
C_x = Amount of the target compound or DMC injected (ng).

Equation 2: Mean Relative Response Factor Calculation

$$\overline{RRF} = \frac{\sum_{i=1}^{n} RRF_i}{n}$$

Where:
n = The number of calibration standards

Equation 3: Percent Relative Standard Deviation Calculation

$$\%RSD = \frac{Standard\ Deviation}{Mean} \times 100$$

$$Standard\ Deviation = \sqrt{\frac{\sum_{i=1}^{n} \left(x_i - \bar{x}\right)^2}{n-1}}$$

Where:
x_i = Each individual value used to calculate the mean
$\bar{x}$ = The mean of n values
n = The total number of values

Equation 4: Relative Response Factor Percent Difference Calculation

$$\%Difference_{RRF} = \frac{RRF_C - \overline{RRF_i}}{\overline{RRFi}} x100$$

Where:
$\overline{RRF_i}$ = Mean Relative Response Factor from the most recent initial calibration meeting technical acceptance criteria
RRF_c = Relative Response Factor from CCV standard.

Equation 6: Percent Moisture Calculation

$$\%Moisture = \frac{grams\ of\ wet\ sample - grams\ of\ dry\ sample}{grams\ of\ wet\ sample} \times 100$$

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Equation 7: Relative Retention Time Calculation

$$Relative\,Retention\,Time(RRT) = \frac{Retention\,Time_{compound}}{Retention\,Time_{internal\ standard}}$$

Equation 8: GPC Factor Calculation

$$GPC\ Factor\ (GPC) = \frac{Volume_{in}}{Volume_{out}}$$

Equation 9: Concentration of Water Samples Calculation

$$Concentration\ ug/L = \frac{(A_x)(I_s)(V_t)(DF)(GPC)}{(A_{is})(\overline{RRF})(V_o)(V_i)}$$

Where:
A_x = Area of the characteristic ion for the compound to be measured.
A_{is} = Area of the characteristic ion for the internal standard.
I_s = Amount of internal standard injected in nanograms (ng).
V_o = Volume of water extracted in milliliters (mL).
V_i = Volume of extract injected in microliters (µL).
V_t = Volume of the concentrated extract in microliters (µL)
$\overline{RRF}$ = Mean Relative response factor determined from the initial calibration standard.
DF = Dilution Factor (If no dilution is performed, DF = 1.0)
GPC = GPC Factor (If no GPC cleanup is performed, GPC = 1.0)

Equation 10: Concentration of Soil and Sediment Samples Calculation

$$Concentration\ ug/Kg\ (Dry\ Weight\ Basis) = \frac{(A_x)(I_s)(V_t)(DF)(GPC)}{(A_{is})(\overline{RRF})(V_i)(W_s)(D)}$$

Where:
A_x = Area of the characteristic ion for the compound to be measured.
A_{is} = Area of the characteristic ion for the internal standard.
I_s = Amount of internal standard injected in nanograms (ng).
W_s = Weight of sample extracted in grams (g)
V_i = Volume of extract injected in microliters (µL).
V_t = Volume of the concentrated extract in microliters (µL)
$\overline{RRF}$ = Mean Relative response factor determined from the initial calibration standard.
DF = Dilution Factor (If no dilution is performed, DF = 1.0)
D = $\frac{100 - \%Moisture}{100}$
GPC = GPC Factor (If no GPC cleanup is performed, GPC = 1.0)

Equation 11: Adjusted LOQ Calculations for Water Samples

$$Adjusted\ LOQ = LOQ \times \frac{(V_x)(V_t)(DF)}{(V_o)(V_c)}$$

Where:
LOQ = The LOQ value reported in Table 1 (µg/L)
V_x = Routine sample volume (1000mL)
V_o = Volume of water extracted in milliliters (mL).
V_t = Volume of the concentrated extract in microliters (µL)
V_c = Routine concentrated extract volume (1000 µL)
DF = Dilution Factor (If no dilution is performed, DF = 1.0)

Equation 12: Adjusted LOQ Calculations for Soil and Sediment Samples

$$Adjusted\ LOQ = LOQ \times \frac{(W_x)(V_t)(DF)}{(W_s)(V_c)(D)}$$

Where:
LOQ = The LOQ value reported in Table 1 (µg/Kg)
W_x = Routine sample weight (40g)
W_s = Weight of sample extracted in grams (g)
V_c = Routine concentrated extract volume (2000 µL)
V_t = Volume of the concentrated extract in microliters (µL)
D = $\frac{100 - \%Moisture}{100}$
DF = Dilution Factor (If no dilution is performed, DF = 1.0)

Equation 13: LCS and Surrogate Percent Recovery Calculation

$$\%\ Recovery = \frac{SSR}{SA} \times 100$$

Where:
SSR = Spike Sample Result
SA = Spike Added

Equation 14: Matrix Spike Percent Recovery Calculation

$$\%\ Recovery = \frac{SSR - SR}{SA} \times 100$$

Where:
SSR = Spike Sample Result
SR = Sample Result
SA = Spike Added

Equation 16: Relative Percent Difference Calculation

$$RPD = \frac{|MSR - MSDR|}{\frac{1}{2}(MSR + MSDR)} \times 100$$

Where:

RPD = Relative Percent Difference
MSR = Matrix Spike Recovery
MSDR = Matrix Spike Duplicate Recovery

Quality Assurance Manual Cover Page

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Facility Distribution No. Electronic **Distributed To: Facility Intranet**

1.0 Title Page

Quality Assurance Manual
Approval Signatures



07/10/2020

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07/10/2020

Quality Assurance Manager – Kristine Dusablon | Date



07/10/2020

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3.0 INTRODUCTION, SCOPE AND APPLICABILITY

3.1 Introduction and Compliance References

Eurofins TestAmerica Burlington's Quality Assurance (QA) Manual is a document prepared to define the overall policies, organization objectives and functional responsibilities for achieving the laboratory's QA Program. Governing SOPS are in place within the organization to ensure the proper execution of this QA Manual (refer to Appendix 1). This manual and referenced documents are required reading for all personnel.

The laboratory is a team of people who work together to serve the health and environmental needs of society through science and technology. We offer comprehensive expertise in environmental laboratory applications and client relations and maintain a national perspective in terms of quality.

As such, this QA Manual has been prepared to assure compliance with The NELAC Institute (TNI) Standard, dated 2009 and 2016; ISO/IEC Guide 17025:2005 and 2017; and the Department of Defense (DoD)/Department of Energy (DOE) Quality Systems Manaual (QSM). Policies and procedures listed in Appendix 1 are compliant with the National Divisional Support Center (NDSC) Quality Management Plan (QMP) for Eurofins TestAmerica; Eurofins (define) and the various accreditation and certification programs which are held by the laboratory to support environmental work (Appendix 2). The QMP provides a summary of Eurofins TestAmerica's quality and data integrity system. It contains requirements and general guidelines under which all Eurofins TestAmerica facilities shall conduct their operations.

Refer to Appendix 3 for a list of additional references for which this QA Manual is compliant; and Appendix 4 for the compliance crosswalk of this manual to the TNI & ISO/IEC Guide 17025 requirements.

3.2 Terms and Definitions

A QA Program is a system designed to ensure that data produced by the laboratory conforms to the standards set by state and/or federal regulations. The program functions at the local management level through company goals, from guidance at the executive management level, and at the analytical level through Standard Operating Procedures (SOPs) and quality control. Our program is designed to minimize systematic error, encourage constructive, documented problem solving, and provide a framework for continuous improvement within the organization to better serve our clients.

Refer to Appendix 5 for a list of Terms and Acronyms.

3.3 Scope / Fields of Testing

The laboratory analyzes a broad range of environmental and industrial samples. Sample matrices vary among air, drinking water, effluent water, groundwater, hazardous waste, sludge and soils. The QA Program contains specific procedures and methods to test samples of differing matrices for chemical, physical and biological parameters. The Program also contains guidelines on maintaining documentation of analytical processes, reviewing results, servicing clients and tracking samples through the laboratory. The technical and service requirements of all analytical requests are thoroughly evaluated before commitments are made to accept the work. Measurements are made using published reference methods or methods developed and validated by the laboratory.

The methods covered by this manual include the most frequently requested methodologies needed to provide analytical services in the United States and its territories. The specific list of test methods used by the laboratory can be found in the Laboratory Information Management System (TALS). Our areas of expertise include:

Standard Services	Specialty Services
• Volatiles • Semivolatiles • Metals • Pesticides/PCBs • Petroleum Hydrocarbons • Waste Characterization • Non-Potable Water Testing • Drinking Water Testing • Soil and Surface Water Testing • Vapor and Air Analysis • Sediment and Tissue Testing • Method Development	• Perchlorate • 1,4-Dioxane • Perchlorate • PCB Congeners • Explosives • Alkyl PAHs, Alkanes, Biomarkers • PFAS • CLP methodologies

NOTE: **All current certificates and scopes of accreditation are available on the laboratory's website at www.testamericainc.com**

The approach of this manual is to define the minimum level of quality assurance and quality control necessary to meet these requirements. All methods performed by the laboratory shall meet these criteria as appropriate. In some instances, quality assurance project plans (QAPPs), project specific data quality objectives (DQOs) or local regulations may require criteria other than those contained in this manual. In these cases, the laboratory will abide by the requested criteria following review and acceptance of the requirements by the Laboratory President, Laboratory Vice-President, Director of Operations, Laboratory Director and and the Quality Assurance (QA) Manager. In some cases, QAPPs and DQOs may specify less stringent requirements. The Laboratory President, Laboratory Vice-President, Director of Operations, Laboratory Director and the QA Manager must determine if it is in the lab's best interest to follow the less stringent requirements.

3.4 Management of the Manual

3.4.1 Review Process

The template on which this manual is based is reviewed annually by Eurofins National Divisional Support Center (NDSC) which houses the Quality Assurance leadership team for Eurofins Environment Testing America. NDSC QA will assure that the template remains in compliance with Section 3.1. This manual itself is reviewed every two years and annually for DoD/DOE Lab, by senior laboratory management to assure that it reflects current practices and meets the requirements of the laboratory's clients and regulators as well as the QMP. Occasionally, the manual may need changes in order to meet new or changing regulations and operations. The QA Manager will review the changes in the normal course of business and incorporate changes into revised sections of the document. All updates will be reviewed by the senior laboratory management staff. The laboratory updates and approves such changes according to our Document Control & Updating procedures (refer to SOP BR-QA-003, Document Control).

4.0 MANAGEMENT REQUIREMENTS

4.1 Overview

The laboratory is a local operating unit of Eurofins Environment Testing America. The laboratory's operational and support staff have the day-to-day independent operational authority under the direction of the Laboratory President, Laboratory Vice-President, Director of Operations, Laboratory Director and is supported by the NDSC QA team. The laboratory management staff includes managers and group supervisors. The organizational chart of the management staff are presented in Figure 4-1. Individual departmental staff lists are maintained in the laboratory's internal intranet.

4.2 Roles and Responsibilities

In order for the QA Program to function properly, all members of the staff must clearly understand and meet their individual responsibilities as they relate to the quality program. The responsibility for quality resides with every employee of the laboratory. All employees have access to the QA Manual, are trained to this manual, and are responsible for upholding the standards therein. Each person carries out his/her daily tasks impartially and in a manner consistent with the goals and in accordance with the procedures in this manual and the laboratory's SOPs. The following descriptions briefly define each role in its relationship to the Quality Assurance Program.

4.2.1 Vice President of Quality and Environmental Health and Safety (VP-QA/EHS)

The Vice President (VP) of QA/EHS reports directly to Eurofins Environment Testing America Chief Operating Officer (COO). With the aid of the NDSC Quality Team Members, Business Unit Managers, Laboratory Directors, the VP-QA/EHS has the responsibility for the establishment, general overview and maintenance of the Quality Assurance and EH&S Programs within Eurofins Environment Testing America. Additional responsibilities include:

- Review of QA/QC and EHS aspects of NDSC Official Document national projects and expansions or changes in services.
- Work with various organizations outside of the laboratory to further the development of quality standards and represent the laboratory at various trade meetings.
- Prepare monthly reports for quality and EH&S metrics across the environmental testing laboratories and a summary of any quality and EH&S related initiatives and issues.
- With the assistance of the Executive Management, and the EHS Managers, maintenance and implementation of the Eurofins Environment Testing America Environmental, Health and Safety Program.

4.2.2 Quality Directors

There are four (4) Quality Directors within NDSC that report directly to the VP-QA/EHS. These Quality Directors have oversight of the general overview and maintenance of the QA Program within the Eurofins Environment Testing America laboratories. Supported tasks include:

- Monitors laboratory internal audit findings;
- Identifies common laboratory weaknesses and monitors corrective action closures.

- Develops NDSC quality guidance documents and management tools for ensuring and improving compliance;
- Monitors and communicates DoD/DoE requirements;
- Monitors and communicates regulatory and certification requirements;
- Training and OnBoarding
- Laboratory assessments, mentoring, and interventions
- Track/drive root cause investigations and corrective action plans
- Builds knowledge base for preventive actions

4.2.3 Quality Information Manager

The Quality Information works directly with the NDSC Quality Directors and EHS Managers; and reports directly to the VP-QA/EHS. The Quality Information Manager is responsible for the management of:

- NDSC Official Documents
- TALS/LIMS Certification Module Data
- Company's Intranet website
- Company's Regulatory Limits Database
- Subcontract laboratory and approved vendor information
- Internal and External client support for various company groups (e.g., Client Services, EH&S, Legal, IT, Sales) for both quality and operational functions
- Communicate regulatory information and lists

4.2.4 Environmental Health and Safety (EH&S) Managers

There are 3 EH&S Managers within NDSC that report directly to the VP-QA/EHS. These EH&S Managers have oversight of the general overview and maintenance of the EH&S Program within the Eurofins Environment Testing America laboratories. Supported tasks include:

- Consolidation and tracking all safety and health-related information and reports for the company, and managing compliance activities for Eurofins Environment Testing America locations.
- Coordination/preparation of the Environmental, Health and Safety Manual Template that is used by each laboratory to prepare its own laboratory-specific Safety Manual/ CHP.
- Preparation of information and training materials for laboratory EHS Coordinators.
- Assistance in the coordination of employee exposure and medical monitoring programs to insure compliance with applicable safety and health regulations.
- Serving as Department of Transportation (D.O.T.) focal point and providing technical assistance to location management.
- Serving as Hazardous Waste Management main contact and providing technical assistance to location management.

4.2.5 Ethics and Compliance Officers (ECOs)

The NDSC VP-QA/EHS and Corporate Counsel are designated Ethics and Compliance Officer (ECO). Each ECO acts as a back-up to the other ECO and both are involved when data investigations occur. Each ECO has a direct line of communication to the entire executive management personnel and lab management staff.

The ECOs monitor and audit procedures to determine compliance with policies and to make recommendations for policy enhancements to the President, COO, Laboratory Director or other appropriate individuals within the laboratory. The ECO will assist the laboratory QA Manager in the coordination of internal auditing of ethical policy related activities and processes within the laboratory, in conjunction with the laboratory's regular internal auditing function.

The ECOs will also participate in investigations of alleged violations of policies and work with the appropriate internal departments to investigate misconduct, remedy the situation, and prevent recurrence of any such activity.

4.2.6 Laboratory Director

The Laboratory Director is responsible for the overall quality, safety, financial, technical, human resource and service performance of the whole laboratory and reports to their business unit President. The Laboratory Director is also responsible for any service centers connected with their laboratory that perform analytical tests, such as short holding time analyses for pH. The Laboratory Director provides the resources necessary to implement and maintain an effective and comprehensive Quality Assurance and Data Integrity Program.

Specific responsibilities include, but are not limited to:

- Provides one or more technical managers for the appropriate fields of testing. If the Technical Manager is absent for a period of time exceeding 15 consecutive calendar days, the Laboratory Director must designate another full time staff member meeting the qualifications of the Technical Manager to temporarily perform this function. If the absence exceeds 35 consecutive calendar days, the primary accrediting authority must be notified in writing.
- Ensures that all analysts and supervisors have the appropriate education and training to properly carry out the duties assigned to them and ensures that this training has been documented. Works with Eurofins Environment Testing Human Resources for hiring of new personnel.
- Ensures that personnel are free from any commercial, financial and other undue pressures which might adversely affect the quality of their work.
- Ensures company human resource policies are adhered to and maintained.
- Ensures that sufficient numbers of qualified personnel are employed to supervise and perform the work of the laboratory. Assesses laboratory capacity and workload.
- Ensures that appropriate corrective actions are taken to address analyses identified as requiring such actions by internal and external performance or procedural audits. Procedures that do not meet the standards set forth in the QA Manual or laboratory SOPs may be temporarily suspended by the Laboratory Director.
- Reviews and approves all SOPs prior to their implementation and ensures all approved SOPs are implemented and adhered to.

- Pursues and maintains appropriate laboratory certification and contract approvals. Supports ISO 17025 requirements.
- Ensures client specific reporting and quality control requirements are met.
- Contributes to the continuous improvement of the laboratory operations.
- Maintains an awareness of technical developments and regulatory requirements.
- Captains the management team, consisting of the QA Manager, the Technical Manager, and the Operations Manager as direct reports.

4.2.7 Quality Assurance (QA) Manager or Designee

The QA Manager has responsibility and authority to ensure the continuous implementation of the quality system at the laboratory where they work. The QA Manager is also responsible for any service centers connected with their laboratory that perform analytical tests, such as short holding time analyses for pH.

The QA Manager reports directly to the Laboratory Director and their NDSC Quality Director. This position is able to evaluate data objectively and perform assessments without outside (e.g., managerial) influence. The NDSC QA Team may be used as a resource in dealing with regulatory requirements, certifications and other quality assurance related items. The QA Manager directs the activities of the QA office to accomplish specific responsibilities, which include, but are not limited to:

- Serves as the focal point for QA/QC in the laboratory.
- Have functions independent from laboratory operations for which he/she has quality assurance oversight.
- Have documented training and/or experience in QA/QC procedures and the laboratory's Quality System.
- Have a general knowledge of the analytical test methods for which data audit/review is performed (and/or having the means of getting this information when needed).
- Arrange for or conducting internal audits on quality systems and the technical operation
- Notifying laboratory management of deficiencies in the quality system and ensuring corrective action is taken. Procedures that do not meet the standards set forth in the QA Manual or laboratory SOPs shall be investigated following procedures outlined in Section 14 and if deemed necessary may be temporarily suspended during the investigation.
- Maintaining and updating the QA Manual.
- Monitoring and evaluating laboratory certifications; scheduling proficiency testing samples.
- Monitoring and communicating regulatory changes that may affect the laboratory to management.
- Training and advising the laboratory staff on quality assurance/quality control procedures that are pertinent to their daily activities.
- The laboratory QA Manager will maintain records of all ethics-related training, including the type and proof of attendance.
- Maintain, improve, and evaluate the corrective action database and the corrective and preventive action systems.

- Objectively monitor standards of performance in quality control and quality assurance without outside (e.g., managerial) influence.
- Coordinating of document control of SOPs, MDLs, control limits, and miscellaneous forms and information.
- Performing technical data audits and method audits to ensure consistency and compliance with regulatory requirements.
- Review of external audit reports and data validation requests.
- Follow-up with audits to ensure client QAPP requirements are met.
- Establishment of reporting schedule and preparation of various quality reports for the Laboratory Director, clients and/or NDSC QA Team.
- Development of suggestions and recommendations to improve quality systems.
- Research of current state and federal requirements and guidelines.
- Participate in the vendor and supplier approval process, including subcontractors.
- Captains the QA team to enable communication and to distribute duties and responsibilities.
- Communication to the relevant regulatory authorities when there are management or facility changes that impact the laboratory.
- Ensuring communication & monitoring standards of performance to ensure that systems are in place to produce the level of quality as defined in this document.
- Evaluation of the thoroughness and effectiveness of training.
- Compliance with ISO 17025
- Compliance with the DoD/DOE QSM**.** (where applicable)

4.2.8 Technical Manager or Designee

The Technical Manager report directly to the Laboratory Director. He/she is accountable for all analyses and analysts under their experienced supervision and for compliance with the ISO 17025:2017 Standard. The scope of responsibility ranges from the new-hire process and existing technology through the ongoing training and development programs for existing analysts and new instrumentation. Specific responsibilities include, but are not limited to:

- Exercises day-to-day supervision of laboratory operations for the appropriate field of accreditation and reporting of results. Coordinating, writing, and reviewing preparation of all test methods, i. e., SOPs, with regard to quality, integrity, regulatory and optimum and efficient production techniques, and subsequent analyst training and interpretation of the SOPs for implementation and unusual project samples. He/she insures that the SOPs are properly managed and adhered to at the bench. He/she develops standard costing of SOPs to include supplies, labor, overhead, and capacity (design vs. demonstrated versus first-run yield) utilization.
- Reviewing and approving, with input from the QA Manager, proposals from marketing, in accordance with an established procedure for the review of requests and contracts. This procedure addresses the adequate definition of methods to be used for analysis and any limitations, the laboratory's capability and resources, the client's expectations. Differences are resolved before the contract is signed and work begins. A system documenting any

significant changes is maintained, as well as pertinent discussions with the client regarding their requirements or the results of the analyses during the performance of the contract. All work subcontracted by the laboratory must be approved by the client. Any deviations from the contract must be disclosed to the client. Once the work has begun, any amendments to the contract must be discussed with the client and so documented.

- Monitoring the validity of the analyses performed and data generated in the laboratory. This activity begins with reviewing and supporting all new business contracts, insuring data quality, analyzing internal and external non-conformances to identify root cause issues and implementing the resulting corrective and preventive actions, facilitating the data review process (training, development, and accountability at the bench), and providing technical and troubleshooting expertise on routine and unusual or complex problems.
- Providing training and development programs to applicable laboratory staff as new hires and, subsequently, on a scheduled basis. Training includes instruction on calculations, instrumentation management to include troubleshooting and preventive maintenance.
- Enhancing efficiency and improving quality through technical advances and improved LIMS utilization. Capital forecasting and instrument life cycle planning for second generation methods and instruments as well as asset inventory management.
- Coordinating sample management from “cradle to grave,” insuring that no time is lost in locating samples.
- Scheduling all QA/QC-related requirements for compliance, e.g., MDLs, etc.
- Captains department personnel to communicate quality, technical, personnel, and instrumental issues for a consistent team approach.
- Coordinates audit responses with the QA Manager.
- Compliance with ISO 17025.
- Compliance with the DoD/DOE QSM. (where applicable)

4.2.9 Operations Manager

The Operations Manager manages and directs the analytical production sections of the laboratory. He/She reports directly to the Laboratory Director. He/she assists the Technical Manager in determining the most efficient instrument utilization. More specifically, he/she:

- Evaluates the level of internal/external non-conformances for all departments.
- Continuously evaluates production capacity and improves capacity utilization.
- Continuously evaluates turnaround time and addresses any problems that may hinder meeting the required and committed turnaround time from the various departments.
- Develops and improves the training of all analysts in cooperation with the Technical Manager and QA Manager and in compliance with regulatory requirements.
- Works with the Preventive Maintenance Coordinator to ensure that scheduled instrument maintenance is completed.
- Is responsible for efficient utilization of supplies.
- Constantly monitors and modifies the processing of samples through the departments.

- Fully supports the quality system and, if called upon in the absence of the QA Manager, serves as his substitute in the interim.

4.2.10 Project Manager (PM)

Members of the laboratory Client Services/Project Management Group are responsible for organizing and managing client projects. Clients are assigned a project manager who serves as their primary contact at the laboratory. It is the PM's responsibility to act as the client advocate by communicating client requirements to laboratory personnel and ensuring that clients provide complete information needed by the laboratory to meet those requirements – including all verbal communications.

With the overall goal of total client satisfaction, the functions of this position are outlined below:

- Scheduling sample submissions, sample container orders and sample pick-up via the laboratory courier service.
- Confirming certification status
- Coordinating and communicating turnaround time (TAT) requirements for high priority samples/projects.
- Answering common technical questions, facilitating problem resolution and coordinating technical details with the laboratory staff.
- Ensuring that client specifications, when known, are met by communicating project and quality assurance requirements to the laboratory.
- Notifying the supervisors of incoming projects and sample delivery schedules.
- Accountable to clients for communicating sample status reports or results prior to receipt of the final report.
- Monitor the status of all data package projects in-house to ensure timely and accurate delivery of reports.
- Inform clients of data package-related problems and resolve service issues.

4.2.11 Department Supervisors

Supervisors report to the Operations Manager. Each one is responsible to:

- Ensure that analysts in their department adhere to applicable SOPs and the QA Manual. They perform frequent SOP and QA Manual review to determine if analysts are in compliance and if new, modified, and optimized measures are feasible and should be added to these documents.
- With regard to analysts, participates in the selection, training (as documented in Section 5.3), development of performance objectives and standards of performance, appraisal (measurement of objectives), scheduling, counseling, discipline, and motivation of analysts and documents these activities in accordance with systems developed by the QA and Personnel Departments. They evaluate staffing sufficiency and overtime needs. Training consists of familiarization with SOP, QC, Safety, and computer systems.

- Encourage the development of analysts to become cross-trained in various methods and/or operate multiple instruments efficiently while performing maintenance and documentation, self-supervise, and function as a department team.
- Provide guidance to analysts in resolving problems encountered daily during sample prep/analysis in conjunction with the Technical Manager, Operations Manager, and/or QA Manager. Each is responsible for 100% of the data review and documentation, non-conformance and *CPAR* issues, the timely and accurate completion of performance evaluation samples and MDLs, for his department.
- Ensure all logbooks are maintained, current, and properly labeled or archived.
- Report all non-conformance conditions to the QA Manager, Technical Manager, Operations Manager, and Laboratory Director.
- Ensure that preventive maintenance is performed on instrumentation as detailed in the QA Manual or SOPs. He/she is responsible for developing and implementing a system for preventive maintenance, troubleshooting, and repairing or arranging for repair of instruments.
- Maintain adequate and valid inventory of reagents, standards, spare parts, and other relevant resources required to perform daily analysis.
- Achieve optimum turnaround time on analyses and compliance with holding times.
- Conduct efficiency and cost control evaluations on an ongoing basis to determine optimization of labor, supplies, overtime, first-run yield, capacity (designed vs. demonstrated), second- and third-generation production techniques/instruments, and long-term needs for budgetary planning.
- Develop, implement, and enhance calibration programs.
- Provide written responses to external and internal audit issues.

4.2.12 Laboratory Analysts

Laboratory analysts are responsible for conducting analysis and performing all tasks assigned to them by the group leader or supervisor. The responsibilities of the analysts are listed below:

- Perform analyses by adhering to analytical and quality control protocols prescribed by current SOPs, this QA Manual, and project-specific plans honestly, accurately, timely, safely, and in the most cost-effective manner.
- Document standard and sample preparation, instrument calibration and maintenance, data calculations, sample matrix effects, and any observed non-conformance on worklists, benchsheets, lab notebooks and/or the Non-Conformance Database.
- Report all non-conformance situations, instrument problems, matrix problems and QC failures, which might affect the reliability of the data, to their supervisor, the Technical Manager, and/or the QA Manager or member of QA staff.
- Perform 100% review of the data generated prior to entering and submitting for secondary level review.
- Suggest method improvements to their supervisor, the Technical Manager, and the QA Manager. These improvements, if approved, will be incorporated. Ideas for the optimum

performance of their assigned area, for example, through the proper cleaning and maintenance of the assigned instruments and equipment, are encouraged.

- Work cohesively as a team in their department to achieve the goals of accurate results, optimum turnaround time, cost effectiveness, cleanliness, complete documentation, and personal knowledge of environmental analysis.

4.2.13 Environmental Health and Safety Coordinator

The Environmental Health and Safety Coordinator (EHSC) reports to the Laboratory Director and the Region NDSC Environmental Health and Safety Manager and ensures that systems are maintained for the safe operation of the laboratory. The EHSC is responsible to:

- Conduct ongoing, necessary safety training and conduct new employee safety orientation.
- Assist in developing and maintaining the Chemical Hygiene/Safety Manual.
- Administer dispersal of all Material Safety Data Sheet (MSDS) information.
- Perform regular chemical hygiene and housekeeping instruction.
- Give instruction on proper labeling and practice.
- Serve as chairman of the laboratory safety committee.
- Provide and train personnel on protective equipment.
- Oversee the inspection and maintenance of general safety equipment – fire extinguishers, safety showers, eyewash fountains, etc. and ensure prompt repairs as needed.
- Supervise and schedule fire drills and emergency evacuation drills.
- Determine what initial and subsequent exposure monitoring, if necessary to determine potential employee exposure to chemicals used in the laboratory.
- When determined necessary, conduct exposure monitoring assessments.
- Determine when a complaint of possible over-exposure is “reasonable” and should be referred for medical consultation.
- Assist in the internal and external coordination of the medical consultation/monitoring program conducted by Euronfins TestAmerica’s medical consultants.

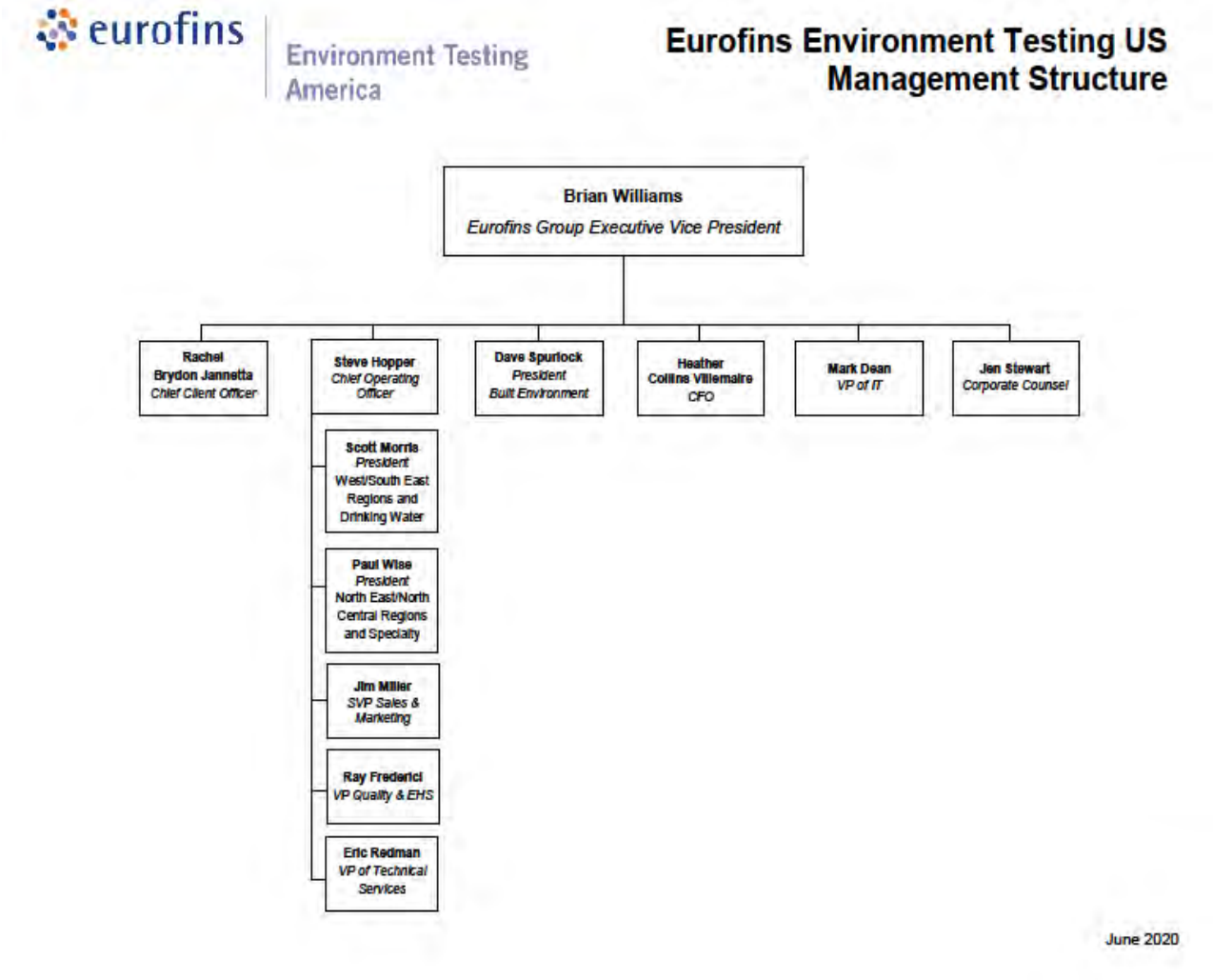
4.3 Business Continuity and Contingency Plans

Various policies and practices are in place to address continuity of business and contingency plans to ensure continued operations or minimal disruption in operations should unplanned events (natural disasters, unexpected management changes, etc.) occur. Deputies are identified for all key management personnel. Deputies would temporarily fill a role if the primary is absent for more than 15 consecutive calendar days. The deputies must meet the same qualifications as the primary person should they be required to take on the responsibilities. The QA Manager communicates to the relevant regulatory authorities when there are management or facility changes that impact the laboratory. Changes in the technical director must be communicated within a period of time and in the manner dictated by each regulatory authority.

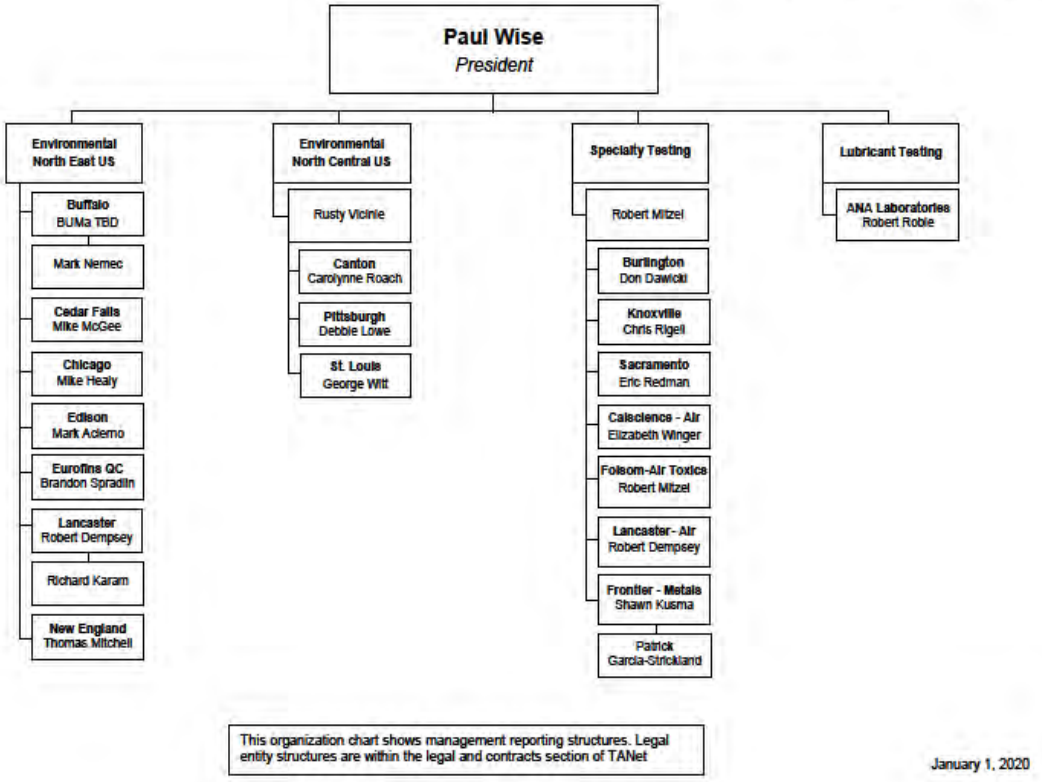
The following table defines who assumes the responsibilities of key personnel in their absence:

Key Personnel	Deputy
Don Dawicki Laboratory Director	Operations Manager Technical Manager
Kristine Dusablon Quality Manager	Laboratory Director Operations Manager
Kristine Dusablon Technical Manager	Laboratory Director Operations Manager
Ryan Hammond Operations Manager/EHS Coordinator	Laboratory Director Technical Manager
William Desjardins Specialty Organics Supervisor	Operations Manager Laboratory Director
Mark Fausel SVOA/LC Supervisor	Operations Manager Laboratory Director
OPEN Inorganic Supervisor	Operations Manager Laboratory Director
Mariah Witas Organic Extraction Supervisor	Operations Manager Laboratory Director
Scott Lavigne Sample Management/Geotechnical Supervisor	Operations Manager Laboratory Director
Dan Helfrich EHS Manager	Laboratory Director EHS Coordinator

Figure 4-1. Laboratory Organization Charts

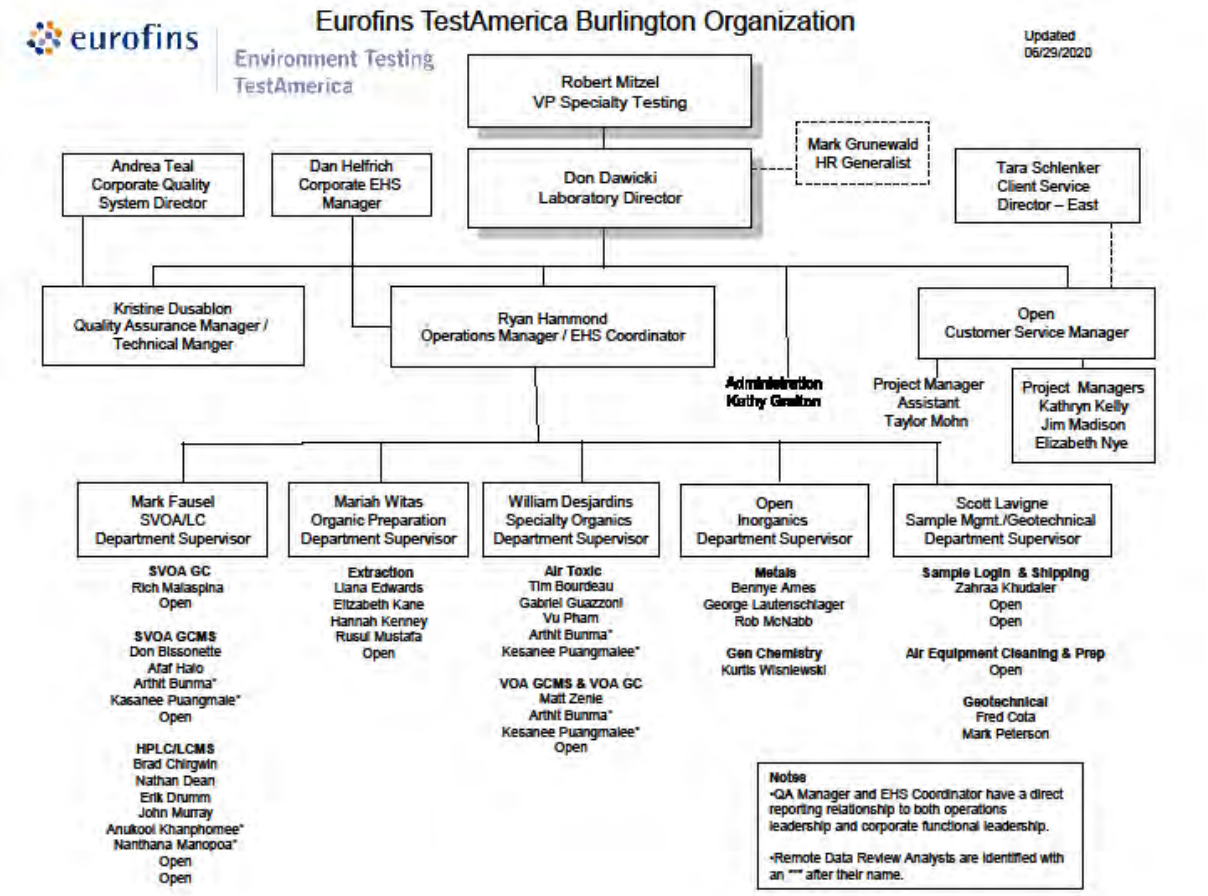


US Environment Testing (North East/North Central Regions & Specialty Services)



This organization chart shows management reporting structures. Legal entity structures are within the legal and contracts section of TANet

January 1, 2020


eurofins
Environment Testing
TestAmerica
Eurofins TestAmerica Burlington Organization
Updated
06/29/2020
Robert Mitzel
VP Specialty Testing
Mark Grunewald
HR Generalist
Andrea Teal
Corporate Quality
System Director
Dan Helfrich
Corporate EHS
Manager
Don Dawicki
Laboratory Director
Tara Schlenker
Client Service
Director – East
Kristine Dusablon
Quality Assurance Manager /
Technical Manger
Ryan Hammond
Operations Manager / EHS Coordinator
Open
Customer Service Manager
Administration
Kathy Gratton
Project Manager
Assistant
Taylor Mohn
Project Managers
Kathryn Kelly
Jim Madison
Elizabeth Nye
Mark Fausel
SVOA/LC
Department Supervisor
Mariah Witas
Organic Preparation
Department Supervisor
William Desjardins
Specialty Organics
Department Supervisor
Open
Inorganics
Department Supervisor
Scott Lavigne
Sample Mgmt./Geotechnical
Department Supervisor
SVOA GC
Rich Malaspina
Open
SVOA GCMS
Don Bissonette
Afaf Halo
Arthit Bunma*
Kasanee Puangmale*
Open
HPLC/LCMS
Brad Chirgwin
Nathan Dean
Erik Drumm
John Murray
Anukool Khanphomee*
Nanthana Manopoa*
Open
Open
Extraction
Liana Edwards
Elizabeth Kane
Hannah Kenney
Rusul Mustafa
Open
Air Toxic
Tim Bourdeau
Gabriel Guazzoni
Vu Pham
Arthit Bunma*
Kesanee Puangmalee*
VOA GCMS & VOA GC
Matt Zenie
Arthit Bunma*
Kesanee Puangmalee*
Open
Metals
Bennye Ames
George Lautenschlager
Rob McNabb
Gen Chemistry
Kurtis Wisniewski
Sample Login & Shipping
Zahraa Khudaier
Open
Open
Air Equipment Cleaning & Prep
Open
Geotechnical
Fred Cota
Mark Peterson
Notes
•QA Manager and EHS Coordinator have a direct reporting relationship to both operations leadership and corporate functional leadership.
•Remote Data Review Analysts are identified with an "*" after their name.

5.0 PERSONNEL

5.1 Overview

The laboratory's management believes that its highly qualified and professional staff is the single most important aspect in assuring a high level of data quality and service. The staff consists of professionals and support personnel.

All personnel must demonstrate competence in the areas where they have responsibility. Any staff that is undergoing training shall have appropriate supervision until they have demonstrated their ability to perform their job function on their own. Staff shall be qualified for their tasks based on appropriate education, training, experience and/or demonstrated skills as required.

The laboratory employs sufficient personnel with the necessary education, training, technical knowledge and experience for their assigned responsibilities.

All personnel are responsible for complying with all QA/QC requirements that pertain to the laboratory and their area of responsibility. Each staff member must have a combination of experience and education to adequately demonstrate a specific knowledge of their particular area of responsibility. Technical staff must also have a general knowledge of lab operations, test methods, QA/QC procedures and records management.

Laboratory management is responsible for formulating goals for lab staff with respect to education, training and skills and ensuring that the laboratory has a policy and procedures for identifying training needs and providing training of personnel. The training shall be relevant to the present and anticipated responsibilities of the lab staff.

The laboratory only uses personnel that are employed by or under contract to, the laboratory. Contracted personnel, when used, must meet competency standards of the laboratory and work in accordance to the laboratory's quality system.

5.2 Education and Experience Requirements for Technical Personnel

The laboratory makes every effort to hire analytical staffs that possess a college degree (AA, BA, BS) in an applied science with some chemistry in the curriculum. Exceptions can be made based upon the individual's experience and ability to learn. Selection of qualified candidates for laboratory employment begins with documentation of minimum education, training, and experience prerequisites needed to perform the prescribed task. Minimum education and training requirements for laboratory employees are outlined in job descriptions maintained by Eurofins Environment Testing America Human Resources.

Experience and specialized training are occasionally accepted in lieu of a college degree (basic lab skills such as using a balance or quantitation techniques, etc., are also considered).

As a general rule for analytical staff:

Specialty	Education	Experience
Extractions, Digestions, some electrode methods (pH, DO, Redox, etc.), or Titrimetric and Gravimetric Analyses	H.S. Diploma	On the job training (OJT)

Specialty	Education	Experience
ICP, ICPMS, Long List or complex chromatography (e.g., Pesticides, PCB, Herbicides, HPLC, etc.), GCMS	A college degree in an applied science or 2 years of college chemistry	or 5 years of prior analytical experience
Spectra Interpretation	A college degree in an applied science or 2 years of college chemistry	And 2 years relevant experience Or 5 years of prior analytical experience
Technical Managers – **General**	Bachelor's Degree in an applied science or engineering with 24 semester hours in chemistry An advanced (MS, PhD.) degree may substitute for one year of experience	And 2 years experience in environmental analysis of representative analytes for which they will oversee
Technical Managers – **Wet Chem** only (no advanced instrumentation)	Associates degree in an applied science or engineering or 2 years of college with 16 semester hours in chemistry	And 2 years relevant experience

When an analyst does not meet these requirements, they can perform a task under the direct supervision of a qualified analyst, peer reviewer, supervisor, or Technical Manager, and are considered an analyst in training. The person supervising an analyst in training is accountable for the quality of the analytical data and must review and approve data and associated corrective actions.

5.3 Training

The laboratory is committed to furthering the professional and technical development of employees at all levels.

Orientation to the laboratory's policies and procedures, in-house method training, and employee attendance at outside training courses and conferences all contribute toward employee proficiency. Below are examples of various areas of required employee training:

Required Training	Time Frame	Employee Type
Environmental Health & Safety	Prior to lab work	All
Ethics – New Hires	1 week of hire	All

Required Training	Time Frame	Employee Type
Ethics – Comprehensive	60 days of hire	All
Data Integrity	60 days of hire	Technical and PMs
Quality Assurance	90 days of hire	All
Ethics – Comprehensive Refresher	Annually	All
Initial Demonstration of Capability (DOC)	Prior to unsupervised method performance	Technical

The laboratory maintains records of relevant authorization/competence, education, professional qualifications, training, skills and experience of technical personnel (including contracted personnel) as well as the date that approval/authorization was given. These records are kept on file at the laboratory. Also refer to "Demonstration of Capability" in Section 19.

The training of technical staff is kept up to date by:

- Each employee must have documentation in their training file that they have read, understood and agreed to follow the most recent version of the laboratory QA Manual and SOPs in their area of responsibility. This documentation is updated as SOPs are updated.
- Documentation from any training courses or workshops on specific equipment, analytical techniques or other relevant topics.
- Documentation of proficiency (refer to Section 19).
- An Ethics Agreement signed by each staff member (renewed each year) and evidence of annual ethics training.
- A Confidentiality Agreement signed by each staff member signed at the time of employment.
- Human Resources maintains documentation and attestation forms on employment status and records; benefit programs; timekeeping/payroll; and employee conduct (e.g., ethics violations). This information is maintained in the employee's secured personnel file.

Evidence of successful training could include such items as:

- Adequate documentation of training within operational areas, including one-on-one technical training for individual technologies, and particularly for people cross-trained.
- Analysts knowledge to refer to QA Manual for quality issues.
- Analysts following SOPs, i.e., practice matches SOPs.
- Analysts regularly communicate to supervisors and QA if SOPs need revision, rather than waiting for auditors to find problems.

Further details of the laboratory's training program are described in the Laboratory Training SOP BR-QA-011.

5.4 Data Integrity and Ethics Training Program

The laboratory's Ethics and Data Integrity Program is discussed in Section 6.2. Employees are trained as to the legal and environmental repercussions that result from data misrepresentation. Key topics covered in the presentation include:

- Organizational mission and its relationship to the critical need for honesty and full disclosure in all analytical reporting.
- Ethics Policy
- How and when to report ethical/data integrity issues. Confidential reporting.
- Record keeping.
- Discussion regarding data integrity procedures.
- Specific examples of breaches of ethical behavior (e.g. peak shaving, altering data or computer clocks, improper macros, etc., accepting/offering kickbacks, illegal accounting practices, unfair competition/collusion)
- Internal monitoring. Investigations and data recalls.
- Consequences for infractions including potential for immediate termination, debarment, or criminal prosecution.
- Importance of proper written narration / data qualification by the analyst and project manager with respect to those cases where the data may still be usable but are in one sense or another partially deficient.

Additionally, a data integrity hotline (1-800-736-9407) is maintained by the NDSC.

6.0 ACCOMMODATIONS AND ENVIRONMENTAL CONDITIONS

6.1 Overview

The laboratory is a 25,000 ft^2 secure laboratory facility with controlled access and designed to accommodate an efficient workflow and to provide a safe and comfortable work environment for employees. All visitors sign in and are escorted by laboratory personnel. Access is controlled by various measures.

The laboratory is equipped with structural safety features. Each employee is familiar with the location, use, and capabilities of general and specialized safety features associated with their workplace. The laboratory provides and requires the use of protective equipment including safety glasses, protective clothing, gloves, etc., OSHA and other regulatory agency guidelines regarding required amounts of bench and fume hood space, lighting, ventilation (temperature and humidity controlled), access, and safety equipment are met or exceeded.

Traffic flow through sample preparation and analysis areas is minimized to reduce the likelihood of contamination. Adequate floor space and bench top area is provided to allow unencumbered sample preparation and analysis space. Sufficient space is also provided for storage of reagents and media, glassware, and portable equipment. Ample space is also provided for refrigerated sample storage before analysis and archival storage of samples after analysis. Laboratory HVAC and deionized water systems are designed to minimize potential trace contaminants.

The laboratory is separated into specific areas for sample receiving, sample preparation, volatile organic sample analysis, non-volatile organic sample analysis, inorganic sample analysis, and administrative functions.

6.2 Environment

Laboratory accommodation, test areas, energy sources, and lighting are adequate to facilitate proper performance of tests. The facility is equipped with heating, ventilation, and air

conditioning (HVAC) systems appropriate to the needs of environmental testing performed at this laboratory.

The environment in which these activities are undertaken does not invalidate the results or adversely affect the required accuracy of any measurements.

The laboratory provides for the effective monitoring, control and recording of environmental conditions that may affect the results of environmental tests as required by the relevant specifications, methods, and procedures. Such environmental conditions include humidity, voltage, temperature, and vibration levels in the laboratory.

When any of the method or regulatory required environmental conditions change to a point where they may adversely affect test results, analytical testing will be discontinued until the environmental conditions are returned to the required levels.

Environmental conditions of the facility housing the computer network and LIMS are regulated to protect against raw data loss.

When the laboratory performs laboratory activities at sites or facilities outside its permanent control, it shall ensure that the requirements related to facilities and environmental conditions of this document are met.

Specific requirements for facility and environmental conditions, as well as periodic monitoring of conditions, are given in the Environmental Health & Safety Manual plus each laboratory's Facility Addendum.

6.3 Work Areas

There is effective separation between neighboring areas when the activities therein are incompatible with each other. Examples include:

- Volatile organic chemical handling areas, including sample preparation and waste disposal, and volatile organic chemical analysis areas.

Access to and use of all areas affecting the quality of analytical testing is defined and controlled by secure access to the laboratory building as described below in the Building Security section.

Adequate measures are taken to ensure good housekeeping in the laboratory and to ensure that any contamination does not adversely affect data quality. These measures include regular cleaning to control dirt and dust within the laboratory. Work areas are available to ensure an unencumbered work area. Work areas include:

- Access and entryways to the laboratory.
- Sample receipt areas.
- Sample storage areas.
- Chemical and waste storage areas.
- Data handling and storage areas.
- Sample processing areas.
- Sample analysis areas.

6.4 Responding to Emergencies

Employees must be aware of procedures to respond to all emergencies that might occur in the workplace. Employees must be familiar with the location and proper operation of all emergency equipment, evacuation routes and designated assembly areas for all areas where they work. Refer to the NDSC EH&S Manual Document No. CW-E-M-001. Sec. 7 and the laboratory's local EH&S addendum for complete details. These documents provide direction for situations where normal operations of the laboratory are not possible (e.g., electrical failures, heating/air conditioning failures, fire/building evacuation, computer failures, hazardous material spills, injury to employees, pandemic flu, disruption of phone service, etc.)

In the event that the building or information technology (IT) systems would be severely challenged, a designated disaster recovery team, which includes Facility Management, Maintenance, Safety, Laboratory/Executive Management, Public Relations, IT, QA and other applicable personnel depending on the scope of the disaster, would assemble at a designated area to assess the situation and formulate a plan.

6.5 Building Security

The laboratory is considered a secure facility. All outside doors (except the main lobby entrance) are locked during normal business hours to prevent unauthorized entry. An attendant monitors this entrance at all times. Building key cards (or key fobs) are distributed to employees as necessary. All visitors to the laboratory must sign in and out in a visitor's logbook that is located in the lobby. A visitor is defined as any person who visits the laboratory who is not an employee of the laboratory. Both visitors and vendors must review and sign specific EH&S forms; and are escorted by laboratory personnel at all times, or the location of the visitor is noted in the visitor's logbook.

7.0 QUALITY SYSTEM

7.1 Quality Policy Statement

The Quality Policy statement gives employees clear requirements for the production of analytical data. As an organization, all personnel are committed to high quality professional practice, testing and data, and service to our clients.

We strive to provide the highest quality data achievable by:

- Reading and understanding all of the quality documents applicable to each position and implementing the process in our work.
- Following all recordkeeping requirements; describing clearly and accurately all activities performed; recording "real time" as the task is carried out; understanding that it is never acceptable to "back date" entries and should additional information be required at a later date, the actual date and by whom the notation is made must be documented.
- Ensuring data integrity through the completeness, consistency, impartiality and accuracy of the data generated. Data is attributable, legible, contemporaneously recorded, original or a true copy, and accurate (ALCOA). This applies to manual paper documentation and electronic records.
- Providing accountability and traceability for each sample analyzed through proper sample handling, labeling, preparation, instrument calibration/qualification/validation, analysis, and reporting; establishing an audit trail (the who, what, when, and why) that identifies date,

time, analyst, instrument used, instrument conditions, quality control samples (where appropriate and/or required by the method), and associated standard material.

- ❖ Emphasizing a total quality management process which provides impartiality, accuracy, and strict compliance with agency regulations and client requirements, giving the highest degree of confidence; understanding that meeting the requirements of the next employee in the work flow process is just as important as meeting the needs of the external client.
- ❖ Providing thorough documentation and explanation to qualify reported data that may not meet all requirements and specifications, but is still of use to the client; understanding this occurs only after discussion with the client on the data limitations and acceptability of this approach.
- ❖ Responding immediately to indications of questionable data, out-of-specification occurrences, equipment malfunctions, and other types of laboratory problems, with investigation and applicable corrective action; documenting these activities completely, including the reasons for the decisions made.
- ❖ Providing a work environment that ensures accessibility to all levels of management and encourages questions and expression of concerns on quality issues to management. Eurofins recognizes that the implementation of a quality assurance program requires management's commitment and support as well as the involvement of the entire staff
- ❖ Continually improve systems and manage risk to support quality improvement efforts in laboratory, administrative and managerial activities

7.2 Ethics and Data Integrity

Eurofins Environment Testing America is committed to ensuring the integrity of its data and meeting the quality needs of its clients. The laboratory operates our Ethics and Data Integrity program under the guidance of Eurofin's Key Guidance Document (KGD). The elements of the our program include:

- An Ethics Policy (NDSC Document No.CW-L-P-004) and Employee Ethics Statements.
- Ethics and Compliance Officer/s (ECOs).
- A Training Program.
- Self-governance through disciplinary action for violations.
- A confidential mechanism for anonymously reporting alleged misconduct and a means for conducting internal investigations of all alleged misconduct. (NDSC Document No. CW-L-S-002).
- Procedures and guidance for recalling data if necessary (NDSC Document No. CW-Q-S-005).
- Effective external and internal monitoring system that includes procedures for internal audits (Section 17).
- Produce results, which are accurate and include QA/QC information that meets client pre-defined Data Quality Objectives (DQOs).
- Present services in a confidential, honest and forthright manner.
- Provide employees with guidelines and an understanding of the Ethical and Quality Standards of our Industry.

- Provide procedures and guidance to ensure the impartiality and confidentiality of all data and customer information.
- Operate our facilities in a manner that protects the environment and the health and safety of employees and the public.
- Obey all pertinent federal, state and local laws and regulations and encourage other members of our industry to do the same.
- Educate clients as to the extent and kinds of services available.
- Assert competency only for work for which adequate personnel and equipment are available and for which adequate preparation has been made.
- Promote the status of environmental laboratories, their employees, and the value of services rendered by them.

7.3 Quality System Documentation

The laboratory's Quality System is communicated through a variety of documents.

- Quality Assurance (QA) Manual – Each laboratory has a lab-specific QA manual.
- NDSC Official Documents – Each laboratory may use the Guidance (instructional use) documents at their discretion. Template documents are process documents that the laboratory's need to implement locally by using the document as is or as an outline to define their internal practices that meet the minimum requirements of the template. Required documents need to be implemented as is and listed in the laboratory's document control list.
- Key Guidance Documents (KGDs) - Documents compiled at the Group Service Centre (GSC) level by Functional Leaders (document owners) aimed at providing specific Eurofins groups of employees with guidelines necessary for the good conduct of their respective work.
- Laboratory SOPs and Policies – General and Technical
- Work Instructions – A subset of procedural steps, tasks or forms associated with an operation of a management system (e.g., checklists, preformatted bench sheets, forms).

7.3.1 Order of Precedence

In the event of a conflict or discrepancy between policies, the order of precedence is as follows:

- Quality Management Plan (QMP)
- NDSC Guidance Documents
- KGDs
- Laboratory Quality Assurance Manual (QA Manual)
- Laboratory SOPs and Policies
- Other (Work Instructions (WI), memos, flow charts, etc.)

NOTE: The laboratory has the responsibility and authority to operate in compliance with regulatory requirements of the jurisdiction in which the work is performed. Where the QMP conflicts with those regulatory requirements, the regulatory requirements of the jurisdiction shall hold primacy. The laboratory's QA Manual shall take precedence over the QMP in those cases.

7.4 QA/QC Objectives for the Measurement of Data

Quality Assurance (QA) is responsible for developing planned activities whose purpose is to provide assurance to all levels of management that a quality program is in place within the laboratory, and that it is functioning in an effective manner that is consistent with the requirements of NELAP, ISO 17025, and DoD and any other regulatory agencies (i.e., states) in which the laboratory maintains accreditation.

Quality Control (QC) is generally understood to be limited to the analyses of samples and to be synonymous with the term *"analytical quality control"*. QC refers to the routine application of statistically based procedures to evaluate and control the accuracy of results from analytical measurements. The QC program includes procedures for estimating and controlling precision and bias and for determining reporting limits.

Request for Proposals (RFPs) and Quality Assurance Project Plans (QAPP) provide a mechanism for the client and the laboratory to discuss the data quality objectives in order to ensure that analytical services closely correspond to client needs. In order to ensure the ability of the laboratory to meet the Data Quality Objectives (DQOs) specified in the QAPP, clients are advised to allow time for the laboratory to review the QAPP before being finalized. The client is responsible for developing the QAPP; however, the laboratory will provide support to the client for developing the sections of the QAPP that concern laboratory activities.

Historically, laboratories have described their QC objectives in terms of precision, accuracy, representativeness, comparability, completeness, selectivity and sensitivity (PARCCSS). Each laboratory SOP defines the required QC indicators.

7.4.1 Precision

The objective is to meet the performance for precision demonstrated for the methods on similar samples and to meet DQOs of the EPA and/or other regulatory programs. Precision is defined as the degree of reproducibility of measurements under a given set of analytical conditions (exclusive of field sampling variability). Precision is documented on the basis of replicate analysis, usually duplicate or matrix spike (MS) duplicate samples.

7.4.2 Accuracy

The objective is to meet the performance for accuracy demonstrated for the methods on similar samples and to meet DQOs of the EPA and/or other regulatory programs. Accuracy is defined as the degree of bias in a measurement system. Accuracy may be documented through the use of laboratory control samples (LCS) and/or MS. A statement of accuracy is expressed as an interval of acceptance recovery about the mean recovery.

7.4.3 Representativeness

The objective is to provide data which is representative of the sampled medium. Representativeness is defined as the degree to which data represent a characteristic of a population or set of samples and is a measurement of both analytical and field sampling precision. The representativeness of the analytical data is a function of the procedures used in procuring and processing the samples. The representativeness can be documented by the relative percent difference between separately procured, but otherwise identical samples or sample aliquots.

The representativeness of the data from the sampling sites depends on both the sampling procedures and the analytical procedures. Refer to laboratory SOPs for subsampling and homogenization techniques appropriate to the analytical method.

7.4.4 Comparability

The objective is to provide analytical data for which the accuracy, precision, representativeness, and reporting limit statistics are similar to these quality indicators generated by other laboratories for similar samples, and data generated by the laboratory over time.

Comparability is documented by inter-laboratory studies carried out by regulatory agencies or carried out for specific projects or contracts, by comparison of periodically generated statements of accuracy, precision, and reporting limits with those of other laboratories.

7.4.5 Completeness

The completeness objective for data is 90% (or as specified by a particular project), expressed as the ratio of the valid data to the total data over the course of the project. Data will be considered valid if they are adequate for their intended use. Data usability will be defined in a QAPP, project scope, or regulatory requirement. Data validation is the process for reviewing data to determine its usability and completeness. If the completeness objective is not met, actions will be taken internally and with the data user to improve performance. This may take the form of an audit to evaluate the methodology and procedures as possible sources for the difficulty or may result in a recommendation to use a different method.

7.4.6 Selectivity

Selectivity is defined as the capability of a test method or instrument to respond to a target substance or constituent in the presence of non-target substances. Target analytes are separated from non-target constituents and subsequently identified/detected through one or more of the following, depending on the analytical method: extractions (separation), digestions (separation), interelement corrections (separation), use of matrix modifiers (separation), specific retention times (separation and identification), confirmations with different columns or detectors (separation and identification), specific wavelengths (identification), specific mass spectra (identification), and specific electrodes (separation and identification).

7.4.7 Sensitivity

Sensitivity refers to the amount of analyte necessary to produce a detector response that can be reliably detected (above the Method Detection Limit) or quantified (above the Reporting Limit).

7.5 Criteria for Quality Indicators

The laboratory maintains tables, housed in LIMS, that summarize the precision and accuracy acceptability limits for performed analyses. This summary includes an effective date, is updated each time new limits are generated, and are managed by the laboratory's QA department. Unless otherwise noted, limits within these tables are laboratory generated. Some acceptability limits are derived from US EPA methods when they are required. Where US EPA method limits are not required, the laboratory has developed limits from evaluation of data from similar matrices. Criteria for development of control limits is contained in BR-QA-013 or Section 26.

7.6 Statistical Quality Control

Statistically-derived precision and accuracy limits are required by selected methods (such as SW-846) and programs. The laboratory routinely utilizes statistically-derived limits to evaluate method performance and determine when corrective action is appropriate. The analysts use the current limits entered into LIMS. The QA department maintains an archive of all limits used within the laboratory. If a method defines the QC limits, the method limits are used.

If a method requires the generation of historical limits, the lab develops such limits from recent data in the QC database of the LIMS following the guidelines described in Section 26. All calculations and limits are documented and dated when approved and effective. On occasion, a client requests contract-specified limits for a specific project.

Current QC limits are entered and maintained in the LIMS analyte database. As sample results and the related QC are entered into LIMS, the sample QC values are compared with the limits in LIMS to determine if they are within the acceptable range. The analyst then evaluates if the sample needs to be rerun or re-extracted/rerun or if a comment should be added to the report explaining the reason for the QC outlier.

7.6.1 QC Charts

The laboratory's procedures for the creation of control charts are described in the laboratory SOP BR-QA-013. Control charts are created from data stored in the LIMS. The QA Manager or technical staff evaluates these to determine if adjustments need to be made or for corrective actions to methods. All findings are documented and kept on file.

7.7 Quality System Metrics

In addition to the QC parameters discussed above, the entire Quality System is evaluated on a monthly basis through the use of specific metrics (refer to Section 18). These metrics are used to drive continuous improvement in the laboratory's Quality System.

8.0 DOCUMENT CONTROL

8.1 Overview

The QA Department is responsible for the control of documents used in the laboratory to ensure that approved, up-to-date documents are in circulation and out-of-date (obsolete) documents are archived or destroyed. The following documents, at a minimum, must be controlled:

- Laboratory Quality Assurance (QA) Manual
- Laboratory Standard Operating Procedures (SOP)
- Laboratory Policies
- Work Instructions and Forms
- NDSC Documents[1]
- KGDs[1]

[1]Includes locally implemented documents that are document controlled within the laboratory's document control system. The NDSC and/or KGD documents are only considered controlled when they are read on the intranet site. Printed copies are considered uncontrolled unless the

laboratory physically distributes them as controlled documents. A detailed description of the procedure for issuing, authorizing, controlling, distributing, and archiving NDSC Official Document is found in Document No. CW-Q-S-001, NDSC Document Control and Archiving. KGD documents are controlled according to BR-QA-003, Document Control. The laboratory's internal document control procedure is defined in SOP No. BR-QA-003, Document Control.

The laboratory QA Department also maintains access to various references and document sources integral to the operation of the laboratory. This includes reference methods and regulations. Instrument manuals (hard or electronic copies) are also maintained by the laboratory.

The laboratory maintains control of records for raw analytical data and supporting records such as audit reports and responses, logbooks, standard logs, training files, MDL studies, Proficiency Testing (PT) studies, certifications and related correspondence, and corrective action reports. Raw analytical data consists of bound logbooks, instrument printouts, any other notes, magnetic media, electronic data, and final reports.

8.2 Document Approval and Issue

The pertinent elements of the document control system includes a unique document title and number, pagination, the total number of pages of the item or an 'end of document' page, the effective date, revision number, and the laboratory's name. The QA personnel are responsible for the maintenance of this system.

Controlled documents are authorized by the QA Department. In order to develop a new document, a responsible manager submits an electronic draft to the QA Department for suggestions and approval before use. Upon approval, QA personnel add the identifying version information to the document and retains that document as the official document on file. That document is then provided to all applicable operational units. Controlled documents are identified as such and records of their distribution are kept by the QA Department. Document control may be achieved by either electronic or hardcopy distribution.

The QA Department maintains a list of the official versions of controlled documents.

Quality System Policies and Procedures will be reviewed at a minimum of every two years, for DW and DOE/DoD every year, and revised as appropriate. Changes to documents occur when a procedural change warrants.

8.3 Procedures for Document Control Policy

For changes to the QA Manual, refer to SOP No. BR-QA-003, Document Control. Uncontrolled copies must not be used within the laboratory. Previous revisions and back-up data are stored by the QA department. Electronic copies are stored on the Public server in the QA folder (\\tafs\Lab\Burlington\Public\QA Public\SOPs Controlled Distribution\Quality Manual) for the applicable revision.

For changes to SOPs, refer to NDSC Document No. CW-Q-S-002, Writing a Standard Operating Procedure) or SOP No. BR-QA-003, Document Control.

Forms, worksheets, work instructions and information are organized by department in the QA office. There is a table of contents. Electronic versions are kept on a hard drive in the QA department; hard copies are kept in QA files. The procedure for the care of these documents is in SOP No. BR-QA-003, Document Control.

8.4 Obsolete Documents

All invalid or obsolete documents are removed, or otherwise prevented from unintended use. The laboratory has specific procedures as described above to accomplish this. In general, obsolete documents are collected from employees according to distribution lists and are marked obsolete on the cover or destroyed. At least one copy of the obsolete document is archived according to SOP No. BR-QA-003, Document Control.

9.0 SERVICE TO THE CLIENT

9.1 Overview

The laboratory has established procedures for the review of work requests and contracts, oral or written. The procedures include evaluation of the laboratory's capability and resources to meet the contract's requirements within the requested time period. All requirements, including the methods to be used, must be adequately defined, documented and understood. For many environmental sampling and analysis programs, testing design is site or program specific and does not necessarily fit into a standard laboratory service or product. It is the laboratory's intent to provide both standard and customized environmental laboratory services to our clients.

A thorough review of technical and QC requirements contained in contracts is performed to ensure project success. The appropriateness of requested methods, and the lab's capability to perform them must be established. Projects, proposals, and contracts are reviewed for adequately defined requirements and the laboratory's capability to meet those requirements. Alternate test methods that are capable of meeting the clients' requirements may be proposed by the lab. A review of the lab's capability to analyze non-routine analytes is also part of this review process.

All projects, proposals and contracts are reviewed for the client's requirements in terms of compound lists, test methodology requested, sensitivity (detection and reporting levels), accuracy, and precision requirements (% Recovery and RPD). The reviewer ensures that the laboratory's test methods are suitable to achieve these requirements and that the laboratory holds the appropriate certifications and approvals to perform the work. The laboratory and any potential subcontract laboratories must be certified, as required, for all proposed tests.

Electronic or hard copy deliverable requirements are evaluated against the laboratory's capacity for production of the documentation.

If the laboratory cannot provide all services but intends to subcontract such services, whether to another Eurofins facility on the same LIMS or to an outside firm, this will be documented and discussed with the client prior to contract approval. (Refer to Section 10 for Subcontracting Procedures.)

The laboratory informs the client of the results of the review if it indicates any potential conflict, deficiency, lack of accreditation, or inability of the lab to complete the work satisfactorily. Any discrepancy between the client's requirements and the laboratory's capability to meet those requirements is resolved in writing before acceptance of the contract. It is necessary that the contract be acceptable to both the laboratory and the client. Amendments initiated by the client and/or Eurofins TestAmerica Burlington are documented in writing.

All contracts, QAPPs, Sampling and Analysis Plans (SAPs), contract amendments, and documented communications become part of the project record.

The same contract review process used for the initial review is repeated when there are amendments to the original contract by the client, and the participating personnel are informed of the changes.

9.2 Review Sequence and Key Personnel

Appropriate personnel will review the work request at each stage of evaluation.

For routine projects and other simple tasks, a review by the Project Manager (PM) is considered adequate. The PM confirms that the laboratory has any required certifications, that it can meet the clients' data quality and reporting requirements and that the lab has the capacity to meet the clients turn around needs. It is recommended that, where there is a sales person assigned to the account, an attempt should be made to contact that sales person to inform them of the incoming samples.

For new, complex or large projects, the proposed contract is given to the Client Relationship Manager or Proposal Team, who will decide which lab will receive the work based on the scope of work and other requirements, including certification, testing methodology, and available capacity to perform the work. The contract review process is outlined in NDSC Document No. CA-L-P-002**,** Contract Compliance Policy.

This review encompasses all facets of the operation. The scope of work is distributed to the appropriate personnel, as needed based on scope of contract, to evaluate all of the requirements shown above (not necessarily in the order below)

- Contract Administrator
- Laboratory Project Manager
- Laboratory Director and/or Technical Manager
- Account Executive
- Quality Manager
- Laboratory Environmental Health and Safety Manager

The Laboratory Director reviews the formal laboratory quote and makes final acceptance for their facility.

The Sales Director, Contract Administrator, Account Executive or Proposal Coordinator then submits the final proposal to the client.

In the event that one of the above personnel is not available to review the contract, his or her back-up will fulfill the review requirements.

9.3 Balancing Laboratory Capacity and Workload

Evaluating laboratory capacity to perform specific projects is the responsibility of the Vice-President, Laboratory Director and Managers, and the Client Services director. Many analysts are cross-trained to perform a variety of tests, and there is redundant equipment available in case of malfunctions. This minimizes the need to evaluate small and medium size projects against capacity available to complete them. Large and complex projects are reviewed against capacity estimates before bids are submitted to ensure that the client's analysis schedule is met. Regularly scheduled meetings are held between laboratory management, PMs, and QA to review progress with current projects, as well as special requirements of new work scheduled for the laboratory. Laboratory capacity and backlog is tracked on a continuous basis using

information from the Laboratory Sample Information System (LIMS) including turnaround time, and work in-house.

9.4 Documentation

The Contracts Department maintains copies of all signed contracts**.** A copy is also retained by the Customer Relations Manager or at the facility by the Project Manager.

Appropriate records are maintained for every contract or work request. All stages of the contract review process are documented and include records of any significant changes. These records are maintained in the LIMS project or other file by CSO if no project ID exists.

The contract will be distributed to and maintained by the appropriate sales/marketing personnel and the Account Executive. A copy of the contract and formal quote will be filed with the laboratory PM and the Laboratory Director.

Records are maintained of pertinent discussions with a client relating to the client's requirements or the results of the work during the period of execution of the contract. The PM keeps a phone log of conversations with the client.

9.4.1 Project-Specific Quality Planning

Communication of contract specific technical and QC criteria is an essential activity in ensuring the success of site specific testing programs. To achieve this goal, a PM is assigned to each client. It is the PM's responsibility to ensure that project-specific technical and QC requirements are effectively evaluated and communicated to the laboratory personnel before and during the project. QA department involvement may be needed to assist in the evaluation of custom QC requirements.

PM's are the primary client contact and they ensure resources are available to meet project requirements, they coordinate opportunities and work with laboratory management and supervisory staff to ensure available resources are sufficient to perform work for the client's project.

Prior to work on a new project, the dissemination of project information and/or project opening meetings may occur to discuss schedules and unique aspects of the project. Items to be discussed may include the project technical profile, turnaround times, holding times, methods, analyte lists, reporting limits, deliverables, sample hazards, or other special requirements. The PM introduces new project information to maximize production and client satisfaction, while maintaining quality. Project notes may be associated with each sample batch as a reminder upon sample receipt and analytical processing.

~~A~~ny change that may occur within an active project is agreed upon between the client/regulatory agency and the PM/laboratory. These changes (e.g., use of a non-standard method or modification of a method) and approvals must be documented prior to implementation. Documentation pertains to any document (e.g., letter, e-mail, variance, contract addendum), which has been signed by both parties.

Such changes are also communicated to the laboratory during production and status meeting. The laboratory staff is then introduced to the modified requirements via the PM, supervisor or the individual laboratory Technical Manager. After the modification is implemented into the laboratory process, documentation of the modification is made in the case narrative of the data report(s).

The laboratory strongly encourages client visits to the laboratory and for formal/informal information sharing session with employees in order to effectively communicate ongoing client needs as well as project specific details for customized testing programs.

9.5 Special Services

The laboratory cooperates with clients and their representatives to monitor the laboratory's performance in relation to work performed for the client. It is the laboratory's goal to meet all client requirements in addition to statutory and regulatory requirements. The laboratory has procedures to ensure confidentiality to clients (Section 17 and 25).

The laboratory's standard procedures for reporting data are described in Section 25. Special services are also available and provided upon request. These services include:

- Reasonable access for our clients or their representatives to the relevant areas of the laboratory for the witnessing of tests performed for the client.
- Assisting client-specified third party data validators as specified in the client's contract.
- Supplemental information pertaining to the analysis of their samples. Note: An additional charge may apply for additional data/information that was not requested prior to the time of sample analysis or previously agreed upon.

When the client requests a statement of conformity to a specification or standard based on the analysis performed by the laboratory (e.g., pass/fail, in-tolerance/out-of-tolerance), the decision rule shall be clearly defined. Unless inherent in the requested specification or standard, the decision rule selected shall be communicated to the client. Associated reporting requirements are addressed in Section 25.2.18.

9.6 Client Communication

PMs are the primary communication link to the clients. They shall inform their clients of any delays in project completion as well as any non-conformances in either sample receipt or sample analysis. Project management will maintain ongoing client communication throughout the entire client project.

Technical Managers are available to discuss any technical questions or concerns that the client may have.

9.7 Reporting

The laboratory works with our clients to produce any special communication reports required by the contract.

9.8 Client Feedback and Surveys

The laboratory assesses both positive and negative client feedback. The results are used to improve overall laboratory quality and client service. Eurofins Sales and Marketing teams periodically develop lab and client specific surveys to assess client satisfaction. Client satisfaction surveys are sent by Sales and Marketing teams periodically. They develop lab and client specific surveys to assess client satisfaction.

When a complaint is received, we determine, to the best of our ability, the extent of the issue and what data is in question. The person receiving the complaint documents this information and promptly forwards it to the appropriate management personnel where the work in question was performed. If a data reporting error is discovered, the final report and/or data must be regenerated with the correct value(s).

The QA Manager is responsible for entering client concerns into iCAT. In some cases, an ICAT / ICAR (Investigation and Corrective Action Report) is initiated to address and document the

situation. While an individual issue may not warrant a formal investigation, QA monitors these issues for potential trends and will issue an ICAR/ICAT if a trend is evident.

10.0 SUBCONTRACTING OF TESTS

The laboratory may subcontract tests to other laboratories if the requested testing is not routinely performed in our laboratory. To a lesser extent, samples may need to be subcontracted to an overflow laboratory to ensure hold times and/or turn-around-times (TAT) are met.

Testing is only subcontracted with the client's knowledge and approval. The Project Manager must notify the client in writing when any of their requested analyses will be subcontracted to another lab. Client approval must be obtained in writing before samples are shipped.

Subcontract laboratories are selected based on their qualifications and accreditations. The subcontractor is requested to sign a Laboratory Analytical Services Subcontract. See form Q-EQA-FRM6867 to review details of the contract terms and information requested from the subcontract laboratory. If projects require a specific agency certification (i.e. individual state agencies, NELAP, DoD, PALA, ISO 17025), only an appropriately accredited laboratory is used. The client may also have a list of laboratories to be used for subcontracting. In these cases, the evaluation of the subcontract laboratory is made by the client.

Data obtained from subcontract laboratories is clearly marked as such when reported by the laboratory. The data are submitted to the client in the format obtained from the subcontractor.

10.1 Overview

For the purpose of this quality manual, the phrase subcontract laboratory refers to a laboratory external to the Eurofins laboratories. The phrase "work sharing" refers to internal transfers of samples between the Eurofins TestAmerica laboratories. The term outsourcing refers to the act of subcontracting tests.

When contracting with our clients, the laboratory makes commitments regarding the services to be performed and the data quality for the results to be generated. When the need arises to outsource testing for our clients because project scope, changes in laboratory capabilities, capacity, or unforeseen circumstances, we must be assured that the subcontractors or work sharing laboratories understand the requirements and will meet the same commitments we have made to the client. Refer to the NDSC Documents on Subcontracting Procedures (CW-L-S-004) and the Work Sharing Process (CA-C-S-001).

When outsourcing analytical services, the laboratory will assure, to the extent necessary, that the subcontract or work sharing laboratory maintains a program consistent with the requirements of this document, the requirements specified in TNI/ISO 17025 and/or the client's Quality Assurance Project Plan (QAPP). All QC guidelines specific to the client's analytical program are transmitted to the subcontractor and agreed upon before sending the samples to the subcontract facility. Additionally, work requiring accreditation will be placed with an appropriately accredited laboratory. The laboratory performing the subcontracted work will be identified in the final report, as will non-TNI accredited work where required.

PMs or other responsible Client Service members, for the Export Lab (i.e., the Eurofins TestAmerica Burlington laboratory that transfers samples to another laboratory) are responsible for obtaining client approval prior to subcontracting any samples. The laboratory will advise the client of a subcontract arrangement in writing and when possible approval from the client shall be obtained and retained in the project folder. Standard Eurofins TestAmerica Burlington Terms

& Conditions include the flexibility to subcontract samples within the Eurofins Burlington TestAmerica laboratories. Therefore, additional advance notification to clients for intra-laboratory subcontracting is not necessary unless specifically required by a client contract.

Note: In addition to the client, some regulating agencies (e.g., USDA) or contracts (e.g., DoD and DOE projects) require notification prior to placing such work**.**

10.2 Qualifying and Monitoring Subcontractors

Whenever a PM, Account Executive (AE) or Client Relationship Manager becomes aware of a client requirement or laboratory need where samples must be outsourced to another laboratory, the other laboratory(s) shall be selected based on the following:

- Subcontractors specified by the client - In these circumstances, the client assumes responsibility for the quality of the data generated from the use of a subcontractor.
- Subcontractors reviewed by Eurofins TestAmerica Burlington – Firms which have been reviewed by the company and are known to meet standards for accreditations (e.g., State, TNI and DoD/DOE); technical specifications; legal and financial information.

A listing of vendors is available on the Eurofins TestAmerica intranet site.

All Eurofins TestAmerica Burlington laboratories are pre-qualified for work sharing provided they hold the appropriate accreditations and can adhere to the project/program requirements. Client approval is not necessary unless specifically required by the contract. In these cases, the client must provide acknowledgement that the samples can be sent to that facility (an e-mail is sufficient documentation or if acknowledgement is verbal, the date, time, and name of person providing acknowledgement must be documented). The originating laboratory is responsible for communicating all technical, quality, and deliverable requirements as well as other contract needs. (NDSC Document No. CA-C-S-001, Work Sharing Process).

10.2.1 When the potential subcontract laboratory has not been previously approved, Account Executives or PM may nominate a laboratory as a subcontractor based on need. The decision to nominate a laboratory must be approved by the Client Relations Manager (CRM) or Laboratory Director***]***. The CRM or Laboratory Director requests that the QA Manager or PM begin the process of approving the subcontract laboratory. Refer to the NDSC Document No. CW-L-S-004, Subcontracting Procedures.

Once the appropriate accreditation and legal information is received by the laboratory, it is evaluated for acceptability and forwarded to the NDSC Quality Information Manager (QIM) for review. After the NDSC QIM reviews the documents for completeness, the information is forwarded to the Finance Department for formal signature and contracting with the laboratory. The approved vendor will be added to the approved subcontractor list on the intranet site, and the finance group is concurrently notified.

The client will assume responsibility for the quality of the data generated from the use of a subcontractor they have requested the lab to use. The qualified subcontractors on the intranet site are known to meet minimal standards. Eurofins TestAmerica Burlington Laboratory does not certify laboratories. The subcontractors on our approved list can only be recommended to the extent that we would use them.

10.3 Oversight and Reporting

The status and performance of qualified subcontractors will be monitored by NDSC and includes an annual review process (NDSC Document No. SOP CW-L-S-004). Any problems

identified will be brought to the attention of NDSC and Procurement personnel.

- Complaints shall be investigated. Documentation of the complaint, investigation, and corrective action will be maintained in the subcontractor's file on the intranet site. Complaints are posted using the Vendor Performance Report.
- Information shall be updated on the intranet when new information is received from the subcontracted laboratories.
- Subcontractors in good standing will be retained on the intranet listing. Client Service personnel will notify all Eurofins TestAmerica Burlington laboratories, NDSC, and Corporate Contracts if any laboratory requires removal from the intranet site. This notification will be posted on the intranet site and e-mailed to all Client Service Personnel, Laboratory Directors, QA Managers, and Sales Personnel.

Prior to initially sending samples to the subcontracted laboratory, the PM confirms their certification status to determine if it's current and scope-inclusive. The information is documented within the project records.

The laboratory's certifications can be viewed on the company's website @ **www.testamericainc.com**

10.3.1 All subcontracted samples must be accompanied by a Eurofins TestAmerica Burlington Chain of Custody (COC). A copy of the original COC sent by the client must be available in LIMS for all samples workshared within Eurofins TestAmerica Burlington Client COCs are only forwarded to external subcontractors when samples are shipped directly from the project site to the subcontractor lab. Under routine circumstances, client COCs are not provided to external subcontractors.

Through communication with the subcontracted laboratory, the PM monitors the status of the subcontracted analyses, facilitates successful execution of the work, and ensures the timeliness and completeness of the analytical report.

Non-TNI accredited work must be identified in the subcontractor's report as appropriate. If TNI accreditation is not required, the report does not need to include this information.

Reports submitted from subcontractor laboratories are not altered and are included in their original form in the final project report. This clearly identifies the data as being produced by a subcontractor facility. If subcontract laboratory data is incorporated into the laboratory's EDD (i.e., imported), the report must explicitly indicate which lab produced the data for which methods and samples.

Note: The results submitted by a Eurofins TestAmerica Burlington work sharing laboratory may be transferred electronically and the results reported by the Eurofins TestAmerica Burlington work sharing lab are identified on the final report. The report must explicitly indicate which lab produced the data for which methods and samples. The final report must include a copy of the completed COC for all work sharing reports.

10.4 Contingency Planning

The full qualification of a subcontractor may be waived to meet emergency needs. This decision and justification must be documented in the project files, and the 'Purchase Order Terms And Conditions For Subcontracted Laboratory Services' must be sent with the samples and COC.

In the event this provision is utilized, the laboratory (e.g., PM) will be required to verify and document the applicable accreditations of the subcontractor. All other quality and accreditation requirements will still be applicable, but the subcontractor need not have signed a subcontract agreement with Eurofins TestAmerica Burlington at this time.

The use of any emergency subcontractor will require the PM to complete a New Vendor Add Form in order to process payment to the vendor and add them to LIMS. This form requires the user to define the subcontractor's category/s of testing and the reason for testing.

10.5 Use of NELAP and A2LA Logo

It is not laboratory policy to use these logos on any company letterhead, including analytical reports.

11.0 PURCHASING SERVICES AND SUPPLIES

11.1 Overview

Evaluation and selection of suppliers and vendors is performed, in part, on the basis of the quality of their products, their ability to meet the demand for their products on a continuous and short term basis, the overall quality of their services, their past history, and competitive pricing. This is achieved through evaluation of objective evidence of quality furnished by the supplier, which can include certificates of analysis, recommendations, and proof of historical compliance with similar programs for other clients. To ensure that quality critical consumables and equipment conform to specified requirements, which may affect quality, all purchases from specific vendors are approved by a member of the supervisory or management staff. Capital expenditures are made in accordance with NDSC Document No. CW-F-S-007, Eurofins TestAmerica's Fixed Asset Acquisition, Retention and Safeguarding.

Contracts will be signed in accordance with the laboratory's authorization matrix in the NDSC Document No. CW-F-P-002, Company-Wide Authorization Matrix Policy. Request for Proposals (RFP's) will be issued where more information is required from the potential vendors than just price. Process details are available in the NDSC Document No. CW-F-P-004, Guidance on Procurement and Contracts Policy. RFP's allow the laboratory to determine if a vendor is capable of meeting requirements such as supplying all of the laboratory facilities, meeting required quality standards and adhering to necessary ethical and environmental standards. The RFP process also allows potential vendors to outline any additional capabilities they may offer.

11.2 Glassware

Glassware used for volumetric measurements must be Class A or verified for accuracy according to laboratory procedure. Pyrex (or equivalent) glass should be used where possible. For safety purposes, thick-wall glassware should be used where available.

11.3 Reagents, Standards & Supplies

Purchasing guidelines for equipment, consumables, and reagents must meet the requirements of the specific method and testing procedures for which they are being purchased. Solvents and acids are pre-tested in accordance with NDSC Document No. CA-Q-S-001, Solvent & Acid Lot Testing & Approval. Approval information for the solvents and acids tested under Document No. CA-Q-S-001 is stored on the laboratory's SharePoint site, under Solvent Approvals. A master list of all tested materials, as well as the certificates of analysis for the materials, is stored in the

same location.

11.3.1 Purchasing

Chemical reagents, solvents, glassware, and general supplies are ordered as needed to maintain sufficient quantities on hand. Materials used in the analytical process must be of a known quality. The wide variety of materials and reagents available makes it advisable to specify recommendations for the name, brand, and grade of materials to be used in any determination. This information is contained in the method SOP. The analyst completes the Material Request Sheet when requesting reagents, standards, or supplies. The analyst may check the item out of the on-site consignment system that contains items approved for laboratory use.

The analyst must provide the master item number (from the master item list that has been approved by the Technical Manager), item description, package size, catalogue page number, and the quantity needed. If an item being ordered is not the exact item requested, approval must be obtained from the Laboratory Directory or Technical Manager prior to placing the order. The purchasing manager places the order.

11.3.2 Receiving

It is the responsibility of the purchasing manager to receive the shipment. It is the responsibility of the analyst who ordered the materials to document the date materials were received. Once the ordered reagents or materials are received, the analyst compares the information on the label or packaging to the original order to ensure that the purchase meets the quality level specified. This is documented through the addition of the received date and initials to the information present on the daily order log.

The purchasing manager verifies the lot numbers of received solvents and acids against the pre-approval lists. If a received material is listed as unapproved, or is not listed, it is sequestered and returned to the vendor. Alternatively, the laboratory may test the material for the intended use, and if it is acceptable, document the approval on the approval list. Records of any testing performed locally are maintained on the shared “public” folder on the computer network.

Materials may not be released for use in the laboratory until they have been inspected, verified as suitable for use, and the inspection/verification has been documented.

Safety Data Sheets (SDSs) are available online through the Company’s intranet website. Anyone may review these for relevant information on the safe handling and emergency precautions of on-site chemicals.

11.3.3 Specifications

Methods used in the laboratory specify the grade of reagent that must be used in the procedure. If the quality of the reagent is not specified, analytical reagent grade will be used. It is the responsibility of the analyst to check the procedure carefully for the suitability of grade of reagent.

Chemicals must not be used past the manufacturer’s expiration date and must not be used past the expiration time noted in a method SOP. If expiration dates are not provided, the laboratory may contact the manufacturer to determine an expiration date.

The laboratory assumes a five year expiration date on inorganic dry chemicals and solvents unless noted otherwise by the manufacturer or by the reference source method.

Chemicals/solvents should not be used past the manufacturer's or SOP expiration date unless verified as outlined below.

- An expiration date **cannot** be extended if the dry chemical/solvent is discolored or appears otherwise physically degraded. In this case, the dry chemical/solvent must be discarded.
- Expiration dates can be extended if the dry chemical/solvent is found to be satisfactory based on acceptable performance of quality control samples (Continuing Calibration Verification (CCV), Blanks, Laboratory Control Sample (LCS), etc.).
- If the dry chemical/solvent is used for the preparation of standards, the expiration dates can be extended 6 months if the dry chemical/solvent is compared to an unexpired independent source in performing the method and the performance of the dry chemical/solvent is found to be satisfactory. The comparison must show that the dry chemical/solvent meets CCV limits. The comparison studies are maintained on-file and available for review by QA.

Wherever possible, standards must be traceable to national or international standards of measurement or to national or international reference materials. Records to that effect are available to the user.

Compressed gases in use are checked for pressure and secure positioning daily. To prevent a tank from going to dryness, or introducing potential impurities, the pressure should be closely watched as it decreases to approximately 15% of the original reading, at which point it should be replaced. For example, a standard sized laboratory gas cylinder containing 3,000 psig of gas should be replaced when it drops to approximately 500 psig. The quality of the gases must meet method or manufacturer specification or be of a grade that does not cause any analytical interference.

Water used in the preparation of samples, standards or reagents must have a specific conductivity of less than 1-µmho/cm (or specific resistivity of greater than 1.0 megohm-cm) at 25^{o}C. The specific conductivity is checked and recorded daily. If the water's specific conductivity is greater than the specified limit, the Facility Manager and appropriate Technical Managers must be notified immediately in order to notify all departments, decide on cessation (based on intended use) of activities, and make arrangements for correction.

The laboratory may purchase reagent grade (or other similar quality) water for use in the laboratory. This water must be certified clean by the supplier for all target analytes or otherwise verified by the laboratory prior to use. This verification is documented.

Standard lots are verified before first time use if the laboratory switches manufacturers or has historically had a problem with the type of standard.

Purchased bottleware used for sampling must be certified clean and the certificates must be maintained. If uncertified sampling bottleware is purchased, all lots must be verified clean prior to use. This verification must be maintained.

Records of manufacturer's certification and traceability statements are maintained in files or binders in each laboratory section. These records include date of receipt, lot number (when applicable), and expiration date (when applicable). Incorporation of the item into the record indicates that the analyst has compared the new certificate with the previous one for the same purpose and that no difference is noted, unless approved and so documented by the Technical Manager or QA Manager.

11.3.4 Storage

Reagent and chemical storage is important from the aspects of both integrity and safety. Light-sensitive reagents may be stored in brown-glass containers. Storage conditions are per the NDSC Environmental Health & Safety Manual, Document No. CW-E-M-001, the local laboratory EH&S manual addendum and method SOPs or manufacturer instructions.

11.4 Purchase of Equipment / Instruments / Software

When a new piece of equipment is needed, either for additional capacity or for replacing inoperable equipment, the analyst or supervisor makes a supply request to the Technical Manager and/or the Laboratory Director. If they agree with the request, the procedures outlined in NDSC Document No. CA-T-P-001**,** Qualified Products List are followed. A decision is made as to which piece of equipment can best satisfy the requirements. The appropriate written requests are completed and purchasing places the order.

Upon receipt of a new or used piece of equipment, an identification name is assigned and added to the equipment list. IT must also be notified so that they can synchronize the instrument for back-ups. Its capability is assessed to determine if it is adequate or not for the specific application. For instruments, a calibration curve is generated, followed by MDLs, Demonstration of Capabilities (DOCs), and other relevant criteria (refer to Section 20). For software, its operation must be deemed reliable and evidence of instrument verification must be retained by the IT Department or QA Department. Software certificates supplied by the vendors are filed with the LIMS Administrator. The manufacturer's operation manual is retained at the bench.

11.5 Services

Service to analytical instruments (except analytical balances) is performed on an as needed basis. Routine preventative maintenance is discussed in Section 22. The need for service is determined by analysts, department superiors, and/or Technical Manager. The service providers that perform the services are approved by the Laboratory Director and/or Technical Manager.

Analytical balances are serviced and calibrated annually in accordance with SOP No. BR-QA-012, Support Equipment Checks. The calibration and maintenance services are performed on-site, and the balances are returned to use immediately following successful calibration. Calibration certificates are filed for reference. If the calibration was unsuccessful, the balance is immediately removed from service and segregated pending either further maintenance or disposal.

Calibration services for support equipment such as thermometers, weight sets, autopipettors, etc., are obtained from vendors with current and valid ISO 17025 accreditation for calibration of the specific piece of equipment. Prior to utilizing the vendor's services, the vendor's accreditation status is verified. Once the equipment has been calibrated, the calibration certificates are reviewed by the QA department, and documentation of the review is filed with the calibration certificates. The equipment is then returned to service within the laboratory

11.6 Suppliers

The laboratory selects vendors through a competitive proposal / bid process, strategic business alliances or negotiated vendor partnerships (contracts)**.**

This process is defined in the NDSC Document No. CW-F-P-004, Procurement & Contracts

Policy. The level of control used in the selection process is dependent on the anticipated spending amount and the potential impact on the laboratory's business. Vendors that provide test and measuring equipment, solvents, standards, certified containers, instrument related service contracts or subcontract laboratory services shall be subject to more rigorous controls than vendors that provide off-the-shelf items of defined quality that meet the end use requirements. The purchasing system includes all suppliers/vendors that have been approved for use.

Evaluation of suppliers is accomplished by ensuring the supplier ships the product or material ordered and that the material is of the appropriate quality. This is documented by signing off on packing slips or other supply receipt documents. The purchasing documents contain the data that adequately describe the services and supplies ordered.

Any issues of vendor performance are to be reported immediately by the laboratory staff to the Purchasing Group by completing a Vendor Performance Report.

The Purchasing Group will work through the appropriate channels to gather the information required to clearly identify the problem and will contact the vendor to report the problem and to make any necessary arrangements for exchange, return authorization, credit, etc.

Suppliers are subject to re-evaluation, as deemed appropriate, through the use of Vendor Performance Reports used to summarize and review to determine corrective action necessary, or service improvements required by vendors.

The laboratory has access to a listing of all approved suppliers of critical consumables, supplies and services. This information is provided through the purchasing system.

11.6.1 New Vendor Procedure

Laboratory employees who wish to request the addition of a new vendor must complete a Vendor Add Request Form.

New vendors are evaluated based upon criteria appropriate to the products or services provided as well as their ability to provide those products and services at a competitive cost. Vendors are also evaluated to determine if there are ethical reasons or potential conflicts of interest with laboratory employees that would make it prohibitive to do business with them as well as their financial stability. The QA Department and/or the Technical Services Director are consulted with vendor and product selection that have an impact on quality.

12.0 COMPLAINTS

12.1 Overview

The laboratory considers an effective client complaint handling processes to be of significant business and strategic value. Listening to and documenting client concerns captures client knowledge that enables our operations to continually improve processes and client satisfaction. An effective client complaint handling process also provides assurance to the data user that the laboratory will stand behind its data, service obligations and products.

A client complaint is any expression of dissatisfaction with any aspect of our business services (e.g., communications, responsiveness, data, reports, invoicing and other functions) expressed by any party, whether received verbally or in written form. Client inquiries, complaints or noted discrepancies are documented, communicated to management, and addressed promptly and thoroughly.

The laboratory has procedures for addressing both external and internal complaints with the goal of providing satisfactory resolution to complaints in a timely and professional manner.

The nature of the complaint is identified, documented and investigated, and an appropriate action is determined and taken. In cases where a client complaint indicates that an established policy or procedure was not followed, the QA Department must evaluate whether a special audit must be conducted to assist in resolving the issue. A written confirmation or letter to the client, outlining the issue and response taken is recommended as part of the overall action taken.

The process of complaint resolution and documentation utilizes the procedures outlined in Section 14 (Corrective Actions) and is documented following SOP No. BR-QA-016, Non-Conformance and CAPA. A copy of this procedure will be made available to any interested party on request.

12.2 External Complaints

An employee that receives a complaint initiates the complaint resolution process by first documenting the complaint according to SOP No. BR-QA-016, Non-Conformance and CAPA.

Complaints fall into two categories: correctable and non-correctable. An example of a correctable complaint would be one where a report re-issue would resolve the complaint. An example of a non-correctable complaint would be one where a client complains that their data was repeatedly late. Non-correctable complaints should be reviewed for preventive action measures to reduce the likelihood of future occurrence and mitigation of client impact.

The general steps in the complaint handling process are:

- Receiving and documenting complaints
- Acknowledging receipt of complaint, whenever possible
- Complaint investigation and service recovery
- Process improvement

The laboratory shall inform the initiator of the complaint of the results of the investigation and the corrective action taken, if any.

12.3 Internal Complaints

Internal complaints include, but are not limited to: errors and non-conformances, training issues, internal audit findings, and deviations from methods. Corrective actions may be initiated by any staff member who observes a nonconformance and shall follow the procedures outlined in Section 14. In addition, Executive Management, Sales and Marketing and IT may initiate a complaint by contacting the laboratory or through the corrective action system described in Section 14.

12.4 Management Review

The number and nature of client complaints is reported by the QA Manager to the Laboratory Director and Quality Director in the QA Monthly report. Monitoring and addressing the overall level and nature of client complaints and the effectiveness of the solutions is part of the Annual Management Systems Review (Section 18).

13.0 CONTROL OF NON-CONFORMING WORK

13.1 Overview

When data discrepancies are discovered or deviations and departures from laboratory SOPs, policies and/or client requests have occurred, corrective action is taken immediately. First, the laboratory evaluates the significance of the nonconforming work. Then, a corrective action plan is initiated based on the outcome of the evaluation. If it is determined that the nonconforming work is an isolated incident, the plan could be as simple as adding a qualifier to the final results and/or making a notation in the case narrative. If it is determined that the nonconforming work is a systematic or improper practices issue, the corrective action plan could include a more in depth investigation and a possible suspension of an analytical method. In all cases, the actions taken are documented using the laboratory's corrective action system (refer to Section 14).

Due to the frequently unique nature of environmental samples, sometimes departures from documented policies and procedures are needed. When an analyst encounters such a situation, the problem is presented to the supervisor for resolution. The supervisor may elect to discuss it with the Technical Manager or have a representative contact the client to decide on a logical course of action. Once an approach is agreed upon, the analyst documents it using the laboratories corrective action system described in Section 14. This information can then be supplied to the client in the form of a footnote or a case narrative with the report.

Project Management may encounter situations where a client may request that a special procedure be applied to a sample that is not standard lab practice. Based on a technical evaluation, the lab may accept or opt to reject the request based on technical or ethical merit. An example might be the need to report a compound that the lab does not normally report. The lab would not have validated the method for this compound following the procedures in Section 21. The client may request that the compound be reported based only on the calibration. Such a request would need to be approved by the Technical Manager and QA Manager, documented and included in the project folder. Deviations **must** also be noted on the final report with a statement that the compound is not reported in compliance with TNI (or the analytical method) requirements and the reason. Data being reported to a non-TNI state would need to note the change made to how the method is normally run.

13.2 Responsibilities and Authorities

Under certain circumstances, the Laboratory Director, a Technical Manager, or QA Manager may authorize departures from documented procedures or policies. The departures may be a result of procedural changes due to the nature of the sample; a one-time procedure for a client; QC failures with insufficient sample to reanalyze, etc. In most cases, the client will be informed of the departure prior to the reporting of the data. Any departures must be well documented using the laboratory's corrective action procedures. This information may also be documented in logbooks and/or data review checklists as appropriate*.* Any impacted data must be referenced in a case narrative and/or flagged with an appropriate data qualifier.

Any misrepresentation or possible misrepresentation of analytical data discovered by any laboratory staff member must be reported to facility Senior Management within 24-hours. The Senior Management staff is comprised of the Laboratory Director, the QA Manager, and the Technical Managers. The reporting of issues involving alleged violations of the company's Data Integrity or Manual Integration procedures must be conveyed to an ECO (e.g., the VP-QA/EHS) and the laboratory's Quality Director within 24 hours of discovery.

Whether an inaccurate result was reported due to calculation or quantitation errors, data entry errors, improper practices, or failure to follow SOPs, the data must be evaluated to determine the possible effect.

The Laboratory Director, QA Manager, ECOs, VP of Operations and the Quality Directors have the authority and responsibility to halt work, withhold final reports, or suspend an analysis for due cause as well as authorize the resumption of work.

13.3 Evaluation of Significance and Actions Taken

For each nonconforming issue reported, an evaluation of its significance and the level of management involvement needed is made. This includes reviewing its impact on the final data, whether or not it is an isolated or systematic issue, and how it relates to any special client requirements.

The NDSC Document entitled Data Recalls (CW-Q-S-005) is the procedure to be followed when it is discovered that erroneous or biased data may have been reported to clients or regulatory agencies.

The NDSC Document entitled Internal Investigations (CW-L-S-002) is the procedure to be followed for investigation and correction of situations involved alleged incidents of misconduct or violation of the company's ethics policy.

Laboratory level decisions are documented and approved using the laboratory's standard nonconformance/corrective action reporting in lieu of the data recall determination form contained in NDSC Document No. CW-Q-S-005.

13.4 Prevention of Nonconforming Work

If it is determined that the nonconforming work could recur, further corrective actions must be made following the laboratory's corrective action system. Periodically as defined by the laboratory's preventive action schedule, the QA Department evaluates non-conformances to determine if any nonconforming work has been repeated multiple times. If so, the laboratory's corrective action process may be followed.

13.5 Method Suspension / Restriction (Stop Work Procedures)

In some cases, it may be necessary to suspend/restrict the use of a method or target analyte which constitutes significant risk and/or liability to the laboratory. Suspension/restriction procedures can be initiated by any of the persons noted in Section 13.2, Paragraph 5.

Prior to suspension/restriction, confidentiality will be respected, and the problem with the required corrective and preventive action will be stated in writing and presented to the Laboratory Director.

The Laboratory Director shall arrange for the appropriate personnel to meet with the QA Manager as needed. This meeting shall be held to confirm that there is a problem, that suspension/restriction of the method is required and will be concluded with a discussion of the steps necessary to bring the method/target or test fully back on line. In some cases, that may not be necessary if all appropriate personnel have already agreed there is a problem and there is agreement on the steps needed to bring the method, target or test fully back on line. The QA Manager will also initiate a corrective action report as described in Section 14 if one has not already been started. A copy of any meeting notes and agreed upon steps should be e-mailed

by the laboratory to their Business Unit President; VP-QA & EHS. This e-mail acts as notification of the incident.

After suspension/restriction, the lab will hold all reports to clients pending review. No faxing, mailing or distributing through electronic means may occur. The report must not be posted for viewing on the internet. It is the responsibility of the Laboratory Director to hold all reporting and to notify all relevant laboratory personnel regarding the suspension/restriction (e.g., Project Management, Log-in, etc.). Clients will NOT generally be notified at this time. Analysis may proceed in some instances depending on the non-conformance issue.

Within 72 hours, the QA Manager will determine if compliance is now met and reports can be released, OR determine the plan of action to bring work into compliance, and release work. A team, with all principals involved (e.g., Laboratory Director, Technical Manager, QA Manager) can devise a start-up plan to cover all steps from client notification through compliance and release of reports. Project Management and the Directors of Client Services and Sales and Marketing must be notified if clients must be notified or if the suspension/restriction affects the laboratory's ability to accept work. The QA Manager must approve start-up or elimination of any restrictions after all corrective action is complete.

14.0 CORRECTIVE ACTION

14.1 Overview

A major component of the laboratory's (QA) Program is the problem investigation and feedback mechanism designed to keep the laboratory staff informed on quality related issues and to provide insight to problem resolution. When nonconforming work or departures from policies and procedures in the quality system or technical operations are identified, the corrective action procedure provides a systematic approach to assess the issues, restore the laboratory's system integrity, and prevent reoccurrence. The laboratory employs two systems to manage non-conformances. Issues suspected of being systematic in nature and for which root cause analysis and a formal Corrective Action Report (CAR) are documented in the Incident Corrective Action Tracking (ICAT) database. Routine batch non-conformances, events that are understood to be isolated in nature, are documented in the LIMS non-conformance memo (NCM) system.

14.2 General

Problems within the quality system or within analytical operations may be discovered in a variety of ways, such as QC sample failures, internal or external audits, proficiency testing (PT) performance, client complaints, staff observation, etc.

The purpose of a corrective action system is to:

- Identify non-conformance events and assign responsibility for investigating.
- Resolve non-conformance events and assign responsibility for any required corrective action.
- Identify systematic problems before they become serious.
- Identify and track client complaints and provide resolution.

14.2.1 Non-Conformance Memo (NCM)

NCMs are used to document the following types of corrective actions:

- Deviations from an established procedure or SOP
- QC outside of limits
- Isolated reporting / calculation errors
- Client complaints
- Discrepancies in materials / goods received vs. manufacturer packing slips (Forms of documentation other than NCMs in LIMS are also acceptable)

14.2.2 Corrective Actions Documented In the ICAT Database

- Internal and external audit findings
- Failed or unacceptable PT results
- Identified poor process or method performance trends
- Systematic reporting / calculation errors
- Data recall investigations
- Questionable trends that are found in the review of NCMs.
- Client complaints
- Excessive revised reports

The ICAT database is used to document background information, track the results of corrective action investigations and root cause analysis, and to provide reports of corrective action plans.

14.3 Closed Loop Corrective Action Process

Any employee in the company can initiate a corrective action. There are four main components to a closed-loop corrective action process once an issue has been identified: Cause Analysis, Selection and Implementation of Corrective Actions (both short and long term), Monitoring of the Corrective Actions, and Follow-up.

14.3.1 Cause Analysis

- Upon discovery of a non-conformance event, the event must be defined and documented. An entry into the ICAT system must be initiated, someone is assigned to investigate the issue and the event is investigated for cause. Table 12-1 provides some general guidelines on determining responsibility for assessment.
- The cause analysis step is the key to the process as a long term corrective action cannot be determined until the cause is determined.
- If the cause is not readily obvious, the Technical Manager, Laboratory Director, or QA Manager (or QA designee) is consulted.

14.3.2 Selection and Implementation of Corrective Actions

- Where corrective action is needed, the laboratory shall identify potential corrective actions. The action(s) most likely to eliminate the problem and prevent recurrence are selected and implemented. Responsibility for implementation is assigned.
- Corrective actions shall be to a degree appropriate to the magnitude of the problem identified through the cause analysis.
- Whatever corrective action is determined to be appropriate, the laboratory shall document and implement the changes. The ICAT record is used for this documentation.

14.3.3 Root Cause Analysis

Root Cause Analysis is a class of problem solving (investigative) methods aimed at identifying the basic or causal factor(s) that underlie variation in performance or the occurrence of a significant failure. The root cause may be buried under seemingly innocuous events, many steps preceding the perceived failure. At first glance, the immediate response is typically directed at a symptom and not the cause. Typically, root cause analysis would be best with three or more incidents to triangulate a weakness. NDSC Document No. CA-Q-S-009, Root Cause Analysis, provides guidance on this procedure.

Systematically analyze and document the root causes of the more significant problems that are reported. Identify, track, and implement the corrective actions required to reduce the likelihood of recurrence of significant incidents. Trend the root cause data from these incidents to identify root causes that, when corrected, can lead to dramatic improvements in performance by eliminating entire classes of problems.

Identify the one event associated with problem and ask why this event occurred. Brainstorm the root causes of failures; for example, by asking why events occurred or conditions existed; and then why the cause occurred consecutive times until you get to the root cause. For each of these sub events or causes, ask why it occurred. Repeat the process for the other events associated with the incident.

Root cause analysis does not mean the investigation is over. Look at technique or other systems outside the normal indicators. Often creative thinking will find root causes that ordinarily would be missed and continue to plague the laboratory or operation.

14.3.4 Monitoring of the Corrective Actions

- The Technical Manager and QA Manager are responsible to ensure that the corrective action taken was effective.
- Ineffective actions are documented and re-evaluated until acceptable resolution is achieved. Technical Managers are accountable to the Laboratory Director to ensure final acceptable resolution is achieved and documented appropriately.
- The QA Manager reviews monthly NCM and ICAT records for trends. Highlights are included in the QA monthly report (refer to Section 18). If a significant trend develops that adversely affects quality, an audit of the area is performed and corrective action implemented.
- Any out-of-control situations that are not addressed acceptably at the laboratory level may be reported to the NDSC Quality Director by the QA Manager, indicating the nature of the out-of-control situation and problems encountered in solving the situation.

14.3.5 Follow-up Audits

- Follow-up audits may be initiated by the QA Manager and shall be performed as soon as possible when the identification of a nonconformance casts doubt on the laboratory's compliance with its own policies and procedures, or on its compliance with state or federal requirements.
- These audits often follow the implementation of the corrective actions to verify effectiveness. An additional audit would only be necessary when a critical issue or risk to business is discovered.

(Also refer to Section 17.1.4, Special Audits.)

14.4 Technical Corrective Actions

In addition to providing acceptance criteria and specific protocols for technical corrective actions in the method SOPs, the laboratory has general procedures to be followed to determine when departures from the documented policies and procedures and quality control have occurred (refer to Section 13). The documentation of these procedures is through the use of an NCM or record in the ICAT system.

Table 12-1 includes examples of general technical corrective actions. For specific criteria and corrective actions, refer to the analytical methods or specific method SOPs. The laboratory may also maintain Work Instructions on these items that are available upon request.

Table 12-1 provides some general guidelines for identifying the individual(s) responsible for assessing each QC type and initiating corrective action. The table also provides general guidance on how a data set should be treated if associated QC measurements are unacceptable. Specific procedures are included in Method SOPs, Work Instructions, QA Manual Sections 19 and 20. All corrective actions are reviewed monthly, at a minimum, by the QA Manager and highlights are included in the QA monthly report.

To the extent possible, samples shall be reported only if all quality control measures are acceptable. If the deficiency does not impair the usability of the results, data will be reported with an appropriate data qualifier and/or the deficiency will be noted in the case narrative. Where sample results may be impaired, the PM is notified by an NCM and appropriate corrective action (e.g., reanalysis) is taken and documented**.**

14.5 Basic Corrections

When mistakes occur in records, each mistake shall be crossed-out, [not obliterated (e.g. no white-out)], and the correct value entered alongside. All such corrections shall be initialed (or signed) and dated by the person making the correction. In the case of records stored electronically, the original uncorrected file must be maintained intact and a second corrected file is created. This same process applies to adding additional information to a record. All additions made later than the initial must also be initialed (or signed) and dated. When corrections are due to reasons other than obvious transcription errors, the reason for the corrections (or additions) shall also be documented.

Table 14-1. Example – General Corrective Action Procedures

QC Activity ***(Individual Responsible for Initiation/Assessment)***	**Acceptance Criteria**	**Recommended Corrective Action**
Initial Instrument Blank *(Analyst)*	- Instrument response < MDL.	- Prepare another blank. - If same response, determine cause of contamination: reagents, environment, instrument equipment failure, etc..
Initial Calibration Standards *(Analyst, Technical Manager(s))*	- Correlation coefficient > 0.99 or standard concentration value. - % Recovery within acceptance range. - See details in Method SOP.	- Reanalyze standards. - If still unacceptable, remake standards and recalibrate instrument.
Independent Calibration Verification (Second Source) *(Analyst, Technical Manager(s))*	- % Recovery within control limits.	- Remake and reanalyze standard. - If still unacceptable, then remake calibration standards or use new primary standards and recalibrate instrument.
Continuing Calibration Standards *(Analyst, Data Reviewer)*	% Recovery within control limits.	- Reanalyze standard. - If still unacceptable, then recalibrate and rerun affected samples.
Matrix Spike / Matrix Spike Duplicate (MS/MSD) *(Analyst, Data Reviewer)*	- % Recovery within limits documented in LIMS	- If the acceptance criteria for duplicates or matrix spikes are not met because of matrix interferences, the acceptance of the analytical batch is determined by the validity of the LCS. - If the LCS is within acceptable limits the batch is acceptable. - The results of the duplicates, matrix spikes and the LCS are reported with the data set. - For matrix spike or duplicate results outside criteria the data for that sample shall be reported with qualifiers.

QC Activity ***(Individual Responsible for Initiation/Assessment)***	**Acceptance Criteria**	**Recommended Corrective Action**
Laboratory Control Sample (LCS) *(Analyst, Data Reviewer)*	- % Recovery within limits specified in LIMS	- Batch must be re-prepared and re-analyzed. This includes any allowable marginal exceedance. When not using marginal exceedances, the following exceptions apply: 1) when the acceptance criteria for the positive control are exceeded high (i.e., high bias) and there are associated samples that are non-detects, then those non-detects may be reported with data qualifying codes; 2) when the acceptance criteria for the positive control are exceeded low (i.e., low bias), those sample results may be reported if they exceed a maximum regulatory limit/decision level with data qualifying codes. **Note:** If there is insufficient sample or the holding time cannot be met, contact client and report with flags.
Surrogates *(Analyst, Data Reviewer)*	- % Recovery within limits of method or within three standard deviations of the historical mean.	- Individual sample must be repeated. Place comment in LIMS. - Surrogate results outside criteria shall be reported with qualifiers.
Method Blank (MB) *(Analyst, Data Reviewer)*	< Reporting Limit [1]	- Reanalyze blank. - If still positive, determine source of contamination. If necessary, reprocess (i.e. digest or extract) entire sample batch. Report blank results. - Qualify the result(s) if the concentration of a targeted analyte in the MB is at or above the reporting limit AND is > 1/10 of the amount measured in the sample.
Proficiency Testing (PT) Samples *(QA Manager, Technical Manager(s))*	- Criteria supplied by PT Supplier.	- Any failures or warnings must be investigated for cause. Failures may result in the need to repeat a PT sample to show the problem is corrected.
Internal / External Audits *(QA Manager, Technical Manager(s), Laboratory Director)*	- Defined in Quality System documentation such as SOPs, QAM, etc.	- Non-conformances must be investigated through CAR system and necessary corrections must be made.

QC Activity ***(Individual Responsible for Initiation/Assessment)***	**Acceptance Criteria**	**Recommended Corrective Action**
Reporting / Calculation Errors (Depends on issue – possible individuals include: Analysts, Data Reviewers, Project Managers, Technical Managers, QA Manager, NDSCQA, Executive Management)	- NDSC Document No. CW-Q-S-005, Data Recall **or** local SOP No.	- Corrective action is determined by type of error. Follow the procedures in NDSC Document No. CW-L-S-002 **or** your lab's CA SOP.
Client Complaints *(Project Managers, Lab Director/Manager, Sales and Marketing)*	-	- Corrective action is determined by the type of complaint. For example, a complaint regarding an incorrect address on a report will result in the report being corrected and then follow-up must be performed on the reasons the address was incorrect (e.g., database needs to be updated).
QA Monthly Report (Refer to Section 16 for an example) (QA Manager, Lab Director/Manager, *Technical Manager(s)*)	- QAM, SOPs.	- Corrective action is determined by the type of issue. For example, CARs for the month are reviewed and possible trends are investigated.
Health and Safety Violation (Safety Officer, Lab Director/Manager, *Technical Manager(s)*)	- Environmental Health and Safety (EHS) Manual.	- Non-conformance is investigated and corrected through CAR system.

Note:
1. Except as noted below for certain compounds, the method blank should be below the detection limit, Concentrations up to five times the reporting limit will be allowed for the ubiquitous laboratory and reagent contaminants: methylene chloride, toluene, acetone, 2-butanone and phthalates **provided** they appear in similar levels in the reagent blank and samples. This allowance presumes that the detection limit is significantly below any regulatory limit to which the data are to be compared and that blank subtraction will not occur. For benzene and ethylene dibromide (EDB) and other analytes for which regulatory limits are extremely close to the detection limit, the method blank must be below the method detection limit.

15.0 PREVENTIVE ACTION / IMPROVEMENT

15.1 Overview

The laboratory's preventive action programs improve or eliminate potential causes of nonconforming product and/or nonconformance to the quality system. This preventive action process is a proactive and continuous process of improvement activities that can be initiated through feedback from clients, employees, business providers, and affiliates. The QA Department has the overall responsibility to ensure that the preventive action process is in place, and that relevant information on actions is submitted for management review.

Dedicating resources to an effective preventive action system emphasizes the laboratory's commitment to its QA Program. It is beneficial to identify and address negative trends before they develop into complaints, problems and corrective actions. Additionally, the laboratory continually strives to improve customer service and client satisfaction through continuous improvements to laboratory systems.

Opportunities for improvement may be discovered through any of the following:

- review of the monthly QA Metrics Report,
- trending NCMs,
- review of control charts and QC results,
- trending proficiency testing (PT) results,
- performance of management system reviews,
- trending client complaints,
- review of processing operations, or
- staff observations.

The monthly Management Systems Metrics Report shows performance indicators in all areas of the laboratory and quality system. These areas include revised reports, corrective actions, audit findings, internal auditing and data authenticity audits, client complaints, PT samples, holding time violations, SOPs, ethics training, etc. The metrics report is reviewed monthly by the laboratory management, NDSC QA Team Local and Executive Mangement. These metrics are used ~~to~~ in evaluating the management and quality system performance on an ongoing basis and provide a tool for identifying areas for improvement.

Items identified as continuous improvement opportunities to the management system may be issued as goals from the annual management systems review, recommendations from internal audits, white papers, Lessons Learned, Technical Services audit report, Technical Best Practices, or as Executive or management initiatives.

The laboratory's corrective action process is integral to implementation of preventive actions. A critical piece of the corrective action process is the implementation of actions to prevent further occurrence of a non-compliance event. Historical review of corrective action and non-conformances provides a valuable mechanism for identifying preventive action opportunities.

15.1.1 The following elements are part of a preventive action/process improvement system:

- Identification of an opportunity for preventive action or process improvement.
- Process for the preventive action or improvement.

- Define the measurements of the effectiveness of the process once undertaken.
- Execution of the preventive action or improvement.
- Evaluation of the plan using the defined measurements.
- Verification of the effectiveness of the preventive action or improvement.
- Close-Out by documenting any permanent changes to the Quality System as a result of the Preventive Action or Process Improvement. Documentation of Preventive Action/process Improvement is incorporated into the monthly QA reports, corrective action process and management review.

15.1.2 Any preventive actions/process improvement undertaken or attempted shall be taken into account during the annual Management Systems Review (Section 16). A highly detailed report is not required; however, a summary of successes and failures within the preventive action program is sufficient to provide management with a measurement for evaluation.

16.0 CONTROL OF RECORDS

The laboratory maintains a records management system appropriate to its needs and that complies with applicable standards or regulations as required. The system produces unequivocal, accurate records that document all laboratory activities. The laboratory retains all original observations, calculations and derived data, calibration records and a copy of the analytical report for a minimum of five years after it has been issued. Exceptions for programs with longer retention requirements are discussed in Section 14.1.2.

16.1 Overview

The laboratory has established procedures for identification, collection, indexing, access, filing, storage, maintenance and disposal of quality and technical records. A record index is listed in Table 14-1. More detailed information on retention of specific records is provided in NDSC Document Nos. CW-L-P-001, Records Retention Policy and CW-L-WI-001, Records Retention/Storage Schedule. Quality records are maintained by the QA department in a database, which is backed up as part of the regular laboratory backup. Records are of two types; either electronic or hard copy paper formats depending on whether the record is computer or hand generated (some records may be in both formats). Technical records are maintained by QA Department

Table 16-1. Record Index

	Record Types [1]:	Retention Time:
Technical Records	- Raw Data - Logbooks[2] - Standards - Certificates - Analytical Records - MDLs/IDLs/DOCs - Lab Reports	5 Years from analytical report issue*
Official Documents	- Quality Assurance Manual (QAM) - Work Instructions - Policies - SOPs - Policy Memorandums - Manuals - Published Methods	Indefinitely
QA Records	- Certifications - Method and Software Validation / Verification Data	Indefinitely
QA Records	- Internal & External Audits/Responses - Corrective/Preventive Actions - Management Reviews - Data Investigation	5 Years from archival* **Data Investigation:** 5 years or the life of the affected raw data storage whichever is greater (beyond 5 years if ongoing project or pending investigation)
Project Records	- Sample Receipt & COC Documents - Contracts and Amendments - Correspondence - QAPP - SAP - Telephone Logbooks - Lab Reports	5 Years from analytical report issue*
Administrative Records	Financial and Business Operations	Refer to NDSC Doc. No. CW-L-WI-001
	EH&S Manual, Permits	Indefinitely
	Disposal Records	Indefinitely
	Employee Handbook	Indefinitely
	Personnel files, Employee Signature & Initials, Administrative Training Records (e.g., Ethics)	Refer to HR Manual
	Administrative Policies	Indefinitely
	Technical Training Records	7 years
	Legal Records	Indefinitely
	HR Records	Refer to NDSC Doc. No. CW-L-WI-001
	IT Records	Refer to NDSC Doc. No. CW-L-WI-001
	NDSC Governance Records	Refer to NDSC Doc. No. CW-L-WI-001
	Sales & Marketing	5 years
	Real Estate	Indefinitely

[1] Record Types encompass hardcopy and electronic records.

[2] Examples of Logbook types: Maintenance, Instrument Run, Preparation (standard and samples), Standard and Reagent Receipt, Archiving, Balance Calibration, Temperature (hardcopy or electronic records).
* Exceptions listed in Table 16-2.

16.1.1 All records are stored and retained in such a way that they are secure and readily retrievable at the laboratory facility or the Iron Mountain data storage facility that provides a suitable environment to prevent damage or deterioration and to prevent loss. All records shall be protected against fire, theft, loss, environmental deterioration, and vermin. In the case of electronic records, electronic or magnetic sources, storage media are protected from deterioration caused by magnetic fields and/or electronic deterioration.

Access to the data is limited to laboratory and company employees and shall be documented with an access log**.** Records archived off-site are stored in a secure location where a record is maintained of any entry into the storage facility. Whether on-site or off-site storage is used, logs are maintained in each storage box to note removal and return of records. Retention of records are maintained on-site at the laboratory for at least 1 year after their generation and moved offsite for the remainder of the required storage time. Records are maintained for a minimum of five years unless otherwise specified by a client or regulatory requirement.

For raw data and project records, record retention shall be calculated from the date the project report is issued. For other records, such as NDSC and/or KGD Documents, QA, or Administrative Records, the retention time is calculated from the date the record is formally retired. Records related to the programs listed in Table 16-2 have lengthier retention requirements and are subject to the requirements in Section 16.1.3.

16.2 Programs with Longer Retention Requirements

Some regulatory programs have longer record retention requirements than the standard record retention time. These are detailed in Table 16-2 with their retention requirements. In these cases, the longer retention requirement is enacted. If special instructions exist such that client data cannot be destroyed prior to notification of the client, the container or box containing that data is marked as to who to contact for authorization prior to destroying the data.

Table 16-2. Example: Special Record Retention Requirements

Program	**[1]Retention Requirement**
Drinking Water – All States	10 years (lab reports and raw data) 10 years - Radiochemistry (project records)
Drinking Water Lead and Copper Rule	12 years (project records)
DOE Hanford	5 years, Written approval must be received from DOE Hanford prior to disposal of analytical data
Commonwealth of MA – All environmental data 310 CMR 42.14	10 years
FIFRA – 40 CFR Part 160	Retain for life of research or marketing permit for pesticides regulated by EPA
Housing and Urban Development (HUD) Environmental Lead Testing	10 years
Alaska	10 years
Louisiana – All	10 years

Program	[1]Retention Requirement
Michigan Department of Environmental Quality – all environmental data	10 years
Navy Facilities Engineering Service Center (NFESC)	10 years
Ohio VAP	10 years and State contacted prior to disposal
OSHA	30 years

[1]Note: Extended retention requirements must be noted with the archive documents or addressed in facility-specific records retention procedures.

16.2.1 The laboratory has procedures to protect and back-up records stored electronically and to prevent unauthorized access to or amendment of these records. All analytical data is maintained as hard copy or in a secure readable electronic format. For analytical reports that are maintained as copies in PDF format, refer to Section 19.15.1 for more information. Also see SOP BR-QA-021, Electronic Data Archive Burlington.

16.2.2 The record keeping system allows for historical reconstruction of all laboratory activities that produced the analytical data, as well as rapid recovery of historical data. Records stored off site should be accessible within 2 days of a request for such records. The history of the sample from when the laboratory took possession of the samples must be readily understood through the documentation. This shall include inter-laboratory transfers of samples and/or extracts.

- The records include the identity of personnel involved in sampling, sample receipt, preparation, or testing. All analytical work contains the initials (at least) of the personnel involved. The laboratory's copy of the COC is stored with the invoice and the work order sheet generated by LIMS. The chain of custody would indicate the name of the sampler. If any sampling notes are provided with a work order, they are kept with this package.
- All information relating to the laboratory facilities' equipment, analytical test methods, and related laboratory activities, such as sample receipt, sample preparation, or data verification are documented.
- The record keeping system facilitates the retrieval of all working files and archived records for inspection and verification purposes (e.g., set format for naming electronic files, set format for what is included with a given analytical data set in laboratory method SOPs. Instrument data is stored sequentially by instrument. A given day's analyses are maintained in the order of the analysis. Run logs are maintained for each instrument or method; a copy of each day's run log or instrument sequence is stored with the data to aid in re-constructing an analytical sequence. Where an analysis is performed without an instrument, bound logbooks or bench sheets are used to record and file data. Standard and reagent information is recorded in logbooks or entered into LIMS for each method as required.
- Changes to hardcopy records shall follow the procedures outlined in Section 12 and 19. Changes to electronic records in LIMS or instrument data are recorded in audit trails.
- The reason for a signature or initials on a document is clearly indicated in the records such as "sampled by," "prepared by," "reviewed by", or "analyzed by".
- All generated data except those that are generated by automated data collection systems, are recorded directly, promptly and legibly in permanent dark ink.
- Hard copy data may be scanned into PDF format for record storage as long as the scanning

process can be verified in order to ensure that no data is lost and the data files and storage media must be tested to verify the laboratory's ability to retrieve the information prior to the destruction of the hard copy that was scanned. The procedure for this verification can be found in SOP BR-QA-003, Document Control.

- Also refer to Section 19.15.1 'Computer and Electronic Data Related Requirements'.

16.3 Technical and Analytical Records

16.3.1 The laboratory retains records of original observations, derived data and sufficient information to establish an audit trail, calibration records, staff records and a copy of each analytical report issued, for a minimum of five years unless otherwise specified by a client or regulatory requirement. The records for each analysis shall contain sufficient information to enable the analysis to be repeated under conditions as close as possible to the original. The records shall include the identity of laboratory personnel responsible for the sampling, performance of each analysis and reviewing results.

16.3.2 Observations, data and calculations are recorded real-time and are identifiable to the specific task.

16.3.3 Changes to hardcopy records shall follow the procedures outlined in Section 12 and 19. Changes to electronic records in LIMS or instrument data are recorded in audit trails.

The essential information to be associated with analysis, such as strip charts, tabular printouts, computer data files, analytical notebooks, and run logs, include:

- Laboratory sample ID code;
- Date of analysis; time of analysis is also required if the holding time is seventy-two (72) hours or less, or when time critical steps are included in the analysis (e.g., drying times, incubations, etc.); instrumental analyses have the date and time of analysis recorded as part of their general operations. Where a time critical step exists in an analysis, location for such a time is included as part of the documentation in a specific logbook or on a benchsheet.
- Instrumentation identification and instrument operating conditions/parameters. Operating conditions/parameters are typically recorded in instrument maintenance logs where available.
- analysis type;
- all manual calculations and manual integrations;
- analyst's or operator's initials/signature;
- sample preparation including cleanup, separation protocols, incubation periods or subculture, ID codes, volumes, weights, instrument printouts, meter readings, calculations, reagents;
- test results;
- standard and reagent origin, receipt, preparation, and use;
- calibration criteria, frequency and acceptance criteria;
- data and statistical calculations, review, confirmation, interpretation, assessment and reporting conventions;
- quality control protocols and assessment;

- electronic data security, software documentation and verification, software and hardware audits, backups, and records of any changes to automated data entries; and
- Method performance criteria including expected quality control requirements. These are indicated both in LIMS and on specific analytical report formats**.**

16.3.4 All logbooks used during receipt, preparation, storage, analysis, and reporting of samples or monitoring of support equipment shall undergo a periodic, documented supervisory or peer review.

16.4 Laboratory Support Activities

In addition to documenting all the above-mentioned activities, the following are retained QA records and project records (previous discussions in this section relate where and how these data are stored):

- all original raw data, whether hard copy or electronic, for calibrations, samples and quality control measures, including analysts' work sheets and data output records (chromatograms, strip charts, and other instrument response readout records)**;**
- a written description or reference to the specific test method used which includes a description of the specific computational steps used to translate parametric observations into a reportable analytical value**;**
- copies of final reports**;**
- archived SOPs**;**
- correspondence relating to laboratory activities for a specific project**;**
- all corrective action reports, audits and audit responses**;**
- proficiency test results and raw data; and
- results of data review, verification, and crosschecking procedures

16.4.1 Sample Handling Records

Records of all procedures to which a sample is subjected while in the possession of the laboratory are maintained. These include but are not limited to records pertaining to:

- sample preservation including appropriateness of sample container and compliance with holding time requirement;
- sample identification, receipt, acceptance or rejection and login;
- sample storage and tracking including shipping receipts, sample transmittal / COC forms; and
- procedures for the receipt and retention of samples, including all provisions necessary to protect the integrity of samples.

16.5 Administrative Records

The laboratory also maintains the administrative records in either electronic or hard copy form. Refer to Table 16-1.

16.6 Records Management, Storage and Disposal

All records (including those pertaining to test equipment), certificates and reports are safely stored, held secure and in confidence to the client. Certification related records are available upon request.

All information necessary for the historical reconstruction of data is maintained by the laboratory. Records that are stored only on electronic media must be supported by the hardware and software necessary for their retrieval.

Records that are stored or generated by computers or personal computers have hard copy, write-protected backup copies, or an electronic audit trail controlling access**.**

The laboratory has a record management system (a.k.a., document control) for control of laboratory notebooks, instrument logbooks, standards logbooks, and records for data reduction, validation, storage and reporting. Laboratory notebooks are issued on a per analysis basis, and are numbered sequentially. All data are recorded sequentially within a series of sequential notebooks. Bench sheets are filed sequentially. Standards are maintained in LIMS – no logbooks are used to record that data**.** Records are considered archived when noted as such in the records management system (a.k.a., document control.)

16.6.1 Transfer of Ownership

In the event that the laboratory transfers ownership or goes out of business, the laboratory shall ensure that the records are maintained or transferred according to client's instructions. Upon ownership transfer, record retention requirements shall be addressed in the ownership transfer agreement and the responsibility for maintaining archives is clearly established. In addition, in cases of bankruptcy, appropriate regulatory and state legal requirements concerning laboratory records must be followed. In the event of the closure of the laboratory, all records will revert to the control of NDSC. Should the entire company cease to exist, as much notice as possible will be given to clients and the accrediting bodies who have worked with the laboratory during the previous 5 years of such action.

16.6.2 Records Disposal

Records are removed from the archive and destroyed after 5 years unless otherwise specified by a client or regulatory requirement. On a project specific or program basis, clients may need to be notified prior to record destruction. Records are destroyed in a manner that ensures their confidentiality such as shredding, mutilation or incineration. (Refer to Tables 14-1 and 14-2).

Electronic copies of records must be destroyed by erasure or physically damaging off-line storage media so no records can be read.

If a third party records management company is hired to dispose of records, a "Certificate of Destruction" is required.

17.0 AUDITS

17.1 Internal Audits

Internal audits are performed to verify that laboratory operations comply with the requirements of the lab's quality system and with the external quality programs under which the laboratory operates. Audits are planned and organized by the QA staff. Personnel conducting the audits should be independent of the area being evaluated. Auditors will have sufficient authority,

access to work areas, and organizational freedom necessary to observe all activities affecting quality and to report the assessments to laboratory management and, when requested, to Executive Management.

Audits are conducted and documented as described in the NDSC Document No. on performing Internal Auditing, SOP No. CW-Q-S-003. The types and frequency of routine internal audits are described in Table 17-1. Special or ad hoc assessments may be conducted as needed under the direction of the QA staff.

Table 17-1. Types of Internal Audits and Frequency

Description	Performed by	Frequency
Quality Systems Audits	QA Department, QA approved designee, or NDSC QA	All areas of the laboratory annually
Method Audits QA Technical Audits	Joint responsibility: a) QA Manager or designee b) Technical Manager or Designee (Refer to NDSC CW-Q-S-003)	QA Technical Audits Frequency: 50% of methods annually
SOP Method Compliance	Joint responsibility: a) QA Manager or designee b) Technical Manager or Designee (Refer to NDSC CW-Q-S-003)	SOP Compliance Review Frequency: • Every 2 years • Every 1 year (Drinking Water Methods) • 100% of SOPs annually (DoD/DOE Labs)
Special	QA Department or Designee	Surveillance or spot checks performed as needed, e.g., to confirm corrective actions from other audits.
Performance Testing	Analysts with QA oversight	Two successful per year for each TNI-field of testing or as dictated by regulatory requirements

17.1.1 Annual Quality Systems Audit

An annual quality systems audit is required to ensure compliance to analytical methods and SOPs, Eurofins Data Integrity and Ethics Policies (see Sec. 7.2), TNI quality systems, client and state requirements, and the effectiveness of the internal controls of the analytical process, including but not limited to data review, quality controls, preventive action and corrective action. The completeness of earlier corrective actions is assessed for effectiveness & sustainability. The audit is divided into sections for each operating or support area of the lab, and each section is comprehensive for a given area. The area audits may be performed on a rotating schedule throughout the year to ensure adequate coverage of all areas. This schedule may change as situations in the laboratory warrant.

17.1.2 QA Technical Audits

QA technical audits assess data authenticity and analyst integrity. These audits are based on client projects, associated sample delivery groups, and the methods performed. Reported results are compared to raw data to verify the authenticity of results. The validity of calibrations and QC results are compared to data qualifiers, footnotes, and case narratives. Documentation is assessed by examining run logs and records of manual integrations. Manual calculations are checked. Where possible, electronic audit miner programs (e.g., Chrom AuditMiner) are used to identify unusual manipulations of the data deserving closer scrutiny. QA technical audits will include all methods within a two-year period.

17.1.3 SOP Method Compliance

Compliance of all SOPs with the source methods and compliance of the operational groups with the SOPs will be assessed by the Technical Manager or qualified designee at least every two years. (Annually for methods and administrative SOPs related to DoD/DOE programs and DW compliance methods. - where applicable) It is also recommended that the work of each newly hired analyst is assessed within 3 months of working independently, (e.g., completion of method IDOC). In addition, as analysts add methods to their capabilities, (new IDOC) reviews of the analyst work products will be performed within 3 months of completing the documented training.

17.1.4 Special Audits

Special audits are conducted on an as needed basis, generally as a follow up to specific issues such as client complaints, corrective actions, PT results, data audits, system audits, validation comments, regulatory audits or suspected ethical improprieties. Special audits are focused on a specific issue, and report format, distribution, and timeframes are designed to address the nature of the issue.

17.1.5 Performance Testing

The laboratory participates semi-annually in performance audits conducted through the analysis of PT samples provided by a third party. The laboratory generally participates in the following types of PT studies: Drinking Water, Nonpotable Water, Soil, and Air.

It is Eurofins policy that PT samples be treated as typical samples in the production process. Furthermore, where PT samples present special or unique problems, in the regular production process they may need to be treated differently, as would any special or unique request submitted by any client. The QA Manager must be consulted and in agreement with any decisions made to treat a PT sample differently due to some special circumstance.

Written investigations for unacceptable PT results are required. In some cases it may be necessary for blind QC samples to be submitted to the laboratory to show a return to control.

17.1.6 Good Laboratory Practice Critical Phase Inspection

Any project that is subject to Good Laboratory Practice (GLP) regulations is audited by the QA Department, as required by the regulations, at intervals adequate to ensure the integrity of the study. Inspections of a GLP project include direct observation of analysts as they perform various phases of the study. Data documentation is reviewed as part of the inspection. The purpose of this type of audit is to ensure that there are no deviations from written methods, procedures, or study protocols.

Results of inspections are documented in a report format and distributed to applicable management personnel responsible for the area(s) under audit. Management is responsible to address all non-conformances found during an inspection. Inspection reports and responses are circulated to applicable laboratory management and an off-site study director, as applicable, to communicate the outcome of the inspection and the proposed plan(s) for corrective action, if warranted.

All records maintained as part of an inspection are kept on file.

17.2 External Audits

External audits are performed when certifying agencies or clients conduct on-site inspections or submit performance testing samples for analysis. It is Eurofins policy to cooperate fully with regulatory authorities and clients. The laboratory makes every effort to provide the auditors with access to personnel, documentation, and assistance. Laboratory supervisors are responsible for providing corrective actions to the QA Manager who coordinates the response. Audit responses are due in the time allotted by the client or agency performing the audit.

The laboratory cooperates with clients and their representatives to monitor the laboratory's performance in relation to work performed for the client. The client may only view data and systems related directly to the client's work. All efforts are made to keep other client information confidential.

17.2.1 Confidential Business Information (CBI) Considerations

During on-site audits, auditors may come into possession of information claimed as business confidential. A business confidentiality claim is defined as "a claim or allegation that business information is entitled to confidential treatment for reasons of business confidentiality or a request for a determination that such information is entitled to such treatment." When information is claimed as business confidential, the laboratory must place on (or attach to) the information at the time it is submitted to the auditor, a cover sheet, stamped or typed legend or other suitable form of notice, employing language such as "trade secret", "proprietary" or "company confidential". Confidential portions of documents otherwise non-confidential must be clearly identified. CBI may be purged of references to client identity by the responsible laboratory official at the time of removal from the laboratory. However, sample identifiers may not be obscured from the information. Additional information regarding CBI can be found in within the 2009 and 2016 TNI standards.

17.3 Audit Findings

Audit findings are documented using the corrective action process and database (see Section 14). The laboratory's corrective action responses may include action plans that could not be completed within a predefined timeframe. In these instances, a completion date must be set and agreed to by operations management and the QA Manager.

Developing and implementing corrective actions to findings is the responsibility of operations management or Technical Manager where the finding originated. Findings that are not corrected by specified due dates are reported monthly to management in the QA monthly report.

If any audit finding casts doubt on the effectiveness of the operations or on the correctness or validity of the laboratory's test results, the laboratory shall take timely corrective action, and shall notify clients in writing if the investigations show that the laboratory results have been

affected. Once corrective action is implemented, a follow-up audit is scheduled to ensure that the problem has been corrected.

Clients must be notified promptly in writing of any event such as the identification of defective measuring or test equipment that casts doubt on the validity of results given in any test report or amendment to a test report. The investigation must begin within 24 hours of discovery of the problem and all efforts are made to notify the client within two weeks after the completion of the investigation.

18.0 MANAGEMENT REVIEWS

18.1 Quality Assurance Report

The QA Department is responsible for preparing a comprehensive monthly metrics report to Management to keep them apprised of current quality issues. This report fosters communication, review, and refinement of the QA system to evaluate the suitability of policies and procedures to meet both regulatory and laboratory quality objectives.

The NDSC QA team compiles information from all of the Environment Testing laboratories monthly metrics reports for the Executive Management team. This report includes notable information and concerns regarding the laboratories QA program and a listing of new regulations that may potentially impact the laboratories.

18.2 Annual Management Review

The Laboratory Management team (Laboratory Director, Operation Manager, Technical Managers, and QA Manager) conducts a review annually of its quality systems to ensure its continuing suitability and effectiveness in meeting client and regulatory requirements and to introduce any necessary changes or improvements. It will also provide a platform for defining goals, objectives and action items that feed into the laboratory planning system. NDSC personnel is be included in this meeting at the discretion of the Laboratory Director. The LIMS review consists of examining any audits, complaints or concerns that have been raised through the year that are related to LIMS. The laboratory will summarize any critical findings that cannot be solved by the lab and report them to Corporate IT.

This management systems review (NDSC Document No. CW-Q-S-004 and Work Instruction No. CW-Q-WI-003) uses information generated during the preceding year to assess the “big picture” by ensuring that routine actions taken and reviewed on a monthly basis are not components of larger systematic concerns. The monthly review should keep the quality systems current and effective, therefore, the annual review is a formal senior management process to review specific existing documentation. Significant issues from the following documentation are compiled or summarized by the QA Manager prior to the review meeting:

- Matters arising from the previous annual review.
- Prior Monthly QA Reports issues.
- Laboratory QA Metrics.
- Review of report reissue requests.
- Review of client feedback and complaints.
- Issues arising from any prior management or staff meetings.

- Minutes from prior senior lab management meetings. Issues that may be raised from these meetings include:
  - Adequacy of staff, equipment and facility resources.
  - Adequacy of policies and procedures.
  - Future plans for resources and testing capability and capacity.
- The annual internal double blind PT program sample performance (if performed),
- Compliance to the Ethics Policy and Data Integrity Plan. Including any evidence/incidents of inappropriate actions or vulnerabilities related to data Integrity.
- For labs analyzing radioactive samples, also include the following:
  - Radiation health and safety
  - Radioactive hazardous waste management
  - Radioactive materials management
- Evaluation of overall risk, including risks to impartiality, confidentiality, reporting statements of conformity, and nonconforming work.

A report is generated by the QA Manager and management. The report is distributed to the Laboratory Director and Corporate Quality Director. The report includes, but is not limited to:

- The date of the review and the names and titles of participants.
- A reference to the existing data quality related documents and topics that were reviewed.
- Quality system or operational changes or improvements that will be made as a result of the review [e.g., an implementation schedule including assigned responsibilities for the changes (Action Table)].

Changes to the quality systems requiring update to the laboratory QA Manual shall be included in the next revision of the QA Manual.

18.3 Potential Integrity Related Managerial Reviews

Potential integrity issues (data or business related) must be handled and reviewed in a confidential manner until such time as a follow-up evaluation, full investigation, or other appropriate actions have been completed and issues clarified. The NDSC Document No. CW-L-S-002, Internal Investigations, shall be followed. All investigations that result in finding of inappropriate activity are documented and include any disciplinary actions involved, corrective actions taken, and all appropriate notifications of clients.

The Eurofins Environment Testing America Presidents, Business Unit Managers, Laboratory Directors, and NDSC Team are informed of any current data integrity or data recall investigations via the monthly metrics report.

19.0 TEST METHODS AND METHOD VALIDATION

19.1 Overview

The laboratory uses methods that are appropriate to meet our clients' requirements and that are within the scope of the laboratory's capabilities. These include sampling, handling, transport,

storage and preparation of samples, and, where appropriate, an estimation of the measurement of uncertainty as well as statistical techniques for analysis of environmental data.

Instructions are available in the laboratory for the operation of equipment as well as for the handling and preparation of samples. All instructions, Standard Operating Procedures (SOPs), reference methods and manuals relevant to the working of the laboratory are readily available to all staff. Deviations from published methods are documented (with justification) in the laboratory's approved SOPs. SOPs are submitted to clients for review at their request. Significant deviations from published methods require client approval and regulatory approval where applicable.

19.2 Standard Operating Procedures (SOPs)

The laboratory maintains SOPs that accurately reflect all phases of the laboratory such as assessing data integrity, corrective actions, handling customer complaints as well as all analytical methods and sampling procedures. The method SOPs are derived from the most recently promulgated/approved, published methods and are specifically adapted to the laboratory facility. Modifications or clarifications to published methods are clearly noted in the SOPs. All SOPs are controlled in the laboratory.

- All SOPs contain a revision number, effective date, and appropriate approval signatures. Controlled copies are available to all staff.
- Procedures for writing an SOP are incorporated by reference to NDSC Document No. CW-Q-S-002, Writing a Standard Operating Procedure.
- SOPs are reviewed at a minimum of every 2 years (annually for Drinking Water and DoD/DOE SOPs), and where necessary, revised to ensure continuing suitability and compliance with applicable requirements.

19.3 Laboratory Methods Manual

For each test method, the laboratory shall have available the published referenced method as well as the laboratory developed SOP.

Note: If more stringent standards or requirements are included in a mandated test method or regulation than those specified in this manual, the laboratory shall demonstrate that such requirements are met. If it is not clear which requirements are more stringent, the standard from the method or regulation is to be followed. Any exceptions or deviations from the referenced methods or regulations are noted in the specific analytical SOP.

The laboratory maintains an SOP Index for both technical and non-technical SOPs. Technical SOPs are maintained to describe a specific test method. Non-technical SOPs are maintained to describe functions and processes not related to a specific test method.

19.4 Selection of Methods

Since numerous methods and analytical techniques are available, continued communication between the client and laboratory is imperative to assure the correct methods are utilized. Once client methodology requirements are established, this and other pertinent information is summarized by the PM. These mechanisms ensure that the proper analytical methods are applied when the samples arrive for log-in. For non-routine analytical services (e.g., special matrices, non-routine compound lists), the method of choice is selected based on client needs and available technology. The methods selected should be capable of measuring the specific

parameter of interest, in the concentration range of interest, and with the required precision and accuracy.

19.4.1 Sources of Methods

Routine analytical services are performed using standard EPA-approved methodology. In some cases, modification of standard approved methods may be necessary to provide accurate analyses of particularly complex matrices. When the use of specific methods for sample analysis is mandated through project or regulatory requirements, only those methods shall be used.

When clients do not specify the method to be used or methods are not required, the methods used will be clearly validated and documented in an SOP and available to clients and/or the end user of the data. Refer to Appendix 6 for a list of the currently accepted U.S. EPA analytical method references used by the laboratory.

The laboratory reviews updated versions to all the aforementioned references for adaptation based upon capabilities, instrumentation, etc., and implements them as appropriate. As such, the laboratory strives to perform only the latest versions of each approved method as regulations allow or require.

Other reference procedures for non-routine analyses may include methods established by specific states (e.g., Underground Storage Tank methods), ASTM or equipment manufacturers. Sample type, source, and the governing regulatory agency requiring the analysis will determine the method utilized.

The laboratory shall inform the client when a method proposed by the client may be inappropriate or out of date. After the client has been informed, and they wish to proceed contrary to the laboratory's recommendation, it will be documented.

19.4.1.1 Client Supplied Methods

Most of the client-supplied method requirements presented to us involve achieving specific quality control criteria, limits of quantitation (LOQ), and/or method detection limits (MDL) using standard EPA methods. These requirements are communicated to the appropriate technical groups prior to the project start up. Each technical group evaluates the scope of work and the requirements to ensure the criteria can be met using the standard EPA method. The data is monitored to ensure the criteria are met throughout the project. The PM notifies the client if there is a more appropriate method available or if the client's criteria cannot be achieved on a certain sample matrix (i.e., due to matrix or dilutions).

Occasionally, we are asked to transfer a non-standardized method from a client into our lab or to develop a new method, when one is not available. In the case of a method transfer, we set up the client's method and perform some initial evaluation. After the initial evaluation, we may make recommendations on how to improve method performance. If the method appears to be adequate, we determine linearity, specificity, precision, accuracy, MDL, and LOQ by performing calibrations, analyzing method blanks, and carrying out method detection limit and IDOC studies.

In the case of method development, we work with the client and/or data user to determine the level of validation required ensuring that the method meets its intended purpose. In addition to the elements above, we also determine standard and sample stability and robustness depending on the scope of the project. Typically, a standard operating procedure is written and submitted to the client with the results of the validation. These steps are completed prior to analysis of field samples. Data related to the setup of the method are archived.

19.4.1.2 Procedural Deviations

Analysts are required to follow a documented method for all tests performed; and any deviations from analytical methods must be documented, approved, and justified in an appropriate and consistent manner. We classify method deviations as either being a planned deviation or an unplanned deviation. In general, the following information is captured to document both types of situations:

- Description of the situation
- Reason or justification for the deviation
- Impact the deviation had on the testing
- Signature/date of analyst performing the test
- Signature/date of QA and Laboratory management approving the deviation
- Signature/date of client approval, if necessary

Deviations to written procedures are documented in raw data records, ICAT or through the ICAR (Investigation and Corrective Action Report) system. Both types of documentation require management and QA review and approval.

NOTE: Deviations to analytical methods are not permitted by PALA. If samples are analyzed for compliance to a regulatory program, deviations may be allowed with approval from the appropriate compliance officer and/or program.

19.4.2 Demonstration of Capability

Before the laboratory may institute a new method and begin reporting results, the laboratory shall confirm that it can properly operate the method. In general, this demonstration does not test the performance of the method in real world samples, but in an applicable and available clean matrix sample. If the method is for the testing of analytes that are not conducive to spiking, demonstration of capability may be performed on quality control samples.

A demonstration of capability (DOC, Lab SOP BR-QA-011) is performed whenever there is a change in instrument type (e.g., new instrumentation), matrix, method or personnel (e.g., analyst has not performed the test within the last 12 months).

Note: The laboratory shall have a DOC for all analytes included in the methods that the laboratory performs, and proficiency DOCs for each analyst shall include all analytes that the laboratory routinely performs. Addition of non-routine analytes does not require new DOCs for all analysts if those analysts are already qualified for routine analytes tested using identical chemistry and instrument conditions.

The initial demonstration of capability must be thoroughly documented and approved by the Technical Manager and QA Manager prior to independently analyzing client samples. All associated documentation must be retained in accordance with the laboratory's archiving procedures.

The laboratory must have an approved SOP, demonstrate satisfactory performance, and conduct an MDL study (when applicable). There may be other requirements as stated within the published method or regulations (e.g., retention time window study).

Note: In some instances, a situation may arise where a client requests that an unusual analyte be reported using a method where this analyte is not normally reported. If the analyte is being reported for regulatory purposes, the method must meet all procedures outlined within this QA

Manual (SOP, MDL, and Demonstration of Capability). If the client states that the information is not for regulatory purposes, the result may be reported as long as the following criteria are met:

- The instrument is calibrated for the analyte to be reported using the criteria for the method and ICV/CCV criteria are met (unless an ICV/CCV is not required by the method or criteria are per project DQOs).
- The laboratory's nominal or default reporting limit (RL) is equal to the quantitation limit (QL), must be at or above the lowest non-zero standard in the calibration curve and must be reliably determined. Project RLs are client specified reporting levels which may be higher than the QL. Results reported below the QL must be qualified as estimated values. Also see Section 19.6.1.3, Relationship of Limit of Detection (LOD) to Quantitation Limit (QL).
- The client request is documented and the lab informs the client of its procedure for working with unusual compounds. The final report must be footnoted: *Reporting Limit based on the low standard of the calibration curve.*

19.4.3 Initial Demonstration of Capability (IDOC) Procedures

The spiking standard used must be prepared independently from those used in instrument calibration.

The analyte(s) shall be diluted in a volume of clean matrix sufficient to prepare four aliquots at the concentration specified by a method or the laboratory SOP.

19.4.3.1 At least four aliquots shall be prepared (including any applicable clean-up procedures) and analyzed according to the test method (either concurrently or over a period of days).

19.4.3.2 Using all of the results, calculate the mean recovery in the appropriate reporting units and the standard deviations for each parameter of interest.

19.4.3.3 When it is not possible to determine the mean and standard deviations, such as for presence, absence and logarithmic values, the laboratory will assess performance against criteria described in the Method SOP.

19.4.3.4 Compare the information obtained above to the corresponding acceptance criteria for precision and accuracy in the test method (if applicable) or in laboratory generated acceptance criteria (LCS or interim criteria) if there is no mandatory criteria established. If any one of the parameters do not meet the acceptance criteria, the performance is unacceptable for that parameter.

19.4.3.5 When one or more of the tested parameters fail at least one of the acceptance criteria, the analyst must proceed according to either option listed below:

- Locate and correct the source of the problem and repeat the test for all parameters of interest beginning with 19.4.3.3 above.
- Beginning with 19.4.3.3 above, repeat the test for all parameters that failed to meet criteria. Repeated failure, however, will confirm a general problem with the measurement system. If this occurs, locate and correct the source of the problem and repeat the test for all compounds of interest beginning with 19.4.3.1 above.

Note: Results of successive LCS analyses can be used to fulfill the DOC requirement.

Methods on line prior to the effective date of this Section shall be updated to the procedures outlined above as new analysts perform their demonstration of capability. A copy of the new record will replace that which was used for documentation in the past. At a minimum, the

precision and accuracy of four mid-level laboratory control samples must have been compared to the laboratory's quality control acceptance limits.

19.5 Laboratory Developed Methods and Non-Standard Methods

Any new method developed by the laboratory must be fully defined in an SOP and validated by qualified personnel with adequate resources to perform the method. Method specifications and the relation to client requirements must be clearly conveyed to the client if the method is a non-standard method (not a published or routinely accepted method). The client must also be in agreement to the use of the non-standard method.

19.6 Validation of Methods

Validation is the confirmation by examination and the provision of objective evidence that the particular requirements for a specific intended use are fulfilled.

All non-standard methods, laboratory designed/developed methods, standard methods used outside of their scope, and major modifications to published methods must be validated to confirm they are fit for their intended use. The validation will be as extensive as necessary to meet the needs of the given application. The results are documented with the validation procedure used and contain a statement as to the fitness for use.

19.6.1 Method Validation and Verification Activities for All New Methods

While method validation can take various courses, the following activities can be required as part of method validation. Method validation records are designated QC records and are archived accordingly.

When changes are made to any validated methods, the influence of such changes shall be documented and, if appropriate, a new validation shall be performed.

19.6.1.1 **Determination of Method Selectivity** – Method selectivity is the demonstrated ability to discriminate the analyte(s) of interest from other compounds in the specific matrix or matrices from other analytes or interference. In some cases to achieve the required selectivity for an analyte, a confirmation analysis is required as part of the method.

19.6.1.2 **Determination of Method Sensitivity** – Sensitivity can be both estimated and demonstrated. Whether a study is required to estimate sensitivity depends on the level of method development required when applying a particular measurement system to a specific set of samples. Detection limit studies are conducted as described in Section 19.7. Where other protocols for estimations and/or demonstrations of sensitivity are required by regulation or client agreement, these shall be followed.

19.6.1.3 **Relationship of Limit of Detection (LOD) to the Limit of Quantitation (LOQ)** – An important characteristic of expression of sensitivity is the distinction between the LOD and the LOQ. The LOD is the minimum level at which the presence of an analyte can be reliably concluded. The LOQ is the minimum concentration of analyte that can be quantitatively determined with acceptable precision and bias, equivalent to the laboratory's routine reporting limit (RL). For most instrumental measurement systems, there is a region where semi-quantitative data is generated around the LOD (both above and below the estimated MDL or LOD) and below the LOQ. In this region, detection of an analyte may be confirmed but quantification of the analyte is unreliable within the accuracy and precision guidelines of the measurement system. When an analyte is detected below the LOQ, and the presence of the analyte is confirmed by meeting the qualitative identification criteria for the analyte, the analyte

can be reliably reported, but the amount of the analyte can only be estimated. If data is to be reported in this region, it must be done so with a qualification that denotes the semi-quantitative nature of the result.

19.6.1.4 **Determination of Interferences** – A determination that the method is free from interferences in a blank matrix is performed.

19.6.1.5 **Determination of Range** – Where appropriate to the method, the quantitation range is determined by comparison of the response of an analyte in a curve to established or targeted criteria. Generally the upper quantitation limit is defined by highest acceptable calibration concentration. The lower quantitation limit or QL cannot be lower than the lowest non-zero calibration level, and can be constrained by required levels of bias and precision.

19.6.1.6 **Determination of Accuracy and Precision** – Accuracy and precision studies are generally performed using replicate analyses, with a resulting percent recovery and measure of reproducibility (standard deviation, relative standard deviation) calculated and measured against a set of target criteria.

19.6.1.7 **Documentation of Method** – The method is formally documented in an SOP. If the method is a minor modification of a standard laboratory method that is already documented in an SOP, an SOP Attachment describing the specific differences in the new method is acceptable in place of a separate SOP.

19.6.1.8 **Continued Demonstration of Method Performance** – Continued demonstration of method performance is addressed in the SOP. Continued demonstration of method performance is generally accomplished by batch specific QC samples such as LCS, method blanks or PT samples.

19.7 Method Detection Limit (MDL) / Limits of Detection (LOD)

The MDL is the minimum measured quantity of a substance that can be reported with 99% confidence that the concentration is distinguishable from method blank results, consistent with 40CFR Part 136 Appendix B, August, 2017. The MDL is equivalent to the TNI LOD or DL, and is also equivalent to the DoD/DOE Quality Systems Manual (QSM) DL. The working or final MDL is the higher of the MDL value determined from spikes (MDLs) and the MDL value determined from blanks (MDLb). An initial MDL study shall be performed during the method validation process and when the method is altered in a way that can reasonably be expected to change its sensitivity. On-going data are collected during each quarter in which samples are being analyzed. At least once every 13 months the MDLs and MDLb are re-calculated and re-evaluated using data collected during the preceding period. Details of the laboratory's procedure for conducting MDL studies are given in NDSC Document No. CA-Q-S-006.

19.8 Verification of Detection Limits

If it is found during the re-evaluation of detection limit results that more than 5% of the spiked samples do not return positive numeric results that meet all method qualitative identification criteria, then then spiking level shall be increased and the initial MDL study pre-performed at the new spiking concentration. For DoD/DOE labs, additional guidance provided in NDSC Document No. No. CA-Q-S-006 - Attachment 2.

19.9 Instrument Detection Limits (IDL)

The IDL is sometimes used to assess the reasonableness of the MDL or in some cases required by the analytical method or program requirements. IDLs are most commonly used in metals analyses but may be useful in demonstration of instrument performance in other areas.

IDLs are calculated to determine an instrument's sensitivity independent of any preparation method. IDLs are calculated either using 7 replicate spike analyses, like MDL but without sample preparation, or by the analysis of 10 instrument blanks and calculating 3 x the absolute value of the standard deviation.

19.10 Limit of Quantitation

The LOQ shall be at a concentration equivalent to the lowest calibration standard concentration, with the exception of methods using a single-point calibration, and shall be greater than the MDL. The LOQ is verified by preparing and analyzing spikes at concentrations 1-2X the selected LOQ, employing the complete analytical process. For DoD/DOE labs, additional requirements provided in NDSC Document No. CA-Q-S-006.

When the laboratory establishes a quantitation limit, it must be initially verified by the analysis of a low level standard or QC sample at 1-2 times the reporting limit or by a DL check samples at or below the LOQ. The LOQ is verified annually thereafter. The annual requirement is waived for methods that have an annually verified MDL. The laboratory will comply with any regulatory requirements.

19.11 Retention Time Windows

Most organic analyses and some inorganic analyses use chromatography techniques for qualitative and quantitative determinations. For every chromatography analysis or as specified in the reference method, each analyte will have a specific time of elution from the column to the detector. This is known as the analyte's retention time. The variance in the expected time of elution is defined as the retention time window. As the key to analyte identification in chromatography, retention time windows must be established on every column for every analyte used for that method. These records are kept on-file and available for review. Complete details are available in the laboratory SOPs.

19.12 Evaluation of Selectivity

The laboratory evaluates selectivity by following the checks within the applicable analytical methods, which include mass spectral tuning, second column confirmation, ICP interelement interference checks, chromatography retention time windows, sample blanks, and spectrochemical.

19.13 Estimation of Uncertainty of Measurement

19.13.1 Uncertainty is "a parameter associated with the result of a measurement, that characterizes the dispersion of the values that could reasonably be attributed to the measurand" (as defined by the International Vocabulary of Basic and General Terms in Metrology, ISO Geneva, 1993, ISBN 92-67-10175-1). Knowledge of the uncertainty of a measurement provides additional confidence in a result's validity. Its value accounts for all the factors which could possibly affect the result, such as adequacy of analyte definition, sampling, matrix effects and interferences, climatic conditions, variances in weights, volumes, and standards, analytical procedure, and random variation. Some national accreditation organizations require the use of

an “expanded uncertainty” defined as the range within which the value of the measurand is believed to lie within at least a 95% confidence level with the coverage factor k=2.

19.13.2 Uncertainty is not error. Error is a single value (i.e., the difference between the true result and the measured result). On environmental samples, the true result is never known. The measurement is the sum of the unknown true value and the unknown error. Unknown error is a combination of systematic error, or bias, and random error. Bias varies predictably, constantly, and independently from the number of measurements. Random error is unpredictable, assumed to be Gaussian in distribution, and reducible by increasing the number of measurements.

19.13.3 The minimum uncertainty associated with results generated by the laboratory can be determined by using the Laboratory Control Sample (LCS) accuracy range for a given analyte. The LCS limits are used to assess the performance of the measurement system since they take into consideration all of the laboratory variables associated with a given test over time (except for variability associated with the sampling and the variability due to matrix effects). The percent recovery of the LCS is compared either to the method-required LCS accuracy limits or to the statistical, historical, in-house LCS accuracy limits.

19.13.4 To calculate the uncertainty for the specific result reported, multiply the result by the decimal of the lower end of the LCS range percent value for the lower end of the uncertainty range, and multiply the result by the decimal of the upper end of the LCS range percent value for the upper end of the uncertainty range. These calculated values represent uncertainties at approximately the 99% confidence level with a coverage factor of k = 3. As an example, for a reported result of 1.0 mg/L with an LCS recovery range of 50 to 150%, the estimated uncertainty in the result would be 1.0 +/- 0.5 mg/L.

19.13.5 In the case where a well-recognized test method specifies limits to the values of major sources of uncertainty of measurement (e.g., 524.2, 525, etc.) and specifies the form of presentation of calculated results, no further discussion of uncertainty is required.

19.14 Sample Reanalysis Guidelines

Because there is a certain level of uncertainty with any analytical measurement, a sample re-preparation (where appropriate) and subsequent analysis (hereafter referred to as ‘reanalysis’) may result in either a higher or lower value from an initial sample analysis. There are also variables that may be present (e.g., sample homogeneity, analyte precipitation over time, etc.) that may affect the results of a reanalysis. Based on the above comments, the laboratory will reanalyze samples at a client’s request with the following caveats. **Client specific Contractual Terms & Conditions for reanalysis protocols may supersede the following items.**

Homogenous samples: If a reanalysis agrees with the original result to within the RPD limits for MS/MSD or Duplicate analyses, or within $\pm$ 1 reporting limit for samples $\leq$ 5x the reporting limit, the original analysis will be reported. At the client’s request, both results may be reported on the same report but not on two separate reports.

- If the reanalysis does not agree (as defined above) with the original result, then the laboratory will investigate the discrepancy and reanalyze the sample a third time for confirmation if sufficient sample is available.
- Any potential charges related to reanalysis are discussed in the contract terms and conditions or discussed at the time of the request. The client will typically be charged for reanalysis unless it is determined that the lab was in error.

- Due to the potential for increased variability, reanalysis may not be applicable to Non-homogenous, Encore, and Sodium Bisulfate preserved samples. See the Area Supervisor or Laboratory Director if unsure.

19.15 Control of Data

The laboratory has policies and procedures in place to ensure the authenticity, integrity, and accuracy of the analytical data generated by the laboratory.

19.15.1 Computer and Electronic Data Related Requirements

The three basic objectives of our computer security procedures and policies are shown below.

The laboratory is currently using the Eurofins LIMS system, which has been highly customized to meet the needs of the laboratory.

19.15.1.1 Maintain the Database Integrity – Assurance that data is reliable and accurate through data verification (review) procedures, password-protecting access, anti-virus protection, data change requirements, as well as an internal LIMS permissions procedure.

- LIMS Database Integrity is achieved through data input validation, internal user controls, documentation of system failures and corrective actions taken, and data change requirements.
- Spreadsheets and other software developed in-house must be verified with documentation through hand calculations prior to use. Cells containing calculations must be lock-protected and controlled.
- Instrument hardware and software adjustments are safeguarded through maintenance logs, audit trails and controlled access.

19.15.1.2 Ensure Information Availability – Protection against loss of information or service is ensured through scheduled back-ups, stable file server network architecture, secure storage of media, line filter, Uninterruptible Power Supply (UPS), and maintaining older versions of software as revisions are implemented.

19.15.1.3 Maintain Confidentiality – Ensure data confidentiality through physical access controls such as password protection or website access approval when electronically transmitting data.

19.15.2 Data Reduction

The complexity of the data reduction depends on the analytical method and the number of discrete operations involved (e.g., extractions, dilutions, instrument readings and concentrations). The analyst calculates the final results from the raw data or uses appropriate computer programs to assist in the calculation of final reportable values.

For manual data entry, e.g., Wet Chemistry and Geotechnical, the data is reduced by the analyst and then verified by the Department Manager or alternate analyst prior to updating the data in LIMS. The spreadsheets, or any other type of applicable documents, are signed by both the analyst and alternate reviewer to confirm the accuracy of the manual entry(s).

Manual integration of peaks will be documented and reviewed and the raw data will be flagged using NDSC Document No. CA-Q-S-002, Acceptable Manual Integration Practices as the guideline for our internal laboratory SOPs.

Analytical results are reduced to appropriate concentration units specified by the analytical method, taking into account factors such as dilution, sample weight or volume, etc. Blank correction will be applied only when required by the method or per manufacturer's indication; otherwise, it should not be performed. Calculations are independently verified by appropriate laboratory staff. Calculations and data reduction steps for various methods are summarized in the respective analytical SOPs or program requirements.

19.15.2.1 All raw data must be retained in the worklist folder, computer file (if appropriate), and/or runlog. All criteria pertinent to the method must be recorded. The documentation is recorded at the time observations or calculations are made and must be signed or initialed/dated (month/day/year). It must be easily identifiable who performed which tasks if multiple people were involved.

19.15.2.2 In general, concentration results are reported in milligrams per liter (mg/l) or micrograms per liter (µg/l) for liquids and milligrams per kilogram (mg/kg) or micrograms per kilogram (µg/kg) for solids. For values greater than 10,000 mg/l, results can be reported in percent, i.e., 10,000 mg/l = 1**%.** Units are defined in each lab SOP.

19.15.2.3 In reporting, the analyst or the instrument output records the raw data result using values of known certainty plus one uncertain digit. If final calculations are performed external to LIMS, the results should be entered in LIMS with at least three significant figures. In general, results are reported to 2 significant figures on the final report.

19.15.2.4 For those methods that do not have an instrument printout or an instrumental output compatible with the LIMS, the raw results and dilution factors are entered directly into LIMS by the analyst, and the software calculates the final result for the analytical report. LIMS has a formatter for significant figure criterion for each analyte.

19.15.2.5 The laboratory strives to import data directly from instruments or calculation spreadsheets to ensure that the reported data are free from transcription and calculation errors. For those analyses with an instrumental output compatible with the LIMS, the raw results and dilution factors are transferred into LIMS, electronically after reviewing the quantitation report, and removing unrequested or poor spectrally-matched compounds. The analyst prints a copy of what has been entered to check for errors. This printout and the instrument's printout of calibrations, concentrations, retention times, chromatograms, and mass spectra, if applicable, are retained with the data file. The data file is stored in a monthly folder on the instrument computer; periodically, this file is transferred to the server and, eventually, to a tape file.

19.15.3 Logbook / Worksheet Use Guidelines

Logbooks and worksheets are filled out 'real time' and have enough information on them to trace the events of the applicable analysis/task. (e.g. calibrations, standards, analyst, sample ID, date, time on short holding time tests, temperatures when applicable, calculations are traceable, etc.)

- Corrections are made following the procedures outlined in Section 14.
- Logbooks are controlled by the QA department. A record is maintained of all logbooks in the lab.
- Unused portions of pages must be "Z"'d out, signed and dated.
- Worksheets are created with the approval of the Technical Manager/QA Manager at the facility. The QA Manager controls all worksheets following the procedures in Section 6.

19.15.4 Review / Verification Procedures

Review procedures are outlined in several SOPs (*Sample Control, Data Review, Project Management, etc.)* to ensure that reported data are free from calculation and transcription errors, that QC parameters have been reviewed and evaluated before data is reported. The laboratory also has an SOP discussing Manual Integrations to ensure the authenticity of the data, NDSC Document No. CA-Q-S-002. The general review concepts are discussed below, more specific information can be found in the SOPs.

19.15.4.1 Log-In Review - The data review process starts at the sample receipt stage. Sample control personnel review chain-of-custody forms and project instructions from the project management group. This is the basis of the sample information and analytical instructions entered into the LIMS. The log-in instructions are reviewed by the personnel entering the information, and a second level review is conducted by the project management staff.

19.15.4.2 First Level Data Review - The next level of data review occurs with the analysts. As data are generated, analysts review their work to ensure that the results meet project and SOP requirements. First level reviews include inspection of all raw data (e.g., instrument output for continuous analyzers, chromatograms, spectra, and manual integrations), evaluation of calibration/calibration verification data in the day's analytical run, evaluation of QC data, and reliability of sample results. The analyst transfers data into LIMS, data qualifiers are added as needed. All first level reviews are documented.

19.15.4.3 Second Level Data Review – All analytical data are subject to review by a second qualified analyst or supervisor. Second level reviews include inspection of all raw data (e.g., instrument output, chromatograms, and spectra) including 100% of data associated with any changes made by the primary analyst, such as manual integrations or reassignment of peaks to different analytes, or elimination of false negative analytes. The second review also includes evaluation of initial calibration/calibration verification data in the day's analytical run, evaluation of QC data, reliability of sample results, qualifiers and NCM narratives. Manual calculations are checked in second level review. All second level reviews are documented.

Issues that deem further review include the following:

- QC data are outside the specified control limits for accuracy and precision
- Reviewed sample data does not match with reported results
- Unusual detection limit changes are observed
- Samples having unusually high results
- Samples exceeding a known regulatory limit
- Raw data indicating some type of contamination or poor technique
- Inconsistent peak integration
- Transcription errors
- Results outside of calibration range

19.15.4.4 Unacceptable analytical results may require reanalysis of the samples. Any problems are brought to the attention of the Laboratory Director, Project Manager, Quality Director/Manager, Technical Manager, or Supervisor for further investigation. Corrective action is initiated whenever necessary.

19.15.4.5 The results are then entered or directly transferred into the computer database and a (.pdf) is printed for the client.

19.15.4.6 As a final review prior to the release of the report, the Project Manager reviews the results for appropriateness and completeness. This review and approval ensures that client requirements have been met and that the final report has been properly completed. The process includes, but is not limited to, verifying that the COC is followed, cover letters / narratives are present, flags are appropriate, and project specific requirements are met. The Project Manager may also evaluate the validity of results for different test methods given expected chemical relationships.

19.15.4.7 Any project that requires a data package is subject to a tertiary data review for transcription errors and acceptable quality control requirements. The Project Manager then signs the final report. The PM also checks the report for any clerical or invoicing errors. When complete, the report is sent out to the client.

19.15.4.8 A brief overview of sample flow and information through the laboratory, as well as data review and validation, is presented in Figure 19-1.

19.15.5 Manual Integrations

Computerized data systems provide the analyst with the ability to re-integrate raw instrument data in order to optimize the interpretation of the data. Though manual integration of data is an invaluable tool for resolving variations in instrument performance and some sample matrix problems, when used improperly, this technique would make unacceptable data appear to meet quality control acceptance limits. Improper re-integrations lead to legally indefensible data, a poor reputation, or possible laboratory decertification. Because guidelines for re-integration of data are not provided in the methods and most methods were written prior to widespread implementation of computerized data systems, the laboratory trains all analytical staff on proper manual integration techniques using NDSC Document No. CA-Q-S-002, Acceptable Manual Integration Practices, as the guideline for our internal laboratory SOPs.

19.15.5.1 The analyst must adjust baseline or the area of a peak in some situations, for example when two compounds are not adequately resolved or when a peak shoulder needs to be separated from the peak of interest. The analyst must use professional judgment to determine when manual integrating is required. Analysts are encouraged to ask for assistance from a senior analyst or manager when in doubt.

19.15.5.2 Analysts shall not increase or decrease peak areas for the sole purpose of achieving acceptable QC recoveries that would have otherwise been unacceptable. The intentional recording or reporting of incorrect information (or the intentional omission of correct information) is against company principles and policy and is grounds for immediate termination.

19.15.5.3 Client samples, performance evaluation samples, and quality control samples are all treated equally when determining whether or not a peak area or baseline should be manually adjusted.

19.15.5.4 All manual integrations receive a second level review. Manual integrations must be indicated on ~~an~~ expanded scale “before” and “after” chromatograms such that the integration performed can be easily evaluated during data review. Expanded scale “before” chromatograms are also required for all manual integrations on QC parameters (calibrations, calibration verifications, laboratory control samples, internal standards, surrogates, etc.) unless the laboratory has another documented NDSC approved procedure in place that can demonstrate an active process for detection and deterrence of improper integration practices.

Figure 19-1. Example – Work Flow

Action	Personnel Involved
Bottle orders generated upon request - Bottles packed and shipped to the client under CoC	- Client Service Personnel / PM - Bottle Preparation Personnel
Samples received at laboratory - Unpacked and reconciled against client paperwork or CoC - Samples acceptance documented against completed (CoC)[1]	Sample Login Personnel
Samples are entered into LIMS - Job # assigned - Methods/Analytes linked from project - Sample labels generated - LIMS backlogs updated immediately	Sample Login Personnel
Sample Storage - Short TATs and hold-time samples are delivered directly to the laboratory for analysis - Samples stored in assigned locations (refrigerated walk-in coolers, freezers, VOA refrigerators; etc.)	Sample Login Personnel
Sample Receipt Acknowledgement sent to client (when requested)	Project Management Personnel
Sample Acquisition/Testing - Analysts collect samples from assigned storage. - Legal CoC samples are signed in-and-out of storage - Remaining sample returned to storage.	Analysts / Login Personnel
Preparation and analysis is performed according to selected analytical method and applicable Project notes - Raw data recorded/reviewed[2] - Data imported to LIMS	Analysts
LIMS performs calculations: - Data reviewed by primary/secondary analyst - Data approved as final	Data Process (LIMS / Analyst)
Generation/review/release of reports (automated through LIMS) - Electronic copy saved in LIMS	Project Management Personnel
Electronic Data Deliverables (EDDs) generated (as applicable)	Project Management Personnel
Hard copy and electronic batch raw data is archived according to data archiving procedures.	Project Management Personnel Analysts / IT

[1]Refer to Sec. 23.3.
[2]Analyses requiring the analyst's interpretation may involve manual data reduction before entry into LIMS.

20.0 EQUIPMENT and CALIBRATIONS

20.1 Overview

The laboratory purchases the most technically advanced analytical instrumentation for sample analyses. Instrumentation is purchased on the basis of accuracy, dependability, efficiency and sensitivity. Each laboratory is furnished with all items of sampling, preparation, analytical testing and measurement equipment necessary to correctly perform the tests for which the laboratory has capabilities. Each piece of equipment is capable of achieving the required accuracy and

complies with specifications relevant to the method being performed. Before being placed into use, the equipment (including sampling equipment) is calibrated and checked to establish that it meets its intended specification. The calibration routines for analytical instruments establish the range of quantitation. Calibration procedures are specified in laboratory SOPs. A list of available laboratory instrumentation is presented in Table 20-1.

Equipment is only operated by authorized and trained personnel. Manufacturer's instructions for equipment use are readily accessible to all appropriate laboratory personnel.

20.2 Preventive Maintenance

The laboratory follows a well-defined maintenance program to ensure proper equipment operation and to prevent the failure of laboratory equipment or instrumentation during use. This program of preventive maintenance helps to avoid delays due to instrument failure.

Routine preventive maintenance procedures and frequency, such as cleaning and replacements, should be performed according to the procedures outlined in the manufacturer's manual. Qualified personnel must also perform maintenance when there is evidence of degradation of peak resolution, a shift in the calibration curve, loss of sensitivity, or failure to continually meet one of the quality control criteria.

Scheduled routine maintenance is defined in each method SOP. It is the responsibility of each Department Manager/Supervisor to ensure that instrument maintenance logs are kept for all equipment in his/her department. Preventative maintenance procedures are also outlined in analytical SOPs or instrument manuals. (Note: for some equipment, the log used to monitor performance is also the maintenance log. Multiple pieces of equipment may share the same log as long as it is clear as to which instrument is associated with an entry.)

Instrument maintenance logs are controlled and are used to document instrument problems, instrument repair and maintenance activities. Maintenance logs shall be kept for all major pieces of equipment. Instrument maintenance logs may also be used to specify instrument parameters.

- Documentation must include all major maintenance activities such as contracted preventive maintenance and service and in-house activities such as the replacement of electrical components, lamps, tubing, valves, columns, detectors, cleaning and adjustments.
- Each entry in the instrument log includes the analyst's initials, the date, a detailed description of the problem (or maintenance needed/scheduled), a detailed explanation of the solution or maintenance performed, and a verification that the equipment is functioning properly (state what was used to determine a return to control. e.g. CCV run on *'date'* was acceptable, or instrument recalibrated on 'date' with acceptable verification, etc.) must also be documented in the instrument records.
- When maintenance or repair is performed by an outside agency, service receipts detailing the service performed can be affixed into the logbooks adjacent to pages describing the maintenance performed. This stapled in page must be signed across the page entered and the logbook so that it is clear that a page is missing if only half a signature is found in the logbook.

If an instrument requires repair (subjected to overloading or mishandling), gives suspect results, or otherwise has shown to be defective or outside of specified limits it shall be taken out of operation and tagged as out-of-service or otherwise isolated until such a time as the repairs have been made and the instrument can be demonstrated as operational by calibration and/or verification or other test to demonstrate acceptable performance. The laboratory shall examine the effect of this defect on previous analyses.

In the event of equipment malfunction that cannot be resolved, service shall be obtained from the instrument vendor manufacturer, or qualified service technician, if such a service can be tendered. If on-site service is unavailable, arrangements shall be made to have the instrument shipped back to the manufacturer for repair. Back up instruments, which have been approved, for the analysis shall perform the analysis normally carried out by the malfunctioning instrument. If the back-up is not available and the analysis cannot be carried out within the needed timeframe, the samples shall be subcontracted.

At a minimum, if an instrument is sent out for service or transferred to another facility, it must be recalibrated and the laboratory MDL verified (using an MDLV) prior to return to lab operations.

20.3 Support Equipment

This section applies to all devices that may not be the actual test instrument, but are necessary to support laboratory operations. These include but are not limited to: balances, ovens, refrigerators, freezers, water baths, field sampling devices, temperature measuring devices, thermal/pressure sample preparation devices and volumetric dispensing devices if quantitative results are dependent on their accuracy, as in standard preparation and dispensing or dilution into a specified volume. All raw data records associated with the support equipment are retained to document instrument performance.

20.3.1 Weights and Balances

The accuracy of the balances used in the laboratory is checked every working day, before use. All balances are placed on stable counter tops.

Each balance is checked prior to initial serviceable use with at least two certified ASTM type 1 weights spanning its range of use (weights that have been calibrated to ASTM type 1 weights may also be used for daily verification). ASTM type 1 weights used only for calibration of other weights (and no other purpose) are inspected for corrosion, damage or nicks at least annually and if no damage is observed, they are calibrated at least every 5 years by an outside calibration laboratory. Any weights (including ASTM Type 1) used for daily balance checks or other purposes are recalibrated/recertified annually to NIST standards (this may be done internally if laboratory maintains “calibration only” ASTM type 1 weights).

All balances are serviced annually by a qualified service representative, who supplies the laboratory with a certificate that identifies traceability of the calibration to the NIST standards.

All of this information is recorded in logs, and the recalibration/recertification certificates are kept on file.

20.3.2 pH, Conductivity, and Turbidity Meters

The pH meters used in the laboratory are accurate to $\pm$ 0.1 pH units, and have a scale readability of at least 0.05 pH units. The meters automatically compensate for the temperature, and are calibrated with at least two working range buffer solutions before each use.

Conductivity meters used in the laboratory are capable or measuring conductivity with an error not exceeding 1% or one umhos/cm, whichever is greater. The meters are also calibrated before each use with a known standard.

Turbidity meters are also calibrated before each use. All of this information is documented in logs.

Consult pH, Conductivity, and Turbidity SOPs for further information.

20.3.3 Thermometers

All thermometers are calibrated on an annual basis with a NIST-traceable thermometer.

- If the temperature measuring device is used over a range of 10°C or less, then a single point verification within the range of use is acceptable;
- If the temperature measuring device is used over a range of greater than 10°C, then the verification must bracket the range of use.

IR thermometers, digital probes and thermocouples are calibrated quarterly. IR Thermometers are calibrated over the full range of use, including ambient, iced (4°C) and frozen (0°C to -5°C), per the Drinking Water Manual.

The digital NIST thermometer is recalibrated every year (unless thermometer has been exposed to temperature extremes or apparent separation of internal liquid) by an approved outside service and the provided certificate of traceability is kept on file. The NIST thermometer(s) have increments of 1 degree, and have ranges applicable to method and certification requirements. The NIST traceable thermometer is used for no other purpose than to calibrate other thermometers.

All of this information is documented in logbooks. Monitoring method-specific temperatures, including heating blocks, water baths, and ovens, is documented in method-specific logbooks. More information on this subject can be found in the method-specific SOPs.

20.3.4 Refrigerators/Freezer Units, Water baths, Ovens and Incubators

The temperatures of all refrigerator units and freezers used for sample and standard storage are monitored each working day. (Sample storage is monitored 7 days a week for DoD/DOE labs).

Ovens, water baths and incubators are monitored on days of use.

All of this equipment has a unique identification number, and is assigned a thermometer for monitoring.

Sample storage refrigerator temperatures are kept between > 0ºC and $\leq$ 6 ºC.

Specific temperature settings/ranges for other refrigerators, ovens, and water baths can be found in method specific SOPs.

All of this information is documented in Daily Temperature Logbooks and method-specific logbooks and discussed in SOP No. BR-QA-012, Support Equipment.

20.3.5 Autopipettors, Dilutors, and Syringes

Mechanical volumetric dispensing devices including burettes (except Class A Glassware and glass microliter syringes) are given unique identification numbers and the delivery volumes are verified gravimetrically, at a minimum, on a quarterly basis.

For those dispensers that are not used for measurements, a label shall be applied to the device stating that it is not calibrated. Any device not regularly verified cannot be used for any quantitative measurements.

Micro-syringes are purchased from Hamilton Company. Each syringe is traceable to NIST. The laboratory keeps on file an “Accuracy and Precision Statement of Conformance” from Hamilton attesting established accuracy.

20.4 Instrument Calibrations

Calibration of analytical instrumentation is essential to the production of quality data. Strict calibration procedures are followed for each method. These procedures are designed to determine and document the method detection limits, the working range of the analytical instrumentation and any fluctuations that may occur from day to day.

Sufficient raw data records are retained to allow an outside party to reconstruct all facets of the initial calibration. Records contain, but are not limited to, the following: calibration date, method, instrument, analyst(s) initials or signatures, analysis date, analytes, concentration, response, and type of calibration (Avg RF, curve, or other calculations that may be used to reduce instrument responses to concentration.)

Sample results must be quantitated from the initial calibration and may not be quantitated from any continuing instrument calibration verification unless otherwise required by regulation, method or program.

If the initial calibration results are outside of the acceptance criteria, corrective action is performed and any affected samples are reanalyzed if possible. If the reanalysis is not possible, any data associated with an unacceptable initial calibration will be reported with appropriate data qualifiers (refer to Section 14).

Note: Instruments are calibrated initially and as needed after that and at least annually ***(the annual requirement does not apply to Isotope dilution).***

20.4.1 Calibration Standards

Calibration standards are prepared using the procedures indicated in the Reagents and Standards section of the determinative method SOP. If a reference method does not specify the number of calibration standards, a minimum of 3 calibration points will be used.

Standards for instrument calibration are obtained from a variety of sources. All standards are traceable to national or international standards of measurement, or to national or international standard reference materials.

The lowest concentration calibration standard that is analyzed during an initial calibration must be at or below the stated reporting limit for the method based on the final volume of extract (or sample).

The other concentrations define the working range of the instrument/method or correspond to the expected range of concentrations found in actual samples that are also within the working range of the instrument/method. Results of samples not bracketed by initial instrument calibration standards (within calibration range to at least the same number of significant figures used to report the data) must be reported as having less certainty, e.g., defined qualifiers or flags (additional information may be included in the case narrative). The exceptions to these rules. ICP and ICPMS methods which define the working range with periodic linear dynamic range studies, rather than through the range of concentrations of daily calibration standards.

All initial calibrations are verified with a standard obtained from a second source and traceable to a national standard, when available (or vendor certified different lot if a second source is not available). For unique situations, such as air analysis where no other source or lot is available, a standard made by a different analyst at a different time or a different preparation would be considered a second source. This verification occurs immediately after the calibration curve has been analyzed, and before the analysis of any samples.

20.4.1.1 Calibration Verification

The calibration relationship established during the initial calibration must be verified initially and at least daily as specified in the laboratory method SOPs in accordance with the referenced analytical methods and in the 2009 and 2016 TNI Standard. The process of calibration verification applies to both external standard and internal standard calibration techniques, as well as to linear and non-linear calibration models. Initial calibration verification (ICV) is with a standard source secondary (second source standard) to the calibration standards, but continuing calibration verifications (CCV) may use the same source standards as the calibration curve.

Note: The process of calibration verification referred to here is fundamentally different from the approach called "calibration" in some methods. As described in those methods, the calibration factors or response factors calculated during calibration are used to update the calibration factors or response factors used for sample quantitation. This approach, while employed in other EPA programs, amounts to a daily single-point calibration.

All target analytes and surrogates, including those reported as non-detects, must be included in periodic calibration verifications for purposes of retention time confirmation and to demonstrate that calibration verification criteria are being met, i.e., RPD, per 2009 and 2016 TNI Std. EL-V1M4 Sec. 1.7.2.

All samples must be bracketed by periodic analyses of standards that meet the QC acceptance criteria (e.g., calibration and retention time). The frequency is found in the determinative methods or SOPs.

Note: If an internal standard calibration is being used then bracketing calibration verification standards may not be are not required, only daily verifications are needed. The results from these verification standards must meet the calibration verification criteria and the retention time criteria (if applicable).

Generally, the calibrations must be verified by an ICV analyzed immediately following initial calibration and before sample analysis. The ICV may be used as the first bracketing CCV, if criteria for both are met.

A continuing instrument calibration verification (CCV) is generally analyzed at the beginning of each 12-hour analytical shift during which samples are analyzed. The 12-hour analytical shift begins with the injection of the calibration verification standard (or the MS tuning standard in MS methods). The shift ends after the completion of the analysis of the last sample, QC, or standard that can be injected within 12-hours of the beginning of the shift. For methods that have quantitation by external calibration models, a CCV is analyzed at the end of each analytical sequence. Some methods have more frequent CCV requirements. See specific SOPs. Most inorganic methods require the CCV to be analyzed after ever 10 samples or injections, including matrix or batch QC samples.

Note: If an internal standard calibration is being used (e.g., GCMS) then bracketing standards are not required, only daily verifications are needed, except as specified by program or method requirements.

If the results of a CCV are outside the established acceptance criteria and analysis of a second consecutive (and immediate) CCV fails to produce results within acceptance criteria, corrective action shall be performed. Once corrective actions have been completed and documented, the laboratory shall demonstrate acceptable instrument / method performance by analyzing two consecutive CCVs, or a new initial instrument calibration shall be performed.

Sample analyses and reporting of data may not occur or continue until the analytical system is calibrated or calibration verified. However, data associated with an unacceptable calibration verification may be fully useable reported based upon discussion and approval of the client under the following special conditions:

a). when the acceptance criteria for the CCV are exceeded high (i.e., high bias) and the associated samples within the batch are non-detects, then those non-detects may be reported case narrative comment explaining the high bias. Otherwise the samples affected by the unacceptable CCV shall be re-analyzed after a new calibration curve has been established, evaluated and accepted; or

b). when the acceptance criteria for the CCV are exceeded low (i.e., low bias), those sample results may be reported if they exceed a maximum regulatory limit/decision level. Otherwise the samples affected by the unacceptable CCV shall be re-analyzed after a new calibration curve has been established, evaluated and accepted.

Samples reported by the 2 conditions identified above will be appropriately flagged.

20.4.1.2 Verification of Linear and Non-Linear Calibrations

Calibration verification for calibrations involves the calculation of the percent drift or the percent difference of the instrument response between the initial calibration and each subsequent analysis of the verification standard. (These calculations are available in the laboratory method SOPs.) Verification standards are evaluated based on the % Difference from the average CF or RF of the initial calibration or based on % Drift or % Recovery if a linear or quadratic curve is used.

Regardless of whether a linear or non-linear calibration model is used, if initial verification criterion is not met, then no sample analyses may take place until the calibration has been verified or a new initial calibration is performed that meets the specifications listed in the method SOPs. If the calibration cannot be verified after the analysis of a single verification standard, then adjust the instrument operating conditions and/or perform instrument maintenance, and analyze another aliquot of the verification standard. If the calibration cannot be verified with the second standard, then a new initial calibration is performed.

- When the acceptance criteria for the calibration verification are exceeded high, i.e., high bias, and there are associated samples that are non-detects, then those non-detects may be reported. Otherwise, the samples affected by the unacceptable calibration verification shall be reanalyzed after a new calibration curve has been established, evaluated and accepted.
- When the acceptance criteria for the calibration verification are exceeded low, i.e., low bias, those sample results may be reported if they exceed a maximum regulatory limit/decision level. Otherwise, the samples affected by the unacceptable verification shall be reanalyzed after a new calibration curve has been established, evaluated and accepted. Alternatively, a reporting limit standard may be analyzed to demonstrate that the laboratory can still support non-detects at their reporting limit.

20.5 Tentatively Identified Compounds (TICs) – GC/MS Analysis

For samples containing components not associated with the calibration standards, a library search may be made for the purpose of tentative identification. The necessity to perform this type of identification will be determined by the purpose of the analyses being conducted. For example, the RCRA permit or waste delisting requirements may require the reporting of non-

target analytes. Only after visual comparison of sample spectra with the nearest library searches may the analyst assign a tentative identification. Data system library search routines should not use normalization routines that would misrepresent the library or unknown spectra when compared to each other. Additional details are provided in NDSC Document No. CA-Q-QM-001, Tentatively Identified Compounds (TICS) - GCMS Analysis.

Note: If the TIC compound is not part of the client target analyte list but is calibrated by the laboratory and is both qualitatively and/or quantitatively identifiable, it should not be reported as a TIC. If the compound is reported on the same form as true TICs, it should be qualified and/or narrated that the reported compound is qualitatively and quantitatively (if verification in control) reported compared to a known standard that is in control (where applicable).

20.6 GC/MS Tuning

Prior to any GC/MS analytical sequence, including calibration, the instrument parameters for the tune and subsequent sample analyses within that sequence must be set.

Prior to tuning/auto-tuning the mass spectrometer, the parameters may be adjusted within the specifications set by the manufacturer or the analytical method. These generally do not need any adjustment but it may be required based on the current instrument performance. If the tune verification does not pass it may be necessary to clean the source or perform additional maintenance. Any maintenance is documented in the maintenance log.

Table 20-1. Example – Instrumentation List

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TestAmerica Burlington Laboratory Instrument List

Area	Laboratory ID	Equipment/ Instrument	Manufacturer	Model Number	Serial Number	Terminal ID	Software and Version	Year Put into Service	Condition When Received
VOA		Purge & Trap	OI Analytical	4760	A708447057			2017	New
VOA	CHAD	GC/MS	Agilent	6890 / 5973	US33220083	TAISAC040	Chemstation E.02.02.1431	2020	USED
VOA	CHD	GC/MS	Agilent	6890N / 5973	CN10439015	TAIBURINSTD	Chemstation E.02.02.1431	2004	NEW
VOA	CHJ	GC/MS	Agilent	6890N / 5973	CN10430052	DBNFZDK1	Chemstation E.01.00.237	2009	USED
VOA	CHAA	GC/MS	Agilent	6890 / 5973	US00035345	TAIBUR1018	Chemstation E.02.01.1177	2013	USED
AIR	CH0001	GC/FID/TCD	Varian	CP-3800	S/N 10328	BTV-VARIAN	Star version 6.20	2003	NEW
AIR	CH1031	GC/FID/TCD	Agilent	7890A	CN10301031	TAIBUR0151	Chemstation B.04.03	2010	NEW
AIR	CHC	GC/MS	Agilent	6890N / 5973	CN10424016	TAIBURINST3	Chemstation E.02.00.493	UNKNOWN	NEW
AIR	CHG	GC/MS	Agilent	6890N / 5973	CN10437065	TAIBURINST1	Chemstation E.02.00.493	UNKNOWN	USED
AIR	CHE	GC/MS	Agilent	6890N / 5973	CN10453004	TAIBURINSTE	Chemstation E.02.02.1431	2005	NEW
AIR	CHF	GC/MS	Agilent	6890N / 5973	CN10531065	BTV-DGX58PH1	Chemstation E.02.00.493	2005	NEW
AIR	CHW	GC/MS	Agilent	7890A / 5975B	CN1231116	TAIBURVOAW	Chemstation E.02.01.1177	2012	USED
AIR	CHX	GC/MS	Agilent	6890N / 5975	CN10526012	TAIBURINSTX	Chemstation D.02.02.1431	2013	USED
AIR	CHAE	GC/MS	Agilent	6890 / 5973	US1853055	TAISAC0076	Chemstation E.02.02.1431	2020	USED
AIR	CHAH	GC/MS	Agilent	6890 / 5973	US30965918	TAISAC0075	Chemstation E.10.36.86.66	2020	USED
GC	CH7424	GC/ECD/ECD	Agilent	6890N	US10332093	TAIBUR0157	ChemStation E.02.01.1177	2003	NEW
GC	CH3283	GC/ECD/ECD	Agilent	6890N	US10805001	TAIBURINST3283	Chemstation D.01.02.16	2008	NEW
GC	CH0825	GC/ECD/ECD	Agilent	6890N	US10202136	BTV-D8X83SH1	Chemstation D.01.02.16	2002	NEW

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TestAmerica Burlington Laboratory Instrument List

Area	Laboratory ID	Equipment/ Instrument	Manufacturer	Model Number	Serial Number	Terminal ID	Software and Version	Year Put into Service	Condition When Received
GC	CH5253	GC/ECD/ECD	Agilent	6890N	CN10723008	TAIBUR0157	ChemStation E.02.01.1177	2007	NEW
GC	CH0911	GC/ECD/ECD	Agilent	6890N	US10230082	TAIBURINST0825	Chemstation D.01.02.16	2002	NEW
GC	CH5005	GC/ECD/ECD	Agilent	6890N	CN10615005	TAIBURINST0825	Chemstation D.01.02.16	2009	USED
SVOA	CHS	GC/MS	Agilent	7890A / 5975	CN10211095	TAIBURINST2	Chemstation E.02.00.493	2010	NEW
SVOA	CHT	GC/MS	Agilent	7890A / 5975	CN10211037	wesweigel	Chemstation E.02.00.493	2010	USED
SVOA	CHP	GC/MS	Hewlett-Packard	5890II / 5971	3203A40985	ICP8-PC	EnviroQuant Chemstation G1701BA Ver: B.01.00	1992	USED
SVOA	CHU	GC/MS	Hewlett-Packard	5890II Plus / 5972	3336A61535	BTV-D1Y58PH1	Chemstation G1701BA Ver B.01.00	1997	NEW
SVOA	CHH	GC/MS	Agilent	6890N / 5975	CN10608102	TAIBUR0138	Chemstation E.02.01.1177	2006	NEW
SVOA	CHZ	GC/MS	Agilent	6890A / 5973	US00036343	TAIBUR0133	Chemstation D.01.02.16	2000	NEW
SVOA	CHAC	GC/MS	Hewlett-Packard	6890 Plus / 5973	US00030199	TAIBURINSTAC	EnviroQuant Chemstation G1701BA Ver: B.01.00	2013	USED
SVOA	CHAG	GC/MS	Agilent	7890A / 5975C	US80618504	TAIBURSL1	Chemstation E.02.02.1431	2020	USED
LCMS	LC812	HPLC	SciEx (812)	HPLC: Nexera XR MSMS: QQQ 5500	HPLC: L204356 ; MSMS: EF221331812	TAIBURLC812	Sciex Analyst 1.6.3 Build 5095	2019	NEW
LCMS	LC410	HPLC	Waters (410)	HPLC: 2795 MSMS: Quattro Premier	HPLC: D00SM9 410M MSMS: VAA260	BTV-HPLC2	Mass Lynx V.4.1 SCN883	2004	NEW
LCMS	CH1208	HPLC/UV/PDA	Waters	600	60004790RP	TAIBURAIR-G	Chemstation A.08.03 [1417]	1988	NEW

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TestAmerica Burlington Laboratory Instrument List

Area	Laboratory ID	Equipment/ Instrument	Manufacturer	Model Number	Serial Number	Terminal ID	Software and Version	Year Put into Service	Condition When Received
LCMS	CH1488	HPLC/UV	Dionex	P680	1680407	TAIBURAIR-G	Chemstation A.09.03 [1417]	1991	UNKNOWN
LCMS	LC3062	HPLC	Waters	616	MX5NM6829M	BTV-D28XRS11	Mass Lynx V.4.1 SCN627	UNKNOWN	NEW
EX		NEVAP1	FMS	05-31-2016	E-0361			2017	NEW
EX		NEVAP2	FMS	10-19-2016	E-0374			2017	NEW
EX		NEVAP3	FMS	10-19-2016	E-0375			2017	NEW
EX		NEVAP4	FMS	10-19-2016	E-0373			2017	NEW
EX		NEVAP5	FMS	05-31-2016	E-0360			2017	NEW
EX	EXTGPCI	GPC	J2 Scientific	Autoinject 110	02D-1030-2.1	TAIEDI0009	Accuprep & Accuchrome 2.1.0.9	2002	NEW
EX	EXTGPCM	GPC	J2 Scientific	573-214-0472	GPC-1555-34-DIIS	uses hub from manufacturer	PrepLink	2019	NEW
EX	EXTGPCO	GPC	J2 Scientific	573-214-0472	GPC-1554-3.4-DIIS	uses hub from manufacturer	PrepLink	2019	NEW
EX	SOXA	Soxtherm	Gerhardt	SE3AS306A	4012396			UNKNOWN	UNKNOWN
EX	SOXB	Soxtherm	Gerhardt	SE3AS306A	4022047			UNKNOWN	UNKNOWN
EX	SOXC	Soxtherm	Gerhardt	SE3AS306A	4022046			UNKNOWN	UNKNOWN
EX	SOXD	Soxtherm	Gerhardt	SE3AS306A	4022045			UNKNOWN	UNKNOWN
EX	SOXE	Soxtherm	Gerhardt	SE3AS306A	4022068			UNKNOWN	UNKNOWN
EX	SOXF	Soxtherm	Gerhardt	SE3AS306A	4022070			UNKNOWN	UNKNOWN
EX		pH Meter	Denver Instruments	UB-5	Sn8213065P			UNKNOWN	UNKNOWN
WC		Automated Distillation Apparatus	Westco	Easy Dist	1090			2002	NEW
WC		Automated Distillation Apparatus	Westco	Easy Dist	1091			2002	NEW
WC	WCCH1	Elemental Analyzer (TOC)	Carlo Erba	NA 1500	220465	TAIBURCHN1	EAS32 version 1.4.1.115	1991	UNKNOWN

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TestAmerica Burlington Laboratory Instrument List

Area	Laboratory ID	Equipment/ Instrument	Manufacturer	Model Number	Serial Number	Terminal ID	Software and Version	Year Put into Service	Condition When Received
WC	WCCH2	Elemental Analyzer (TOC)	Carlo Erba	EA1108	249146	BTV-KRC	EAS32 version 1.4.1.113	1991	UNKNOWN
WC	WCLachat	Flow Injection Analyzer	Lachat	QuikChem 8000	A83000-2167	BTV-BAA	Omnion version 3.0.217	2000	UNKNOWN
WC		Conductivity Meter	Oakton	COND6+	2694637			2017	NEW
MET	MEPCV3 II	CVAA	Leeman Labs	HydraAA112-0064-1	8015	TAIBUR0143	WinHg Runner 1.7 CT Rev 0.286 WinHg Database 1.7	2008	NEW
MET	METICPMS2	ICP-MS	Thermo Elemental (METICPMS2)	X7	X0288	Windows.R5VR6 RM	PlasmaLab 2.6.2_337	2003	NEW
MET	METICPMS4	ICP-MS	Agilent	7800 ICP-MS	SG19425029	TAIBUR0162	Masshunter 4.6 C.01.06	2020	NEW
MET	METICP7	ICP-OES	Thermo Electron Corp (METICP7)	iCAP 6500	ICP 20063302	BTV-ICAP6500B	iTeva version 2.8.0.97	2006	NEW

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21.0 MEASUREMENT TRACEABILITY

21.1 Overview

Traceability of measurements shall be assured using a system of documentation, calibration, and analysis of reference standards. Laboratory equipment that are peripheral to analysis and whose calibration is not necessarily documented in a test method analysis or by analysis of a reference standard shall be subject to ongoing certifications of accuracy. At a minimum, these must include procedures for checking specifications of ancillary equipment: balances, thermometers, temperature, Deionized (DI) and Reverse Osmosis (RO) water systems, automatic pipettes and other volumetric measuring devices. (Refer to Section 20.3). With the exception of Class A Glassware and glass microliter syringes, quarterly accuracy checks are performed for all mechanical volumetric devices. Wherever possible, subsidiary or peripheral equipment is checked against standard equipment or standards that are traceable to national or international standards. Class A Glassware and glass microliter syringes should be routinely inspected for chips, acid etching or deformity (e.g., bent needle). If the Class A glassware or syringe is suspect, the accuracy of the glassware will be assessed prior to use.

21.2 NIST-Traceable Weights and Thermometers

Reference standards of measurement shall be used for calibration only and for no other purpose, unless it can be shown that their performance as reference standards would not be invalidated.

For NIST-traceable weights and thermometers, the laboratory requires that all calibrations be conducted by a calibration laboratory accredited by A2LA, NVLAP (National Voluntary Laboratory Accreditation Program), or another accreditation organization that is a signatory to a MRA (Mutual Recognition Arrangement) of one or more of the following cooperations – ILAC (International Laboratory Accreditation Cooperation) or APLAC (Asia–Pacific Laboratory Accreditation Cooperation). A calibration certificate and scope of accreditation is kept on file at the laboratory.

21.3 Reference Standards / Materials

Reference standards/materials, where commercially available, are traceable to certified reference materials. Commercially prepared reference standards, to the extent available, are purchased from vendors that are accredited to ISO Guide 34 and ISO/IEC Guide 17025. All reference standards from commercial vendors shall be accompanied with a certificate that includes at least the following information:

- Manufacturer
- Analytes or parameters calibrated
- Identification or lot number
- Calibration method
- Concentration with associated uncertainties
- Purity

If a standard cannot be purchased from a vendor that supplies a Certificate of Analysis, the purity of the standard is documented by analysis. The receipt of all reference standards must be documented. Reference standards are labeled with a unique Standard Identification Number

and expiration date. All documentation received with the reference standard is retained as a QC record and references the Standard Identification Number.

All reference, primary and working standards/materials, whether commercially purchased or laboratory prepared, must be checked regularly to ensure that the variability of the standard or material from the true value does not exceed method requirements. The accuracy of calibration standards is checked by comparison with a standard from a second source. In cases where a second standard manufacturer is not available, a vendor certified different lot is acceptable for use as a second source. For unique situations, such as air analysis where no other source or lot is available, a standard made by a different analyst would be considered a second source. The appropriate Quality Control (QC) criteria for specific standards are defined in laboratory SOPs. In most cases, the analysis of an Initial Calibration Verification (ICV) or LCS (where there is no sample preparation) is used as the second source confirmation. These checks are generally performed as an integral part of the analysis method (e.g. calibration checks, laboratory control samples).

All standards and materials must be stored and handled according to method or manufacturer's requirements in order to prevent contamination or deterioration. Refer to the Environmental Health & Safety Manual or laboratory SOPs. For safety requirements, please refer to method SOPs and the laboratory's Environmental Health and Safety Manual.

Standards and reference materials shall not be used after their expiration dates unless their reliability is verified by the laboratory and their use is approved by the Quality Assurance Manager. The laboratory must have documented contingency procedures for re-verifying expired standards.

21.4 Documentation and Labeling of Standards, Reagents, and Reference Materials

Reagents must be at a minimum the purity required in the test method. The date of reagent receipt and the expiration date are documented. The lots for most of the common solvents and acids are tested for acceptability prior to company-wide purchase. Refer to NDSC Document No. CA-Q-S-001, Solvent and Acid Lot Testing and Approval.

All manufacturer or vendor supplied Certificate of Analysis or Purity must be retained, stored appropriately, and readily available for use and inspection. These records are maintained in the LIMS. Records must be kept of the date of receipt and date of expiration of standards, reagents and reference materials. In addition, records of preparation of laboratory standards, reagents, and reference materials must be retained, stored appropriately, and be readily available for use and inspection. For detailed information on documentation and labeling, please refer to method specific SOPs.

Commercial materials purchased for preparation of calibration solutions, spike solutions, etc.., are usually accompanied with an assay certificate or the purity is noted on the label. If the assay purity is 96% or better, the weight provided by the vendor may be used without correction. If the assay purity is less than 96% a correction will be made to concentrations applied to solutions prepared from the stock commercial material. Blended gas standard cylinders use a nominal concentration if the certified value is within +/-15%, otherwise the certified values is used for the canister concentration.

21.4.1 All standards, reagents, and reference materials must be labeled in an unambiguous manner. Standards are logged into the laboratory's LIMS, and are assigned a unique identification number. The following information is typically recorded in the electronic database within the LIMS.

- Standard ID
- Description of Standard
- Department
- Preparer's name
- Final volume and number of vials prepared
- Solvent type and lot number
- Preparation Date
- Expiration Date
- Standard source type (stock or daughter)
- Standard type (spike, surrogate, other)
- Parent standard ID (if applicable)
- Parent Standard Analyte Concentration (if applicable)
- Parent Standard Amount used (if applicable)
- Component Analytes
- Final concentration of each analyte
- Comment box (text field)

Records are maintained electronically for standard and reference material preparation. These records show the traceability to purchased stocks or neat compounds. These records also include method of preparation, date of preparation, expiration date and preparer's name or initials. Preparation procedures are provided in the Method SOPs.

21.4.2 All standards, reagents, and reference materials must be clearly labeled with a minimum of the following information:

- Expiration Date (include prep date for reagents)
- Standard ID (from LIMS or logbook)
- Special Health/Safety warnings if applicable

Records must also be maintained of the date of receipt for commercially purchased items or date of preparation for laboratory prepared items. Special Health/Safety warnings must also be available to the analyst. This information is maintained in LIMS.

21.4.3 In addition, the following information may be helpful:

- Date opened (for multi-use containers, if applicable)
- Description of standard (if different from manufacturer's label or if standard was prepared in the laboratory)
- Recommended Storage Conditions
- Concentration (if applicable)
- Initials of analyst preparing standard or opening container

All containers of prepared reagents must include an expiration date and an ID number to trace back to preparation.

Procedures for preparation of reagents can be found in the Method SOPs.

Standard ID numbers must be traceable through associated logbooks, worksheets and preparation/analytical batch records.

All reagents and standards must be stored in accordance to the following priority: 1) with the manufacturer's recommendations; 2) with requirements in the specific analytical methods as specified in the laboratory SOP.

22.0 SAMPLING

22.1 Overview

The laboratory does not provide sampling services. The laboratory's responsibility in the sample collection process lies in supplying the sampler with the necessary coolers, reagent water, sample containers, preservatives, sample labels, custody seals, COC forms, ice, and packing materials required to properly preserve, pack, and ship samples to the laboratory

22.2 Sampling Containers

The laboratory offers clean sampling containers for use by clients. These containers are obtained from reputable container manufacturers and meet EPA specifications as required. Certificates of cleanliness for bottles and preservatives are provided by the supplier and are maintained at the laboratory. Alternatively, the certificates may be maintained by the supplier and available to the laboratory on-line.

22.2.1 Preservatives

Upon request, preservatives are provided to the client in pre-cleaned sampling containers. In some cases containers may be purchased pre-preserved from the container supplier. Whether prepared by the laboratory or bought pre-preserved, the grades of the preservatives are at a minimum:

- Hydrochloric Acid – Reagent ACS (Certified VOA Free) or equivalent
- Methanol – Purge and Trap grade
- Nitric Acid – Instra-Analyzed or equivalent
- Sodium Bisulfate – ACS Grade or equivalent
- Sodium Hydroxide – Instra-Analyzed or equivalent
- Sulfuric Acid – Instra-Analyzed or equivalent
- Sodium Thiosulfate – ACS Grade or equivalent

22.3 Definition of Holding Time

The date and time of sampling documented on the COC form establishes the day and time zero. As a general rule, when the maximum allowable holding time is expressed in days (e.g., 14 days, 28 days), the holding time is based on calendar day measured. Holding times expressed in hours (e.g., 6 hours, 24 hours, etc.) are measured from date and time zero. Holding times for analysis include any necessary reanalysis. However, there are some programs that determine holding time compliance based on the date and specific time of analysis compared to the time of sampling regardless of how long the holding time is.

22.4 Sampling Containers, Preservation Requirements, Holding Times

The preservation and holding time criteria specified in the laboratory SOPs are derived from the source documents for the methods. If method required holding times or preservation requirements are not met, the reports will be qualified using a flag, footnote or case narrative. As soon as possible or "ASAP" is an EPA designation for tests for which rapid analysis is advised, but for which neither EPA nor the laboratory have a basis for a holding time.

22.5 Sample Aliquots / Subsampling

Taking a representative sub-sample from a container is necessary to ensure that the analytical results are representative of the sample collected in the field. The size of the sample container, the quantity of sample fitted within the container, and the homogeneity of the sample need consideration when sub-sampling for sample preparation. It is the laboratory's responsibility to take a representative subsample or aliquot of the sample provided for analysis.

Analysts should handle each sample as if it is potentially dangerous. At a minimum, safety glasses, gloves, and lab coats must be worn when preparing aliquots for analysis.

Guidelines on taking sample aliquots and subsampling are located in method-specific SOPs or SOP BR-QA-020, Subsampling.

23.0 HANDLING OF SAMPLES

It is the responsibility of the client to send us representative and/or homogeneous and properly preserved samples of the system from which they are drawn. The laboratory assumes that all multiple sample containers with the same designator/description and bottle type contain a homogeneous, representative sample.

The laboratory provides the appropriate sample containers, required preservative, chain-of-custody (COC) forms, shipping containers, labels, and custody seals. The laboratory also provides trip blanks and analyte-free water for field blanks. Preparation of methanol containers for field preservation of volatile soil samples is available.

Sample management procedures at the laboratory ensure that sample integrity and custody are maintained and documented from sampling/receipt through disposal.

23.1 Chain of Custody (COC)

The COC form is the written documented history of any sample and is initiated when bottles are sent to the field, or at the time of sampling. This form is completed by the sampling personnel and accompanies the samples to the laboratory where it is received and stored under the laboratory's custody. The purpose of the COC form is to provide a legal written record of the handling of samples from the time of collection until they are received at the laboratory. It also serves as the primary written request for analyses from the client to the laboratory. The COC form acts as a purchase order for analytical services when no other contractual agreement is in effect.

23.1.1 Field Documentation

The information the sampler needs to provide at the time of sampling on the container label is:

- Sample identification
- Date and time

- Preservative

During the sampling process, the COC form is completed and must be legible. This form includes information such as:

- Client name, address, phone number and fax number (if available)
- Project name and/or number
- The sample identification
- Date, time and location of sampling
- Sample collectors name
- The matrix description
- The container description
- The total number of each type of container
- Preservatives used
- Analysis requested
- Requested turnaround time (TAT)
- Any special instructions
- Purchase Order number or billing information (e.g. quote number) if available
- The date and time that each person received or relinquished the sample(s), including their signed name.

When the sampling personnel deliver the samples directly to the laboratory personnel, the samples are stored in a cooler with ice, as applicable, and remain solely in the possession of the client's field technician until the samples are delivered to the laboratory personnel. The sample collector must assure that each container is in his/her physical possession or in his/her view at all times, or stored in such a place and manner to preclude tampering. The field technician relinquishes the samples in writing on the COC form to the sample control personnel at the laboratory or to a laboratory courier. When sampling personnel deliver the samples through a common carrier (Fed-Ex, UPS), the CoC relinquished date/time is completed by the field personnel and samples are released to the carrier. Samples are only considered to be received by the laboratory when personnel at the fixed laboratory facility have physical contact with the samples.

Note: Independent couriers are not required to sign the COC form. The COC is usually kept in the sealed sample cooler. The receipt from the courier is stored in log-in by date; it lists all receipts each date.

23.1.2 Legal / Evidentiary Chain-of-Custody

Samples being tested for litigation may require locked storage and documentation of the time and personnel responsible when the sample was not in storage. This level of documentation is available upon client request and procedures to define these activities are in place and include the following:

- A chain-of-custody document is initiated for each bottle type submitted by the client.

- The chain of custody is signed each time the sample is stored, removed from storage, or changes hands.
- Clients requesting legal internal chain-of-custody documentation receive the completed forms after the analysis is complete.

23.2 Sample Receipt

Samples are received at the laboratory by designated sample receiving personnel and a unique laboratory project identification number is assigned. Each sample container shall be assigned a unique sample identification number that is cross-referenced to the client identification number such that traceability of test samples is unambiguous and documented. Each sample container is affixed with a durable sample identification label. Sample acceptance, receipt, tracking and storage procedures are detailed in the laboratory's SOP No. BR_SM-001, Sample Management and summarized in the following sections.

23.2.1 Laboratory Receipt

When samples arrive at the laboratory, sample receiving personnel inspect the coolers and samples. The integrity of each sample must be determined by comparing sample labels or tags with the COC and by visual checks of the container for possible damage. Any non-conformance, irregularity, or compromised sample receipt must be documented on a NCM (Non Conformance Form) and brought to the immediate attention of the client. The COC, shipping documents, documentation of any non-conformance, irregularity, or compromised sample receipt, record of client contact, and resulting instructions become part of the project record.

Sample Registration personnel check and document preservation of non-volatile liquid samples after the samples have been entered into the LIMS and before they are released to the laboratory for testing or placed into storage. Any checks of volatile samples are performed and documented at the time of analysis.

23.3 Sample Acceptance Policy

The laboratory has a written sample acceptance policy (Figure 23-1) that clearly outlines the circumstances under which samples shall be accepted or rejected. These include:

- a COC filled out completely;
- samples must be properly labeled;
- proper sample containers with adequate volume for the analysis (Sampling Guide) and necessary QC;
- samples must be preserved according to the requirements of the requested analytical method (Sampling Guide);
- sample holding times must be adhered to (Sampling Guide);
- the project manager will be notified if any sample is received in damaged condition.

Data from samples which do not meet these criteria are flagged and the nature of the variation from policy is defined.

23.3.1 After inspecting the samples, the sample receiving personnel sign and date the COC form, make any necessary notes of the samples' conditions and store them in appropriate refrigerators or storage locations.

23.3.2 Any deviations from these checks that question the suitability of the sample for analysis, or incomplete documentation as to the tests required will be resolved by consultation with the client. If the sample acceptance policy criteria are not met, the laboratory shall either:

- Retain all correspondence and/or records of communications with the client regarding the disposition of rejected samples, or
- Fully document any decision to proceed with sample analysis that does not meet sample acceptance criteria.

Note: North Carolina requires that they be notified when samples are processed that do not meet sample acceptance criteria.

Once sample acceptance is verified, the samples are logged into LIMS according SOP No. BR-SM-001, Sample Management.

23.4 Sample Storage

In order to avoid deterioration, contamination or damage to a sample during storage and handling, from the time of receipt until all analyses are complete, samples are stored in refrigerators, freezers or protected locations suitable for the sample matrix. In addition, samples to be analyzed for volatile organic parameters are stored in separate refrigerators designated for volatile organic parameters only. Samples are never to be stored with reagents, standards or materials that may create contamination.

To ensure the integrity of the samples during storage, refrigerator blanks are maintained in the volatile sample refrigerators and analyzed at least every two weeks in project logins.

Analysts and technicians retrieve the sample container allocated to their analysis from the designated refrigerator and place them on carts, analyze the sample, and return the remaining sample or empty container to the refrigerator from which it originally came. All unused portions of samples, including empty sample containers, are returned to the secure sample control area. All samples are kept in the refrigerators for two to four weeks after analysis, which meets or exceeds most sample holding times. After two to four weeks the samples can be moved to dry room temperature, sample archive area where they are stored for an additional four weeks before they are disposed of. This eight week holding period allows samples to be checked if a discrepancy or question arises. Special arrangements may be made to store samples for longer periods of time. This extended holding period allows additional metal analyses to be performed on the archived sample and assists clients in dealing with legal matters or regulatory issues.

Access to the laboratory is controlled such that sample storage need not be locked at all times unless a project specifically demands it. Samples are accessible to laboratory personnel only. Visitors to the laboratory are prohibited from entering the refrigerator and laboratory areas unless accompanied by an employee of Eurofins TestAmerica Burlington.

23.5 Hazardous Samples and Foreign Soils

To minimize exposure to personnel and to avoid potential accidents, hazardous and foreign soil samples are stored in an isolated area designated for hazardous waste only. For any sample that is known to be hazardous at the time of receipt or, if after completion of analysis the result

exceeds the acceptable regulatory levels, a Hazardous Sample Notice must be completed by the analyst. This form may be completed by Sample Control, Project Managers, or analysts and must be attached to the report. The sample itself is clearly marked with a red stamp, stamped on the sample label reading “HAZARDOUS” or “FOREIGN SOIL” and placed in a colored and/or marked bag to easily identify the sample. The date, log number, lab sample number, and the result or brief description of the hazard are all written on the Hazardous & Foreign Soil Sample Notice. A copy of the form must be included with the original COC and Work Order and the original must be given to the Sample Control Custodian. Analysts will notify Sample Control of any sample determined to be hazardous after completion of analysis by completing a Hazardous Sample Notice. All hazardous samples are either returned to the client or disposed of appropriately through a hazardous waste disposal firm that lab-packs all hazardous samples and removes them from the laboratory. Foreign soil samples are sent out for incineration by a USDA-approved waste disposal facility.

23.6 Sample Shipping

In the event that the laboratory needs to ship samples, the samples are placed in a cooler with enough ice to ensure the samples remain just above freezing and at or below 6.0°C transit. The samples are carefully surrounded by packing material to avoid breakage (yet maintain appropriate temperature). A trip blank is enclosed for those samples requiring water/solid volatile organic analyses (see Note). The chain-of-custody form is signed by the sample control technician and attached to the shipping paperwork. Samples are generally shipped overnight express or hand-delivered by a Eurofins TestAmerica Burlington courier to maintain sample integrity. All personnel involved with shipping and receiving samples must be trained to maintain the proper chain-of-custody documentation and to keep the samples intact and on ice. The laboratory’s Environmental, Health and Safety Manual contains additional shipping requirements.

Note: If a client does not request trip blank analysis on the COC or other paperwork, the laboratory will not analyze the trip blanks that were supplied. However, in the interest of good client service, the laboratory will advise the client at the time of sample receipt that it was noted that they did not request analysis of the trip blank; and that the laboratory is providing the notification to verify that they are not inadvertently omitting a key part of regulatory compliance testing.

23.7 Sample Disposal

Samples should be retained for a minimum of 30 days after the project report is sent, however, provisions may be made for earlier disposal of samples once the holding time is exceeded. Some samples are required to be held for longer periods based on regulatory or client requirements (e.g., 60 days after project report is sent). The laboratory must follow the longer sample retention requirements where required by regulation or client agreement. Several possibilities for sample disposal exist: the sample may be consumed completely during analysis, the sample may be returned to the customer or location of sampling for disposal, or the sample may be disposed of in accordance with the laboratory’s waste disposal procedures (SOP: BR_EH-001). All procedures in the laboratory’s Environmental Health and Safety Manual are followed during disposal. Samples are normally maintained in the laboratory no longer than two months from receipt unless otherwise requested. Unused portions of samples found or suspected to be hazardous according to state or federal guidelines may be returned to the client upon completion of the analytical work.

If a sample is part of a known litigation, the affected legal authority, sample data user, and/or submitter of the sample must participate in the decision about the sample's disposal. All documentation and correspondence concerning the disposal decision process must be kept on file. Pertinent information includes the date of disposal, nature of disposal (such as sample depletion, hazardous waste facility disposal, return to client), names of individuals who conducted the arrangements and physically completed the task. The laboratory will remove or deface sample labels prior to disposal unless this is accomplished through the disposal method (e.g., samples are incinerated). A Waste Disposal Record should be completed.

Figure 23-1. Example: Sample Acceptance Policy

All incoming work will be evaluated against the criteria listed below. Where applicable, data from any samples that do not meet the criteria listed below will be noted on the laboratory report defining the nature and substance of the variation. In addition the client will be notified either by telephone, fax or e-mail ASAP after the receipt of the samples.

Per State and/or Federal Regulation, the client is responsible to ensure that samples are shipped in accordance with DOT/IATA requirements, and that radioactive materials may only be delivered to licensed facilities. Any samples containing (or suspected to contain) Source, Byproduct, or Special Nuclear Material as defined by 10 CFR should be delivered directly to facilities licensed to handle such radioactive material. Natural material or ores containing naturally occurring radionuclides may be delivered to any Eurofins TestAmerica Burlington facility or courier as long as the activity concentration of the material does not exceed 270 pCi/g alpha or 2700 pCi/g beta (49 CFR Part 173).

1) Samples must arrive with labels intact with a Chain of Custody filled out completely. The following information must be recorded.
    - Client name, address, phone number and fax number (if available)
    - Project name and/or number
    - The sample identification
    - Date, time and location of sampling
    - The collectors name
    - The matrix description
    - The container description
    - The total number of each type of container
    - Preservatives used
    - Analysis requested
    - Requested turnaround time (TAT)
    - Any special instructions
    - Purchase Order number or billing information (e.g. quote number) if available
    - The date and time that each person received or relinquished the sample(s), including their signed name.
    - The date and time of receipt must be recorded between the last person to relinquish the samples and the person who receives the samples in the lab, and they must be

exactly the same.

- Information must be legible

2) Samples must be properly labeled.
   - Use durable labels (labels provided by Eurofins TestAmerica Burlington are preferred)
   - Include a unique identification number
   - Include sampling date and time & sampler ID
   - Include preservative used.
   - Use indelible ink
   - Information must be legible
3) Proper sample containers with adequate volume for the analysis and necessary QC are required for each analysis requested. See Lab Sampling Guide.
4) Samples must be preserved according to the requirements of the requested analytical method (See Sampling Guide**.**
5) Most analytical methods require chilling samples to 4^{o} C (other than water samples for metals analysis). For these methods, the criteria are met if the samples are chilled to below 6^{o} C and above freezing (0^{o}C). For methods with other temperature criteria (e.g. some methods require $\leq$ 10 ^{o}C), the samples must arrive within $\pm$ 2^{o} C of the required temperature or within the method specified range.

   5i.) Samples that are delivered to the laboratory on the same day they are collected may not meet the requirements of Section 5. In these cases, the samples shall be considered acceptable if the samples were received on ice.

   5ii.)If sample analysis is begun within fifteen (15) minutes of collection, thermal preservation is not required.

   5iii.)Thermal preservation is not required in the field if the laboratory receives and refrigerates the sample within fifteen (15) minutes of collection.

   - Chemical preservation (pH) will be verified prior to analysis and documented, either in sample control or at the analyst's level. The project manager will be notified immediately if there is a discrepancy. If analyses will still be performed, all affected results will be flagged to indicate improper preservation.
   - For Organic analyses in drinking water, Residual chlorine must be neutralized prior to preservation. If there is prior knowledge that the samples are not chlorinated, state it on the COC and use the VOA vials pre-preserved with HCl. The following are other options for a sampler and laboratory where the presence of chlorine is not known:
     - 1. Test for residual chlorine in the field prior to sampling.
       - If no chlorine is present, the samples are to be preserved using HCl as usual.
       - If chlorine is present, add either ascorbic acid or sodium thiosulfate prior to adding HCl.

- 2. Use VOA vials pre-preserved with sodium thiosulfate or ascorbic acid and add HCl after filling the VOA vial with the sample.

Sample Holding Times

- Eurofins TestAmerica Burlington will make every effort to analyze samples within the regulatory holding time. Samples must be received in the laboratory with enough time to perform the sample analysis. Except for short holding time samples (< 48hr HT) sample must be received with at least 48 hrs (2 working days) remaining on the holding time for us to ensure analysis.
- Analyses that are designated as "field" analyses (Odor, pH, Dissolved Oxygen, Disinfectant Residual; a.k.a. Residual Chlorine, and Redox Potential) should be analyzed ASAP by the field sampler prior to delivering to the lab (within 15 minutes). However, if the analyses are to be performed in the laboratory, Eurofins TestAmerica Burlington will make every effort to analyze the samples within 24 hours from receipt of the samples in the testing laboratory. Samples for "field" analyses received after 4:00 pm on Friday or on the weekend will be analyzed no later than the next business day after receipt (Monday unless a holiday). Samples will remain refrigerated and sealed until the time of analysis. The actual times of all "field" sample analyses are noted on the "Short Hold Time Detail Report" in the final report. Samples analyzed in the laboratory will be qualified on the final report with an 'H' to indicate holding time exceedance.

6) All samples submitted for Volatile Organic analyses must have a Trip Blank submitted at the same time. Eurofins TestAmerica Burlington will supply a blank with the bottle order.
7) The project manager will be notified if any sample is received in damaged condition. Eurofins TestAmerica Burlington will request that a sample be resubmitted for analysis.
8) Recommendations for packing samples for shipment.
   - Pack samples in Ice rather than "Blue" ice packs.
   - Soil samples should be placed in plastic zip-lock bags. The containers often have dirt around the top and do not seal very well and are prone to intrusion from the water from melted ice.
   - Water samples would be best if wrapped with bubble-wrap or paper (newspaper, or paper towels work) and then placed in plastic zip-lock bags.
   - Fill extra cooler space with bubble wrap.

24.0 ASSURING THE QUALITY OF TEST RESULTS

24.1 Overview

In order to assure our clients of the validity of their data, the laboratory continuously evaluates the quality of the analytical process. The analytical process is controlled not only by instrument calibration as discussed in Section 20, but also by routine process quality control measurements (e.g. Blanks, Laboratory Control Samples (LCS), Matrix Spikes (MS), duplicates (DUP), surrogates, Internal Standards (IS)). These quality control checks are performed as required by the method or regulations to assess precision and accuracy. Quality control samples are to be treated in the exact same manner as the associated field samples being tested. In addition to

the routine process quality control samples, Proficiency Testing (PT) Samples (concentrations unknown to laboratory) are analyzed to help ensure laboratory performance.

24.2 Controls

Sample preparation or pre-treatment is commonly required before analysis. Typical preparation steps include homogenization, grinding, solvent extraction, sonication, acid digestion, distillation, reflux, evaporation, drying and ashing. During these pre-treatment steps, samples are arranged into discreet manageable groups referred to as preparation (prep) batches. Prep batches provide a means to control variability in sample treatment. Control samples are added to each prep batch to monitor method performance and are processed through the entire analytical procedure with investigative/field samples.

24.3 Negative Controls

Table 24-1. Example – Negative Controls

Control Type	Details
Method Blank (MB)	are used to assess preparation and analysis for possible contamination during the preparation and processing steps.
	The specific frequency of use for method blanks during the analytical sequence is defined in the specific standard operating procedure for each analysis. Generally it is 1 for each batch of samples; not to exceed 20 environmental samples.
	The method blank is prepared from a clean matrix similar to that of the associated samples that is free from target analytes (e.g., Reagent water, Ottawa sand, glass beads, etc.) and is processed along with and under the same conditions as the associated samples. The method blank goes through all of the steps of the process (including as necessary: filtration, clean-ups, etc.).
	Reanalyze or qualify associated sample results when the concentration of a targeted analyte in the blank is at or above the reporting limit as established by the method or by regulation, AND is greater than 1/10 of the amount measured in the sample.
Calibration Blanks	are prepared and analyzed along with calibration standards where applicable. They are prepared using the same reagents that are used to prepare the standards. In some analyses the calibration blank may be included in the calibration curve.
Instrument Blanks	are blank reagents or reagent water that may be processed during an analytical sequence in order to assess contamination in the analytical system. In general, instrument blanks are used to differentiate between contamination caused by the analytical system and that caused by the sample handling or sample prep process. Instrument blanks may also be inserted throughout the analytical sequence to minimize the effect of carryover from samples with high analyte content.
Trip Blank [1]	are required to be submitted by the client with each shipment of samples requiring aqueous and solid volatiles analyses (or as specified in the client's project plan). Additionally, trip blanks may be prepared and analyzed for volatile analysis of air samples, when required by the client. A trip blank may be purchased (certified clean) or is prepared by the laboratory by filling a clean container with pure deionized water that has been purged to remove any volatile compounds. Appropriate preservatives are also added to the container. The trip blank is sent with the bottle order and is intended to reflect the environment that the containers are subjected to throughout shipping and handling and help identify possible sources if contamination is found. The field sampler returns the trip blank in the cooler with the field samples.

Table 24-1. Example – Negative Controls

Control Type	Details
Field Blanks [1]	are sometimes used for specific projects by the field samplers. A field blank prepared in the field by filling a clean container with pure reagent water and appropriate preservative, if any, for the specific sampling activity being undertaken. (EPA OSWER)
Equipment Blanks [1]	are also sometimes created in the field for specific projects. An equipment blank is a sample of analyte-free media which has been used to rinse common sampling equipment to check effectiveness of decontamination procedures. (TNI)
Holding Blanks	also referred to as refrigerator or freezer blanks, are used to monitor the sample storage units for volatile organic compounds during the storage of VOA samples in the laboratory

[1] When known, these field QC samples should not be selected for matrix QC as it does not provide information on the behavior of the target compounds in the field samples. Usually, the client sample ID will provide information to identify the field blanks with labels such as "FB", "EB", or "TB."

Evaluation criteria and corrective action for these controls are defined in the specific standard operating procedure for each analysis.

24.4 Positive Controls

Control samples (e.g., QC indicators) are analyzed with each batch of samples to evaluate data based upon (1) Method Performance (Laboratory Control Sample (LCS) or Blank Spike (BS)), which entails both the preparation and measurement steps; and (2) Matrix Effects (Matrix Spike (MS) (Matrix spikes are not applicable to air) or Sample Duplicate (MD, DUP), which evaluates field sampling accuracy, precision, representativeness, interferences, and the effect of the matrix on the method performed. Each regulatory program and each method within those programs specify the control samples that are prepared and/or analyzed with a specific batch

Note that frequency of control samples vary with specific regulatory, methodology and project specific criteria. Complete details on method control samples are as listed in each analytical SOP.

24.4.1 Method Performance Control - Laboratory Control Sample (LCS)

The LCS measures the accuracy of the method in a blank matrix and assesses method performance independent of potential field sample matrix affects in a laboratory batch.

The LCS is prepared from a clean matrix similar to that of the associated samples that is free from target analytes (for example: Reagent water, Ottawa sand, glass beads, etc.) and is processed along with and under the same conditions as the associated samples. The LCS is spiked with verified known amounts of analytes or is made of a material containing known and verified amounts of analytes, taken through all preparation and analysis steps along with the field samples. Where there is no preparation taken for an analysis (such as in aqueous volatiles), or when all samples and standards undergo the same preparation and analysis process, a calibration verification standard is reported as the LCS. In some instances where there is no practical clean solid matrix available, aqueous LCS's may be processed for solid matrices; final results may be calculated as mg/kg or ug/kg, assuming 100% solids and a weight equivalent to the aliquot used for the corresponding field samples, to facilitate comparison with the field samples.

Certified pre-made reference material purchased from a NIST/A2LA accredited vendor may also be used for the LCS when the material represents the sample matrix or the analyte is not easily spiked (e.g. solid matrix LCS for metals, TDS, etc.).

The specific frequency of use for LCS during the analytical sequence is defined in the specific standard operating procedure for each analysis. It is generally 1 for each batch of samples; not to exceed 20 environmental samples.

If the mandated or requested test method, or project requirements, do not specify the spiking components, the laboratory shall spike all reportable components to be reported in the Laboratory Control Sample (and Matrix Spike) where applicable (e.g. no spike of pH). However, in cases where the components interfere with accurate assessment (such as simultaneously spiking chlordane, Toxaphene and PCBs in Method 608), the test method has an extremely long list of components or components are incompatible, at a minimum, a representative number of the listed components (see below) shall be used to control the test method. The selected components of each spiking mix shall represent all chemistries, elution patterns and masses, permit specified analytes and other client requested components. However, the laboratory shall ensure that all reported components are used in the spike mixture within a two-year time period.

- For methods that have 1-10 target analytes, spike all components.
- For methods that include 11-20 target analytes, spike at least 10 or 80%, whichever is greater.
- For methods with more than 20 target analytes, spike at least 16 components.
- Exception: Due to analyte incompatibility in pesticides, Toxaphene and Chlordane are only spiked at client request based on specific project needs.
- Exception: Due to analyte incompatibility between the various PCB aroclors, aroclors 1016 and 1260 are used for spiking as they cover the range of all of the aroclors. Specific aroclors may be used by request on a project specific basis.

24.5 Sample Matrix Controls

Table 24-3. Sample Matrix Control

Control Type	Details	
Matrix Spikes (MS)	Use	used to assess the effect sample matrix of the spiked sample has on the precision and accuracy of the results generated by the method used;
	Typical Frequency [1]	At a minimum, with each matrix-specific batch of samples processed, an MS is carried through the complete analytical procedure. Unless specified by the client, samples used for spiking are randomly selected and rotated between different client projects. If the mandated or requested test method does not specify the spiking components, the laboratory shall spike all reportable components to be reported in the Laboratory Control Sample and Matrix Spike. Refer to the method SOP for complete details
	Description	essentially a sample fortified with a known amount of the test analyte(s).
Surrogate	Use	Measures method performance to sample matrix (organics only).

Table 24-3. Sample Matrix Control

Control Type	Details	
	Typical Frequency [1]	Are added to all samples, standards, and blanks, for all organic chromatography methods except when the matrix precludes its use or when a surrogate is not available. The recovery of the surrogates is compared to the acceptance limits for the specific method. Poor surrogate recovery may indicate a problem with sample composition and shall be reported, with data qualifiers, to the client whose sample produced poor recovery.
	Description	Are similar to matrix spikes except the analytes are compounds with properties that mimic the analyte of interest and are unlikely to be found in environment samples.
Duplicates[2]	Use	For a measure of analytical precision, with each matrix-specific batch of samples processed, a matrix duplicate (MD or DUP) sample, matrix spike duplicate (MSD), or LCS duplicate (LCSD) is carried through the complete analytical procedure.
	Typical Frequency [1]	Duplicate samples are usually analyzed with methods that do not require matrix spike analysis.
	Description	Performed by analyzing two aliquots of the same field sample independently or an additional LCS.
Internal Standards	Use	Are spiked into all environmental and quality control samples (including the initial calibration standards) to monitor the qualitative aspect of organic and some inorganic analytical measurements.
	Typical Frequency [1]	All organic and ICP methods as required by the analytical method.
	Description	Used to correct for matrix effects and to help troubleshoot variability in analytical response and are assessed after data acquisition. Possible sources of poor internal standard response are sample matrix, poor analytical technique or instrument performance.

[1] See the specific analytical SOP for type and frequency of sample matrix control samples.
[2] LCSD's are normally not performed except when regulatory agencies or client specifications require them. The recoveries for the spiked duplicate samples must meet the same laboratory established recovery limits as the accuracy QC samples. If an LCSD is analyzed both the LCS and LCSD must meet the same recovery criteria and be included in the final report. The precision measurement is reported as "Relative Percent Difference" (RPD). Poor precision between duplicates (except LCS/LCSD) may indicate non-homogeneous matrix or sampling.

24.6 Acceptance Criteria (Control Limits)

As mandated by the test method and regulation, each individual analyte in the LCS, MS, or Surrogate Spike is evaluated against the control limits published in the test method. Where there are no established acceptance criteria, the laboratory calculates in-house control limits with the use of control charts or, in some cases, utilizes client project specific control limits. When this occurs, the regulatory or project limits will supersede the laboratory's in-house limits.

Note: For methods, analytes and matrices with very limited data (e.g., unusual matrices not analyzed often), interim limits are established using available data or by analogy to similar methods or matrices.

Once control limits have been established, they are verified, reviewed, and updated if necessary on an annual basis unless the method requires more frequent updating. Control limits are established per method (as opposed to per instrument) regardless of the number of instruments utilized.

Laboratory generated % Recovery acceptance (control) limits are generally established by taking $\pm$ 3 Standard Deviations (99% confidence level) from the average recovery of a minimum of 20-30 data points (more points are preferred).

- Regardless of the calculated limit, the limit should be no tighter than the Calibration Verification (ICV/CCV). (Unless the analytical method specifies a tighter limit).
- In-house limits cannot be any wider than those mandated in a regulated analytical method. Client or contract required control limits are evaluated against the laboratory's statistically derived control limits to determine if the data quality objectives (DQOs) can be achieved. If laboratory control limits are not consistent with DQOs, then alternatives must be considered, such as method improvements or use of an alternate analytical method.
- The lowest acceptable recovery limit will be 10% (the analyte must be detectable and identifiable). Exception: The lowest acceptable recovery limit for Benzidine will be 5% and the analyte must be detectable and identifiable.
- The maximum acceptable recovery limit will be 150%.
- The maximum acceptable RPD limit will be 35% for waters and 40% for soils. The minimum RPD limit is 10%.
- If either the high or low end of the control limit changes by $\leq$ 5% from previous, the control chart is visually inspected and, using professional judgment, they may be left unchanged if there is no affect on laboratory ability to meet the existing limits.

24.6.1 The lab must be able to generate a current listing of their control limits and track when the updates are performed. In addition, the laboratory must be able to recreate historical control limits, SOP No BR-QA-013, Control Chart.

The QA department or department supervisor generates a Quality Control Limit Summary that contains tables that summarize the precision and accuracy acceptability limits for analyses performed at Eurofins TestAmerica Burlington. This summary includes an effective date, is updated each time new limits are generated and is located in LIMS. Unless otherwise noted, limits within these tables are laboratory generated. The analysts are instructed to use the current limits in the laboratory (dated and approved by the Technical Manager and QA Manager) and entered into LIMS. The Quality Assurance department maintains an archive of all limits used within the laboratory.

24.6.2 A LCS that is within the acceptance criteria establishes that the analytical system is in control and is used to validate the process. Samples that are analyzed with an LCS with recoveries outside of the acceptance limits may be determined as out of control and should be reanalyzed if possible. If reanalysis is not possible, then the results for all affected analytes for samples within the same batch must be qualified when reported. The internal corrective action process (see Section 12) is also initiated if an LCS exceeds the acceptance limits. Sample results may be qualified and reported without reanalysis if:

- The analyte results are below the reporting limit and the LCS is above the upper control limit.
- If the analytical results are above the relevant regulatory limit and the LCS is below the lower control limit.

For TNI and DoD/DOE work, there are an allowable number of Marginal Exceedances (ME):

<11 analytes	0 marginal exceedances are allowed.
11 – 30 Analytes	1 marginal exceedance is allowed
31-50 Analytes	2 marginal exceedances are allowed

51-70 Analytes	3 marginal exceedances are allowed
71-90 Analytes	4 marginal exceedances are allowed
> 90 Analytes	5 marginal exceedances are allowed

- Marginal exceedances are recovery exceedances between 3 SD and 4 SD from the mean recovery limit (TNI).
- Marginal exceedances must be random. If the same analyte exceeds the LCS control limit repeatedly, it is an indication of a systematic problem. The source of the error must be located and corrective action taken. The laboratory has a system to monitor marginal exceedances to ensure that they are random.

Though marginal exceedences may be allowed, the data must still be qualified to indicate it is outside of the normal limits.

24.6.3 If the MS/MSDs do not meet acceptance limits, the MS/MSD and the associated spiked sample is reported with a qualifier for those analytes that do not meet limits. If obvious preparation errors are suspected, or if requested by the client, unacceptable MS/MSDs are reprocessed and reanalyzed to prove matrix interference. A more detailed discussion of acceptance criteria and corrective action can be found in the lab's method SOPs and in Section 12.

24.6.4 If a surrogate standard falls outside the acceptance limits, and if there is not obvious chromatographic matrix interference, reanalyze the sample to confirm a possible matrix effect. If the recoveries confirm or there was obvious chromatographic interference, results are reported from the original analysis and a qualifier is added. If the reanalysis meets surrogate recovery criteria, the second run is reported (or both are reported if requested by the client). Under certain circumstances, where all of the samples are from the same location and share similar chromatography, the reanalysis may be performed on a single sample rather than all of the samples and if the surrogate meets the recovery criteria in the reanalysis, all of the affected samples would require reanalysis.

24.7 Additional Procedures to Assure Quality Control

The laboratory has written and approved method SOPs to assure the accuracy of the test method including calibration (see Section 20), use of certified reference materials (see Section 21) and use of PT samples (see Section 17).

A discussion regarding MDLs, Limit of Detection (LOD) and Limit of Quantitation (LOQ) can be found in Section 19.

- Use of formulae to reduce data is discussed in the method SOPs and in Section 20.
- Selection of appropriate reagents and standards is included in Section 11 and 21.
- A discussion on selectivity of the test is included in Section 7.
- Constant and consistent test conditions are discussed in Section 6.
- The laboratories sample acceptance policy is included in Section 23.

25.0 REPORTING RESULTS

25.1 Overview

The results of each test are reported accurately, clearly, unambiguously, and objectively in accordance with State and Federal regulations as well as client requirements. Analytical results are issued in a format that is intended to satisfy customer and laboratory accreditation requirements as well as provide the end user with the information needed to properly evaluate the results. Where there is conflict between client requests and laboratory ethics or regulatory requirements, the laboratory's ethical and legal requirements are paramount, and the laboratory will work with the client during project set up to develop an acceptable solution. Refer to Section 9.

A variety of report formats are available to meet specific needs.

In cases where a client asks for simplified reports, there must be a written request from the client. There still must be enough information that would show any analyses that were out of conformance (QC out of limits) and there should be a reference to a full report that is made available to the client. Review of reported data is included in Section 19.

25.2 Test Reports

Analytical results are reported in a format that is satisfactory to the client and meets all requirements of applicable accrediting authorities and agencies. A variety of report formats are available to meet specific needs. The report is printed on laboratory letterhead, reviewed, and signed by the appropriate project manager. At a minimum, the standard laboratory report shall contain the following information:

25.2.1 A report title (e.g., Analytical Report)

25.2.2 The cover page shall include the laboratory name, address and telephone number.

25.2.3 A unique identification of the report (e.g., Eurofins TestAmerica Burlington Job ID #) and on each page an identification in order to ensure the page is recognized as part of the report and a clear identification of the end.

Note: Page numbers of report are represented as page # of ##. Where the first number is the page number and the second is the total number of pages.

25.2.4 A copy of the chain of custody (COC).

- Any COCs involved with Subcontracting are included.

25.2.5 The name and address of client and a project name/number, if applicable.

25.2.6 Client project manager or other contact

25.2.7 Description and unambiguous identification of the tested sample(s) including the client identification code.

25.2.8 Date of receipt of sample, date and time of collection, and date(s) of test preparation and performance, and time of preparation or analysis if the required holding time for either activity is less than or equal to 72 hours.

25.2.9 Date reported or date of revision, if applicable.

25.2.10 Method of analysis including method code (EPA, Standard Methods, etc.).

25.2.11 Practical quantitation limits or reporting limit.

25.2.12 Method detection limits (if requested)

25.2.13 Definition of Data qualifiers and reporting acronyms (e.g. ND).

25.2.14 Sample results.

25.2.15 QC data consisting of method blank, surrogate, LCS, and MS/MSD recoveries and control limits.

25.2.16 Condition of samples at receipt including temperature. This may be accomplished in a narrative or by attaching sample login sheets (Refer to Sec. 25.2.4 – Item 3 regarding additional addenda).

25.2.17 A statement expressing the validity of the results, that the source methodology was followed and all results were reviewed for error.

25.2.18 A statement to the effect that the results relate only to the items tested and the sample as received by the laboratory, except when information is provided by the client. When data is provided by the client there shall be a clear identification of it, and a disclaimer shall be put in the report when the client supplied data can affect the validity of the test.

25.2.19 A statement that the report shall not be reproduced except in full, without prior express written approval by the laboratory coordinator.

25.2.20 A signature and title of the person(s) accepting responsibility for the content of the report and date of issue. Authorized signatories are qualified Project Managers appointed by the Manager of Project Managers.

25.2.21 When TNI accreditation is required, the lab shall certify that the test results meet all requirements of TNI or provide reasons and/or justification if they do not.

25.2.22 The laboratory includes a cover letter.

25.2.23 Where applicable, a narrative to the report that explains the issue(s) and corrective action(s) taken in the event that a specific accreditation or certification requirement was not met.

25.2.24 When soil samples are analyzed, a specific identification as to whether soils are reported on a “wet weight” or “dry weight” basis.

25.2.25 Appropriate laboratory certification number for the state of origin of the sample, if applicable.

25.2.26 If only part of the report is provided to the client (client requests some results before all of it is complete), it must be clearly indicated on the report (e.g., partial report, or how your lab identifies it). A complete report must be sent once all of the work has been completed.

25.2.27 Any non-Euronfins TestAmerica Burlington subcontracted analysis results are provided as a separate report on the official letterhead of the subcontractor. All Euronfins TestAmerica Burlington subcontracting is clearly identified on the report as to which laboratory performed a specific analysis.

25.2.28 A Certification Summary Report, where required, will document that, unless otherwise noted, all analytes tested and reported by the laboratory were covered by the noted certifications.

Note: Refer to the NDSC Document No. CA-I-P-002, Electronic Reporting and Signature Policy, for details on internally applying electronic signatures of approval.

25.2.29 Reports for Ohio VAP work require a VAP affidavit be completed and included with the report.

25.2.30 Where the laboratory is responsible for the sampling stage, in addition to the requirements listed above, reports containing the results of sampling shall include the following, where necessary for the interpretation of test results:

- the date of sampling;
- unambiguous identification of the material sampled;
- the location of sampling plan and procedures, and deviations, addition to or exclusions from the sample procedures;
- a reference to the sampling plan and procedure, and deviations, additions to or exclusions from the sample procedures;
- details of any environmental conditions during sampling that affect the interpretation of test results;
- information required to evaluate measurement uncertainty for subsequent testing

25.3 Reporting Level or Report Type

The laboratory offers four levels of quality control reporting. Each level, in addition to its own specific requirements, contains all the information provided in the preceding level. The packages provide the following information in addition to the information described above:

- Level 1 is a report with all of the elements outlined in Section 25.2 above, excluding 25.2.15 (QC data).
- Level II is a Level I report plus summary information, including results for the method blank reported to the laboratory MDL, percent recovery for laboratory control samples and matrix spike samples, and the RPD values for all MSD and sample duplicate analyses.
- Level III contains all the information supplied in Level II, but presented on the CLP-like summary forms, and relevant calibration information. A Level II report is not included, unless specifically requested. No raw data is provided.
- Level IV is the same as Level III with the addition of all raw supporting data.

In addition to the various levels of QC packaging, the laboratory also provides reports in EDD (see below). Initial reports may be provided to clients by facsimile. Procedures used to ensure client confidentiality are outlined in Section 25.6.

25.4 Electronic Data Deliverables (EDDs)

EDDs are routinely offered as part of Eurofins TestAmerica Burlington services in addition to the test report as described in Section 25.2. When NELAP accreditation is required and both a test report and EDD are provided to the client, the official version of the test report will be the combined information of the report and the EDD. Eurofins TestAmerica Burlington offers a variety of EDD formats including Environmental Restoration Information Management System (ERPIMS), Staged Electronic Data Deliverable (SEDD) Environmental Quality Information System (EQuIS), Electronic Deliverable Format (EDF), Excel and custom files.

EDD specifications are submitted to the IT department by the PM for review and undergo the contract review process. Once the facility has committed to providing data in a specific electronic format, the coding of the format may need to be performed. This coding is documented and validated. The validation of the code is retained by the IT staff coding the EDD.

EDDs shall be subject to a review to ensure their accuracy and completeness. If EDD generation is automated, review may be reduced to periodic screening if the laboratory can demonstrate that it can routinely generate that EDD without errors. Any revisions to the EDD format must be reviewed until it is demonstrated that it can routinely be generated without errors. If the EDD can be reproduced accurately and if all subsequent EDDs can be produced error-free, each EDD does not necessarily require a review.

25.5 Supplemental Information for Test

The lab identifies any unacceptable QC analyses or any other unusual circumstances or observations such as environmental conditions and any non-standard conditions that may have affected the quality of a result. This is typically in the form of a footnote or a qualifier and/or a narrative explaining the discrepancy in the front of the report.

Numeric results with values outside of the calibration range, either high or low are qualified as estimated.

Where quality system requirements are not met, a statement of compliance/non-compliance with requirements and/or specifications is required, including identification of test results derived from any sample that did not meet TNI sample acceptance requirements such as improper container, holding time, or temperature.

Where applicable, a statement on the estimated uncertainty of measurements; information on uncertainty is needed when a client's instructions so require.

When, as requested by the client and agreed to by Eurofins TestAmerica Burlington the report includes a statement of conformity to specification or standard (see Special Services, Section 7.4), the report shall clearly identify:

- to which results the statement applies,
- which specifications, standard or parts thereof are met or not, and
- the decision rule that was applied (unless the decision rule is inherent in the requested specification or standard, taking into account the level of risk (such as false accept and false reject and statistical assumptions) associated with the decision rule.

Opinions and Interpretations - The test report contains objective information, and generally does not contain subjective information such as opinions and interpretations. If such information is required by the client, the Laboratory Director will determine if a response can be prepared. If so, the Laboratory Director will designate the appropriate member of the management team to prepare a response. The response will be fully documented, and reviewed by the Laboratory Director, before release to the client. There may be additional fees charged to the client at this time, as this is a non-routine function of the laboratory.

Note: Review of data deliverable packages for submittal to regulatory authorities requires responses to non-conforming data concerning potential impact on data quality. This necessitates a limited scope of interpretation, and this work is performed by the QA Department. This is the only form of "interpretation" of data that is routinely performed by the laboratory.

When opinions or interpretations are included in the report, the laboratory provides an explanation as to the basis upon which the opinions and interpretations have been made. Opinions and interpretations are clearly noted as such and where applicable, a comment should be added suggesting that the client verify the opinion or interpretation with their regulator.

25.6 Environmental Testing Obtained From Subcontractors

If the laboratory is not able to provide the client the requested analysis, the samples would be subcontracted following the procedures outlined in the NDSC Document No. CW-L-S-004, Subcontracting,

Data reported from analyses performed by a subcontractor laboratory are clearly identified as such on the analytical report provided to the client. Results from a subcontract laboratory outside of Eurofins TestAmerica Burlington are reported to the client on the subcontract laboratory's original report stationary and the report includes any accompanying documentation.

25.7 Client Confidentiality

The laboratory will ensure the highest standards of quality and integrity of the data and services provided to our clients

The laboratory is responsible for maintaining in confidence all client information obtained or created. In situations involving the transmission of environmental test results by telephone, facsimile or other electronic means, client confidentiality must be maintained.

The laboratory will not intentionally divulge to any person (other than the client or any other person designated by the client in writing) any information regarding the services provided by the laboratory or any information disclosed to the laboratory by the client. Furthermore, information known to be potentially endangering to national security or an entity's proprietary rights will not be released.

Information about the client obtained from sources other than the client (e.g., complainant, regulators) shall be confidential between client and the laboratory. The source of this information shall be confidential to the laboratory and shall not be shared with the client, unless agreed by the source.

Note: This shall not apply to the extent that the information is required to be disclosed by the laboratory under the compulsion of legal process. The laboratory will, to the extent feasible, provide reasonable notice to the client before disclosing the information.

Note: Authorized representatives of an accrediting authority are permitted to make copies of any analyses or records relevant to the accreditation process, and copies may be removed from the laboratory for purposes of assessment.

25.7.1 Report deliverable formats are discussed with each new client. If a client requests that reports be e-mailed, the reports are to meet all requirements of this document.

25.8 Format of Reports

The format of reports is designed to accommodate each type of environmental test carried out and to minimize the possibility of misunderstanding or misuse.

25.9 Amendments to Test Reports

Corrections, additions, or deletions to reports are only made when justification arises through supplemental documentation. Justification is documented using the laboratory's corrective action system (refer to Section 12).

The revised report is retained on the Archive data server, as is the original report. The revised report is stored in the Archive data server under the sample number followed by "R". The

revised report will have the word “revised” or “amended” next to the date rather than the word “reported”.

When the report is re-issued, a notation of “report re-issue“ is placed on the cover/signature page of the report *or at the top of the narrative page* with a brief explanation of reason for the re-issue and a reference back to the last final report generated. *For Example*: *Report was revised on 3/8/20 to include toluene in sample NQA1504 per client’s request. This final report replaces the final report generated on 1/27/20 at 10:47am.*

25.10 Policies on Client Requests for Amendments

25.10.1 Policy on Data Omissions or Reporting Limit Increases

Fundamentally, our policy is simply to not omit previously reported results (including data qualifiers) or to not raise reporting limits and report sample results as ND. This policy has few exceptions. Exceptions are:

- Laboratory error.
- Sample identification is indeterminate (confusion between COC and sample labels).
- An incorrect analysis (not analyte) was requested (e.g., COC lists 8315 but client wanted 8310). A written request for the change is required.
- Incorrect limits reported based on regulatory requirements.
- The requested change has absolutely ***no possible*** impact on the interpretation of the analytical results and there is ***no possibility*** of the change being interpreted as misrepresentation by anyone inside or outside of our company.

25.10.2 Multiple Reports

The laboratory does not issue multiple reports for the same work order where there is different information on each report (this does not refer to copies of the same report) unless required to meet regulatory needs and approved by QA.

Appendix 1. List of Governing Documents applicable to the QA Manual

(NDSC, KDG and Laboratory SOPs and Policies)

NDSC Doc. No.	Title
CA-C-S-001	Work Sharing Process
CA-I-P-002	Electronic Reporting and Signature Policy
CA-L-P-002	Contract Compliance Policy
CA-Q-M-002	Quality Management Plan
CA-Q-QM-001	Guidance on Tentatively Identified Compounds (TICs) – GC/MS Analysis
CA-Q-S-001	Acid and Solvent-Lot Testing and Approval Program
CA-Q-S-002	Manual Integrations
CA-Q-S-006	Detection and Quantitation Limits
CA-Q-S-009	Root Cause Analysis
CA-T-P-001	Qualified Products List
CW-E-M-001	Environmental Health & Safety Manual
CW-F-P-002	Company-Wide Authorization Matrix
CW-F-P-004	Procurement and Contracts Policy
CW-F-S-007	Fixed Asset Acquisition, Retention and Safeguarding
CW-I-M-001	IT Change Control Procedure Manual
CW-L-P-001	Records Retention Policy
CW-L-P-004	Ethics Policy
CW-L-S-002	Internal Investigation
CW-L-S-004	Subcontracting
CW-Q-S-001	Document Control and Archiving
CW-Q-S-002	Writing a Standard Operating Procedure (SOPs)
CW-Q-S-003	Internal Auditing
CW-Q-S-004	Management Systems Review
CW-Q-S-005	Data Recall Process
CW-Q-S-001	Document Control and Archiving
Local Laboratory Document No.	**Title**
BR-QA-003	Document Control
BR-QA-011	Lab Training & DOC
BR-QA-012	Support Equipment Checks
BR-QA-013	Control Chart
BR-QA-016	Preventive and Corrective Action
BR-QA-020	Subsampling and Homogenization
BR-QA-021	IT
BR-SM-001	Sample Receipt / Login

Appendix 2. List of Laboratory Certifications, Accreditations, Validations

The laboratory maintains accreditations, certifications, and approvals with numerous state and national entities. Programs vary but may include on-site audits, reciprocal agreements with another entity, performance testing evaluations, review of the QA Manual, Standard Operating Procedures, Method Detection Limits, training records, etc. At the time of this QA Manual revision, the laboratory has accreditation/ certification/licensing with the following organizations:

Laboratory	Program	Authority	Lab ID
Burlington	DoD ELAP	ANAB	L2336
Burlington	NELAP	Florida	E87467
Burlington	NELAP	Minnesota	050-999-436
Burlington	NELAP	New Hampshire	2006
Burlington	NELAP	New Jersey	VT972
Burlington	NELAP	New York	10391
Burlington	NELAP	Pennsylvania	68-00489
Burlington	NELAP	Virginia	460209
Burlington	State Program	Connecticut	PH-0751
Burlington	State Program	DE Haz. Subst. Cleanup Act (HSCA)	NA
Burlington	State Program	Rhode Island	LAO00298
Burlington	State Program	Vermont	VT-4000
Burlington	State Program	Wisconsin	399133350

The certificates and accredited parameter lists are available for each State/Program at www.testamericainc.com under Analytical Services Search – Certifications.

Appendix 3. References used to prepare the QA Manual

The QAM has been prepared to be consistent with the requirements of the following documents:

- *ANSI/ASQC, E4-1994, "Specifications and Guidelines for Quality Management Systems for Environmental Data Collection and Environmental Technology Programs" (American National Standard, January 5, 1995, or most recent version)*
- *"EPA Requirements for Quality Management Programs" (QA/R-2) (EPA/240/B-01/002, May 31, 2006).*
- *EPA 600/4-88/039, Methods for the Determination of Organic Compounds in Drinking Water, EPA, Revised July 1991.*
- *EPA 600/R-95/131, Methods for the Determination of Organic Compounds in Drinking Water, Supplement III, EPA, August 1995.*
- *EPA 600/4-79-019, Handbook for Analytical Quality Control in Water and Wastewater Laboratories, EPA, March 1979.*
- *Test Methods for Evaluating Solid Waste Physical/Chemical Methods (SW846), Third Edition, September 1986, Final Update I, July 1992, Final Update IIA, August 1993, Final Update II, September 1994; Final Update IIB, January 1995; Final Update III, December 1996; Final Update IV, January 2008; Final Update V, August 2015.*
- *U.S. Department of Defense (DoD)/Department of Energy (DOE) Consolidated Quality Systems Manual (QSM) for Environmental Laboratories, Version 5.1.1, dated 2017*
- *Federal Register, 40 CFR Parts 136, 141, 172, 173, 178, 179 and 261.*
- *Manual for the Certification of Laboratories Analyzing Drinking Water (EPA 815-R-05-004, January 2005) (DW labs only)*
- *Statement of Work for Inorganics & Organics Analysis, SOM and ISM, current versions, USEPA Contract Laboratory Program Multi-media, Multi-concentration.*
- *APHA, Standard Methods for the Examination of Water and Wastewater, 18th Edition, 19th, 20th, 21st, 22nd and on-line Editions.*
- *U.S. Department of Energy Order 414.1B, Quality Assurance, Approved April 29, 2004.*
- *U.S. Department of Energy Order 414.1C, Quality Assurance, June 17, 2005.*
- *U.S. Department of Energy Order 414.1D, Quality Assurance, April, 25, 2011.*
- *Nuclear Regulatory Commission (NRC) Quality Assurance Requirements.*
- *Marine Protection, Research, and Sanctuaries Act (MPRSA).*
- *Toxic Substances Control Act (TSCA).*

Appendix 4. QA Manual Crosswalk with TNI and ISO/IEC 17025 Standards

Sec. No.	Title	2009 and 2016 TNI Standard Reference	ISO/IEC 17025:2005(E) Reference	ISO/IEC 17025:2017(E) Reference
-	QUALITY ASSURANCE MANUAL COVER PAGE	V1M2 Sec. 4.2.8.3		
1.0	Title Page			
2.0	Table of Contents	V1M2 Secs. 4.2.8.3-4.2.8.4		8.1.2, 8.2.1
3.0	Introduction, Scope and Applicability	V1M2 Sec. 4.2.8.4		
3.1	Introduction and Compliance References	V1M2 Secs. 1.1; 1.2; 2.0; 3.2; 4.1.2; 4.2.4	4.1.2; 4.2.4	5.3; 5.4; 8.2.4; 8.3.1
3.2	Terms and Definitions	V1M2 Secs. 3.0; 4.2.4	4.2.4	
3.3	Scope /Field of Testing	V1M2 Secs. 1.2; 4.2.4	4.1.2; 4.2.4	5.3; 5.4; 8.2.1; 8.2.4
3.4	Management of The Manual	V1M2 Secs. 4.2.1; 4.2.7; 4.3.3.2; 4.3.3.3	4.2.1; 4.2.7; 4.3.3.2; 4.3.3.3	5.3
4.0	Management and Responsibilities	V1M2 Sec. 4		8.2.4; 8.2.5
4.1	Overview	V1M2 Secs. 4.1.1, 4.1.3; 4.1.5	4.1.1; 4.1.3; 4.1.5; 4.2.6	5.1; 5.2; 5.5; 5.6 ; 6.2.1; 6.2.4
4.2	Roles and Responsibilities	V1M2 Secs. 4.1.4; 4.1.5; 4.1.6; 4.2.1; 4.2.6; 5.2.4	4.1.3; 4.1.5; 4.1.6; 4.2.1; 4.2.6; 5.2.4	4.1.1 to 4.1.3; 4.1.5; 5.5; 5.6; 6.2.1; 6.2.4; 6.2.6 8.2.2;
4.3	Business Continuity and Contingency Plan *(Prev. Deputies)*	V1M2 Secs. 4.1.5; 4.1.7.2; 4.2.7	4.1.5; 4.2.7	
5.0	PERSONNEL	V1M2 Secs. 5.2; 5.2.1	5.2.1	6.1; 6.2.3
5.1	Overview	V1M2 Secs. 5.2.2; 5.2.3; 5.2.5	5.2.2; 5.2.3; 5.2.5	6.2.2
5.2	Education and Experience Requirements for Technical Staff	V1M2 Secs. 5.2.1; 5.2.3; 5.2.4	5.2.1; 5.2.3; 5.2.4	6.2.2 to 6.2.4
5.3	Training	V1M2 Sec. 5.2.5	5.2.5	4.2.1; 6.2.2; 6.2.4; 6.2.5
5.4	Data Integrity and Ethics Training Program	V1M2 Sec. 4.2.8.1; 5.2.7		4.1.1
6.0	ACCOMMODATIONS AND ENVIRONMENTAL CONDITIONS	V1M2 Sec. 5.3		6.1; 6.3.1
6.1	Overview	V1M2 Secs. 5.3.1; 5.3.3; 5.3.4; 5.3.5	5.3.1; 5.3.3; 5.3.4; 5.3.5	6.3.1
6.2	Environment	V1M2 Secs. 5.3.1; 5.3.2; 5.3.3; 5.3.4; 5.3.5	5.3.1; 5.3.2; 5.3.3; 5.3.4; 5.3.5	6.3.1 to 6.3.5
6.3	Work Area	V1M2 Secs. 5.3.3; 5.3.4; 5.3.5	5.3.3; 5.3.4; 5.3.5	6.3.1
6.4	Responding to Emergencies			
6.5	Building Security	V1M2 Sec. 5.3.4	5.3.4	6.3.4
7.0	QUALITY SYSTEM			6.1; 8.2.4
7.1	Quality Policy	V1M2 Secs. 4.1.5; 4.2.2; 4.2.3; 4.2.8.3	4.1.5; 4.2.2; 4.2.3	8.2.3; 8.6.1
7.2	Ethics and Data Integrity	V1M2 Secs. 4.1.5; 4.16; 4.2.2; 4.2.8.1; 5.2.7	4.1.5; 4.2.2	4.1.1 to 4.1.3; 4.2.1; 6.2.1; 8.2.2; 8.2.3
7.3	Quality System Documentation	V1M2 Secs. 4.1.5; 4.2.2; 4.2.5	4.2.2; 4.2.5	8.2.4

Sec. No.	Title	2009 and 2016 TNI Standard Reference	ISO/IEC 17025:2005(E) Reference	ISO/IEC 17025:2017(E) Reference
7.4	QA/QC Objectives for the Measurement of Data	V1M2 Sec. 4.2.2	4.1.5; 4.2.2	6.2.4
7.5	Criteria for Quality Indicators			
7.6	Statistical Quality Control			
7.7	Quality System Metrics			
8.0	Document Control	V1M2 Secs. 4.2.7; 4.3.1; 4.3.2.2 ; 4.3.3.3; 4.3.3.4	4.2.7; 4.3.1; 4.3.2.2; 4.3.3.3; 4.3.3.4	8.2.4; 8.3.1
8.1	Overview			8.2.5; 8.3.1; 8.3.2
8.2	Document Approval and Issue	V1M2 Secs. 4.3.2; 4.3.2.1-4.3.2.3; 4.3.3.1	4.3.2.1; 4.3.2.2; 4.3.2.3; 4.3.3.1	8.2.5; 8.3.2
8.3	Procedures for Document Control Policy	V1M2 Secs. 4.3.2.1–4.3.2.2; 4.3.3.1	4.3.2.1; 4.3.2.2; 4.3.3.1	8.2.5; 8.3.2
8.4	Obsolete Documents	V1M2 Secs. 4.3.2.1–4.3.2.2	4.3.2.1; 4.3.2.2	8.2.5; 8.3.2
9.0	SERVICE TO THE CLIENT	V1M2 Secs. 4.4.1 - 4.4.4	4.4.1; 4.4.2; 4.4.3; 4.4.4	7.1.1; 7.1.1.4; 7.1.1.5; 7.1.1.8; 7.1.2.1
9.1	Overview	V1M2 Secs. 4.4.5; 4.5.5; 5.7.1	4.4.5; 5.7.1	
9.2	Review Sequence and Key Personnel	V1M2 Sec. 4.4.5	4.4.5	7.1.1.6
9.3	Balancing Laboratory Work Load and Capacity			
9.4	Documentation	V1M2 Sec. 5.7.1	5.7.1	
9.5	Special Services	V1M2 Secs. 4.7.1-4.7.2	4.7.1; 4.7.2	7.1.1.3; 7.1.1.7
9.6	Client Communication	V1M2 Secs. 4.7.1-4.7.2	4.7.1; 4.7.2	7.1.1.7
9.7	Reporting	V1M2 Secs. 4.7.1-4.7.2	4.7.1; 4.7.2	7.1.1.7
9.8	Client Feedback and Surveys	V1M2 Secs. 4.7.1-4.7.2	4.7.1; 4.7.2	7.1.1.7; 8.6.2
10.0	SUBCONTRACTING OF TESTS	V1M2 Secs. 4.4.3; 4.5.4	4.4.3; 4.5.4	
10.1	Overview	V1M2 Secs. 4.5.1 - 4.5.3; 4.5.5; 5.3.1	4.5.1; 4.5.2; 4.5.3; 5.3.1	6.6.1; 7.1.2.1; 7.1.2.2
10.2	Qualifying and Monitoring Subcontractors	V1M2 Secs. 4.5.1; 4.5.2; 4.5.3; 4.5.5	4.5.1; 4.5.2; 4.5.3	6.6.1; 7.1.2.1; 7.1.2.2
10.3	Oversight and Reporting	V1M2 Sec. 4.5.5		
10.4	Contingency Planning			
10.5	Use of NELAP and A2LA Logo			
11.0	PURCHASING SERVICES AND SUPPLIES	V1M2 Sec. 4.6.1	4.6.1	
11.1	Overview	V1M2 Secs. 4.6.2; 4.6.3; 4.6.4	4.6.2; 4.6.3; 4.6.4	6.6.1; 6.6.2
11.2	Glassware	V1M2 Sec. 5.5.13.1		
11.3	Reagents, Standards & Supplies	V1M2 Secs. 4.6.2; 4.6.3; 4.6.4 2016 V1M4 1.7.2.5	4.6.2; 4.6.3; 4.6.4	6.6.1 to 6.6.3
11.4	Purchase of Equipment / Instruments / Software			
11.5	Services			
11.6	Suppliers			
12.0	COMPLAINTS	V1M2 Sec. 4.8	4.8	8.6.1; 8.6.2
12.1	Overview			7.9.1 to 7.9.3 8.6.1; 8.6.2;
12.2	External Complaints			7.9.2 to 7.9.7 8.6.1; 8.6.2;
12.3	Internal Complaints			8.6.1; 8.6.2

Sec. No.	Title	2009 and 2016 TNI Standard Reference	ISO/IEC 17025:2005(E) Reference	ISO/IEC 17025:2017(E) Reference
12.4	Management Review			8.6.1; 8.6.2
13.0	CONTROL OF NON-CONFORMING WORK	V1M2 Secs. 4.9.1; 5.10.5	4.9.1; 5.10.5	7.10.1
13.1	Overview	V1M2 Secs. 4.9.1; 4.11.3; 4.11.5	4.9.1; 4.11.3; 4.11.5	7.10.1
13.2	Responsibilities and Authorities	V1M2 Secs. 4.9.1; 4.11.3; 4.11.5; 5.2.7	4.9.1; 4.11.3; 4.11.5	7.10.1
13.3	Evaluation of Significance and Action Taken	V1M2 Secs. 4.9.1; 4.11.3; 4.11.5	4.9.1; 4.11.3; 4.11.5	4.1.5; 7.10.1; 7.10.2; 8.5.3
13.4	Prevention of Non-Conforming Work	V1M2 Secs. 4.9.4; 4.11.2	4.9.2; 4.11.2	7.10.2; 7.10.3; 8.5.3
13.5	Method Suspension / Restriction (Stop Work Procedures)	V1M2 Secs. 4.9.1; 4.9.2; 4.11.5	4.9.1; 4.9.2; 4.11.5	7.10.1; 7.10.2
14.0	CORRECTIVE ACTION	V1M2 Sec. 4.11		4.1.4; 4.1.5
14.1	Overview	V1M2 Secs. 4.9.2; 4.11.1; 4.11.2	4.9.2; 4.11.1; 4.11.2; 8.7.1; 8.7.3	7.10.2 8.7.1; 8.7.3;
14.2	General	V1M2 Sec. 4.11.2; 4.11.3	4.11.2; 4.11.3	7.7.2; 8.5.3; 8.7.1;
14.3	Closed Loop Correction Action Process	V1M2 Sec. 4.11.2; 4.11.3; 4.11.4; 4.11.6; 4.11.7; 4.12.2	4.11.2; 4.11.3; 4.11.4; 4.12.2	8.5.3; 8.6.1; 8.7.2
14.4	Technical Corrective Actions	V1M2 Sec. 4.11.6		8.7.1
14.5	Basic Corrections	V1M2 Secs. 4.11.1; 4.13.2.3	4.11.1; 4.13.2.3	7.5.2; 8.7.1
15.0	PREVENTIVE ACTION / IMPROVEMENT	V1M2 Secs. 4.10; 4.12.1; 4.12.2	4.10; 4.12.1; 4.12.2	4.1.4
15.1	Overview	V1M2 Secs. 4.15.1; 4.15.2	4.15.1; 4.15.2	8.6.2
15.2	Management of Change			
16.0	CONTROL OF RECORDS	V1M2 Secs. 4.2.7; 4.13.1.1; 4.13.3	4.2.7; 4.13.1.1	8.4.2
16.1	Overview	V1M2 Secs. 4.13.1.1 - 4.13.1.4; 4.13.2.1-4.13.2.3; 4.13.3	4.13.1.1 - 4.13.1.4; 4.13.2.1-4.13.2.3	8.4.1; 8.4.2
16.2	Programs with Longer Retention Requirements			
16.3	Technical and Analytical Records	V1M2 Sec. 4.13.2.2 - 4.13.2.3	4.13.2.2; 4.13.2.3	7.5.1; 8.4.2
16.4	Laboratory Support Activities			7.5.2; 8.4.2
16.5	Administrative Records			8.4.2
16.6	Records Management, Storage and Disposal	V1M2 Sec. 4.13.3		4.2.1; 8.4.2
17.0	AUDITS			
17.1	Internal Audits	V1M2 Sec. 4.2.8.1; 4.14; 4.14.1; 4.14.2 ; 4.14.3; 4.14.5; 5.9.1; 5.9.2	4.14.1; 4.14.2; 4.14.3; 5.9.1; 5.9.2	8.6.1; 8.8.1; 8.8.2
17.2	External Audits	V1M2 Secs.4.14.2; 4.14.3	4.14.2; 4.14.3; 4.14.4	4.2.1; 8.6.1
17.3	Audit Findings	V1M2 Secs. 4.14.2; 4.14.3; 4.14.5		8.6.1
18.0	MANAGEMENT REVIEWS	V1M2 Sec. 4.1.6; 4.15; 4.15.1; 4.15.2	4.1.6; 4.15.1; 4.15.2	4.1.4; 8.5.1; 8.6.1; 8.9.1; 8.9.2
18.1	Quality Assurance Report			8.5.1

Sec. No.	Title	2009 and 2016 TNI Standard Reference	ISO/IEC 17025:2005(E) Reference	ISO/IEC 17025:2017(E) Reference
18.2	Annual Management Review	V1M2 Sec. 4.2.2; 4.15.3	4.2.2	4.1.1, 4.1.4; 4.2.1; 7.1.1.3; 8.2.2; 8.5.1 to 8.5.3; 8.6.1; 8.9.3
18.3	Potential Integrity Related Managerial Reviews			4.1.5; 8.5.1; 8.6.1
19.0	TEST METHODS AND METHOD VALIDATION	V1M2 Sec. 5.4.1	5.4.1	7.2.1.1; 8.2.5
19.1	Overview	V1M2 Sec. 5.4.1	5.4.1; 5.4.5.1	6.2.3; 7.2.1.1 to 7.2.1.3
19.2	Standard Operating Procedures (SOPs)	V1M2 Secs. 4.2.8.5; 4.3.3.1; 5.4.2	4.3.3.1; 5.4.2	7.2.1.4
19.3	Laboratory Methods Manual	V1M2 Sec. 4.2.8.5 (2019 5.4.4.2)		6.2.3
19.4	Selection of Methods	V1M2 Secs. 4.13.3; 5.4.1; 5.4.2; 5.4.3. (2019 5.4.4) V1M4 Secs. 1.4; 1.5.1; 1.6.1; 1.6.2; 1.6.2.1; 1.6.2.2	5.4.1; 5.4.2; 5.4.3; 5.4.4; 5.4.5.1; 5.4.5.2; 5.4.5.3	7.1.1.2; 7.2; 7.2.1.2; 7.2.1.3; 7.1.2.4 to 7.2.1.7; 7.2.2.1 to 7.2.1.7; 7.2.2.1
19.5	Laboratory Developed Methods and and Non-Standard Methods	V1M2 Sec. 5.4.2. (2019 5.4.4.1, 5.4.4.2) V1M4 Sec. 1.5.1	5.4.2; 5.4.4; 5.4.5.2; 5.4.5.3	7.1.1.2; 7.2.1.4 to 7.2.1.7 7.2.2.1; 7.2.2.3; 8.2.5
19.6	Validation of Methods	V1M2 Sec. 5.4.2. (2019 5.4.5.4) V1M4 Secs. 1.5.1; 1.5.2; 1.5.2.1; 1.5.2.2; 1.5.3	5.4.2; 5.4.4; 5.4.5.2; 5.4.5.3	7.1.1.2; 7.1.2.4; 7.2.1.6; 7.2.2.1 to 7.2.2.4
19.7	Method Detection Limits (MDLs) / Limit of Detection (LOD)	V1M2 Sec. 5.9.3. V1M4 Secs. 1.5.2; 1.5.2.1; 1.5.2.2	5.4.5.3	7.2.2.3
19.8	Verification of Detection Limits	V1M2 Sec. 5.9.3		
19.9	Instrument Detection Limits (IDLs)	V1M2 Sec. 5.9.3. V1M4 Sec. 1.5.2.1		
19.10	Limit of Quantitation	V1M2 Sec. 5.9.3		
19.11	Retention Windows	V1M2 Sec. 5.9.3. V1M4 Sec. 1.5.4; (2009 1.7.3.6) (2016 1.7.2.6)		
19.12	Evaluation of Selectivity	V1M2 Sec. 5.9.3. V1M4 Sec. 1.5.4; (2009 1.7.3.6) (2016 1.7.2.6)		
19.13	Estimation of Uncertainty of Measurement	V1M2 Sec. 5.1.1; 5.1.2; 5.4.6	5.1.1; 5.1.2; 5.4.6.1; 5.4.6.2; 5.4.6.3	7.6.1; 7.6.2; 7.6.3
19.14	Sample Reanalysis Guidelines	V1M2 Sec 5.9.1	5.9.1	
19.15	Control of Data	V1M2 Secs. 5.4.7.1; 5.4.7.2; 5.9.1	5.4.7.1; 5.4.7.2; 5.9.1	7.11.1 to 7.11.6
20.0	EQUIPMENT AND CALIBRATIONS	V1M2 Secs. 5.5.4; 5.5.5; 5.5.6	5.5.4; 5.5.5; 5.5.6; 5.6.1	6.1; 6.4.3; 6.4.6; 6.4.9
20.1	Overview	V1M2 Secs. 5.5.1; 5.5.2; 5.5.3; 5.5.5; 5.5.10	5.5.1; 5.5.2; 5.5.3; 5.5.5; 5.5.10; 5.6.1	6.4.1; 6.4.4 to 6.4.6; 6.4.9; 6.4.11
20.2	Preventive Maintenance	V1M2 Secs. 5.5.1; 5.5.3; 5.5.7; 5.5.9	5.5.1; 5.5.3; 5.5.7; 5.5.9; 5.6.1	6.4.1 to 6.4.3; 6.4.6; 6.4.10

Sec. No.	Title	2009 and 2016 TNI Standard Reference	ISO/IEC 17025:2005(E) Reference	ISO/IEC 17025:2017(E) Reference
20.3	Support Equipment	V1M2 Secs. 5.5.10; 5.5.11; 5.5.13.1	5.5.10; 5.5.11; 5.6.2.1.2; 5.6.2.2.1; 5.6.2.2.2	6.4.11; 6.4.12; 6.5.1; 6.5.2; 6.5.3
20.4	Instrument Calibrations	V1M2 Secs. 5.5.8; 5.5.10; 5.6.3.1. V1M4 Sec. 1.7.1.1; (2009 1.7.2) (2016 1.7.1.2)	5.5.8; 5.5.9; 5.5.10; 5.6.1; 5.6.2; 5.6.3.1	6.4.2; 6.4.3; 6.4.6 to 6.4.8; 6.4.11; 6.4.13; 6.4.14; 6.5.1; 6.5.2
20.5	Tentatively Identified Compounds (TICs) – GC/MS Analysis			
20.6	GC/MS Tuning			
21.0	MEASUREMENT OF TRACEABILITY			
21.1	Overview	V1M2 Sec. 5.6.3.1	5.6.2.1.2; 5.6.2.2.2; 5.6.3.1	6.4.14; 6.5.1; 6.5.2; 6.5.3
21.2	NIST-Traceable Weights and Thermometers	V1M2 Secs. 5.5.13.1; 5.6.3.1; 5.6.3.2	5.6.3.1; 5.6.3.2	6.4.14
21.3	Reference Standards / Materials	V1M2 Secs. 5.6.3.1; 5.6.3.2; 5.6.3.3; 5.6.3.4; 5.6.4.1; 5.6.4.2; 5.9.1; 5.9.3	5.6.3.1; 5.6.3.2; 5.6.3.3; 5.6.3.4; 5.9.1	6.4.14
21.4	Documentation and Labeling of Standards, Reagents, and Reference Materials	V1M2 Secs. 5.6.4.2; 5.9.3		
22.0	SAMPLING			
22.1	Overview	V1M2 Secs. 5.7.1; 5.7.3	5.7.1; 5.7.3	7.3.1; 7.3.2; 7.3.3
22.2	Sampling Containers			
22.3	Definition of Holding Time			
22.4	Sampling Containers, Preservation Requirements, Holding Times			
22.5	Sample Aliquots / Subsampling	V1M2 Sec. 5.7.1	5.7.1	7.3.1; 7.3.2
23.0	HANDLING OF SAMPLES	V1M2 Sec. 5.8.1	5.8.1	7.4.1
23.1	Chain of Custody (CoC)	V1M2 Secs. 5.7.2; 5.7.4; 5.8.4; 5.8.7.5; 5.8.8; 5.9.1	5.7.2; 5.8.4; 5.9.1	7.1.1.6; 7.4.1
23.2	Sample Receipt	V1M2 Secs. 5.8.1; 5.8.2; 5.8.3; 5.8.5; 5.8.7.3; 5.8.7.4; 5.8.7.5	5.8.2; 5.8.3	7.4.3
23.3	Sample Acceptance Policy	V1M2 Secs. 5.8.6; 5.8.7.2		
23.4	Sample Storage	V1M2 Secs. 5.7.4; 5.8.4	5.8.4	7.4.1; 7.4.4
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23.6	Sample Shipping	V1M2 Sec. 5.8.2	5.8.2	7.4.2
23.7	Sample Disposal			
24.0	ASSURING THE QUALITY OF TEST RESULTS			
24.1	Overview	V1M2 Secs. 5.9.2; 5.9.3	5.9.2	7.7.2 to 7.7.3
24.2	Controls	V1M2 Secs. 5.9.2; 5.9.3	5.9.2	7.7.1; 7.7.3
24.3	Negative Controls	V1M2 Secs. 5.9.2; 5.9.3 V1M4 Secs. (2019 1.7.3; 1.7.3.1; 1.7.4.1) (2016 1.7.2, 1.7.2.1, 1.7.3.1)	5.9.2	7.7.1; 7.7.3

Sec. No.	Title	2009 and 2016 TNI Standard Reference	ISO/IEC 17025:2005(E) Reference	ISO/IEC 17025:2017(E) Reference
24.4	Positive Controls	V1M2 Secs 5.9.2; 5.9.3. V1M4 Secs.(2009 1.7.3; 1.7.3.2; 1.7.3.2.1; 1.7.3.2.2; 1.7.3.2.3) (2016 1.7.2, 1.7.2.2, 1.7.3.2)	5.9.2	7.7.1; 7.7.3
24.5	Sample Matrix Controls	V1M2 Secs. 5.9.2; 5.9.3. V1M4 Secs. (2009 1.7.3 ; 1.7.3.3; 1.7.3.3.1; 1.7.3.3.2; 1.7.3.3.3) (2016 1.7.2, 1.7.2.3, 1.7.3.3)	5.9.2	7.7.1; 7.7.3
24.6	Acceptance Criteria (Control Limits)	V1M2 Sec. 5.9.3. V1M4 Secs. 1.7.4.2; 1.7.4.3		7.7.1; 7.7.3
24.7	Additional Procedures to Assure Quality Control	V1M2 Sec. 5.9.3. V1M4 Sec. (2009 1.7.3.4), (2016 1.7.2.4)		7.7.1; 7.7.3
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25.2	Test Reports	V1M2 Secs. 5.10.1; 5.10.2; 5.10.3.1; 5.10.3.2; 5.10.5; 5.10.6; 5.10.7; 5.10.8; 5.10.10; 5.10.11	5.10.1; 5.10.2; 5.10.3.1; 5.10.3.2; 5.10.5; 5.10.6; 5.10.7; 5.10.8	7.8.2.1; 7.8.2.2; 7.8.3; 7.8.5
25.3	Reporting Level or Report Type	V1M2 Secs. 5.10.1; 5.10.7; 5.10.8	5.10.1; 5.10.7; 5.10.8	
25.4	Electronic Data Deliverables (EDDs)			
25.5	Supplemental Information for Test	V1M2 Secs. 5.10.1; 5.10.3.1; 5.10.5	5.10.1; 5.10.3.1; 5.10.5	7.1.1.3; 7.8.6.1; 7.8.6.2; 7.8.7.1 to 7.8.7.3
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25.7	Client Confidentiality	V1M2 Secs. 4.1.5; 5.10.7	4.1.5; 5.10.7	4.2.1 to 4.2.4
25.8	Format of Reports	V1M2 Sec. 5.10.8	5.10.8	
25.9	Amendments to Reports	V1M2 Sec. 5.10.9	5.10.1; 5.10.9	7.8.8.1 to 7.8.8.3
25.10	Policies on Client Requests for Amendments	V1M2 Secs. 5.9.1; 5.10.9	5.9.1; 5.10.1; 5.10.5; 5.10.9	

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Appendix 5. Terms/Glossary and Acronyms (EL-V1M2 Sec. 3.1)

Terms/Glossary:

Acceptance Criteria: Specified limits placed on characteristics of an item, process, or service defined in requirement documents. (ASQC)

Accreditation: The process by which an agency or organization evaluates and recognizes a laboratory as meeting certain predetermined qualifications or standards, thereby accrediting the laboratory.

Accuracy: The degree of agreement between an observed value and an accepted reference value. Accuracy includes a combination of random error (precision) and systematic error (bias) components which are due to sampling and analytical operations; a data quality indicator. (QAMS)

Analyst: The designated individual who performs the "hands-on" analytical methods and associated techniques and who is the one responsible for applying required laboratory practices and other pertinent quality controls to meet the required level of quality.

Analytical Uncertainty: A subset of Measurement Uncertainty that includes all laboratory activities performed as part of the analysis. (TNI)

Anomaly: A condition or event, other than a deficiency, that may affect the quality of the data, whether in the laboratory's control or not.

Assessment: The evaluation process used to measure or establish the performance, effectiveness, and conformance of an organization and/or its systems to defined criteria (to the standards and requirements of laboratory accreditation). (TNI)

Audit: A systematic and independent examination of facilities, equipment, personnel, training, procedures, record-keeping, data validation, data management, and reporting aspects of a system to determine whether QA/QC and technical activities are being conducted as planned and whether these activities will effectively achieve quality objectives. (TNI)

Batch: Environmental samples that are prepared and/or analyzed together with the same process and personnel, using the same lot(s) of reagents. A **preparation batch** is composed of one (1) to twenty (20) environmental samples of the same quality systems matrix, meeting the above mentioned criteria and with a maximum time between the start of processing of the first and last sample in the batch to be twenty-four (24) hours. An **analytical batch** is composed of prepared environmental samples (extracts, digestates or concentrates) which are analyzed together as a group. An analytical batch can include prepared samples originating from various quality system matrices and can exceed twenty (20) samples. (TNI)

Bias: The systematic or persistent distortion of a measurement process, which causes errors in one direction (i.e., the expected sample measurement is different from the sample's true value). (TNI)

Blank: A sample that has not been exposed to the analyzed sample stream in order to monitor contamination during sampling, transport, storage or analysis. The blank is subjected to the usual analytical and measurement process to establish a zero baseline or background value and is sometimes used to adjust or correct routine analytical results. (ASQC)

Calibration: A set of operations that establish, under specified conditions, the relationship between values of quantities indicated by a measuring instrument or measuring system, or values represented by a material measure or a reference material, and the corresponding values realized by standards. (TNI)

1) In calibration of support equipment the values realized by standards are established through the use of reference standards that are traceable to the International System of Units (SI).

2) In calibration according to methods, the values realized by standards are typically established through the use of Reference Materials that are either purchased by the laboratory with a certificate of analysis or purity, or prepared by the laboratory using support equipment that has been calibrated or verified to meet specifications.

Calibration Curve: The mathematical relationship between the known values, such as concentrations, of a series of calibration standards and their instrument response. (TNI)

Calibration Standard: A substance or reference material used to calibrate an instrument (QAMS)

Certified Reference Material (CRM): A reference material accompanied by a certificate, having a value, measurement uncertainty, and stated metrological traceability chain to a national metrology institute. (TNI)

Chain of Custody (COC) Form: Record that documents the possession of the samples from the time of collection to receipt in the laboratory. This record generally includes: the number and types of containers; the mode of collection; the collector; time of collection; preservation; and requested analyses. (TNI)

Compromised Samples: Those samples which are improperly sampled, insufficiently documented (chain of custody and other sample records and/or labels), improperly preserved, collected in improper containers, or exceeding holding times when delivered to a laboratory. Under normal conditions, compromised samples are not analyzed. If emergency situation require analysis, the results must be appropriately qualified.

Confidential Business Information (CBI): Information that an organization designates as having the potential of providing a competitor with inappropriate insight into its management, operation or products. TNI and its representatives agree to safeguard identified CBI and to maintain all information identified as such in full confidentiality.

Confirmation: Verification of the identity of a component through the use of an approach with a different scientific principle from the original method. These may include, but are not limited to Second Column Confirmation; Alternate wavelength; Derivatization; Mass spectral interpretation; Alternative detectors or Additional Cleanup procedures. (TNI)

Conformance: An affirmative indication or judgment that a product or service has met the requirements of the relevant specifications, contract, or regulation; also the state of meeting the requirements. (ANSI/ASQC E4-1994)

Correction: Actions necessary to correct or repair analysis specific non-conformances. The acceptance criteria for method specific QC and protocols as well as the associated corrective actions. The analyst will most frequently be the one to identify the need for this action as a result of calibration checks and QC sample analysis**.** No significant action is taken to change behavior, process or procedure.

Corrective Action: The action taken to eliminate the causes of an existing nonconformity, defect or other undesirable situation in order to prevent recurrence. (ISO 8402)

Data Integrity: The condition that exists when data are sound, correct, and complete, and accurately reflect activities and requirements. (TNI)

Data Audit: A qualitative and quantitative evaluation of the documentation and procedures associated with environmental measurements to verify that the resulting data re of acceptable quality (i.e., that they meet specified acceptance criteria).

Data Reduction: The process of transforming the number of data items by arithmetic or statistical calculations, standard curves, and concentration factors, and collation into a more useable form. (TNI)

Deficiency: An unauthorized deviation from acceptable procedures or practices, or a defect in an item (ASQC), whether in the laboratory's control or not.

Demonstration of Capability: A procedure to establish the ability of the analyst to generate analytical results of acceptable accuracy and precision. (TNI)

Document Control: The act of ensuring that documents (and revisions thereto) are proposed, reviewed for accuracy, approved for release by authorized personnel, distributed properly, and controlled to ensure use of the correct version at the location where the prescribed activity if performed. (ASQC)

Duplicate Analyses: The analyses or measurements of the variable of interest performed identically on two subsamples of the same sample. The results from duplicate analyses are used to evaluate analytical or measurement precision but not the precision of sampling, preservation or storage internal to the laboratory. (EPA-QAD)

Equipment Blank: Sample of analyte-free media which has been used to rinse common sampling equipment to check effectiveness of decontamination procedures.

External Standard Calibration: Calibrations for methods that do not utilize internal standards to compensate for changes in instrument conditions.

Field Blank: Blank prepared in the field by filing a clean container with pure de-ionized water and appropriate preservative, if any, for the specific sampling activity being undertaken (EPA OSWER)

Field of Accreditation: Those matrix, technology/method, and analyte combinations for which the accreditation body offers accreditation.

Holding Times: The maximum time that samples may be held prior to analyses and still be considered valid or not compromised. (40 CFR Part 136)

Internal Standard: A known amount of standard added to a test portion of a sample as a reference for evaluating and controlling the precision and bias of the applied analytical test method. (TNI)

Internal Standard Calibration: Calibrations for methods that utilize internal standards to compensate for changes in instrument conditions.

Instrument Blank: A clean sample (e.g., distilled water) processed through the instrumental steps of the measurement process; used to determine instrument contamination. (EPA-QAD)

Instrument Detection Limit (IDL): The minimum amount of a substance that can be measured with a specified degree of confidence that the amount is greater than zero using a specific instrument. The IDL is associated with the instrumental portion of a specific method only, and sample preparation steps are not considered in its derivation. The IDL is a statistical estimation at a specified confidence interval of the concentration at which the relative uncertainty is ± 100%. The IDL represents a range where qualitative detection occurs on a specific instrument. Quantitative results are not produced in this range.

Laboratory Control Sample (however named, such as laboratory fortified blank, spiked blank, or QC check sample): A sample matrix, free from the analytes of interest, spiked with verified known amounts of analytes or a material containing known and verified amounts of analytes, taken through all preparation and analysis steps of the procedure unless otherwise noted in a reference method. It is generally used to establish intra-laboratory or analyst specific precision and bias or to assess the performance of all or a portion of the measurement system.

An LCS shall be prepared at a minimum of 1 per batch of 20 or less samples per matrix type per sample extraction or preparation method except for analytes for which spiking solutions are not available such as total suspended solids, total dissolved solids, total volatile solids, total solids, pH, color, odor, temperature, dissolved oxygen or turbidity. The results of these samples shall be used to determine batch acceptance.

Least Squares Regression (1st Order Curve): The least squares regression is a mathematical calculation of a straight line over two axes. The y axis represents the instrument response (or Response ratio) of a standard or sample and the x axis represents the concentration. The regression calculation will generate a correlation coefficient (r) that is a measure of the "goodness of fit" of the regression line to the data. A value of 1.00 indicates a perfect fit. In order to be used for quantitative purposes, r must be greater than or equal to 0.99 for organics and 0.995 for inorganics.

Limit(s) of Detection (LOD) [a.k.a., Method Detection Limit (MDL)]: The MDL is the minimum measured quantity of a substance that can be reported with 99% confidence that the concentration is distinguishable from method blank results, consistent with 40CFR Part 136 Appendix B, August, 2017.

Limit(s) of Quantitation (LOQ) [a.k.a., Reporting Limit]: The minimum levels, concentrations, or quantities of a target variable (e.g., target analyte) that can be reported with a specified degree of confidence.

(QS) Matrix: The component or substrate that contains the analyte of interest. For purposes of batch and QC requirement determinations, the following matrix distinctions shall be used:

Aqueous: Any aqueous sample excluded from the definition of Drinking Water or Saline/Estuarine. Includes surface water, groundwater effluents, and TCLP or other extracts.

Drinking Water: Any aqueous sample that has been designated as a potable or potential potable water source.

Saline/Estuarine: Any aqueous sample from an ocean or estuary, or other salt water source such as the Great Salt Lake.

Non-Aqueous Liquid: Any organic liquid with <15% settleable solids.

Biological Tissue: Any sample of a biological origin such as fish tissue, shellfish, or plant material. Such samples shall be grouped according to origin.

Solids: Includes soils, sediments, sludges, and other matrices with >15% settleable solids.

Chemical Waste: A product or by-product of an industrial process that results in a matrix not previously defined.

Air & Emissions: Whole gas or vapor samples including those contained in flexible or rigid wall containers and the extracted concentrated analytes of interest from a gas or vapor that are collected with a sorbant tube, impinger solution, filter, or other device. (TNI)

Matrix Spike (spiked sample or fortified sample): A sample prepared, taken through all sample preparation and analytical steps of the procedure unless otherwise noted in a referenced method, by adding a known amount of target analyte to a specified amount of sample for which an independent test result of target analyte concentration is available. Matrix spikes are used, for example, to determine the effect of the matrix on a method's recovery efficiency.

Matrix Spike Duplicate (spiked sample or fortified sample duplicate): A replicate matrix spike prepared and analyzed to obtain a measure of the precision of the recovery for each analyte.

Method Blank: A sample of a matrix similar to the batch of associated samples (when available) that is free from the analytes of interest and is processed simultaneously with and under the same conditions as samples through all steps of the analytical procedures, and in which no target analytes or interferences are present at concentrations that impact the analytical results for sample analyses.

Method Detection Limit: See Limit of Detection (LOD)

Negative Control: Measures taken to ensure that a test, its components, or the environment do not cause undesired effects, or produce incorrect test results.

Non-conformance: An indication, judgment, or state of not having met the requirements of the relevant specifications, contract, or regulation.

Observation: A record of phenomena that (1) may assist in evaluation of the sample data; (2) may be of importance to the project manager and/or the client, and yet not at the time of the observation have any known effect on quality.

Performance Audit: The routine comparison of independently obtained qualitative and quantitative measurement system data with routinely obtained data in order to evaluate the proficiency of an analyst or laboratory.

Positive Control: Measures taken to ensure that a test and/or its components are working properly and producing correct or expected results from positive test subjects.

Precision: The degree to which a set of observations or measurements of the same property, obtained under similar conditions, conform to themselves; a data quality indicator. Precision is usually expressed as standard deviation, variance or range, in either absolute or relative terms. (TNI)

Preservation: Any conditions under which a sample must be kept in order to maintain chemical and/or biological integrity prior to analysis. (TNI)

Proficiency Testing: A means of evaluating a laboratory's performance under controlled conditions relative to a given set of criteria through analysis of unknown samples provided by an external source. (TNI)

Proficiency Testing Program: The aggregate of providing rigorously controlled and standardized environmental samples to a laboratory for analysis, reporting of results, statistical evaluation of the results and the collective demographics and results summary of all participating laboratories. (TNI)

Proficiency Test Sample (PT): A sample, the composition of which is unknown to the laboratory and is provided to test whether the laboratory can produce analytical results within specified acceptance criteria. (TNI)

Quality Assurance: An integrated system of management activities involving planning, implementation, assessment, reporting and quality improvement to ensure that a process, item or service is of the type of quality needed and expected by the client. (TNI)

Quality Assurance [Project] Plan (QAPP): A formal document describing the detailed quality control procedures by which the quality requirements defined for the data and decisions pertaining to a specific project are to be achieved. (EAP-QAD)

Quality Control: The overall system of technical activities that measures the attributes and performance of a process, item, or service against defined standards to verify that they meet the stated requirements established by the customer; operational techniques and activities that are used to fulfill requirements for quality; also the system of activities and checks used to ensure that measurement systems are maintained within prescribed limits, providing protection against "out of control" conditions and ensuring that the results are of acceptable quality. (TNI)

Quality Control Sample: A sample used to assess the performance of all or a portion of the measurement system. One of any number of samples, such as Certified Reference Materials, a quality system matrix fortified by spiking, or actual samples fortified by spiking, intended to demonstrate that a measurement system or activity is in control. (TNI)

Quality Manual: A document stating the management policies, objectives, principles, organizational structure and authority, responsibilities, accountability, and implementation of an agency, organization, or laboratory, to ensure the quality of its product and the utility of its product to its users. (TNI)

Quality System: A structured and documented management system describing the policies, objectives, principles, organizational authority, responsibilities, accountability, and implementation plan of an organization for ensuring quality in its work processes, products (items), and services. The quality system provides the framework for planning, implementing, and assessing work performed by the organization and for carrying out required QA and QC activities. (TNI)

Raw Data: The documentation generated during sampling and analysis. This documentation includes, but is not limited to, field notes, electronic data, magnetic tapes, untabulated sample results, QC sample results, print outs of chromatograms, instrument outputs, and handwritten records. (TNI)

Record Retention: The systematic collection, indexing and storing of documented information under secure conditions.

Reference Material: Material or substance one or more properties of which are sufficiently homogeneous and well established to be used for the calibration of an apparatus, the assessment of a measurement method, or for assigning values to materials. (TNI)

Reference Standard: Standard used for the calibration of working measurement standards in a given organization or a given location. (TNI)

Sampling: Activity related to obtaining a representative sample of the object of conformity assessment, according to a procedure.

Second Order Polynomial Curve (Quadratic): The 2^{nd} order curves are a mathematical calculation of a slightly curved line over two axis. The y axis represents the instrument response (or Response ratio) of a standard or sample and the x axis represents the concentration. The 2^{nd} order regression will generate a coefficient of determination (COD or r^2) that is a measure of the "goodness of fit" of the quadratic curvature the data. A value of 1.00 indicates a perfect fit. In order to be used for quantitative purposes, r^2 must be greater than or equal to 0.99.

Selectivity: The ability to analyze, distinguish, and determine a specific analyte or parameter from another component that may be a potential interferent or that may behave similarly to the target analyte or parameter within the measurement system. (TNI)

Sensitivity: The capability of a method or instrument to discriminate between measurement responses representing different levels (e.g., concentrations) of a variable of interest. (TNI)

Spike: A known mass of target analyte added to a blank, sample or sub-sample; used to determine recovery efficiency or for other quality control purposes.

Standard: The document describing the elements of laboratory accreditation that has been developed and established within the consensus principles of standard setting and meets the approval requirements of standard adoption organizations procedures and policies. (TNI)

Standard Operating Procedures (SOPs): A written document which details the method for an operation, analysis, or action, with thoroughly prescribed techniques and steps. SOPs are officially approved as the methods for performing certain routine or repetitive tasks. (TNI)

Storage Blank: A blank matrix stored with field samples of a similar matrix (volatiles only) that measures storage contribution to any source of contamination.

Surrogate: A substance with properties that mimic the analyte of interest. It is unlikely to be found in environment samples and is added to them for quality control purposes.

Surrogate compounds must be added to all samples, standards, and blanks, for all organic chromatography methods except when the matrix precludes its use or when a surrogate is not available. Poor surrogate recovery may indicate a problem with sample composition and shall be reported to the client whose sample produced poor recovery. (QAMS)

Systems Audit (also Technical Systems Audit): A thorough, systematic, qualitative on-site assessment of the facilities, equipment, personnel, training, procedures, record keeping, data validation, data management, and reporting aspects of a total measurement system. (EPA-QAD)

Technical Manager: A member of the staff of an environmental laboratory who exercises actual day-to-day supervision of laboratory operations for the appropriate fields of accreditation and reporting of results

Technology: A specific arrangement of analytical instruments, detection systems, and/or preparation techniques.

Traceability: The ability to trace the history, application, or location of an entity by means of recorded identifications. In a calibration sense, traceability relates measuring equipment to national or international standards, primary standards, basic physical constants or properties, or reference materials. In a data collection sense, it relates calculations and data generated throughout the project back to the requirements for the quality of the project. (TNI)

Trip Blank: A blank matrix placed in a sealed container at the laboratory that is shipped, held unopened in the field, and returned to the laboratory in the shipping container with the field samples.

Uncertainty: A parameter associated with the result of a measurement that characterizes the dispersion of the value that could reasonably be attributed to the measured value.

Acronyms:

CAR – Corrective Action Report

CCV – Continuing Calibration Verification

CF – Calibration Factor

CFR – Code of Federal Regulations

COC – Chain of Custody

DOC – Demonstration of Capability

DQO – Data Quality Objectives

DUP - Duplicate

EHS – Environment, Health and Safety

EPA – Environmental Protection Agency

GC - Gas Chromatography

GC/MS - Gas Chromatography/Mass Spectrometry

HPLC - High Performance Liquid Chromatography

ICP - Inductively Coupled Plasma Atomic Emission Spectroscopy

ICP/MS – ICP/Mass Spectrometry

ICV – Initial Calibration Verification

IDL – Instrument Detection Limit

IH – Industrial Hygiene

IS – Internal Standard
LCS – Laboratory Control Sample
LCSD – Laboratory Control Sample Duplicate
LIMS – Laboratory Information Management System
LOD – Limit of Detection
LOQ – Limit of Quantitation
MDL – Method Detection Limit
MDLCK – MDL Check Standard
MDLV – MDL Verification Check Standard
MRL – Method Reporting Limit Check Standard
MS – Matrix Spike
MSD – Matrix Spike Duplicate
SDS - Safety Data Sheet
NELAP - National Environmental Laboratory Accreditation Program
PT – Performance Testing
TNI – The NELAC Institute
QAM – Quality Assurance Manual
QA/QC – Quality Assurance / Quality Control
QAPP – Quality Assurance Project Plan
RF – Response Factor
RPD – Relative Percent Difference
RSD – Relative Standard Deviation
SD – Standard Deviation
SOP – Standard Operating Procedure
TAT – Turn-Around-Time
VOA – Volatiles
VOC – Volatile Organic Compound

Appendix 6. Analytical Method References

Reference methods include:

- *Methods for Measuring the Acute Toxicity of Effluents and Receiving Waters to Freshwater and Marine Organisms, Fourth Edition, EPA/600/4-90/027F, August 1993.*
- *Methods for Measuring the Acute Toxicity of Effluents and Receiving Waters to Freshwater and Marine Organisms, Fifth Edition, EPA-821-R-02-012, October 2002.*
- *Compendium of Methods for the Determination of Toxic Organic Compounds in Ambient Air, US EPA, January 1996.*
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- *Methods for the Determination of Inorganic Substances in Environmental Samples, EPA-600/R-93/100, August 1993.*
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- *Methods for the Determination of Organic Compounds in Drinking Water, EPA-600/4-88-039, December 1988, Revised, July 1991, Supplement I, EPA-600-4-90-020, July 1990, Supplement II, EPA-600/R-92-129, August 1992. Supplement III EPA/600/R-95/131 - August 1995 (EPA 500 Series) (EPA 500 Series methods)*
- *Technical Notes on Drinking Water Methods, EPA-600/R94-173, October 1994*
- *NIOSH Manual of Analytical Methods, 4th ed., August 1994.*
- *Statement of Work for Inorganics & Organics Analysis, SOM and ISM, current versions, USEPA Contract Laboratory Program Multi-media, Multi-concentration.*
- *Standard Methods for the Examination of Water and Wastewater, 18th/19th /20th/ on-line edition; Eaton, A.D. Clesceri, L.S. Greenberg, A.E. Eds; American Water Works Association, Water Pollution Control Federation, American Public Health Association: Washington, D.C.*
- *Test Methods for Evaluating Solid Waste Physical/Chemical Methods (SW846), Third Edition, September 1986, Final Update I, July 1992, Final Update IIA, August 1993, Final Update II, September 1994; Final Update IIB, January 1995; Final Update III, December 1996; Final Update IV, January 2008; Final Update V, August 2015.*
- *Annual Book of ASTM Standards, American Society for Testing & Materials (ASTM), Philadelphia, PA.*
- *National Status and Trends Program, National Oceanographic and Atmospheric Administration, Volume I-IV, 1985-1994.*
- *Code of Federal Regulations (CFR) 40, Parts 136, 141, 172, 173, 178, 179 and 261*

Title: Total Organic Carbon
Lloyd Kahn Method

Approvals (Signature):

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1.0 Scope and Application

This SOP describes the laboratory procedure for the determination of total organic carbon (TOC) in soil and solid materials.

1.1 Analytes, Matrix(s), and Reporting Limits

This procedure may be used to determine total organic carbon in soil and solid materials. This procedure may not be amenable to oily matrices.

The routine reporting limit is 1000 mg/kg based on an initial sample weight of 10 mg.

On occasion clients may request modifications to this SOP. These modifications are handled following the procedures outlined in the Quality Assurance Manual.

2.0 Summary of Method

10 mg of dried sample is transferred to a tin capsule, treated with phosphoric acid and dried in an oven at a temperature 105°C for 30-60 minutes to separate the organic carbon from inorganic carbonates and bicarbonates. The sample is analyzed on an instrument where it is pyrolyzed in an inductive type furnace. The carbon is converted to carbon dioxide and measured by a differential thermal conductivity detector.

This procedure is based on the following reference documents:

- EPA Region II Document Determination of Total Organic Carbon in Sediment, July 27, 1988, authored by Lloyd Kahn, Quality Assurance Specialist.

If the laboratory's SOP has been modified from the above referenced document, a list of modifications is provided in Section 15.0 of this SOP.

3.0 Definitions

Refer to the Laboratory's Quality Assurance Manual (QAM) for the Glossary of Terms, Definitions and Acronyms except as follows:

A list of general laboratory terms and definitions are provided in Appendix A.

4.0 Interferences

Volatile organics in the sediments may be lost in the de-carbonation step resulting in a low bias.

5.0 Safety

Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual (CW-E-M-001) and this document. This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toe,

nonabsorbent shoes are a minimum.

5.1 Specific Safety Concerns or Requirements

None

5.2 Primary Materials Used

Table 1 lists those materials used in this procedure that have a serious or significant hazard rating along with the exposure limits and primary hazards associated with that material as identified in the SDS. **Note: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the SDS for each of the materials listed in the table.** A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the SDS for each material before using it for the first time or when there are major changes to the SDS.

6.0 Equipment and Supplies

- Drying Oven: Capable of maintaining a temperature of 105 ±2°C.
- Carlo Erba Elemental Analyzer Model EA1108 and Model NA 1500 or equivalent.
- Costech Elemental Analyzer: Model 4010 or equivalent.
- Analytical Balance: Capable of weighing to the nearest 0.0001g.
- Aluminum Weigh Boats.
- Tweezers
- 5mm X 9mm tin capsules
- Quartz Columns: Costech Analytical or equivalent.
- Quartz wool: for segregating and containing column materials
- Copper Wire, Reduced: Costech Analytical or equivalent.
- Tungsten on Alumina: Costech Analytical or equivalent.
- High Temperature Gloves
- Clear Plastic Sample Trays: Costech Analytical or equivalent.
- 100ul Hamilton syringe or similar

7.0 Reagents and Standards

7.1 Reagents

- Reagent water
- Phosphoric Acid, Concentrated: Reagent Grade, J.T. Baker recommended.

Phosphoric Acid Solution (1:19): Add approximately 100 mL of reagent water to a 200 mL volumetric flask. Add 18.34 g of concentrated phosphoric acid to the volumetric flask then adjust

to volume with reagent water. Mix the solution well then transfer the solution to a 250 mL polyethylene bottle. Assign an expiration date of six months from date made and store the solution at room temperature.

7.2 Standards

- Potassium Hydrogen Phthalate (KHP) (Primary Standard Grade) Used to calibrate the instrument. 47.05% Carbon by weight

- Laboratory Control Samples (LCS) Material, Organic Material of known Carbon percentage: Purchased from LECO Corporation.

1% Carbon KHP Solution (10,000 mg Carbon/L): Add 50 mL of reagent water to a 100 mL volumetric flask. Add 2.128 g of KHP and dissolve completely. Adjust to final volume with reagent water. To mix the solution, cap the flask and invert. Allow the air bubble to reach the top of the flask. Repeat 9 times. Assign an expiration of 6 months from the date prepared and store at room temperature.

0.1% Carbon KHP Solution (1000mg Carbon/L): Add approximately 25 mL of reagent water to a 50 mL volumetric flask. Add 5 mL of 1 % Carbon KHP solution to the flask and adjust to final volume with reagent water. To mix the solution, cap the flask and invert. Allow the air bubble to reach the top of the flask. Repeat 9 times. Assign an expiration date of 6 months from the date prepared so long as the parent solution does not expire sooner, in which case use the earliest expiration date. Store the solution at room temperature.

0.01% Carbon KHP Solution (100mg Carbon/L): Add approximately 25 mL of reagent water to a 50 mL volumetric flask. Add 0.5 mL of 1% Carbon KHP Solution and adjust to final volume with reagent water. To mix the solution, cap the flask and invert. Allow the air bubble to reach the top of the flask. Repeat 9 times. Assign an expiration date of 6 months from the date prepared so long as the parent solution does not expire sooner, in which case use the earliest expiration date. Store the solution at room temperature.

Note: Alternatively a 10,000mg/L TOC standard may be purchased from a reputable vendor (Spex Certiprep, SCP Science or ERA) and diluted appropriately to prepare the intermediate solutions above.

8.0 Sample Collection, Preservation, Shipment and Storage

The laboratory does not perform sample collection so sampling procedures are not included in this SOP. Sampling requirements may be found in the published reference method. Sample container, preservation techniques and holding times may vary and are dependent on sample matrix, method of choice, regulatory compliance, and/or specific contract or client requests. Listed below are the holding times and the references that include preservation requirements.

Matrix	Sample Container	Minimum Sample Size	Preservation	Holding Time	Reference
Solids	Amber glass	10 g	Chilled to ≤ 4°C	14 Days	Lloyd Kahn Method

Holding time is calculated from date of sample collection. Unless otherwise specified by client or regulatory program, after analysis, samples and extracts are retained for a minimum of 30 days after provision of the project report and then disposed of in accordance with applicable regulations.

9.0 Quality Control

9.1 Sample QC

The following QC samples are analyzed with each batch:

QC Item	Frequency	Acceptance Criteria
Method Blank (MB)	1 in 20 or fewer samples	< RL
Laboratory Control Sample (LCS)	1 in 20 or fewer samples**	%R (75-125) **Quad RSD 18%
Sample Quadruplicate	1 in 20 or fewer samples (Only when site specific P&A has been requested)	SD (≤ 3x SD of site specific P&A study)
Matrix Spike	Client request	%R (75-125)
Duplicate/Matrix Spike Duplicate	Client request	RPD 20%

**If a site specific P&A study not requested analyze the LCS in quadruplicate as a measure of method precision.

9.2 Instrument QC

The laboratory analyzes the following instrument check standards:

QC Item	Frequency	Acceptance Criteria
Initial Calibration (ICAL)	Initial Method Set-Up, after combustion chamber is changed (approx. every 200 drops)	Correlation coefficient must be >0.995
Initial Calibration Verification (ICV)	Following Calibration	%R (75-125)
Calibration Verification (CCV)	Every 20 drops and at the end of the analytical sequence	%R (75-125)
Calibration Blank (CCB)	After every CCV	<RL

10.0 Procedure

10.1 Calibration

Analyze a calibration curve each time the combustion column is changed. Change the column after 200 drops or when you experience QC recovery issues, odd peak shapes or baseline issues. The column change procedure is provided in Appendix B.

The recommended formulations for each calibration level are provided in the following table:

Calibration Standards	1.0% C KHP uL	0.1% C KHP uL	0.01%C KHP uL	% Carbon KHP	Carbon (mg)	mg/Kg of Carbon (10mg sample)
Level 1	0	0	0	47.05	0	0
Level 2	0	0	100	47.05	0.010	1000
Level 3	0	40	0	47.05	0.040	4000
Level 4	25	0	0	47.05	0.25	25000
Level 5	50	0	0	47.05	0.500	50000
Level 6	75	0	0	47.05	1.000	75000

Using a volumetric Hamilton or similar syringe, measure the specified volume of standard into a tin capsule. Dry the calibration levels completely by placing in the oven at 105°C for 60 minutes. Fold the capsules. A blank (empty) tin must be dropped for the calibration blank. Proceed to Section 10.5 for analysis instructions.

The instrument software system plots peak area against mg of Carbon and calculates a correlation coefficient using standard linear regression. The correlation coefficient (r) must be $\geq$ 0.995 for the calibration to be considered acceptable. If it is not, repeat the calibration prior to analysis.

10.2 Troubleshooting

- Calibration passes at > 0.995 correlation, but LCS fails abnormally low: Re-calibrate.
- Large peak before Carbon peak; Indicates leak in system, perform leak test, isolate and repair leak.
- Carbon peak “maxes out” at instrument 1200mv (peak has flat top): Reanalyze sample at lower weight.
- No peaks on any chromatograms, no results: Gases to instrument may be off. Turn on all gasses at valve manifold.
- Autosampler will not work at all: Gasses to instrument may be off. Turn on all gasses at valve manifold.
- Carry over; Clean autosampler slide, if persists reduce sample mass. Note sample carryover in NCM. Oily samples may not be amenable to this test.
- Single chromatogram shows results at bottom of page, but no peak or baseline in chromatogram window: Re-print single chromatogram.
- Some or all chromatograms show carbon peak at same retention time as CCV, but peak is not identified as carbon, or is identified as another element: Retention time shifted. Adjust retention time in calibration window, and reprint chromatograms.
- Peaks shift later through the run, or reduced recovery of samples with known concentration. Check oxygen tank for sufficient pressure, and replace if necessary.
- Upon recalibration, peaks are not being identified as carbon: In calibration window, general tab, adjust retention time to match peaks. Starting at level 1, “Open Standard”, open level1

curve pt. in calibration directory, click “Add Peak” button, click on peak itself. Increase level #, opening standard for each curve pt and add each peak. Carbon Tab should have all five calibration points on curve, if done correctly.

- Peaks in chromatograms identified as carbon, but all results in summary table below chromatogram are zero: Current calibration not associated with run when started. Open current calibration, copy first two columns for all points (5 rows) in small table in general tab. Then, open calibration that was associated with run (should be empty) and paste into table in calibration tab. Reprint all chromatograms on run.
- Software crashes during analysis: Boot up software normally. Chromatograms already printed/analyzed are ok, but, sample that was analyzing during shutdown is lost. Restart table at next sample by un-checking “run” box for samples already run and sample that was lost.
- Autosampler error causes few samples to remain in autosampler tray after run has finished: Identify samples that got stuck. Create a new run and analyze stuck samples (with initial weights) with bracketing QC. No MB/LCS needed.
- Autosampler error causes many sequential samples to remain in autosampler tray after run has finished (usually end of run): Add rows onto existing table. Identify samples that did not get analyzed and repeat Ids and weights into added rows. Restart table. All analyzed samples’ status should be blue (analyzed), added rows should be green (not analyzed yet).

10.3 Sample Preparation

Homogenize the sample using the procedure described in SOP BR-QA-020. Refer to Appendix D for Marine Sediment Processing, and Appendix E for Black Carbon Processing

Dry approximately 5-10 g of sample in at 105 ^{o}C for 12-24 hrs. (The sample from the moisture fraction may be used for this step.) Disaggregate the sample to break up clumps to ensure exposure to acidification in next step. Do not grind the sample.
Due to the sample size required by the instrumentation, if the sample matrix contains particles that are too large for analysis, the sample will be sieved using a 200 micron (#35) sieve.

For each field sample prepare a tin for analysis. Using tweezers, and working directly from the box, place a tin capsule on the analytical balance and tare the balance. Using the small sample scoop, add approximately 10 mg (or the project specified sample weight) of sample to a tin capsule. Record the actual sample weight used on sample preparation log. Remove the capsule from the balance and place into one of the aluminum holding trays.

To prepare the method blank, set an empty tin capsule into an aluminum holding tray (this tin must be acidified with the remainder of the samples).

To prepare the LCS, weigh 9 to 11 mg of the LECO LCS material into two tin capsules and set them in sequence in an aluminum holding tray. Prepare the LCS in quadruplicate if site specific P&A study has not been requested in the project.

For the matrix spike, weigh out an additional sample aliquot and record its weight. Add 35 uL of 1% KHP calibration stock.

For the sample duplicate, weigh out an additional sample aliquot.

Add one to two drops of 1:19 phosphoric acid to each tin capsule (enough to sufficiently cover the sample until reaction ceases). Place the aluminum trays into a drying oven set to a temperature of 105 ±2 ºC for 30-60 minutes or until all samples appear dry.

Using tweezers pinch the top of each tin capsule closed and compress the capsule around the material inside. Work carefully so as not to tear the capsule, but crush it down to the smallest size. Set the prepared samples in line in a clear plastic sample tray for storage, or place directly into an autosampler tray for analysis. For the latter, leave positions open for the CCV check standards and associated calibration blanks.

Prepare the ICV, CCV standards and blanks as follows:

Prepare an ICV for each sequence. To prepare the ICV, weigh 9 to11 mg of the LECO LCS material into a tin capsule.

For each CCV, transfer 35uL of 1% KHP solution into a tin capsule. Dry the capsules in a drying oven set to a temperature of 105 ±2 ºC for 30-60 minutes or until all samples appear dry. Fold the capsule up and compress down to the smallest size possible. Prepare enough CCVs to ensure a frequency of every 20 drops and the end of the analytical sequence. For each associated calibration blank, leave an empty position in the auto-sampler tray (a tin must be analyzed for calibration blanks).

10.4 Preparation of the ICV, CCV and Blanks

For each ICV weigh ~9-11 mg of the LECO LCS material into a tin capsule.

For each CCV, transfer 35uL of 1% KHP to a tin capsule. Dry the capsules in a drying oven at 105 ^{o}C for 30-60 minutes or until dry. Fold the capsule and compress down to the smallest size possible. Prepare enough CCVs to ensure a CCV frequency of every 20 drops and at the beginning and end of the analytical sequence. Use a folded empty tin for each calibration blank(not acidified).

10.5 Software Set-up and Analysis of a Curve

If a there is not a valid curve or the valid curve is not listed in the software, create a calibration curve in the analytical software. Enter the standard type, level, and mg of KHP used for each calibration level. Enter each sample ID and their respective weights into the instrument software, enter a weight of 10 mg for the method blank and calibration blanks and save the sample table. Enter the weight for the LCS. Add the tin capsules to the autosampler tray in sequence and set the tray into the autosampler carriage.

An example analytical sequence that includes ICAL is as follows:

Initial Calibration (calibration blank and 5 calibration standards)
ICV
ICB(blank tin)
CCV
CCB(blank tin)
MB(acidified tin)
LCS (Routine=4 reps; Dixon=2reps)

Samples (Routine=1 rep; Dixon=2reps)
CCV
CCB(blank tin)
Samples (Routine=1 rep; Dixon=2reps)
CCV
CCB(blank tin)

Click the “start” icon to begin the analysis.

11.0 Corrective Action

When an out-of-control situation occurs that is not delineated in this corrective action table or the corrective actions listed do not adequately address the circumstances, a Corrective Action Report (CAR) (NCM), etc., must be developed (see SOP BR-QA-016) and the analyst must use his/her best analytical judgment and available resources to determine the corrective action to be taken. The out-of-control situation may be caused by more than one variable. The analyst should seek the assistance of his/her immediate supervisor, QA manager or other experienced staff if they are uncertain of the cause of the out-of-control situation. The analysis must not be resumed until the source of the problem and an in-control status is re-established. All samples associated with the out-of-control situation must be reanalyzed after in-control status has been re-established or if authorization is received from the supervisor or QA Manager for release with data qualification.

12.0 Calculations / Data Reduction

12.1 Calculations

The instrument software calculates TOC using the area response from the calibration curve and sample mass used. The determination of TOC is performed by the laboratory’s LIMS system. See Appendix C for equations for percent recovery and relative percent difference.

12.2 Data Review

12.2.1 Primary Data Review

Enter the results of the quadruplicate analysis into the EXCEL spreadsheet designated for this purpose. Compare the standard deviation derived from the quadruplicate sample or LCS against the standard deviation from the appropriate P&A study. If the SD of the sample quadruplicate is greater than 3 times the SD of the study, initiate a nonconformance memo and notify the PM to determine if further action is necessary.

Upload the instrument data to the laboratory information system (LIMS) “TALS”. Evaluate the sequence against the acceptance criteria given in Table 2. Perform the recommended corrective action as necessary. If corrective action is not taken or is not successful, initiate a nonconformance memo (NCM) to document the situation. Set the Batch to 1st level reviewed. For Dixon analysis, an additional 2 reps must be performed if the original reps yielded a RSD greater than 40. Then the four different replicates must be entered into the Dixon Outlier Template (FWC200A for 4 reps or FWC200B for 3 reps) from the lowest to the highest result.

Assemble supporting documents including the quad sample calculation spreadsheet and forward to secondary review staff.

12.2.2 Secondary Data Review

Spot-check the analytical results. Verify that acceptance criteria were met and if the results do not fall within the established limits verify the recommended corrective actions were performed. If corrective action was not taken or is unsuccessful, ensure the situation is documented with a nonconformance memo (NCM) and ensure data is qualified accordingly.

Set the batch to second level review. Scan and attach copies of supporting documentation such as; run logs, raw data and quad sample spreadsheet to the batch. Set the job to lab complete, review the form set, correct any problems then forward supporting documentation to report management.

12.2.3 Data Reporting

The report format, application of data qualifiers and creation of the data deliverable is performed by the LIMS using the formatter set by the project manager during log-in.

Retain, manage and archive electronic and hardcopy data as specified in laboratory SOP BR-QA-014 Laboratory Records.

13.0 Method Performance

13.1 Method Detection Limit Study (MDL)

The method detection limit (MDL) is the lowest concentration that can be detected for a given analytical method and sample matrix with 99% confidence that the analyte is present. MDLs reflect a calculated (statistical) value determined under ideal laboratory conditions in a clean matrix, and may not be achievable in all environmental matrices. An initial method detection limit study is performed in accordance with SOP No. BR-QA-005. The laboratory maintains MDL studies for analyses performed; these are verified at least annually unless method or program requirements require a greater frequency.

13.2 Demonstration of Capabilities

All personnel are required to perform an initial demonstration of proficiency (IDOC) on the instrument they will be using for analysis prior to testing samples. On-going proficiency must be demonstrated annually. IDOCs and on-going proficiency demonstrations are conducted as follows.

13.2.1 Four aliquots of the QC check sample are analyzed using the same procedures used to analyze samples, including sample preparation. The concentration of the QC check sample may be equivalent to a mid- level calibration.

13.2.2 Calculate the average recovery and standard deviation of the recovery for each analyte of interest.

13.2.3 If any analyte does not meet the acceptance criteria, the test must be repeated. Only those analytes that did not meet criteria in the first test need to be evaluated. TNI 2016 requires consecutive passing results. Repeated failure for any analyte indicates the need for the laboratory to evaluate the analytical procedure and take corrective action.

13.2.4 Until the IDOC is approved by the QA Manager (or designee); the trainer and trainee must be identified in the batch record.

13.3 Training Requirements

The Group Leader is responsible for ensuring that this procedure is performed by an associate who has been properly trained in its use and has the required experience. A new analyst must be working under documented supervision prior to approval of the IDOC. Documentation that a new analyst is performing under supervision must be entered into the batch record (View Batch Information) until that analyst's IDOC has been approved by the QA Manager (or designee). See requirements for demonstration of analyst proficiency in SOP No. BR-QA-011.

14.0 Pollution Control

It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage and reagent stability). Employees must abide by the policies in Section 13 of the Corporate Safety Manual for "Waste Management and Pollution Prevention."

15.0 Waste Management

Waste management practices are conducted consistent with all applicable rules and regulations. Excess reagents, samples and method process wastes are disposed of in an accepted manner. Waste description rules and land disposal restrictions are followed. Waste disposal procedures are incorporated by reference to BR-EH-001 *Hazardous Waste*.

The following waste streams are produced when this method is carried out.

- Caustic waste – 2.5 L glass satellite container.
- Acidic Waste - 2.5L glass satellite container

The satellite containers are labeled "Hazardous Waste" along with the type of waste category generated. Authorized personnel routinely transfer the contents of the satellite containers to the hazardous waste storage room for future disposal in accordance with Federal, State and Local regulations.

16.0 References / Cross-References

- EPA Region II Document Determination of Total Organic Carbon in Sediment, July 27, 1998, authored by Lloyd Kahn, Quality Assurance Specialist.
- Corporate SOP CW-E-M-001 Corporate Environmental Health and Safety Manual
- Laboratory SOP BR-QA-005, Procedures for the Determination of Limits of Detection (LOD), Limits of Quantitation (LOQ) and Reporting Limits (RL).
- Laboratory SOP BR-QA-011 Employee Training
- Laboratory SOP BR-EH-011 Hazardous Waste
- Laboratory SOP BR-QA-014 Laboratory Records
- Laboratory Quality Assurance Manual (QAM)

17.0 Method Modifications

The laboratory procedure is modified from the reference method as follows:

Modification Number	Method Reference	Modification
1	TOC by Lloyd Kahn	The Laboratory uses 1:19 (w/w) phosphoric acid to decarbonize the sample (to reduce sample loss from effervescence) and dries the sample in an oven at 105^{o}C.
2	TOC by Lloyd Kahn	Due to the small sample size the laboratory dries and disaggregates the sample prior to analysis. This step improves precision associated with high moisture or clay type matrices.
3	TOC by Lloyd Kahn	P&A studies are not performed per the reference method due to variability in project sites. See section 12.2 for the P&A procedure that the laboratory uses.

18.0 Attachments

- Table 1: Primary Materials Used
- Table 2: QC Summary & Recommended Corrective Action
- Appendix A: Terms and Definitions
- Appendix B: Column change procedure
- Appendix C: Equations

19.0 Revision History

BR-WC-008, Revision 17.0

- Updated Title Page
- Updated Headers and Footers Throughout
- Section 10.2: Updated troubleshooting section to add peaks shift later through the run, or reduced recovery of samples with known concentration. Check oxygen tank for sufficient pressure, and replace if necessary.

Previous revisions are retained by the QA Department.

Table 1: Primary Materials Used

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Phosphoric Acid	Corrosive	1 Mg/M3 TWA	Inhalation is not an expected hazard unless misted or heated to high temperatures. May cause redness, pain, and severe skin burns. May cause redness, pain, blurred vision, eye burns, and permanent eye damage.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

Table 2: QC Summary, Frequency, Acceptance Criteria and Recommended Corrective Action (TOC Lloyd Kahn)

QC Item	Frequency	Acceptance Criteria	Recommended Corrective Action[1]
ICAL	Following each column change and when CCV failure indicates calibration may no longer be valid.	correlation coefficient $\geq$ 0.995	Standards check, re-calibration
CCV	Every 20 drops and at the end of the analytical run	%R (75-125)	Re-prepare and reanalyze samples not bracketed by passing standard. If CCV fails high, and TOC is not detected in any of the bracketed samples, the samples without TOC may be reported without reanalysis.
CCB	Following each CCV	< RL	Re-prepare and reanalyze batch.
Method Blank (MB)	Once per batch of 20 samples	< RL	Re-prepare and reanalyze batch.
LCS	Once per batch of 20 samples. Prepared in quadruplicate unless site P&A study is performed, then analyze as single injection.	%R (75-125)	Re-prepare and reanalyze batch.
Sample Quadruplicate	When site specific P&A study is specified. If no quad specified for any jobs included in the batch analyze the LCS in quadruplicate.	SD ≤ 3X annual P&A SD	If sample quad, notify PM. If LCS quad, reanalyze the entire batch.

[1]*The recommended corrective action may include some or all of the items listed in this column. The corrective action taken may be dependent on project data quality objectives and/or analyst judgment but must be sufficient to ensure that results will be valid. If corrective action is not taken or is not successful, data must be flagged with appropriate qualifiers.*

Appendix A: Terms and Definitions

Batch: environmental samples, which are prepared and/or analyzed together with the same process, using the same lot(s) of reagents. A preparation/digestion batch is composed of one to 20 environmental samples of similar matrix, meeting the above criteria.

Calibration: the establishment of an analytical curve based on the absorbance, emission intensity or other measured characteristic of known standard.

Calibration Standards: a series of known standard solutions used to calibrate the instrument response with respect to analyte concentration.

Demonstration of Capability (DOC): procedure to establish the ability to generate acceptable accuracy and precision.

Holding Time: the maximum time that a sample may be held before preparation and/or analysis as promulgated by regulation or as specified in a test method.

Laboratory Control Sample (LCS): a blank matrix spiked with a known amount of analyte(s) processed simultaneously with and under the same conditions as samples through all steps of the procedure.

Method Blank (MB): a blank matrix processed simultaneously with and under the same conditions as samples through all steps of the procedure. Also known as the preparation blank (PB).

Non-conformance: an indication, judgment, or state of not having met the requirements of the relevant specification, contract or regulation.

Preservation: refrigeration and/or reagents added at the time of sample collection to maintain the chemical, physical, and/or biological integrity of the sample.

Reporting Limit (RL): the level to which data is reported for a specific test method and/or sample.

Appendix B: Column Change Procedure:

Turn off the helium and oxygen supplies to the instrument.

Dial the left furnace temperature to a reading of 052 (this equates to 520˚C). Wait until the temperature drops below 600˚C to remove the column.

Remove the panel covering the furnace and unscrew the autosampler connection from the top of the column.

Unscrew the fitting at the bottom of the column and remove.

Lift the column up and out of the furnace using high temperature gloves.

CAUTION: The column will still be 500-600˚C. Do not touch the center portion of the column. Place the spent column in the metal can designated for this purpose.

Lay a new quartz column on the bench top, measure and mark off for the following:

- One inch up from the bottom and add a ½ inch plug of quartz wool. Note: pack the quartz wool tightly enough for it to stay in place.
- Pour in 2 ½ inches of copper wire
- Pack another ½ inch quartz wool plug on top of the copper
- Pour in 3 inches of tungsten
- Pack a final ½ inch quartz wool plug on top of the tungsten

Place the new column into the furnace and reconnect the top and bottom fittings. Snug these up, but don't over tighten.
Replace the panel covering the furnace, dial the furnace temperature back to 102 (this equates to

1020˚C), and turn the helium and oxygen supplies back on.

When the instrument comes up to operating temperature, it is ready to calibrate.

Appendix C: Equations

Percent Recovery (%R) LCS and CCVs

$$\%R = \frac{SR}{SA} \times 100\%$$

Where:
SR= Sample Result
SA=Concentration of Spike Added

Relative Percent Difference (%RPD)

$$\%RPD = \frac{|D_1 - D_2|}{\frac{D_1 + D_2}{2}} \times 100$$

Where:
D1 = Sample result
D2 = Duplicate Result

Appendix D: Marine Sediments High in Inorganic Carbon

Sample Preparation

Transfer approximately 10 g of a thoroughly mixed sample to an aluminum weigh dish, and dry in the 105°C oven. Grind the sample with the pink mortar and pestle to a fine powder. Record the weight of a 250 mL Teflon beaker then transfer ~ 5 g of the ground sample to this beaker.

If the sample is to be spiked, weigh the beaker to the nearest 0.1mg and record the weight. Likewise determine and record the weight of the added sample. Add 0.1g of NIST 1632b Trace Elements in Coal (80.11% Carbon) to the sample. Record the weight added. Evenly distribute the spike over the sample and use a glass stir rod to mix the spike with the sample. Do not use that stir rod with any other sample.

Use Talc-free latex gloves from this point on to minimize the risk of acid burns. Add several drops of 1:1 HCL to each sample and stir each sample with its own glass stir rod. Carefully rinse the stir rod and beaker walls with DI water using a fine-tipped squirt bottle. Use only what is needed to bring the entire sample to the bottom of the beaker. ***When adding water to acid use necessary precautions to avoid splashing!*** Samples with high concentrations of inorganic carbon may effervesce to the point of overflowing the beaker, so take care to add the acid in small aliquots and stir vigorously. If the sample "boils over" it must be re-prepared. Continue to add 1:1 HCL in small aliquots until there is no further reaction, taking sample to dryness after each addition of acid in a 105-degree oven.

Dry the treated samples in the oven after each acid/water addition. Do not add more than a total of 200 mL of 1:1 HCL to any sample.

***NOTE**: Samples are hygroscopic and will absorb water if they are exposed to air for too long.*

Weigh beaker with residue and record the residue weight measurement. After the sample is thoroughly dry, scrape the sample residue from the beaker and grind to a powder using the pink mortar and pestle. Transfer the ground sample to a clean, dry 40-mL vial reserved for this analysis.

***NOTE:** Depending on the nature of the sample, it may be difficult to completely remove the dried residue from the beaker or to grind it to a homogenous powder. Where difficulties are encountered, make a note on the preparation worksheet.*

Analysis

Perform TOC analysis on processed sample material as outlined in section 10.0 of this SOP.

Appendix E: Determination of Black Carbon in Sediment Procedure

1. Obtain a representative subsample of the sediment. Weight 10 grams of sample into a clean pre-tared aluminum drying pan or equivalent.

2. Dry the sample at 105^{o}C for at least 12 hours.

3. Grind the sample using a mortar and pestle.

4. Sieve the sample using a number 35 sieve (500 um).

5. Treat the sample with phosphoric acid. Add acid drop wise until effervescence is no longer observed.

6. Dry the sample at 105^{o}C for 1 hour.

7. Set aside an aliquot of the sample at this stage for direct TOC analysis, reported without correction for the IN623 percent solids. Continue with the sample for Black Carbon.

8. Place the dried sample into a clean crucible and cover the sample.

9. Bake the samples at 375^{o}C in a muffle for 24 hours or until the LCS is +/- 50% of the true value.

10. Allow the samples to cool and transfer approximately 10.0 mg into each of two tin capsules.

11. Transfer the sample (in the tin capsules) to the TOC analyzer for analysis by the Lloyd Kahn Method.

12. The sample is pyrolyzed in an inductive type furnace, where the carbon is converted to carbon dioxide, which is measured using a differential thermal conductivity detector.

13. The results will be reported as mg/Kg Black Carbon.

Note: Black carbon LCS material: NIST Standard Reference Material 1944 New York-New Jersey Waterways Sediment.

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Quality Assurance Manual Cover Page

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Quality Assurance Manual
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3.0 INTRODUCTION, SCOPE AND APPLICABILITY

3.1 Introduction and Compliance References

Eurofins Knoxville's Quality Assurance (QA) Manual is a document prepared to define the overall policies, organization objectives and functional responsibilities for achieving the laboratory's QA Program. Governing SOPs are in place within the organization to ensure the proper execution of this QA Manual (refer to Appendix 1). This manual and referenced documents are required reading for all personnel.

The laboratory is a team of people who work together to serve the health and environmental needs of society through science and technology. We offer comprehensive expertise in environmental laboratory applications *OR* maintain a local perspective in its scope of services and client relations and maintain a national perspective in terms of quality.

As such, this QA Manual has been prepared to assure compliance with The NELAC Institute (TNI) Standard, dated 2009 and 2016; ISO/IEC Guide 17025:2005 and 2017; the Department of Defense (DoD)/Department of Energy (DOE) Quality Systems Manual (QSM). Policies and procedures listed in Appendix 1 are compliant with the National Divisional Support Center (NDSC) Quality Management Plan (QMP) for Eurofins TestAmerica and the various accreditation and certification programs which are held by the laboratory to support environmental work (Appendix 2). The QMP provides a summary of Eurofins TestAmerica's quality and data integrity system. It contains requirements and general guidelines under which all Eurofins TestAmerica facilities shall conduct their operations.

Refer to Appendix 3 for a list of additional references for which this QA Manual is compliant; and Appendix 4 for the compliance crosswalk of this manual to the TNI & ISO/IEC Guide 17025 requirements.

3.2 Terms and Definitions

A QA Program is a system designed to ensure that data produced by the laboratory conforms to the standards set by state and/or federal regulations. The program functions at the local management level through company goals, from guidance at the executive management level, and at the analytical level through Standard Operating Procedures (SOPs) and quality control. Our program is designed to minimize systematic error, encourage constructive, documented problem solving, and provide a framework for continuous improvement within the organization to better serve our clients.

Refer to Appendix 5 for a list of Terms and Acronyms.

3.3 Scope / Fields of Testing

The laboratory analyzes a broad range of environmental and industrial samples. Sample matrices vary among air (source and ambient), drinking water, effluent water, groundwater, soils, sediment, biological, hazardous waste, and sludge. The QA Program contains specific procedures and methods to test samples of differing matrices for chemical, physical and biological parameters. The Program also contains guidelines on maintaining documentation of analytical processes, reviewing results, servicing clients and tracking samples through the laboratory. The technical and service requirements of all analytical requests are thoroughly evaluated before

commitments are made to accept the work. Measurements are made using published reference methods or methods developed and validated by the laboratory.

The methods covered by this manual include the most frequently requested methodologies needed to provide analytical services in the United States and its territories. The specific list of test methods used by the laboratory can be found in Appendix 6. Our areas of expertise include:

Standard Services	Specialty Services
• Volatiles • Semivolatiles • Metals • Pesticides/PCBs/Herbicides • Waste Characterization • Non-Potable Water Testing • Drinking Water Testing • Soil and Surface Water Testing • Vapor and Air Analysis • Sediment and Tissue Testing • Method Development	• Dioxins & Furans • PCB Congeners • Alkyl PAHs • PFAS • Source Air Analysis including PFAS • Metals – Sequential Extraction Procedure

NOTE: All current certificates and scopes of accreditation are available on the laboratory's website at www.testamericainc.com

The approach of this manual is to define the minimum level of quality assurance and quality control necessary to meet these requirements. All methods performed by the laboratory shall meet these criteria as appropriate. In some instances, quality assurance project plans (QAPPs), project specific data quality objectives (DQOs) or local regulations may require criteria other than those contained in this manual. In these cases, the laboratory will abide by the requested criteria following review and acceptance of the requirements by the Laboratory Director and/or Business Unit Manager and the Quality Assurance (QA) Manager. In some cases, QAPPs and DQOs may specify less stringent requirements. The Laboratory Director and/or Business Unit Manager and the QA Manager must determine if it is in the lab's best interest to follow the less stringent requirements.

Note: Eurofins TestAmerica Knoxville must comply with the information provided in its VAP approved QA Manual and SOPs.

3.4 Management of the Manual

3.4.1 Review Process

The template on which this manual is based is reviewed annually by Eurofins National Divisional Support Center (NDSC) which houses the Quality Assurance leadership team for Eurofins Environment Testing America. NDSC QA will assure that the template remains in compliance with Section 3.1. This manual itself is reviewed annually by senior laboratory management to assure that it reflects current practices and meets the requirements of the laboratory's clients and regulators as well as the QMP. Occasionally, the manual may need changes in order to meet new or changing regulations and operations. The QA Manager will review the changes in the normal course of business and incorporate changes into revised sections of the document. All updates will be reviewed by the senior laboratory management staff. The laboratory updates

and approves such changes according to our Document Control and Distribution procedures (refer to SOP No. KNOX-QA-0011).

4.0 MANAGEMENT REQUIREMENTS

4.1 Overview

The laboratory is a local operating unit of Eurofins Environment Testing America. The laboratory's operational and support staff have the day-to-day independent operational authority under the direction of the Laboratory President and the Laboratory Director and/or Business Unit Manager and is supported by the NDSC QA team. The laboratory management staff includes directors, managers and group leaders. The organizational chart of the management staff are presented in Figure 4-1.

4.2 Roles and Responsibilities

In order for the QA Program to function properly, all members of the staff must clearly understand and meet their individual responsibilities as they relate to the quality program. The responsibility for quality resides with every employee of the laboratory. All employees have access to the QA Manual, are trained to this manual, and are responsible for upholding the standards therein. Each person carries out his/her daily tasks impartially and in a manner consistent with the goals and in accordance with the procedures in this manual and the laboratory's SOPs. The following descriptions briefly define each role in its relationship to the Quality Assurance Program.

4.2.1 Vice President of Quality and Environmental Health and Safety (VP-QA/EHS)

The Vice President (VP) of QA/EHS reports directly to Eurofins Environment Testing America Chief Operating Officer (COO). With the aid of the NDSC Quality Team Members, Business Unit Managers, Laboratory Directors, the VP-QA/EHS has the responsibility for the establishment, general overview and maintenance of the Quality Assurance and EH&S Programs within Eurofins Environment Testing America. Additional responsibilities include:

- Review of QA/QC and EHS aspects of NDSC Official Document, national projects and expansions or changes in services.
- Work with various organizations outside of the laboratory to further the development of quality standards and represent the laboratory at various trade meetings.
- Prepare monthly reports for quality and EH&S metrics across the environmental testing laboratories and a summary of any quality and EH&S related initiatives and issues.
- With the assistance of the Executive Management, Corporate Senior Management Teams and the EHS Managers, maintenance and implementation of the Eurofins Environment Testing America Environmental, Health and Safety Program.

4.2.2 Quality Directors

There are four (4) Quality Directors within NDSC that report directly to the VP-QA/EHS. These Quality Directors have oversight of the general overview and maintenance of the QA Program within the Eurofins Environment Testing America laboratories. Supported tasks include:

- Monitors laboratory internal audit findings;
- Identifies common laboratory weaknesses and monitors corrective action closures.
- Develops NDSC quality guidance documents and management tools for ensuring and improving compliance;

- Monitors and communicates DoD/DoE requirements;
- Monitors and communicates regulatory and certification requirements;
- Training and Onboarding
- Laboratory assessments, mentoring, and interventions
- Track/drive root cause investigations and corrective action plans
- Builds knowledge base for preventive actions

4.2.3 Quality Information Manager

The Quality Information works directly with the NDSC Quality Directors and EHS Managers; and reports directly to the VP-QA/EHS. The Quality Information Manager is responsible for the management of:

- NDSC Official Documents
- TALS/LIMS Certification Module Data
- Company's Intranet website
- Company's Regulatory Limits Database
- Subcontract laboratory and approved vendor information
- Internal and External client support for various company groups (e.g., Client Services, EH&S, Legal, IT, Sales) for both quality and operational functions
- Communicate regulatory information and lists

4.2.4 Environmental Health and Safety (EH&S) Managers

There are 3 EH&S Managers within NDSC that report directly to the VP-QA/EHS. These EH&S Managers have oversight of the general overview and maintenance of the EH&S Program within the Eurofins Environment Testing America laboratories. Supported tasks include:

- Consolidation and tracking all safety and health-related information and reports for the company, and managing compliance activities for Eurofins Environment Testing America locations.
- Coordination/preparation of the Environmental, Health and Safety Manual Template that is used by each laboratory to prepare its own laboratory-specific Safety Manual/ CHP.
- Preparation of information and training materials for laboratory EHS Coordinators.
- Assistance in the coordination of employee exposure and medical monitoring programs to insure compliance with applicable safety and health regulations.
- Serving as Department of Transportation (D.O.T.) focal point and providing technical assistance to location management.
- Serving as Hazardous Waste Management main contact and providing technical assistance to location management.

4.2.5 Ethics and Compliance Officers (ECOs)

The NDSC VP-QA/EHS and Corporate Counsel are designated Ethics and Compliance Officer (ECO). Each ECO acts as a back-up to the other ECO and both are involved when data investigations occur. Each ECO has a direct line of communication to the entire executive management personnel and lab management staff.

The ECOs monitor and audit procedures to determine compliance with policies and to make recommendations for policy enhancements to the President, COO, Laboratory Director or other

appropriate individuals within the laboratory. The ECO will assist the laboratory QA Manager in the coordination of internal auditing of ethical policy related activities and processes within the laboratory, in conjunction with the laboratory's regular internal auditing function.

The ECOs will also participate in investigations of alleged violations of policies and work with the appropriate internal departments to investigate misconduct, remedy the situation, and prevent recurrence of any such activity.

4.2.6 Laboratory Director

The Laboratory Director is responsible for the overall quality, safety, financial, technical, human resource and service performance of the whole laboratory and reports to their business unit President. The Laboratory Director provides the resources necessary to implement and maintain an effective and comprehensive Quality Assurance and Data Integrity Program.

Specific responsibilities include, but are not limited to:

- Implementing and ensuring adherence to the Quality System as described in this QAM and in the supporting laboratory policies and procedures.
- Provides one or more technical managers for the appropriate fields of testing. If the Technical Manager is absent for a period of time exceeding 15 consecutive calendar days, the Laboratory Director must designate another full time staff member meeting the qualifications of the Technical Manager to temporarily perform this function. If the absence exceeds 35 consecutive calendar days, the primary accrediting authority must be notified in writing.
- Ensures that all analysts and supervisors have the appropriate education and training to properly carry out the duties assigned to them and ensures that this training has been documented. Works with Eurofins Environment Testing Human Resources for hiring of new personnel.
- Ensures that personnel are free from any commercial, financial and other undue pressures which might adversely affect the quality of their work.
- Ensures company human resource policies are adhered to and maintained.
- Ensures that sufficient numbers of qualified personnel are employed to supervise and perform the work of the laboratory. Assesses laboratory capacity and workload.
- Ensures that appropriate corrective actions are taken to address analyses identified as requiring such actions by internal and external performance or procedural audits. Procedures that do not meet the standards set forth in the QA Manual or laboratory SOPs may be temporarily suspended by the Laboratory Director.
- Reviews and approves all SOPs prior to their implementation and ensures all approved SOPs are implemented and adhered to.
- Pursues and maintains appropriate laboratory certification and contract approvals. Supports ISO 17025 requirements.
- Ensures client specific reporting and quality control requirements are met.
- Contributes to the continuous improvement of the laboratory operations.
- Maintains an awareness of technical developments and regulatory requirements.
- Captains the management team, consisting of the QA Manager, the Technical Manager(s), and the Operations Manager as direct reports.

4.2.7 Quality Assurance (QA) Manager or Designee

The QA Manager has responsibility and authority to ensure the continuous implementation of the quality system at the laboratory where they work.

The QA Manager reports directly to the Laboratory Director and their NDSC Quality Director. This position is able to evaluate data objectively and perform assessments without outside (e.g., managerial) influence. The NDSC QA Team may be used as a resource in dealing with regulatory requirements, certifications and other quality assurance related items. The QA Manager directs the activities of the QA office to accomplish specific responsibilities, which include, but are not limited to:

- Serves as the focal point for QA/QC in the laboratory.
- Have functions independent from laboratory operations for which he/she has quality assurance oversight.
- Have documented training and/or experience in QA/QC procedures and the laboratory's Quality System.
- Maintaining and updating the QA Manual.
- Monitoring and evaluating laboratory certifications; scheduling proficiency testing samples.
- Monitoring and communicating regulatory changes that may affect the laboratory to management.
- Training and advising the laboratory staff on quality assurance/quality control procedures that are pertinent to their daily activities.
- Have a general knowledge of the analytical test methods for which data audit/review is performed (and/or having the means of getting this information when needed).
- Arrange for or conducting internal audits on quality systems and the technical operation.
- Notifying laboratory management of deficiencies in the quality system and ensuring corrective action is taken. Procedures that do not meet the standards set forth in the QA Manual or laboratory SOPs shall be investigated following procedures outlined in Section 14 and if deemed necessary may be temporarily suspended during the investigation.
- The laboratory QA Manager will maintain records of all ethics-related training, including the type and proof of attendance.
- Maintain, improve, and evaluate the corrective action database and the corrective and preventive action systems.
- Objectively monitor standards of performance in quality control and quality assurance without outside (e.g., managerial) influence.
- Coordinating of document control of SOPs, MDLs, control limits, and miscellaneous forms and information.
- Performing technical data audits and method audits to ensure consistency and compliance with regulatory requirements.
- Review of external audit reports and data validation requests.
- Follow-up with audits to ensure client QAPP requirements are met.
- Establishment of reporting schedule and preparation of various quality reports for the Laboratory Director, clients and/or NDSC QA Team.

- Development of suggestions and recommendations to improve quality systems.
- Research of current state and federal requirements and guidelines.
- Participate in the vendor and supplier approval process, including subcontractors.
- Captains the QA team to enable communication and to distribute duties and responsibilities.
- Communication to the relevant regulatory authorities when there are management or facility changes that impact the laboratory.
- Ensuring communication & monitoring standards of performance to ensure that systems are in place to produce the level of quality as defined in this document.
- Evaluation of the thoroughness and effectiveness of training.
- Compliance with ISO 17025
- Compliance with the DoD/DOE QSM.
- Asssuring compliance with The NELAC Institute (TNI) Standard.

4.2.8 Technical Manager or Designee

The Technical Manager is responsible for the technical operation of the laboratory and reports directly to the Laboratory Director. He/she is accountable for all analyses and analysts under their experienced supervision and for compliance with the ISO 17025:2017 Standard. The scope of responsibility ranges from the new-hire process and existing technology through the ongoing training and development programs for existing analysts and new instrumentation. Specific responsibilities include, but are not limited to:

- Exercises day-to-day supervision of laboratory operations for the appropriate field of accreditation and reporting of results. Coordinating, writing, and reviewing preparation of all test methods, i.e., SOPs, with regard to quality, integrity, regulatory and optimum and efficient production techniques, and subsequent analyst training and interpretation of the SOPs for implementation and unusual project samples. He/she insures that the SOPs are properly managed and adhered to at the bench. He/she develops standard costing of SOPs to include supplies, labor, overhead, and capacity (design vs. demonstrated versus first-run yield) utilization.
- Reviewing and approving, with input from the QA Manager, proposals from marketing, in accordance with an established procedure for the review of requests and contracts. This procedure addresses the adequate definition of methods to be used for analysis and any limitations, the laboratory's capability and resources, the client's expectations. Differences are resolved before the contract is signed and work begins. A system documenting any significant changes is maintained, as well as pertinent discussions with the client regarding their requirements or the results of the analyses during the performance of the contract. All work subcontracted by the laboratory must be approved by the client. Any deviations from the contract must be disclosed to the client. Once the work has begun, any amendments to the contract must be discussed with the client and so documented.
- Monitoring the validity of the analyses performed and data generated in the laboratory. This activity begins with reviewing and supporting all new business contracts, insuring data quality, analyzing internal and external non-conformances to identify root cause issues and implementing the resulting corrective and preventive actions, facilitating the data review process (training, development, and accountability at the bench), and providing technical and troubleshooting expertise on routine and unusual or complex problems.

- Providing training and development programs to applicable laboratory staff as new hires and, subsequently, on a scheduled basis. Training includes instruction on calculations, instrumentation management to include troubleshooting and preventive maintenance.
- Enhancing efficiency and improving quality through technical advances and improved LIMS utilization. Capital forecasting and instrument life cycle planning for second generation methods and instruments as well as asset inventory management.
- Coordinating sample management from “cradle to grave,” insuring that no time is lost in locating samples.
- Scheduling all QA/QC-related requirements for compliance, e.g., MDLs, etc.
- Captains department personnel to communicate quality, technical, personnel, and instrumental issues for a consistent team approach.
- Coordinates audit responses with the QA Manager.
- Compliance with ISO 17025.
- Compliance with the DoD/DOE QSM.
- Compliance with The NELAC Institute (TNI) Standard.

4.2.9 Operations Manager

The Operations Manager manages and directs the analytical production sections of the laboratory. He/She reports directly to the Laboratory Director. He/She assists the Technical Manager in determining the most efficient instrument utilization. More specifically, he/she:

- Scheduling analytical operations.
- Supervising QC activities performed as a part of routine analytical operations.
- Implementing data review procedures.
- Supervising the preparation and maintenance of laboratory records.
- Supervising maintenance of instruments and scheduling of repairs.
- Supervising the daily activities of the Report Production Group.
- Working with the Project Managers and Group Leaders to assure the requirements of projects are met in a timely manner.
- Ensuring that corrective actions are implemented and effective.
- Evaluates the level of internal/external non-conformances for all departments.
- Continuously evaluates production capacity and improves capacity utilization.
- Continuously evaluates turnaround time and addresses any problems that may hinder meeting the required and committed turnaround time from the various departments.
- Develops and improves the training of all analysts in cooperation with the Technical Manager and QA Manager and in compliance with regulatory requirements.
- Is responsible for efficient utilization of supplies.
- Constantly monitors and modifies the processing of samples through the departments.
- Fully supports the quality system and, if called upon in the absence of the QA Manager, serves as his substitute in the interim.

- Complying with ISO17025:2017, The NELAC Institute (TNI) Standard, DoD/DOE QSM and the various QC programs implemented at the laboratory.

4.2.10 Project Manager (PM)

Members of the laboratory Project Management Group are responsible for organizing and managing client projects. Clients are assigned a project manager who serves as their primary contact at the laboratory. It is the PM's responsibility to act as the client advocate by communicating client requirements to laboratory personnel and ensuring that clients provide complete information needed by the laboratory to meet those requirements – including all verbal communcations.

The Project Manager's responsibilities include:

- Scheduling sample submissions, sample container orders and sample pick-up via the laboratory courier service.
- Confirming certification status
- Coordinating and communicating turnaround time (TAT) requirements for high priority samples/projects.
- Answering common technical questions, facilitating problem resolution and coordinating technical details with the laboratory staff.
- Ensuring that client specifications, when known, are met by communicating project and quality assurance requirements to the laboratory.
- Notifying laboratory personnel of incoming projects and sample delivery schedules.
- Coordinating and communicating with EH&S staff regarding new/unusual sample matrices and/or analyses in advance of the arrival of the work.
- Monitor the status of all data package projects in-house to ensure timely and accurate delivery of reports.
- Coordinating client requests for sample containers and other services.
- Scheduling sample pick-ups from client offices or project sites and notifying the laboratory staff of incoming samples.
- Coordinating subcontract work.
- Respond to client inquires and resolve service issues.

4.2.11 Group Leaders

Group Leaders report to the Operations Manager. Each one is responsible to:

- Ensure that analysts in their department adhere to applicable SOPs and the QA Manual. They perform frequent SOP and QA Manual review to determine if analysts are in compliance and if new, modified, and optimized measures are feasible and should be added to these documents.
- With regard to analysts, participates in the selection, training (as documented in Section 5.3), development of performance objectives and standards of performance, appraisal (measurement of objectives), scheduling, counseling, discipline, and motivation of analysts and documents these activities in accordance with systems developed by the QA and Personnel Departments. They evaluate staffing sufficiency and overtime needs. Training consists of familiarization with SOP, QC, Safety, and computer systems.

- Encourage the development of analysts to become cross-trained in various methods and/or operate multiple instruments efficiently while performing maintenance and documentation, self-supervise, and function as a department team.
- Provide guidance to analysts in resolving problems encountered daily during sample prep/analysis in conjunction with the Technical Manager, Operations Manager, and/or QA Manager. Each is responsible for 100% of the data review and documentation, non-conformance and *CPAR* issues, the timely and accurate completion of performance evaluation samples and MDLs, for his department.
- Ensure all logbooks are maintained, current, and properly labeled or archived.
- Report all non-conformance conditions to the QA Manager, Technical Manager, Operations Manager, and/or BU / Laboratory Director.
- Ensure that preventive maintenance is performed on instrumentation as detailed in the QA Manual or SOPs. He is responsible for developing and implementing a system for preventive maintenance, troubleshooting, and repairing or arranging for repair of instruments.
- Maintain adequate and valid inventory of reagents, standards, spare parts, and other relevant resources required to perform daily analysis.
- Achieve optimum turnaround time on analyses and compliance with holding times.
- Conduct efficiency and cost control evaluations on an ongoing basis to determine optimization of labor, supplies, overtime, first-run yield, capacity (designed vs. demonstrated), second- and third-generation production techniques/instruments, and long-term needs for budgetary planning.
- Develop, implement, and enhance calibration programs.
- Provide written responses to external and internal audit issues.
- Complying with ISO17025:2017, The NELAC Institute (TNI) Standard, DoD/DOE QSM and the various QC programs implemented at the laboratory.

4.2.12 Laboratory Analysts

Laboratory analysts are responsible for conducting analysis and performing all tasks assigned to them by the group leader or supervisor. The responsibilities of the analysts are listed below:

- Perform analyses by adhering to analytical and quality control protocols prescribed by current SOPs, this QA Manual, and project-specific plans honestly, accurately, timely, safely, and in the most cost-effective manner.
- Document standard and sample preparation, instrument calibration and maintenance, data calculations, sample matrix effects, and any observed non-conformance on worklists, benchsheets, lab notebooks and/or the Non-Conformance Database.
- Report all non-conformance situations, instrument problems, matrix problems and QC failures, which might affect the reliability of the data, to their supervisor, the Technical Manager, and/or the QA Manager or member of QA staff.
- Perform 100% review of the data generated prior to entering and submitting for secondary level review.
- Suggest method improvements to their supervisor, the Technical Manager, and the QA Manager. These improvements, if approved, will be incorporated. Ideas for the optimum

performance of their assigned area, for example, through the proper cleaning and maintenance of the assigned instruments and equipment, are encouraged.

- Work cohesively as a team in their department to achieve the goals of accurate results, optimum turnaround time, cost effectiveness, cleanliness, complete documentation, and personal knowledge of environmental analysis.

4.2.13 Environmental Health and Safety Coordinator and Radiation Safety Officer

The Environmental Health and Safety Coordinator reports directly to the Laboratory Director. Responsibilities include, but are not limited to:

- Staying current with the hazardous waste regulations.
- Continuing training on hazardous waste issues.
- Reviewing and updating annually the Hazardous Waste Contingency Plan.
- Auditing the staff with regard to compliance with the TestAmerica Knoxville Specific Emergency Contingency Plan and the Corporate Environmental Health and Safety Manual (CW-E-M-001).
- Contacting the hazardous waste subcontractors for review of procedures and opportunities for minimization of waste.
- Conducting ongoing, necessary safety training and conduct new employee safety orientation.
- Assisting in developing and maintaining the Chemical Hygiene/Safety Manual.
- Administering dispersal of all Material Safety Data Sheet (MSDS) information.
- Performing regular chemical hygiene and housekeeping instruction.
- Giving instruction on proper labeling and practice; Serving as chairman of the laboratory safety committee.
- Overseeing the inspection and maintenance of general safety equipment – fire extinguishers, safety showers, eyewash fountains, etc. and ensure prompt repairs as needed.
- Supervising and scheduling fire drills and emergency evacuation drills.

As Radiation Safety Officer (RSO), responsibilities include but are not limited to:

- Ensuring that all uses of radioactive materials and radiation sources are conducted safely, in accordance with the conditions of the license and applicable regulations, and result in exposures to personnel which are as low as reasonably achievable (ALARA).

- Apprising and informing management of the radiation safety status of the facility and for keeping them aware of their responsibility in maintaining an adequate radiation protection program.

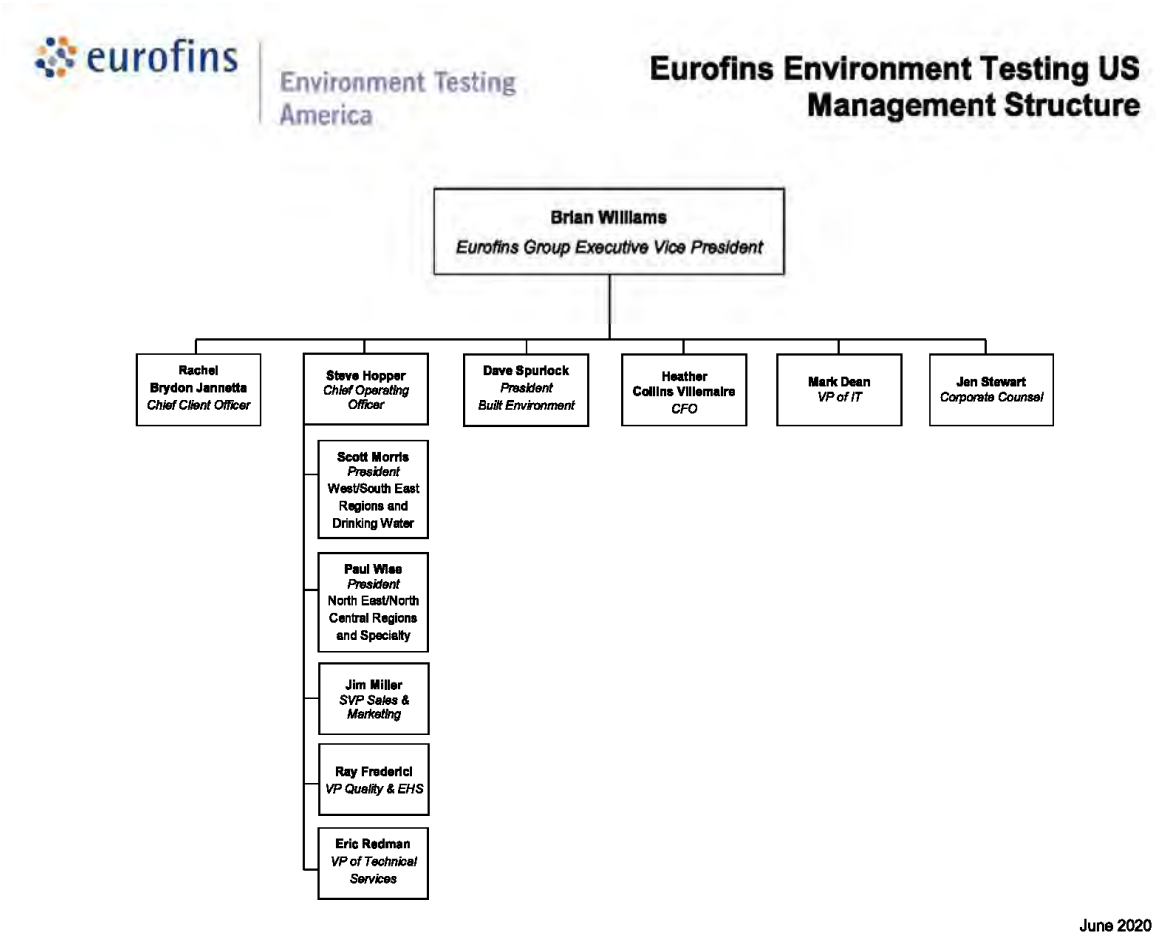
4.3 Business Continuity and Contingency Plans

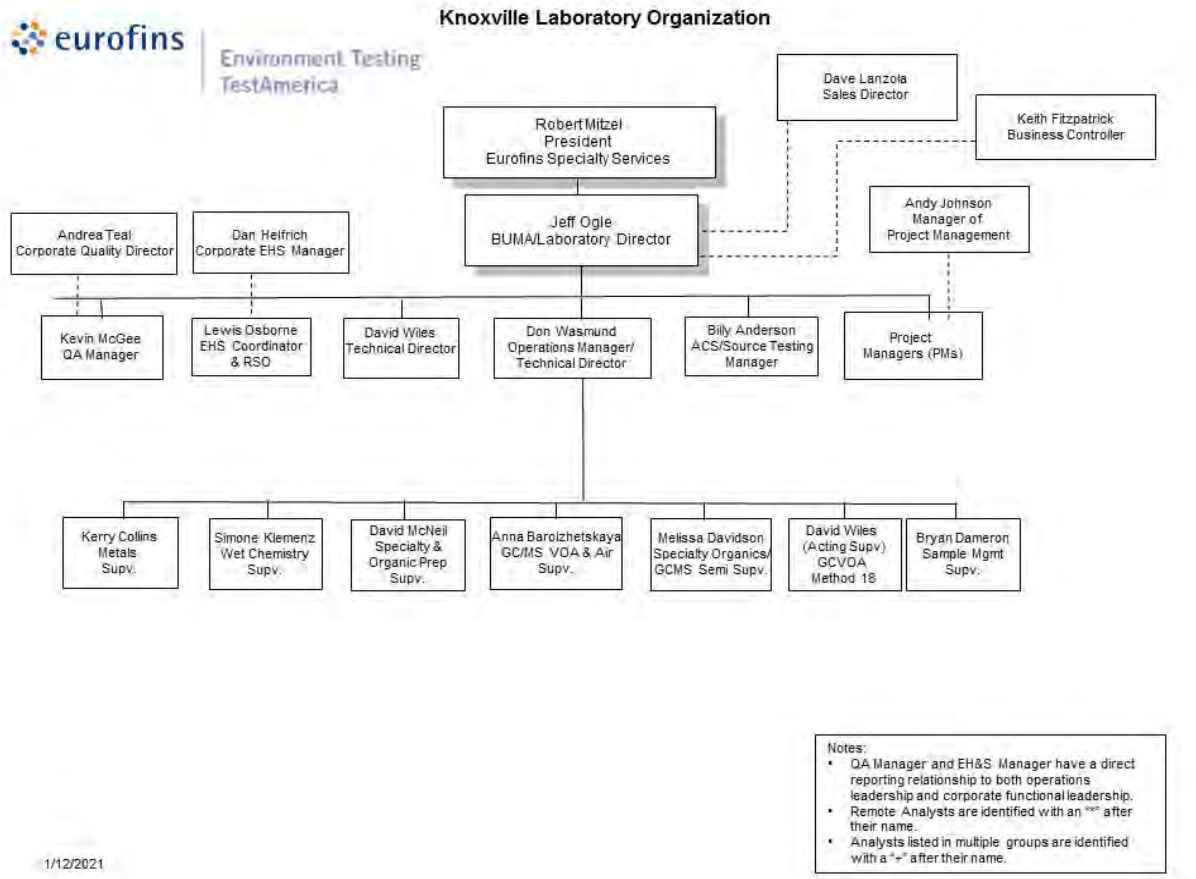
Various policies and practices are in place to address continuity of business and contingency plans to ensure continued operations or minimal disruption in operations should unplanned events (natural disasters, unexpected management changes, etc.) occur. Deputies are identified for all key management personnel. Deputies would temporarily fill a role if the primary is absent for more than 15 consecutive calendar days. The deputies must meet the same qualifications as the primary person should they be required to take on the responsibilities. The QA Manager communicates to the relevant regulatory authorities when there are management or facility changes that impact the laboratory. Changes in the technical director must be communicated within a period of time and in the manner dictated by each regulatory authority.

The following table defines who assumes the responsibilities of key personnel in their absence:

Key Personnel	**Deputy**
Laboratory Director: Jeff Ogle	Don Wasmund
Quality Assurance Manager: Kevin McGee	Annette Ribet
Technical Manager: David Wiles	Melissa Davidson
EHS Coordinator: Lewis Osborne	Kevin McGee
Radiation Safety Officer (RSO): Lewis Osborne	Mike Ridenhower

Figure 4-1. Laboratory Organization Charts




eurofins
Environment Testing
TestAmerica
Knoxville Laboratory Organization
Dave Lanzola
Sales Director
Keith Fitzpatrick
Business Controller
Robert Mitzel
President
Eurofins Specialty Services
Andy Johnson
Manager of
Project Management
Andrea Teal
Corporate Quality Director
Dan Helfrich
Corporate EHS Manager
Jeff Ogle
BUMA/Laboratory Director
Kevin McGee
QA Manager
Lewis Osborne
EHS Coordinator
& RSO
David Wiles
Technical Director
Don Wasmund
Operations Manager/
Technical Director
Billy Anderson
ACS/Source Testing
Manager
Project
Managers (PMs)
Kerry Collins
Metals
Supv.
Simone Klemenz
Wet Chemistry
Supv.
David McNeil
Specialty &
Organic Prep
Supv.
Anna Barolzhetskaya
GC/MS VOA & Air
Supv.
Melissa Davidson
Specialty Organics/
GCMS Semi Supv.
David Wiles
(Acting Supv)
GCVOA
Method 18
Bryan Dameron
Sample Mgmt
Supv.
1/12/2021
Notes:
• QA Manager and EH&S Manager have a direct reporting relationship to both operations leadership and corporate functional leadership.
• Remote Analysts are identified with an "*" after their name.
• Analysts listed in multiple groups are identified with a "+" after their name.

5.0 PERSONNEL

5.1 Overview

The laboratory's management believes that its highly qualified and professional staff is the single most important aspect in assuring a high level of data quality and service. The staff consists of professionals and support personnel.

All personnel must demonstrate competence in the areas where they have responsibility. Any staff that is undergoing training shall have appropriate supervision until they have demonstrated their ability to perform their job function on their own. Staff shall be qualified for their tasks based on appropriate education, training, experience and/or demonstrated skills as required.

The laboratory employs sufficient personnel with the necessary education, training, technical knowledge and experience for their assigned responsibilities.

All personnel are responsible for complying with all QA/QC requirements that pertain to the laboratory and their area of responsibility. Each staff member must have a combination of experience and education to adequately demonstrate a specific knowledge of their particular area of responsibility. Technical staff must also have a general knowledge of lab operations, test methods, QA/QC procedures and records management.

Laboratory management is responsible for formulating goals for lab staff with respect to education, training and skills and ensuring that the laboratory has a policy and procedures for identifying training needs and providing training of personnel. The training shall be relevant to the present and anticipated responsibilities of the lab staff.

The laboratory only uses personnel that are employed by or under contract to, the laboratory. Contracted personnel, when used, must meet competency standards of the laboratory and work in accordance to the laboratory's quality system.

5.2 Education and Experience Requirements for Technical Personnel

The laboratory makes every effort to hire analytical staffs that possess a college degree (AA, BA, BS) in an applied science with some chemistry in the curriculum. Exceptions may be made based upon the technical nature of the position and the individual's experience and abilities. Selection of qualified candidates for laboratory employment begins with documentation of minimum education, training, and experience prerequisites needed to perform the prescribed task. Minimum education and training requirements for laboratory employees are outlined in job descriptions maintained by Eurofins Environment Testing America Human Resources.

Experience and specialized training are occasionally accepted in lieu of a college degree (basic lab skills such as using a balance, colony counting, aseptic or quantitation techniques, etc., are also considered).

As a general rule for analytical staff:

Specialty	Education	Experience
Extractions, Digestions, some electrode methods (pH, DO, Redox, etc.), or Titrimetric and Gravimetric Analyses	H.S. Diploma	On the job training (OJT)

Specialty	Education	Experience
CVAA, Single component or short list Chromatography (e.g., Fuels, BTEX-GC, IC)	A college degree in an applied science or 2 years of college and at least 1 year of college chemistry	Or 2 years prior analytical experience is required
ICP, ICPMS, Long List or complex chromatography (e.g., Pesticides, PCB, Herbicides, HPLC, etc.), GCMS	A college degree in an applied science or 2 years of college chemistry	or 5 years of prior analytical experience
Spectra Interpretation	A college degree in an applied science or 2 years of college chemistry	And 2 years relevant experience Or 5 years of prior analytical experience
Technical Managers – **General**	Bachelor's Degree in an applied science or engineering with 24 semester hours in chemistry An advanced (MS, PhD.) degree may substitute for one year of experience	And 2 years experience in environmental analysis of representative analytes for which they will oversee
Technical Managers – **Wet Chem** only (no advanced instrumentation)	Associates degree in an applied science or engineering or 2 years of college with 16 semester hours in chemistry	And 2 years relevant experience

When an analyst does not meet these requirements, they can perform a task under the direct supervision of a qualified analyst, peer reviewer or Group Leader, and are considered an analyst in training. The person supervising an analyst in training is accountable for the quality of the analytical data and must review and approve data and associated corrective actions.

5.3 Training

The laboratory is committed to furthering the professional and technical development of employees at all levels.

Orientation to the laboratory's policies and procedures, in-house method training, and employee attendance at outside training courses and conferences all contribute toward employee proficiency. Below are examples of various areas of required employee training:

Required Training	Time Frame	Employee Type
Environmental Health & Safety	Prior to lab work	All
Ethics – New Hires	1 week of hire	All
Ethics – Comprehensive	60 days of hire	All

Required Training	Time Frame	Employee Type
Data Integrity	60 days of hire	Technical and PMs
Quality Assurance	90 days of hire	All
Ethics – Comprehensive Refresher	Annually	All
Initial Demonstration of Capability (DOC)	Prior to unsupervised method performance	Technical

The laboratory maintains records of relevant authorization/competence, education, professional qualifications, training, skills and experience of technical personnel (including contracted personnel) as well as the date that approval/authorization was given. These records are kept on file at the laboratory. Also refer to "Demonstration of Capability" in Section 19.

The training of technical staff is kept up to date by:

- Each employee must have documentation in their training file that they have read, understood and agreed to follow the most recent version of the laboratory QA Manual and SOPs in their area of responsibility. This documentation is updated as SOPs are updated.
- Documentation from any training courses or workshops on specific equipment, analytical techniques or other relevant topics.
- Documentation of proficiency (refer to Section 19).
- An Ethics Agreement signed by each staff member (renewed each year) and evidence of annual ethics training.
- A Confidentiality Agreement signed by each staff member signed at the time of employment.
- Human Resources maintains documentation and attestation forms on employment status and records; benefit programs; timekeeping/payroll; and employee conduct (e.g., ethics violations). This information is maintained in the employee's secured personnel file.

Evidence of successful training could include such items as:

- Adequate documentation of training within operational areas, including one-on-one technical training for individual technologies, and particularly for people cross-trained.
- Analysts knowledge to refer to QA Manual for quality issues.
- Analysts following SOPs, i.e., practice matches SOPs.
- Analysts regularly communicate to supervisors and QA if SOPs need revision, rather than waiting for auditors to find problems.

The QA Manager is responsible for QA and Ethics Orientation, Initial and On-Going Ethics and Data Integrity Training and QA training. The EH&S Coordinator is responsible for EH&S Orientation and Training. Group Leaders and Technical Managers are responsible for technical training.

The QA Manager evaluates the effectiveness of training through the review of demonstration of capability documentation, on-going proficiency test results, nonconformances and corrective action reports, and internal and external audit reports.

Further details of the laboratory's training program are described in Personnel Orientation and Training SOP (KNOX-QA-0009).

5.4 Data Integrity and Ethics Training Program

The laboratory's Ethics and Data Integrity Program is discussed in Section 6.2. Employees are trained as to the legal and environmental repercussions that result from data misrepresentation. Key topics covered in the presentation include:

- Organizational mission and its relationship to the critical need for honesty and full disclosure in all analytical reporting.
- Ethics Policy
- How and when to report ethical/data integrity issues. Confidential reporting.
- Record keeping.
- Discussion regarding data integrity procedures.
- Specific examples of breaches of ethical behavior (e.g. peak shaving, altering data or computer clocks, improper macros, etc., accepting/offering kickbacks, illegal accounting practices, unfair competition/collusion)
- Internal monitoring. Investigations and data recalls.
- Consequences for infractions including potential for immediate termination, debarment, or criminal prosecution.
- Importance of proper written narration / data qualification by the analyst and project manager with respect to those cases where the data may still be usable but are in one sense or another partially deficient.

Additionally, a data integrity hotline (1-800-736-9407) is maintained by the NDSC.

6.0 ACCOMMODATIONS AND ENVIRONMENTAL CONDITIONS

6.1 Overview

The laboratory is a 28,000 ft^2 secure laboratory facility with controlled access and designed to accommodate an efficient workflow and to provide a safe and comfortable work environment for employees. All visitors sign in and are escorted by laboratory personnel. Access is controlled by various measures.

The laboratory is equipped with structural safety features. Each employee is familiar with the location, use, and capabilities of general and specialized safety features associated with their workplace. The laboratory provides and requires the use of protective equipment including safety glasses, protective clothing, gloves, etc., OSHA and other regulatory agency guidelines regarding required amounts of bench and fume hood space, lighting, ventilation (temperature and humidity controlled), access, and safety equipment are met or exceeded.

Traffic flow through sample preparation and analysis areas is minimized to reduce the likelihood of contamination. Adequate floor space and bench top area is provided to allow unencumbered sample preparation and analysis space. Sufficient space is also provided for storage of reagents and media, glassware, and portable equipment. Ample space is also provided for refrigerated sample storage before analysis and archival storage of samples after analysis. Laboratory HVAC and deionized water systems are designed to minimize potential trace contaminants.

The laboratory is separated into specific areas for sample receiving, sample preparation, volatile organic sample analysis, non-volatile organic sample analysis, specialty organics sample preparation, specialty organics analysis, metals sample preparation, metals analysis, inorganic sample analysis, and administrative functions.

6.2 Environment

Laboratory accommodation, test areas, energy sources, and lighting are adequate to facilitate proper performance of tests. The facility is equipped with heating, ventilation, and air conditioning (HVAC) systems appropriate to the needs of environmental testing performed at this laboratory.

The environment in which these activities are undertaken does not invalidate the results or adversely affect the required accuracy of any measurements.

The laboratory provides for the effective monitoring, control and recording of environmental conditions that may affect the results of environmental tests as required by the relevant specifications, methods, and procedures. Such environmental conditions include humidity controls, temperature controls, and voltage conditioning with an uninterruptible backup power supply.

When any of the method or regulatory required environmental conditions change to a point where they may adversely affect test results, analytical testing will be discontinued until the environmental conditions are returned to the required levels.

Environmental conditions of the facility housing the computer network and LIMS are regulated to protect against raw data loss.

When the laboratory performs laboratory activities at sites or facilities outside its permanent control, it shall ensure that the requirements related to facilities and environmental conditions of this document are met.

Specific requirements for facility and environmental conditions, as well as periodic monitoring of conditions, are given in the Environmental Health & Safety Manual plus each laboratory's Facility Addendum.

6.3 Work Areas

There is effective separation between neighboring areas when the activities therein are incompatible with each other. Examples include:

- Volatile organic chemical handling areas, including sample preparation and waste disposal, and volatile organic chemical analysis areas.

Access to and use of all areas affecting the quality of analytical testing is defined and controlled by secure access to the laboratory building as described below in the Building Security section.

Adequate measures are taken to ensure good housekeeping in the laboratory and to ensure that any contamination does not adversely affect data quality. These measures include regular cleaning to control dirt and dust within the laboratory.

Work areas are available to ensure an unencumbered work area. Work areas include:

- Access and entryways to the laboratory.
- Sample receipt areas.
- Sample storage areas.
- Chemical and waste storage areas.
- Data handling and storage areas.
- Sample processing areas.
- Sample analysis areas.

6.4 Responding to Emergencies

Employees must be aware of procedures to respond to all emergencies that might occur in the workplace. Employees must be familiar with the location and proper operation of all emergency equipment, evacuation routes and designated assembly areas for all areas where they work. Refer to the NDSC EH&S Manual Document No. CW-E-M-001. Sec. 7 and the laboratory's local EH&S addendum for complete details. These documents provide direction for situations where normal operations of the laboratory are not possible (e.g., electrical failures, heating/air conditioning failures, fire/building evacuation, computer failures, hazardous material spills, injury to employees, pandemic flu, disruption of phone service, etc.)

In the event that the building or information technology (IT) systems would be severely challenged, a designated disaster recovery team, which includes Facility Management, Maintenance, Safety, Laboratory/Executive Management, Public Relations, IT, QA and other applicable personnel depending on the scope of the disaster, would assemble at a designated area to assess the situation and formulate a plan.

6.5 Building Security

Building access codes and alarm codes are distributed to employees as necessary.

Visitors to the laboratory sign in and out in a visitor's logbook. A visitor is defined as any person who visits the laboratory who is not an employee of the laboratory. In addition to signing into the laboratory, the Environmental, Health and Safety Manual contains requirements for visitors and vendors. There are specific safety forms that must be reviewed and signed.

Visitors (with the exception of company employees) are escorted by laboratory personnel at all times, or the location of the visitor is noted in the visitor's logbook.

7.0 QUALITY SYSTEM

7.1 Quality Policy Statement

The Quality Policy statement gives employees clear requirements for the production of analytical data. As an organization, all personnel are committed to high quality professional practice, testing and data, and service to our clients.

We strive to provide the highest quality data achievable by:

- Reading and understanding all of the quality documents applicable to each position and implementing the process in our work.
- Following all recordkeeping requirements; describing clearly and accurately all activities performed; recording “real time” as the task is carried out; understanding that it is never acceptable to “back date” entries and should additional information be required at a later date, the actual date and by whom the notation is made must be documented.
- Ensuring data integrity through the completeness, consistency, impartiality and accuracy of the data generated. Data is attributable, legible, contemporaneously recorded, original or a true copy, and accurate (ALCOA). This applies to manual paper documentation and electronic records.
- Providing accountability and traceability for each sample analyzed through proper sample handling, labeling, preparation, instrument calibration/qualification/validation, analysis, and reporting; establishing an audit trail (the who, what, when, and why) that identifies date, time, analyst, instrument used, instrument conditions, quality control samples (where appropriate and/or required by the method), and associated standard material.
- Emphasizing a total quality management process which provides impartiality, accuracy, and strict compliance with agency regulations and client requirements, giving the highest degree of confidence; understanding that meeting the requirements of the next employee in the work flow process is just as important as meeting the needs of the external client.
- Providing thorough documentation and explanation to qualify reported data that may not meet all requirements and specifications, but is still of use to the client; understanding this occurs only after discussion with the client on the data limitations and acceptability of this approach.
- Responding immediately to indications of questionable data, out-of-specification occurrences, equipment malfunctions, and other types of laboratory problems, with investigation and applicable corrective action; documenting these activities completely, including the reasons for the decisions made.
- Providing a work environment that ensures accessibility to all levels of management and encourages questions and expression of concerns on quality issues to management. Eurofins recognizes that the implementation of a quality assurance program requires management’s commitment and support as well as the involvement of the entire staff
- Continually improve systems and manage risk to support quality improvement efforts in laboratory, administrative and managerial activities

7.2 Ethics and Data Integrity

Eurofins Environment Testing America is committed to ensuring the integrity of its data and meeting the quality needs of its clients. The laboratory operates our Ethics and Data Integrity program under the guidance of Eurofin's Key Guidance Document (KGD). The elements of the our program include:

- An Ethics Policy (NDSC Document No. CW-L-P-004;) and Employee Ethics Statements.
- Ethics and Compliance Officer/s (ECOs).
- A Training Program.
- Self-governance through disciplinary action for violations.
- A confidential mechanism for anonymously reporting alleged misconduct and a means for conducting internal investigations of all alleged misconduct. (NDSC Document No. CW-L-S-002,
- Procedures and guidance for recalling data if necessary (NDSC Document No. CW-Q-S-005).
- Effective external and internal monitoring system that includes procedures for internal audits (Section 17).
- Produce results, which are accurate and include QA/QC information that meets client pre-defined Data Quality Objectives (DQOs).
- Present services in a confidential, honest and forthright manner.
- Provide employees with guidelines and an understanding of the Ethical and Quality Standards of our Industry.
- Provide procedures and guidance to ensure the impartiality and confidentiality of all data and customer information.
- Operate our facilities in a manner that protects the environment and the health and safety of employees and the public.
- Obey all pertinent federal, state and local laws and regulations and encourage other members of our industry to do the same.
- Educate clients as to the extent and kinds of services available.
- Assert competency only for work for which adequate personnel and equipment are available and for which adequate preparation has been made.
- Promote the status of environmental laboratories, their employees, and the value of services rendered by them.

7.3 Quality System Documentation

The laboratory's Quality System is communicated through a variety of documents.

- Quality Assurance (QA) Manual – Each laboratory has a lab-specific QA manual.
- NDSC Official Documents – Each laboratory may use the Guidance (instructional use) documents at their discretion. Template documents are process documents that the laboratory's need to implement locally by using the document as is or as an outline to define their internal practices that meet the minimum requirements of the template. Required documents need to be implemented as is and listed in the laboratory's document control list.

- Key Guidance Documents (KGDs) - Documents compiled at the Group Service Centre (GSC) level by Functional Leaders (document owners) aimed at providing specific Eurofins groups of employees with guidelines necessary for the good conduct of their respective work.
- Laboratory SOPs and Policies – General and Technical
- Work Instructions – A subset of procedural steps, tasks or forms associated with an operation of a management system (e.g., checklists, preformatted bench sheets, forms).

7.3.1 Order of Precedence

In the event of a conflict or discrepancy between policies, the order of precedence is as follows:

- Quality Management Plan (QMP)
- NDSC Guidance Documents
- KGDs
- Laboratory Quality Assurance Manual (QA Manual)
- Laboratory SOPs and Policies
- Other (Work Instructions (WI), memos, flow charts, etc.)

NOTE: The laboratory has the responsibility and authority to operate in compliance with regulatory requirements of the jurisdiction in which the work is performed. Where the QMP conflicts with those regulatory requirements, the regulatory requirements of the jurisdiction shall hold primacy. The laboratory's QA Manual shall take precedence over the QMP in those cases. Any regulatory requirements (e.g.; Ohio EPA VAP, CT RCP, etc) provided in the laboratory specific documents (i.e., QAM and SOPs) take precedence over any policies provided in corporate documents.

7.4 QA/QC Objectives for the Measurement of Data

Quality Assurance (QA) is responsible for developing planned activities whose purpose is to provide assurance to all levels of management that a quality program is in place within the laboratory, and that it is functioning in an effective manner that is consistent with the requirements of NELAP, ISO 17025, DoD, and any other regulatory agencies (i.e., states) in which the laboratory maintains accreditation.

Quality Control (QC) is generally understood to be limited to the analyses of samples and to be synonymous with the term *"analytical quality control"*. QC refers to the routine application of statistically based procedures to evaluate and control the accuracy of results from analytical measurements. The QC program includes procedures for estimating and controlling precision and bias and for determining reporting limits.

Request for Proposals (RFPs) and Quality Assurance Project Plans (QAPP) provide a mechanism for the client and the laboratory to discuss the data quality objectives in order to ensure that analytical services closely correspond to client needs. In order to ensure the ability of the laboratory to meet the Data Quality Objectives (DQOs) specified in the QAPP, clients are advised to allow time for the laboratory to review the QAPP before being finalized. The client is responsible for developing the QAPP; however, the laboratory will provide support to the client for developing the sections of the QAPP that concern laboratory activities.

Note: TestAmerica Knoxville must comply with the information provided in its VAP approved QAM and SOPs.

Historically, laboratories have described their QC objectives in terms of precision, accuracy, representativeness, comparability, completeness, selectivity and sensitivity (PARCCSS). Each laboratory SOP defines the required QC indicators.

7.4.1 Precision

The objective is to meet the performance for precision demonstrated for the methods on similar samples and to meet DQOs of the EPA and/or other regulatory programs. Precision is defined as the degree of reproducibility of measurements under a given set of analytical conditions (exclusive of field sampling variability). Precision is documented on the basis of replicate analysis, usually duplicate or matrix spike (MS) duplicate samples.

7.4.2 Accuracy

The objective is to meet the performance for accuracy demonstrated for the methods on similar samples and to meet DQOs of the EPA and/or other regulatory programs. Accuracy is defined as the degree of bias in a measurement system. Accuracy may be documented through the use of laboratory control samples (LCS) and/or MS. A statement of accuracy is expressed as an interval of acceptance recovery about the mean recovery.

7.4.3 Representativeness

The objective is to provide data which is representative of the sampled medium. Representativeness is defined as the degree to which data represent a characteristic of a population or set of samples and is a measurement of both analytical and field sampling precision. The representativeness of the analytical data is a function of the procedures used in procuring and processing the samples. The representativeness can be documented by the relative percent difference between separately procured, but otherwise identical samples or sample aliquots.

The representativeness of the data from the sampling sites depends on both the sampling procedures and the analytical procedures. Refer to laboratory SOPs for subsampling and homogenization techniques appropriate to the analytical method.

7.4.4 Comparability

The objective is to provide analytical data for which the accuracy, precision, representativeness, and reporting limit statistics are similar to these quality indicators generated by other laboratories for similar samples, and data generated by the laboratory over time.

Comparability is documented by inter-laboratory studies carried out by regulatory agencies or carried out for specific projects or contracts, by comparison of periodically generated statements of accuracy, precision, and reporting limits with those of other laboratories.

7.4.5 Completeness

The completeness objective for data is 90% (or as specified by a particular project), expressed as the ratio of the valid data to the total data over the course of the project. Data will be considered valid if they are adequate for their intended use. Data usability will be defined in a QAPP, project scope, or regulatory requirement. Data validation is the process for reviewing

data to determine its usability and completeness. If the completeness objective is not met, actions will be taken internally and with the data user to improve performance. This may take the form of an audit to evaluate the methodology and procedures as possible sources for the difficulty or may result in a recommendation to use a different method.

7.4.6 Selectivity

Selectivity is defined as the capability of a test method or instrument to respond to a target substance or constituent in the presence of non-target substances. Target analytes are separated from non-target constituents and subsequently identified/detected through one or more of the following, depending on the analytical method: extractions (separation), digestions (separation), interelement corrections (separation), use of matrix modifiers (separation), specific retention times (separation and identification), confirmations with different columns or detectors (separation and identification), specific wavelengths (identification), specific mass spectra (identification), and specific electrodes (separation and identification).

7.4.7 Sensitivity

Sensitivity refers to the amount of analyte necessary to produce a detector response that can be reliably detected (above the Method Detection Limit, Estimated Detection Limit, or Limit of Detection) or quantified (above the Reporting Limit, Minimum Level, or Limit of Quantitation).

7.5 Criteria for Quality Indicators

The laboratory can prepare upon request a Quality Control Limit Summary from the Laboratory Information Management System (LIMS) that summarize the precision and accuracy acceptability limits for performed analyses. This summary includes an effective date, is updated each time new limits are generated and is located in the LIMS. Current limits are controlled through the LIMS. The limits in effect for a given date are archived in the LIMS with the associated sample data. Unless otherwise noted, limits within these tables are laboratory generated. Some acceptability limits are derived from US EPA methods when they are required. Where US EPA method limits are not required, the laboratory has developed limits from evaluation of data from similar matrices. Criteria for development of control limits is contained in SOP KNOX-QA-0004.

7.6 Statistical Quality Control

Statistically-derived precision and accuracy limits are required by selected methods (such as SW-846) and programs. The laboratory routinely utilizes statistically-derived limits to evaluate method performance and determine when corrective action is appropriate. The laboratory analysts are instructed to use the current limits in the laboratory (dated and approved by the Technical Manager/Group Leader and QA Manager) and entered into TALS. The Quality Assurance department maintains an archive of all limits used within the laboratory. These limits are maintained in the LIMS as part of the analytical historical record. If a method defines the QC limits, the method limits are used.

If a method requires the generation of historical limits, the lab develops such limits from recent data in the QC database of the LIMS following the guidelines described in Section 26. All calculations and limits are documented and dated when approved and effective. On occasion, a client requests contract-specified limits for a specific project.

Current QC limits are entered and maintained in TALS analyte database. As sample results and the related QC are entered into TALS, the sample QC values are compared with the limits

in TALS to determine if they are within the acceptable range. The laboratory analyst then evaluates if the sample needs to be rerun or re-extracted/rerun or if a comment should be added to the report explaining the reason for the QC outlier.

7.6.1 QC Charts

As the QC limits are calculated, QC charts are generated showing warning and control limits for the purpose of evaluating trends. The QA Manager evaluates these to determine if adjustments need to be made or for corrective actions to methods. All findings are documented and kept on file. The generation and use of QC Charts (Control Charts) are described in the laboratory SOP KNOX-QA-0004.

7.7 Quality System Metrics

In addition to the QC parameters discussed above, the entire Quality System is evaluated on a monthly basis through the use of specific metrics (refer to Section 18). These metrics are used to drive continuous improvement in the laboratory's Quality System.

8.0 DOCUMENT CONTROL

8.1 Overview

The QA Department is responsible for the control of documents used in the laboratory to ensure that approved, up-to-date documents are in circulation and out-of-date (obsolete) documents are archived or destroyed. The following documents, at a minimum, must be controlled:

- Laboratory Quality Assurance (QA) Manual
- Laboratory Standard Operating Procedures (SOP)
- Laboratory Policies
- Work Instructions and Forms
- NDSC Documents[1]

[1]Includes locally implemented documents that are document controlled within the laboratory's document control system.

The NDSC documents are only considered controlled when they are read on the intranet site. Printed copies are considered uncontrolled unless the laboratory physically distributes them as controlled documents. A detailed description of the procedure for issuing, authorizing, controlling, distributing, and archiving NDSC Official Documents is found in Document No. CW-Q-S-001, NDSC Document Control and Archiving. The laboratory's internal document control procedure is defined in KNOX-QA-0011, Document Control and Distribution.

The laboratory QA Department also maintains access to various references and document sources integral to the operation of the laboratory. This includes reference methods and regulations. Instrument manuals (hard or electronic copies) are also maintained by the laboratory.

The laboratory maintains control of records for raw analytical data and supporting records such as audit reports and responses, logbooks, standard logs, training files, MDL studies, Proficiency Testing (PT) studies, certifications and related correspondence, and nonconformance reports (NCMs). Raw analytical data consists of bound logbooks, instrument printouts, any other notes, magnetic media, electronic data, and final reports.

8.2 Document Approval and Issue

The pertinent elements of the document control system includes a unique document title and number, pagination, the total number of pages of the item or an 'end of document' page, the effective date, revision number, and the laboratory's name. The QA personnel are responsible for the maintenance of this system.

Controlled documents are authorized by the QA Department and other laboratory management. In order to develop a new document, a responsible manager submits a draft to the QA Department by way of the group leader/Technical Manager. Upon approval, QA personnel add the identifying version information to the document and retains that document as the official document on file. That document is then provided to all applicable operational units (may include electronic access). Controlled documents are identified as such and records of their distribution are kept by the QA Department. Document control may be achieved by either electronic or hardcopy distribution.

The QA Department maintains a list of the official versions of controlled documents.

Quality System Policies and Procedures are reviewed at a minimum of every two years and revised as appropriate. Technical procedures used in support of work done for the Department of Defense (DOD) and Department of Energy (DOE), and the Eurofins TestAmerica Knoxville Quality Assurance Manual must be reviewed at a one year frequency. Changes to documents occur when a procedural change warrants a revision of the document.

8.3 Procedures for Document Control Policy

For changes to the QAM, requests for change or revision may be made to the TestAmerica QA Manager. Changes affecting the text of the Corporate Quality Management Plan are forwarded to the Corporate QA Director for review and approval. If the proposed changes are accepted, they are incorporated into the next revision of the appropriate section of the QAM. Each revision to the QAM is approved by the same authorized signatories as shown on the cover page of the QAM. Uncontrolled copies must not be used within the laboratory. Previous revisions are archived by the QA department.

For changes to SOPs, refer to SOP No. KNOX-QA-0018, Preparation and Management of Standard Operating Procedures.

Forms, worksheets, work instructions and information are organized by form number and revision number on the QAQC shared directory on a local area network server.

8.4 Obsolete Documents

All invalid or obsolete documents are removed, or otherwise prevented from unintended use. The laboratory has specific procedures as described above to accomplish this. In general, obsolete documents are collected from employees according to distribution lists and are marked obsolete on the cover or destroyed. At least one copy of the obsolete document is archived according to KNOX-QA-0011, Document Control and Distribution.

9.0 SERVICE TO THE CLIENT

9.1 Overview

The laboratory has established procedures for the review of work requests and contracts, oral or written. The procedures include evaluation of the laboratory's capability and resources to meet the contract's requirements within the requested time period. All requirements, including the methods to be used, must be adequately defined, documented and understood. For many environmental sampling and analysis programs, testing design is site or program specific and does not necessarily fit into a standard laboratory service or product. It is the laboratory's intent to provide both standard and customized environmental laboratory services to our clients.

A thorough review of technical and QC requirements contained in contracts is performed to ensure project success. The appropriateness of requested methods, and the lab's capability to perform them must be established. Projects, proposals, and contracts are reviewed for adequately defined requirements and the laboratory's capability to meet those requirements. Alternate test methods that are capable of meeting the clients' requirements may be proposed by the lab. A review of the lab's capability to analyze non-routine analytes is also part of this review process.

All projects, proposals and contracts are reviewed for the client's requirements in terms of compound lists, test methodology requested, sensitivity (detection and reporting levels), accuracy, and precision requirements (% Recovery and RPD). The reviewer ensures that the laboratory's test methods are suitable to achieve these requirements and that the laboratory holds the appropriate certifications and approvals to perform the work. The laboratory and any potential subcontract laboratories must be certified, as required, for all proposed tests.

Electronic or hard copy deliverable requirements are evaluated against the laboratory's capacity for production of the documentation.

If the laboratory cannot provide all services but intends to subcontract such services, whether to another Eurofins facility on the same LIMS or to an outside firm, this will be documented and discussed with the client prior to contract approval. (Refer to Section 10 for Subcontracting Procedures.)

The laboratory informs the client of the results of the review if it indicates any potential conflict, deficiency, lack of accreditation, or inability of the lab to complete the work satisfactorily. Any discrepancy between the client's requirements and the laboratory's capability to meet those requirements is resolved in writing before acceptance of the contract. It is necessary that the contract be acceptable to both the laboratory and the client. Amendments initiated by the client and/or Eurofins TestAmerica Knoxville are documented in writing.

All contracts, QAPPs, Sampling and Analysis Plans (SAPs), contract amendments, and documented communications become part of the project record.

The same contract review process used for the initial review is repeated when there are amendments to the original contract by the client, and the participating personnel are informed of the changes.

9.2 Review Sequence and Key Personnel

Appropriate personnel will review the work request at each stage of evaluation.

For routine projects and other simple tasks, a review by the Project Manager (PM) is considered adequate. The PM confirms that the laboratory has any required certifications, that it can meet the clients' data quality and reporting requirements and that the lab has the capacity to meet the clients turn around needs.

It is recommended that, where there is a sales person assigned to the account, an attempt should be made to contact that sales person to inform them of the incoming samples.

For new, complex or large projects, the proposed contract is given to the Client Relationship Manager or Proposal Team, who will decide which lab will receive the work based on the scope of work and other requirements, including certification, testing methodology, and available capacity to perform the work. The contract review process is outlined in NDSC Document No. CA-L-P-002**,** Contract Compliance Policy.

This review encompasses all facets of the operation. The scope of work is distributed to the appropriate personnel, as needed based on scope of contract, to evaluate all of the requirements shown above (not necessarily in the order below) Contract Administrator

- Laboratory Project Manager
- Laboratory Directors and/or Technical Managers
- Account Executives
- Quality Managers
- Laboratory Environmental Health and Safety Managers/Directors

The Laboratory Director reviews the formal laboratory quote and makes final acceptance for their facility.

The Sales Director, Contract Administrator, Account Executive or Proposal Coordinator then submits the final proposal to the client.

In the event that one of the above personnel is not available to review the contract, his or her back-up will fulfill the review requirements.

9.3 Balancing Laboratory Capacity and Workload

Evaluating laboratory capacity to perform specific projects is the responsibility of the Vice-President, Laboratory Director and Operations Manager. Many analysts are cross-trained to perform a variety of tests, and there is redundant equipment available in case of malfunctions. This minimizes the need to evaluate small and medium size projects against capacity available to complete them. Large and complex projects are reviewed against capacity estimates before bids are submitted to ensure that the client's analysis schedule is met. Regularly scheduled meetings are held between laboratory management, PMs, Client Services and QA personnel to review progress with current projects, as well as special requirements of new work scheduled for the laboratory. Laboratory capacity and backlog is tracked on a continuous basis using information from the Laboratory Sample Information System (LIMS) including turnaround time, and work in-house.

9.4 Documentation

The Contracts Department maintains copies of all signed contracts

Appropriate records are maintained for every contract or work request. All stages of the contract review process are documented and include records of any significant changes. This information is archived in the project files

The contract is distributed to and maintained by the appropriate sales/marketing personnel and the Account Executive. A copy of the contract and formal quote will be filed with the laboratory PM and the Laboratory Director.

Records are maintained of pertinent discussions with a client relating to the client's requirements or the results of the work during the period of execution of the contract. The PM keeps a phone log of conversations with the client. This information is archived in the project file as appropriate.

9.4.1 Project-Specific Quality Planning

Communication of contract specific technical and QC criteria is an essential activity in ensuring the success of site specific testing programs. To achieve this goal, a PM is assigned to each client. It is the PM's responsibility to ensure that project-specific technical and QC requirements are effectively evaluated and communicated to the laboratory personnel before and during the project. QA department involvement may be needed to assist in the evaluation of custom QC requirements.

PM's are the primary client contact and they ensure resources are available to meet project requirements, they coordinate opportunities and work with laboratory management and supervisory staff to ensure available resources are sufficient to perform work for the client's project.

Prior to work on a new project, the dissemination of project information and/or project opening meetings may occur to discuss schedules and unique aspects of the project. Items to be discussed may include the project technical profile, turnaround times, holding times, methods, analyte lists, reporting limits, deliverables, sample hazards, or other special requirements. The PM introduces new project information to the laboratory staff through project kick-off meetings or to the supervisory staff during production meetings. These meetings provide direction to the laboratory staff in order to maximize production and client satisfaction, while maintaining quality.

Any change that may occur within an active project is agreed upon between the client/regulatory agency and the PM/laboratory. These changes (e.g., use of a non-standard method or modification of a method) and approvals must be documented prior to implementation. Documentation pertains to any document (e.g., letter, e-mail, variance, contract addendum), which has been signed by both parties.

Project specific instructions are communicated to the laboratory through the use of notes, comments and attachments in TALS or by reference to a Quality Assurance Summary.

The laboratory strongly encourages client visits to the laboratory and for formal/informal information sharing session with employees in order to effectively communicate ongoing client needs as well as project specific details for customized testing programs.

9.5 Special Services

The laboratory cooperates with clients and their representatives to monitor the laboratory's performance in relation to work performed for the client. It is the laboratory's goal to meet all client requirements in addition to statutory and regulatory requirements. The laboratory has procedures to ensure confidentiality to clients (Section 17 and 25).

The laboratory's standard procedures for reporting data are described in Section 25. Special services are also available and provided upon request. These services include:

- Reasonable access for our clients or their representatives to the relevant areas of the laboratory for the witnessing of tests performed for the client.
- Assisting client-specified third party data validators as specified in the client's contract.
- Supplemental information pertaining to the analysis of their samples. Note: An additional charge may apply for additional data/information that was not requested prior to the time of sample analysis or previously agreed upon.

When the client requests a statement of conformity to a specification or standard based on the analysis performed by the laboratory (e.g., pass/fail, in-tolerance/out-of-tolerance), the decision rule shall be clearly defined. Unless inherent in the requested specification or standard, the decision rule selected shall be communicated to the client. Associated reporting requirements are addressed in Section 25.2.18.

9.6 Client Communication

PMs are the primary communication link to the clients. They shall inform their clients of any delays in project completion as well as any non-conformances in either sample receipt or sample analysis. Project management will maintain ongoing client communication throughout the entire client project.

Technical Managers are available to discuss any technical questions or concerns that the client may have.

9.7 Reporting

The laboratory works with our clients to produce any special communication reports required by the contract.

9.8 Client Feedback and Surveys

The laboratory assesses both positive and negative client feedback. The results are used to improve overall laboratory quality and client service.

Eurofins Sales and Marketing teams periodically develop lab and client specific surveys to assess client satisfaction.

When a complaint is received, we determine, to the best of our ability, the extent of the issue and what data is in question. The person receiving the complaint documents this information and promptly forwards it to the appropriate management personnel where the work in question was performed. If a data reporting error is discovered, the final report and/or data must be regenerated with the correct value(s).

The project manager or QA is responsible for entering client concerns into iCAT In some cases, an ICAT / ICAR (Investigation and Corrective Action Report) is initiated to address and document the situation. While an individual issue may not warrant a formal investigation, QA monitors these issues for potential trends and will issue an ICAR/ICAT if a trend is evident.

10.0 SUBCONTRACTING OF TESTS

10.1 Overview

For the purpose of this quality manual, the phrase subcontract laboratory refers to a laboratory external to the Eurofins TestAmerica laboratories. The phrase “work sharing” refers to internal transfers of samples between the Eurofins TestAmerica laboratories. The term outsourcing refers to the act of subcontracting tests.

When contracting with our clients, the laboratory makes commitments regarding the services to be performed and the data quality for the results to be generated. When the need arises to outsource testing for our clients because project scope, changes in laboratory capabilities, capacity, or unforeseen circumstances, we must be assured that the subcontractors or work sharing laboratories understand the requirements and will meet the same commitments we have made to the client. Refer to the <NDSC Documents on Subcontracting Procedures (CW-L-S-004) and the Work Sharing Process (CA-C-S-001).

When outsourcing analytical services, the laboratory will assure, to the extent necessary, that the subcontract or work sharing laboratory maintains a program consistent with the requirements of this document, the requirements specified in TNI/ISO 17025 and/or the client’s Quality Assurance Project Plan (QAPP). All QC guidelines specific to the client’s analytical program are transmitted to the subcontractor and agreed upon before sending the samples to the subcontract facility. Additionally, work requiring accreditation will be placed with an appropriately accredited laboratory. The laboratory performing the subcontracted work will be identified in the final report, as will non-TNI accredited work where required.

PMs or other responsible Client Service members, for the Export Lab (i.e., the Eurofins TestAmerica laboratory that transfers samples to another laboratory) are responsible for obtaining client approval prior to subcontracting any samples. The laboratory will advise the client of a subcontract arrangement in writing and when possible approval from the client shall be obtained and retained in the project folder. Standard Eurofins TestAmerica Terms & Conditions include the flexibility to subcontract samples within the Eurofins TestAmerica laboratories. Therefore, additional advance notification to clients for intra-laboratory subcontracting is not necessary unless specifically required by a client contract.

Note: In addition to the client, some regulating agencies (e.g., USDA) or contracts (e.g., DoD and DOE projects) require notification prior to placing such work**.** Unless the work is done directly for the USDA, this notification is made to the client who is responsible for ensuring notification of the regulating agency.

10.2 Qualifying and Monitoring Subcontractors

Whenever a PM or Client Relationship Manager becomes aware of a client requirement or laboratory need where samples must be outsourced to another laboratory, the other laboratory(s) shall be selected based on the following:

- Subcontractors specified by the client - In these circumstances, the client assumes responsibility for the quality of the data generated from the use of a subcontractor.
- Subcontractors reviewed by Eurofins TestAmerica Knoxville – Firms which have been reviewed by the company and are known to meet standards for accreditations (e.g., State,

TNI and DoD/DOE); technical specifications; legal and financial information.

A listing of vendors is available on the Eurofins TestAmerica intranet site.

All Eurofins TestAmerica laboratories are pre-qualified for work sharing provided they hold the appropriate accreditations and can adhere to the project/program requirements. Client approval is not necessary unless specifically required by the contract. In these cases, the client must provide acknowledgement that the samples can be sent to that facility (an e-mail is sufficient documentation or if acknowledgement is verbal, the date, time, and name of person providing acknowledgement must be documented). The originating laboratory is responsible for communicating all technical, quality, and deliverable requirements as well as other contract needs. (NDSC Document No. CA-C-S-001, Work Sharing).

10.2.1 When the potential subcontract laboratory has not been previously approved, PMs may nominate a laboratory as a subcontractor based on need. The decision to nominate a laboratory must be approved by the Laboratory Director. The Laboratory Director requests that the PM begin the process of approving the subcontract laboratory. Refer to the NDSC Document No. CW-L-S-004, Subcontracting Procedures

Once the appropriate accreditation and legal information is received by the laboratory, it is evaluated for acceptability and forwarded to the NDSC Quality Information Manager (QIM) for review. After the NDSC QIM reviews the documents for completeness, the information is forwarded to the Finance Department for formal signature and contracting with the laboratory. The approved vendor will be added to the approved subcontractor list on the intranet site, and the finance group is concurrently notified.

The client will assume responsibility for the quality of the data generated from the use of a subcontractor they have requested the lab to use. The qualified subcontractors on the intranet site are known to meet minimal standards. Eurofins TestAmerica does not certify laboratories. The subcontractors on our approved list can only be recommended to the extent that we would use them.

10.3 Oversight and Reporting

The status and performance of qualified subcontractors will be monitored by NDSC and includes an annual review process (ref.: NDSC Document No. SOP CW-L-S-004). Any problems identified will be brought to the attention of NDSC.

- Complaints shall be investigated. Documentation of the complaint, investigation, and corrective action will be maintained in the subcontractor's file on the intranet site. Complaints are posted using the Vendor Performance Report.
- Information shall be updated on the intranet when new information is received from the subcontracted laboratories.
- Subcontractors in good standing will be retained on the intranet listing. Client Service personnel will notify all Eurofins Testamerica laboratories, NDSC, and Corporate Contracts if any laboratory requires removal from the intranet site. This notification will be posted on the intranet site and e-mailed to all Client Service Personnel, Laboratory Directors, QA Managers, and Sales Personnel.

Prior to initially sending samples to the subcontracted laboratory, the PM confirms their certification status to determine if it's current and scope-inclusive. The information is documented within the project records.

The laboratory's certifications can be viewed on the company's website http://www.testamericainc.com.

10.3.1 All subcontracted samples must be accompanied by a Eurofins TestAmerica Chain of Custody (COC). A copy of the original COC sent by the client must be available in LIMS for all samples workshared within Eurofins TestAmerica. Client COCs are only forwarded to external subcontractors when samples are shipped directly from the project site to the subcontractor lab. Under routine circumstances, client COCs are not provided to external subcontractors.

Through communication with the subcontracted laboratory, the PM monitors the status of the subcontracted analyses, facilitates successful execution of the work, and ensures the timeliness and completeness of the analytical report.

Non-TNI accredited work must be identified in the subcontractor's report as appropriate. If TNI accreditation is not required, the report does not need to include this information.

Reports submitted from subcontractor laboratories are not altered and are included in their original form in the final project report. This clearly identifies the data as being produced by a subcontractor facility. If subcontract laboratory data is incorporated into the laboratory's EDD (i.e., imported), the report must explicitly indicate which lab produced the data for which methods and samples.

Note: The results submitted by a Eurofins TestAmerica work sharing laboratory may be transferred electronically and the results reported by the Eurofins TestAmerica work sharing lab are identified on the final report. The report must explicitly indicate which lab produced the data for which methods and samples. The final report must include a copy of the completed COC for all work sharing reports.

10.4 Contingency Planning

The full qualification of a subcontractor may be waived to meet emergency needs. This decision and justification must be documented in the project files, and the 'Purchase Order Terms And Conditions For Subcontracted Laboratory Services' must be sent with the samples and COC.

In the event this provision is utilized, the laboratory (e.g., PM) will be required to verify and document the applicable accreditations of the subcontractor. All other quality and accreditation requirements will still be applicable, but the subcontractor need not have signed a subcontract agreement with Eurofins TestAmerica at this time.

The use of any emergency subcontractor will require the PM to complete a New Vendor Add Form in order to process payment to the vendor and add them to LIMS. This form requires the user to define the subcontractor's category/s of testing and the reason for testing.

10.5 Use of NELAP and A2LA Logo

It is not laboratory policy to use these logos on any company letterhead, including analytical reports.

11.0 PURCHASING SERVICES AND SUPPLIES

11.1 Overview

Evaluation and selection of suppliers and vendors is performed, in part, on the basis of the quality of their products, their ability to meet the demand for their products on a continuous and short term basis, the overall quality of their services, their past history, and competitive pricing. This is achieved through evaluation of objective evidence of quality furnished by the supplier, which can include certificates of analysis, recommendations, and proof of historical compliance with similar programs for other clients. To ensure that quality critical consumables and equipment conform to specified requirements, which may affect quality, all purchases from specific vendors are approved by a member of the supervisory or management staff. Capital expenditures are made in accordance with NDSC Document No. CW-F-S-007, Eurofins TestAmerica's Fixed Asset Acquisition, Retention and Safeguarding.

Contracts are signed in accordance with NDSC Document No. CW-F-P-002, Company-Wide Authorization Matrix Policy. Request for Proposals (RFP's) will be issued where more information is required from the potential vendors than just price. Process details are available in the NDSC Document No. CW-F-P-004, Guidance on Procurement and Contracts Policy. RFP's allow the laboratory to determine if a vendor is capable of meeting requirements such as supplying all of the laboratory facilities, meeting required quality standards and adhering to necessary ethical and environmental standards. The RFP process also allows potential vendors to outline any additional capabilities they may offer.

11.2 Glassware

Glassware used for volumetric measurements must be Class A or verified for accuracy according to laboratory procedure. Pyrex (or equivalent) glass should be used where possible. For safety purposes, thick-wall glassware should be used where available.

11.3 Reagents, Standards & Supplies

Purchasing guidelines for equipment, consumables, and reagents must meet the requirements of the specific method and testing procedures for which they are being purchased. Solvents and acids are pre-tested in accordance with NDSC Document No. CA-Q-S-001, Solvent & Acid Lot Testing & Approval. Approval information for the solvents and acids tested under Document No. CA-Q-S-001 is stored on the laboratory's SharePoint site, under Solvent Approvals. A master list of all tested materials, as well as the certificates of analysis for the materials, is stored in the same location.

11.3.1 Purchasing

Chemical reagents, solvents, glassware, and general supplies are ordered as needed to maintain sufficient quantities on hand. Materials used in the analytical process must be of a known quality. The wide variety of materials and reagents available makes it advisable to specify recommendations for the name, brand, and grade of materials to be used in any determination. This information is contained in the method SOP.

Many consumable items are maintained in an on-site consignment system, where the items in inventory have been pre-approved for laboratory use. On-site consignment is an inventory of

commonly used items maintained at the laboratory by a laboratory supply company where they track the supply of items and restock as needed. If an item is not included in the on-site consignment system but is needed for laboratory operations, the group leader or designee enters the request into the procurement system. The vendor item catalogue number is entered into the system to specify the required quality. Each request is reviewed and approved by the Laboratory Director or the Technical Manager (or designee) prior to purchase.

11.3.2 Receiving

It is the responsibility of the laboratory associate (typically sample receiving personnel) to receive the shipment. Once the ordered reagents or materials are received, the analyst compares the information on the label or packaging to the original order to ensure that the purchase meets the quality level specified and documents the date the materials were received. Refer to SOP KNOX-QA-0001 for further details.

The laboratory associate verifies the lot numbers of received solvents and acids against the pre-approval lists. If a received material is listed as unapproved, or is not listed, it is sequestered and returned to the vendor. Alternatively, the laboratory may test the material for the intended use, and if it is acceptable, document the approval on the approval list. Records of any testing performed locally are maintained on the shared “public” folder on the computer network.

Materials may not be released for use in the laboratory until they have been inspected, verified as suitable for use, and the inspection/verification has been documented.

Safety Data Sheets (SDSs) are available online through the Company’s intranet website. Anyone may review these for relevant information on the safe handling and emergency precautions of on-site chemicals.

11.3.3 Specifications

Methods used in the laboratory specify the grade of reagent that must be used in the procedure. If the quality of the reagent is not specified, analytical reagent grade will be used. It is the responsibility of the analyst to check the procedure carefully for the suitability of grade of reagent.

Chemicals must not be used past the manufacturer’s expiration date and must not be used past the expiration time noted in a method SOP. If expiration dates are not provided, the laboratory may contact the manufacturer to determine an expiration date.

The laboratory assumes a five year expiration date on inorganic dry chemicals and solvents unless noted otherwise by the manufacturer or by the reference source method. Chemicals/solvents should not be used past the manufacturer’s or SOP expiration date unless verified as outlined below.

- An expiration date **cannot** be extended if the dry chemical/solvent is discolored or appears otherwise physically degraded. In this case, the dry chemical/solvent must be discarded.
- Expiration dates can be extended if the dry chemical/solvent is found to be satisfactory based on acceptable performance of quality control samples (Continuing Calibration Verification (CCV), Blanks, Laboratory Control Sample (LCS), etc.).
- If the dry chemical/solvent is used for the preparation of standards, the expiration dates can be extended 6 months if the dry chemical/solvent is compared to an unexpired independent source in performing the method and the performance of the dry chemical/solvent is found to be satisfactory. The comparison must show that the dry chemical/solvent meets CCV

limits. The comparison studies are maintained on-file and available for review in the group share on the local area network with the associated scanned calibration files.

Wherever possible, standards must be traceable to national or international standards of measurement or to national or international reference materials. Records to that effect are available to the user.

Compressed gases in use are checked for pressure and secure positioning daily. To prevent a tank from going to dryness, or introducing potential impurities, the pressure should be closely watched as it decreases to approximately 15% of the original reading, at which point it should be replaced. For example, a standard sized laboratory gas cylinder containing 3,000 psig of gas should be replaced when it drops to approximately 500 psig. The quality of the gases must meet method or manufacturer specification or be of a grade that does not cause any analytical interference.

Water used in the preparation of samples, standards or reagents must have a specific conductivity of less than 1-µmho/cm (or specific resistivity of greater than 1.0 megohm-cm) at 25°C. The specific conductivity is checked and recorded daily. If the water's specific conductivity is greater than the specified limit, the Facility Manager and appropriate Technical Managers must be notified immediately in order to notify all departments, decide on cessation (based on intended use) of activities, and make arrangements for correction.

The laboratory may purchase reagent grade (or other similar quality) water for use in the laboratory. This water must be certified clean by the supplier for all target analytes or otherwise verified by the laboratory prior to use. This verification is documented.

Standard lots are verified before first time use if the laboratory switches manufacturers or has historically had a problem with the type of standard. Spiking standards are verified before use and instrument calibration standards are verified with a second source calibration verification standard. (Note: For Ohio EPA VAP, the continuing calibration verification standard must be from the same source used to prepare the standards used for the calibration curve, unless the analytical method states otherwise.)

Purchased bottleware used for sampling must be certified clean and the certificates must be maintained. If uncertified sampling bottleware is purchased, all lots must be verified clean prior to use. This verification must be maintained.

Tedlar bags purchased by the laboratory must be certified clean and the certificates must be maintained. Tedlar bags are not recommended for trace level analyses as the lab default reporting limits are 20x higher for Tedlar bags than for Summa Canisters.

Required records for solvents and standards are specified in the laboratory SOP KNOX-QA-0001, "Standard/Reagent Labeling and Documentation."

11.3.4 Storage

Reagent and chemical storage is important from the aspects of both integrity and safety. Light-sensitive reagents may be stored in brown-glass containers. Storage conditions are per the NDSC Environmental Health & Safety Manual, Document No. CW-E-M-001, and method SOPs or manufacturer instructions.

11.4 Purchase of Equipment / Instruments / Software

When a new piece of equipment is needed, either for additional capacity or for replacing

inoperable equipment, the analyst or supervisor makes a supply request to the Technical Manager and/or the Laboratory Director. If they agree with the request, the procedures outlined in NDSC Document No. CA-T-P-001, Qualified Products List, are followed. A decision is made as to which piece of equipment can best satisfy the requirements. The appropriate written requests are completed and purchasing places the order.

Upon receipt of a new or used piece of equipment, an identification name is assigned in the LIMS and added to the equipment list. IT must also be notified so that they can synchronize the instrument for back-ups. Its capability is assessed to determine if it is adequate or not for the specific application. For instruments, a calibration curve is generated, followed by MDLs, Demonstration of Capabilities (DOCs), and other relevant criteria (refer to Section 20). For software, its operation must be deemed reliable and evidence of instrument verification must be retained by the IT Department. Software certificates supplied by the vendors are filed with the LIMS Administrator. The manufacturer's operation manual is retained at the bench.

11.5 Services

Service to analytical instruments (except analytical balances) is performed on an as needed basis. Routine preventative maintenance is discussed in Section 22. The need for service is determined by analysts and/or Group Leaders. The service providers that perform the services are approved by the Group Leader/Technical Manager.

Analytical balances are serviced and calibrated annually in accordance with SOP KNOX-QA-0005, "Balance Calibration and Weight Verification." The calibration and maintenance services are performed on-site, and the balances are returned to use immediately following successful calibration. Calibration certificates are filed for reference. If the calibration was unsuccessful, the balance is immediately removed from service and segregated pending either further maintenance or disposal.

Calibration services for support equipment such as thermometers, weight sets, autopipettors, etc., are obtained from vendors with current and valid ISO 17025 accreditation for calibration of the specific piece of equipment. Prior to utilizing the vendor's services, the vendor's accreditation status is verified. Once the equipment has been calibrated, the calibration certificates are reviewed by the QA department, and documentation of the review is filed with the calibration certificates. The equipment is then returned to service within the laboratory

11.6 Suppliers

The laboratory selects vendors through a competitive proposal / bid process, strategic business alliances or negotiated vendor partnerships (contracts). This process is defined in the NDSC Document No. CW-F-P-004, Procurement & Contracts Policy. The level of control used in the selection process is dependent on the anticipated spending amount and the potential impact on the laboratory's business. Vendors that provide test and measuring equipment, solvents, standards, certified containers, instrument related service contracts or subcontract laboratory services shall be subject to more rigorous controls than vendors that provide off-the-shelf items of defined quality that meet the end use requirements. The purchasing system includes all suppliers/vendors that have been approved for use.

Evaluation of suppliers is accomplished by ensuring the supplier ships the product or material ordered and that the material is of the appropriate quality. This is documented by signing off on packing slips or other supply receipt documents. The purchasing documents contain the data that adequately describe the services and supplies ordered.

Any issues of vendor performance are to be reported immediately by the laboratory staff to the Purchasing Group by completing a Vendor Performance Report.

The Purchasing Group will work through the appropriate channels to gather the information required to clearly identify the problem and will contact the vendor to report the problem and to make any necessary arrangements for exchange, return authorization, credit, etc.

Suppliers are subject to re-evaluation, as deemed appropriate, through the use of Vendor Performance Reports used to summarize and review to determine corrective action necessary, or service improvements required by vendors

The laboratory has access to a listing of all approved suppliers of critical consumables, supplies and services. This information is provided through the purchasing system.

11.6.1 New Vendor Procedure

Laboratory employees who wish to request the addition of a new vendor must complete a Vendor Add Request Form.

New vendors are evaluated based upon criteria appropriate to the products or services provided as well as their ability to provide those products and services at a competitive cost. Vendors are also evaluated to determine if there are ethical reasons or potential conflicts of interest with laboratory employees that would make it prohibitive to do business with them as well as their financial stability. The QA Department and/or the Technical Services Director are consulted with vendor and product selection that have an impact on quality.

12.0 COMPLAINTS

12.1 Overview

The laboratory considers an effective client complaint handling processes to be of significant business and strategic value. Listening to and documenting client concerns captures client knowledge that enables our operations to continually improve processes and client satisfaction. An effective client complaint handling process also provides assurance to the data user that the laboratory will stand behind its data, service obligations and products.

A client complaint is any expression of dissatisfaction with any aspect of our business services (e.g., communications, responsiveness, data, reports, invoicing and other functions) expressed by any party, whether received verbally or in written form. Client inquiries, complaints or noted discrepancies are documented, communicated to management, and addressed promptly and thoroughly.

The laboratory has procedures for addressing both external and internal complaints with the goal of providing satisfactory resolution to complaints in a timely and professional manner.

The nature of the complaint is identified, documented and investigated, and an appropriate action is determined and taken. In cases where a client complaint indicates that an established policy or procedure was not followed, the QA Department must evaluate whether a special audit must be conducted to assist in resolving the issue. A written confirmation or letter to the client, outlining the issue and response taken is recommended as part of the overall action taken.

The process of complaint resolution and documentation utilizes the procedures outlined in Section 14 (Corrective Actions) and is documented following the procedure for Nonconformance and Corrective Action System (SOP KNOX-QA-0008). Client complaints are recorded as a type of nonconformance memo (NCM) in TestAmerica LIMS (i.e., TALS), and are summarized in the monthly QA Reports to Management. It is the laboratory's goal to provide a satisfactory resolution to complaints in a timely and professional manner.

12.2 External Complaints

An employee that receives a complaint initiates the complaint resolution process by first documentation of the complaint.

Complaints fall into two categories: correctable and non-correctable. An example of a correctable complaint would be one where a report re-issue would resolve the complaint. An example of a non-correctable complaint would be one where a client complains that their data was repeatedly late. Non-correctable complaints should be reviewed for preventive action measures to reduce the likelihood of future occurrence and mitigation of client impact.

The general steps in the complaint handling process are:

- Receiving and documenting complaints
- Acknowledging receipt of complaint, whenever possible
- Complaint investigation and service recovery
- Process improvement

The laboratory informs the initiator of the complaint of the results of the investigation and the corrective action taken, if any.

12.3 Internal Complaints

Internal complaints include, but are not limited to: errors and non-conformances, training issues, internal audit findings, and deviations from methods. Corrective actions may be initiated by any staff member who observes a nonconformance and shall follow the procedures outlined in Section 14. In addition, Executive Management, Sales and Marketing and IT may initiate a complaint by contacting the laboratory or through the corrective action system described in Section 14.

12.4 Management Review

The number and nature of client complaints is reported by the QA Manager to the Laboratory Director and Quality Director in the QA Monthly report. Monitoring and addressing the overall level and nature of client complaints and the effectiveness of the solutions is part of the Annual Management Systems Review (Section 18).

13.0 CONTROL OF NON-CONFORMING WORK

13.1 Overview

When data discrepancies are discovered or deviations and departures from laboratory SOPs, policies and/or client requests have occurred, corrective action is taken immediately. First, the laboratory evaluates the significance of the nonconforming work. Then, a corrective action plan is initiated based on the outcome of the evaluation. If it is determined that the nonconforming work is an isolated incident, the plan could be as simple as adding a qualifier to the final results and/or making a notation in the case narrative. If it is determined that the nonconforming work is a systematic or improper practices issue, the corrective action plan could include a more in depth investigation and a possible suspension of an analytical method. In all cases, the actions taken are documented using the laboratory's corrective action system (refer to Section 14).

Due to the frequently unique nature of environmental samples, sometimes departures from documented policies and procedures are needed. When a laboratory analyst encounters such a situation, the problem is typically discussed with the group leader. The group leader or laboratory analyst may elect to discuss it with the Technical Manager or have the Project Manager contact the client to decide on a logical course of action. Once an approach is agreed upon, the laboratory analyst documents it using the laboratory's corrective action system described in Section 14. This information is supplied to the client in the form of a footnote or a case narrative with the report.

Note: For Ohio EPA VAP work, the laboratory must implement corrective action procedures to resolve the deviation and limit qualification of the final results. The laboratory is not permitted to deviate from its VAP approved SOP for nonconformances under the laboratory's control if it intends to attest under affidavit that the "results" are VAP certified. When all corrective actions listed in the SOP have been exhausted for nonconformances under the laboratory's control, data still considered qualified is noted as not VAP certified on the affidavit. The SOPs must include exceptions to taking corrective actions for situations outside the laboratory's control. For example, this includes but is not limited to samples prepared or analyzed outside holding times due to no fault of the laboratory and insufficient sample amount. The positive or negative bias associated with the situation outside the laboratory's control must be narrated as described in the SOP and the data is reported as VAP certified on the affidavit.

Project Management may encounter situations where a client may request that a special procedure be applied to a sample that is not standard lab practice. Based on a technical evaluation, the lab may accept or opt to reject the request based on technical or ethical merit. If accepted, such a request is documented in project specific instructions to the laboratory.

13.2 Responsibilities and Authorities

Under certain circumstances the Laboratory Director, Technical Manager, Group Leader or the QA Manager may exceptionally authorize departures from documented procedures or policies depending on the nature and extent of the departure. The departures may be a result of procedural changes due to the nature of the sample; a one-time procedure for a client; QC failures with insufficient sample to reanalyze, etc. In most cases, the client is informed of the departure prior to the reporting of the data. Any departures must be well documented using the laboratory's corrective action procedures. This information may also need to be documented on

data review checklists as appropriate. Any impacted data must be referenced in a case narrative and/or flagged with an appropriate data qualifier.

Any misrepresentation or possible misrepresentation of analytical data discovered by any laboratory staff member must be reported to facility Senior Management within 24-hours. The Senior Management staff is comprised of the Laboratory Director, the QA Manager, Technical Manager(s) and the Operations Manager. The Laboratory Director and QA Manager must be included in the notification. The reporting of issues involving alleged violations of the company's Data Integrity or Manual Integration procedures must be conveyed to an Ethics and Compliance Officer (ECO) (e.g., the VP-QA/EHS) and the laboratory's Quality Director within 24 hours of discovery.

Whether an inaccurate result was reported due to calculation or quantitation errors, data entry errors, improper practices, or failure to follow SOPs, the data must be evaluated to determine the possible effect.

The Laboratory Director, QA Manager, ECOs, VP of Operations and the Quality Directors have the authority and responsibility to halt work, withhold final reports, or suspend an analysis for due cause as well as authorize the resumption of work.

13.3 Evaluation of Significance and Actions Taken

For each nonconforming issue reported, an evaluation of its significance and the level of management involvement needed is made. This includes reviewing its impact on the final data, whether or not it is an isolated or systematic issue, and how it relates to any special client requirements.

The NDSC Document entitled Data Recalls (CW-Q-S-005) is the procedure to be followed when it is discovered that erroneous or biased data may have been reported to clients or regulatory agencies.

The NDSC Document entitled Internal Investigations (CW-L-S-002) is the procedure to be followed for investigation and correction of situations involved alleged incidents of misconduct or violation of the company's ethics policy.

Laboratory level decisions are documented and approved using the laboratory's standard nonconformance/corrective action reporting in lieu of the data recall determination form contained in NDSC Document No CW-Q-S-005.

13.4 Prevention of Nonconforming Work

If it is determined that the nonconforming work could recur, further corrective actions must be made following the laboratory's corrective action system. Periodically as defined by the laboratory's preventive action schedule, the QA Department evaluates non-conformances to determine if any nonconforming work has been repeated multiple times. If so, the laboratory's corrective action process may be followed.

13.5 Method Suspension / Restriction (Stop Work Procedures)

In some cases, it may be necessary to suspend/restrict the use of a method or target analyte which constitutes significant risk and/or liability to the laboratory. Suspension/restriction procedures can be initiated by any of the persons noted in Section 13.2, Paragraph 5.

Prior to suspension/restriction, confidentiality will be respected, and the problem with the required corrective and preventive action will be stated in writing and presented to the Laboratory Director.

The Laboratory Director shall arrange for the appropriate personnel to meet with the QA Manager as needed. This meeting shall be held to confirm that there is a problem, that suspension/restriction of the method is required and will be concluded with a discussion of the steps necessary to bring the method/target or test fully back on line. In some cases, that may not be necessary if all appropriate personnel have already agreed there is a problem and there is agreement on the steps needed to bring the method, target or test fully back on line. The QA Manager will also initiate a corrective action report as described in Section 14 if one has not already been started. A copy of any meeting notes and agreed upon steps should be e-mailed by the laboratory to their Business Unit President; VP-QA & EHS. This e-mail acts as notification of the incident.

After suspension/restriction, the lab will hold all reports to clients pending review. No faxing, mailing or distributing through electronic means may occur. The report must not be posted for viewing on the internet. It is the responsibility of the Laboratory Director to hold all reporting and to notify all relevant laboratory personnel regarding the suspension/restriction (e.g., Project Management, Log-in, etc.). Clients will NOT generally be notified at this time. Analysis may proceed in some instances depending on the non-conformance issue.

Within 72 hours, the QA Manager will determine if compliance is now met and reports can be released, OR determine the plan of action to bring work into compliance, and release work. A team, with all principals involved (e.g., Laboratory Director, Technical Manager, QA Manager, Project Manager, Group Leader) can devise a start-up plan to cover all steps from client notification through compliance and release of reports. Project Management and the Directors of Client Services and Sales and Marketing must be notified if clients must be notified or if the suspension/restriction affects the laboratory's ability to accept work. The QA Manager must approve start-up or elimination of any restrictions after all corrective action is complete.

14.0 CORRECTIVE ACTION

14.1 Overview

A major component of the laboratory's (QA) Program is the problem investigation and feedback mechanism designed to keep the laboratory staff informed on quality related issues and to provide insight to problem resolution. When nonconforming work or departures from policies and procedures in the quality system or technical operations are identified, the corrective action procedure provides a systematic approach to assess the issues, restore the laboratory's system integrity, and prevent reoccurrence. The laboratory employs two systems to manage non-conformances. Issues suspected of being systematic in nature and for which root cause analysis and a formal Corrective Action Report (CAR) are documented in the Incident Corrective Action Tracking (ICAT) database. Routine batch non-conformances, events that are understood to be isolated in nature, are documented in the LIMS non-conformance memo (NCM) system.

14.2 General

Problems within the quality system or within analytical operations may be discovered in a variety of ways, such as QC sample failures, internal or external audits, proficiency testing (PT) performance, client complaints, staff observation, etc.

The purpose of a corrective action system is to:

- Identify non-conformance events and assign responsibility for investigating.
- Resolve non-conformance events and assign responsibility for any required corrective action.
- Identify systematic problems before they become serious.
- Identify and track client complaints and provide resolution.

14.2.1 Non-Conformance Memo (NCM)

NCMs are used to document the following types of corrective actions:

- Deviations from an established procedure or SOP
- QC outside of limits
- Isolated reporting / calculation errors
- Client complaints
- Equipment Failures (Equipment Tag-out)
- Discrepancies in materials / goods received vs. manufacturer packing slips (Forms of documentation other than NCMs in LIMS are also acceptable)

See SOP KNOX-QA-0008, Nonconformance and Corrective Action System, current revision, for details.

14.2.2 Corrective Actions Documented In the ICAT Database

- Internal and external audit findings

- Failed or unacceptable PT results
- Identified poor process or method performance trends
- Systematic reporting / calculation errors
- Data recall investigations
- Questionable trends that are found in the review of NCMs.
- Client complaints
- Excessive revised reports

The ICAT database is used to document background information, track the results of corrective action investigations and root cause analysis, and to provide reports of corrective action plans.

14.3 Closed Loop Corrective Action Process

Any employee in the company can initiate a corrective action. There are four main components to a closed-loop corrective action process once an issue has been identified: Cause Analysis, Selection and Implementation of Corrective Actions (both short and long term), Monitoring of the Corrective Actions, and Follow-up.

14.3.1 Cause Analysis

- Upon discovery of a non-conformance event, the event must be defined and documented. An entry into the ICAT system must be initiated, someone is assigned to investigate the issue and the event is investigated for cause. Table 12-1 provides some general guidelines on determining responsibility for assessment.
- The cause analysis step is the key to the process as a long term corrective action cannot be determined until the cause is determined.
- If the cause is not readily obvious, the Technical Manager, Laboratory Director, or QA Manager (or QA designee) is consulted.

14.3.2 Selection and Implementation of Corrective Actions

- Where corrective action is needed, the laboratory shall identify potential corrective actions. The action(s) most likely to eliminate the problem and prevent recurrence are selected and implemented. Responsibility for implementation is assigned.
- Corrective actions shall be to a degree appropriate to the magnitude of the problem identified through the cause analysis.
- Whatever corrective action is determined to be appropriate, the laboratory shall document and implement the changes. The ICAT record is used for this documentation.

14.3.3 Root Cause Analysis

Root Cause Analysis is a class of problem solving (investigative) methods aimed at identifying the basic or causal factor(s) that underlie variation in performance or the occurrence of a significant failure. The root cause may be buried under seemingly innocuous events, many steps preceding the perceived failure. At first glance, the immediate response is typically directed at a symptom and not the cause. Typically, root cause analysis would be best with

three or more incidents to triangulate a weakness. NDSC Document No. CA-Q-S-009, Root Cause Analysis, provides guidance on this procedure.

Systematically analyze and document the root causes of the more significant problems that are reported. Identify, track, and implement the corrective actions required to reduce the likelihood of recurrence of significant incidents. Trend the root cause data from these incidents to identify root causes that, when corrected, can lead to dramatic improvements in performance by eliminating entire classes of problems.

Identify the one event associated with problem and ask why this event occurred. Brainstorm the root causes of failures; for example, by asking why events occurred or conditions existed; and then why the cause occurred consecutive times until you get to the root cause. For each of these sub events or causes, ask why it occurred. Repeat the process for the other events associated with the incident.

Root cause analysis does not mean the investigation is over. Look at technique or other systems outside the normal indicators. Often creative thinking will find root causes that ordinarily would be missed and continue to plague the laboratory or operation.

14.3.4 Monitoring of the Corrective Actions

- The Group Leader, Operations Manager, Technical Manager and QA Manager are responsible to ensure that the corrective action taken was effective.
- Ineffective actions are documented and re-evaluated until acceptable resolution is achieved. Technical Managers are accountable to the Laboratory Director to ensure final acceptable resolution is achieved and documented appropriately.
- The QA Manager reviews monthly NCM and ICAT records for trends. Highlights are included in the QA monthly report (refer to Section 18). If a significant trend develops that adversely affects quality, an audit of the area is performed and corrective action implemented.
- Any out-of-control situations that are not addressed acceptably at the laboratory level may be reported to the NDSC Quality Director by the QA Manager, indicating the nature of the out-of-control situation and problems encountered in solving the situation.

14.3.5 Follow-up Audits

- Follow-up audits may be initiated by the QA Manager and shall be performed as soon as possible when the identification of a nonconformance casts doubt on the laboratory's compliance with its own policies and procedures, or on its compliance with state or federal requirements.
- These audits often follow the implementation of the corrective actions to verify effectiveness. An additional audit would only be necessary when a critical issue or risk to business is discovered.

(Also refer to Section 17.1.4, Special Audits.)

14.4 Technical Corrective Actions

In addition to providing acceptance criteria and specific protocols for technical corrective actions in the method SOPs, the laboratory has general procedures to be followed to determine when departures from the documented policies and procedures and quality control have occurred

(refer to Section 13). The documentation of these procedures is through the use of an NCM or record in the ICAT system.

Table 14-1 includes examples of general technical corrective actions. For specific criteria and corrective actions, refer to the analytical methods or specific method SOPs. The laboratory may also maintain Work Instructions on these items that are available upon request.

Table 14-1 provides some general guidelines for identifying the individual(s) responsible for assessing each QC type and initiating corrective action. The table also provides general guidance on how a data set should be treated if associated QC measurements are unacceptable. Specific procedures are included in Method SOPs, Work Instructions, QA Manual Sections 19 and 20. All corrective actions are reviewed monthly, at a minimum, by the QA Manager and highlights are included in the QA monthly report.

To the extent possible, samples shall be reported only if all quality control measures are acceptable. If the deficiency does not impair the usability of the results, data will be reported with an appropriate data qualifier and/or the deficiency will be noted in the case narrative. Where sample results may be impaired, the PM is notified by an NCM and appropriate corrective action (e.g., reanalysis) is taken and documented.

14.5 Basic Corrections

When mistakes occur in records, each mistake shall be crossed-out, [not obliterated (e.g. no white-out)], and the correct value entered alongside. All such corrections shall be initialed (or signed) and dated by the person making the correction. In the case of records stored electronically, the original uncorrected file must be maintained intact and a second corrected file is created.

This same process applies to adding additional information to a record. All additions made later than the initial must also be initialed (or signed) and dated.

When corrections are due to reasons other than obvious transcription errors, the reason for the corrections (or additions) shall also be documented.

Table 14-1. Example – General Corrective Action Procedures

QC Activity ***(Individual Responsible for Initiation/Assessment)***	**Acceptance Criteria**	**Recommended Corrective Action**
Initial Instrument Blank *(Analyst)*	- Instrument response < Reporting Limit (RL)[1]	- Prepare another instrument blank. - If same response, determine cause of contamination: reagents, environment, instrument equipment failure, etc.
Initial Calibration Standards *(Analyst, Group Leader)*	- Correlation coefficient r>0.990 for organics and r>0.995 for inorganics or - %RSD within acceptance range. - See details in Method SOP.	- Reanalyze standards. - If still unacceptable, remake standards and recalibrate instrument.

QC Activity *(Individual Responsible for Initiation/Assessment)*	Acceptance Criteria	Recommended Corrective Action
Independent Calibration Verification (Second Source) *(Analyst, Group Leaders)*	- % Recovery within control limits.	- Remake and reanalyze standard. - If still unacceptable, then remake calibration standards or use new primary standards and recalibrate instrument.
Continuing Calibration Standards *(Analyst, Data Reviewer)*	% Difference (or %Drift) within acceptance limits.	- Reanalyze standard. - If still unacceptable, then recalibrate and rerun affected samples.
Matrix Spike / Matrix Spike Duplicate (MS/MSD) *(Analyst, Data Reviewer)*	- % Recovery within limits documented in TALS.	- If the acceptance criteria for duplicates or matrix spikes are not met because of matrix interferences, the acceptance of the analytical batch is determined by the validity of the LCS. - If the LCS is within acceptable limits the batch is acceptable. - The results of the duplicates, matrix spikes and the LCS are reported with the data set. - For matrix spike or duplicate results outside criteria the data for that sample shall be reported with qualifiers.
Laboratory Control Sample (LCS) and LCSD (where applicable) *(Analyst, Data Reviewer)*	- % Recovery within limits specified in TALS. This includes any allowable marginal exceedance (Marginal Exceedances are not allowed for Ohio VAP).	- Batch must be re-prepared and re-analyzed. Note: See Method SOPs for further details. For example, if there is insufficient sample or the holding time cannot be met, contact client and report with flags.
Surrogates *(Analyst, Data Reviewer)*	- % Recovery within limits of method or within three standard deviations of the historical mean.	- Individual sample must be repeated. Place comment in LIMS. - Surrogate results outside criteria shall be reported with qualifiers. Note: See Method SOPs for further details. For example, if there is insufficient sample or the holding time cannot be met, contact client and report with flags.
Method Blank (MB) *(Analyst, Data Reviewer)*	< Reporting Limit [1]	- Reanalyze blank. - If still positive, determine source of contamination. If necessary, reprocess (i.e. digest or extract) entire sample batch. Report blank results. - Qualify the result(s) if the concentration of a targeted analyte in the MB is at or above the reporting limit AND is > 1/10 of the amount measured in the sample.

QC Activity ***(Individual Responsible for Initiation/Assessment)***	**Acceptance Criteria**	**Recommended Corrective Action**
Proficiency Testing (PT) Samples *(QA Manager, Technical Manager(s))*	- Criteria supplied by PT Supplier.	- Any failures or warnings must be investigated for cause. Failures may result in the need to repeat a PT sample to show the problem is corrected.
Internal / External Audits *(QA Manager, Technical Manager(s), Laboratory Director)*	- Defined in Quality System documentation such as SOPs, QAM, etc..	- Non-conformances must be investigated through CAR system and necessary corrections must be made.
Reporting / Calculation Errors (Depends on issue – possible individuals include: Analysts, Data Reviewers, Project Managers, Technical Managers, QA Manager, NDSC QA, Executive Management)	- NDSC Document No. CW-Q-S-005, Data Recall **or** local SOP No.	- Corrective action is determined by type of error. Follow the procedures in NDSC Document No. CW-L-S-002 **or** your lab's CA SOP.
Client Complaints *(Project Managers, Lab Director/Manager, Sales and Marketing)*	-	- Corrective action is determined by the type of complaint. For example, a complaint regarding an incorrect address on a report will result in the report being corrected and then follow-up must be performed on the reasons the address was incorrect (e.g., database needs to be updated).
QA Monthly Report (Refer to Section 16 for an example) (QA Manager, Lab Director/Manager, *Technical Manager(s)*)	- QAM, SOPs.	- Corrective action is determined by the type of issue. For example, CARs for the month are reviewed and possible trends are investigated.
Health and Safety Violation (Safety Officer, Lab Director/Manager, *Technical Manager(s)*)	- Environmental Health and Safety (EHS) Manual.	- Non-conformance is investigated and corrected through CAR system.

Note:

1. See specific method SOPs for acceptance criteria. Concentrations up to five times the reporting limit may be allowed for the common laboratory and reagent contaminants: methylene chloride, toluene, acetone, 2-butanone and phthalates. Some program requirements (e.g. DOD QSM and Ohio EPA VAP) have more stringent requirements that must be followed when performing work for those programs. For example, DOD QSM requires the Method Blanks < ½ Reporting Limit with common lab contaminants <Reporting Limit. (Note: Ohio EPA VAP requires all analytes of concern in Method Blanks to be less than the RL.)

15.0 PREVENTIVE ACTION / IMPROVEMENT

15.1 Overview

The laboratory's preventive action programs improve or eliminate potential causes of nonconforming product and/or nonconformance to the quality system. This preventive action process is a proactive and continuous process of improvement activities that can be initiated through feedback from clients, employees, business providers, and affiliates. The QA Department has the overall responsibility to ensure that the preventive action process is in place, and that relevant information on actions is submitted for management review.

Dedicating resources to an effective preventive action system emphasizes the laboratory's commitment to its QA Program. It is beneficial to identify and address negative trends before they develop into complaints, problems and corrective actions. Additionally, the laboratory continually strives to improve customer service and client satisfaction through continuous improvements to laboratory systems.

Opportunities for improvement may be discovered through any of the following:

- review of the monthly QA Metrics Report,
- trending NCMs,
- review of control charts and QC results,
- trending proficiency testing (PT) results,
- performance of management system reviews,
- trending client complaints,
- review of processing operations, or
- staff observations.

The monthly Management Systems Metrics Report shows performance indicators in all areas of the laboratory and quality system. These areas include revised reports, corrective actions, audit findings, internal auditing and data authenticity audits, client complaints, PT samples, holding time violations, SOPs, ethics training, etc. The metrics report is reviewed monthly by the laboratory management, NDSC QA Team, Local and Executive Mangement. These metrics are used in evaluating the management and quality system performance on an ongoing basis and provide a tool for identifying areas for improvement.

Items identified as continuous improvement opportunities to the management system may be issued as goals from the annual management systems review, recommendations from internal audits, white papers, Lessons Learned, Technical Services audit report, Technical Best Practices, or as Executive or management initiatives.

The laboratory's corrective action process is integral to implementation of preventive actions. A critical piece of the corrective action process is the implementation of actions to prevent further occurrence of a non-compliance event. Historical review of corrective action and non-conformances provides a valuable mechanism for identifying preventive action opportunities.

15.1.1 The following elements are part of a preventive action/process improvement system:

- Identification of an opportunity for preventive action or process improvement.

- Process for the preventive action or improvement.
- Define the measurements of the effectiveness of the process once undertaken.
- Execution of the preventive action or improvement.
- Evaluation of the plan using the defined measurements.
- Verification of the effectiveness of the preventive action or improvement.
- Close-Out by documenting any permanent changes to the Quality System as a result of the Preventive Action or Process Improvement. Documentation of Preventive Action/process Improvement is incorporated into the monthly QA reports, corrective action process and management review.

15.1.2 Any preventive actions/process improvement undertaken or attempted shall be taken into account during the annual Management Systems Review (Section 16). A highly detailed report is not required; however, a summary of successes and failures within the preventive action program is sufficient to provide management with a measurement for evaluation.

15.2 Management of Change

The Management of Change process is designed to manage significant events and changes that occur within the laboratory. This is routinely managed by the Laboratory Director with input from the General Manager, the QA Manager, Operations Manager, Technical Managers, Group Leaders, and IT support staff. Through these procedures, the potential risks inherent with a new event or change are identified and evaluated. The risks are minimized or eliminated through pre-planning and the development of preventive measures. The types of changes covered under this system include: Facility Changes, Major Accreditation Changes, Addition or Deletion to Division's Capabilities or Instrumentation, Key Personnel Changes, Laboratory Information Management System (LIMS) changes.

16.0 CONTROL OF RECORDS

The laboratory maintains a records management system appropriate to its needs and that complies with applicable standards or regulations as required. The system produces unequivocal, accurate records that document all laboratory activities. The laboratory retains all original observations, calculations and derived data, calibration records and a copy of the analytical report for a minimum of five years after it has been issued. Exceptions for programs with longer retention requirements are discussed in Section 16.1.2.

16.1 Overview

The laboratory has established procedures for identification, collection, indexing, access, filing, storage, maintenance and disposal of quality and technical records. A record index is listed in Table 16-1. More detailed information on retention of specific records is provided in NDSC Document Nos. CW-L-P-001, Records Retention Policy and CW-L-WI-001, Records Retention/Storage Schedule. Quality records are maintained by the QA department in a database, which is backed up as part of the regular laboratory backup. Records are of two types; either electronic or hard copy paper formats depending on whether the record is computer or hand generated (some records may be in both formats). Technical records are maintained by Group Leaders and the local IT staff.

Table 16-1. Record Index

	Record Types [1]:	**Retention Time:**
Technical Records	- Raw Data - Logbooks[2] - Standards - Certificates - Analytical Records - MDLs/IDLs/DOCs - Lab Reports	5 Years from analytical report issue*
Official Documents	- Quality Assurance Manual (QAM) - Work Instructions - Policies - SOPs - Policy Memorandums - Manuals - Published Methods	Indefinitely
QA Records	- Certifications - Method and Software Validation / Verification Data	Indefinitely
QA Records	- Internal & External Audits/Responses - Corrective/Preventive Actions - Management Reviews - Data Investigation	5 Years from archival* **Data Investigation:** 5 years or the life of the affected raw data storage whichever is greater (beyond 5 years if ongoing project or pending investigation)

	Record Types [1]:	Retention Time:
Project Records	- Sample Receipt & COC Documents - Contracts and Amendments - Correspondence - QAPP - SAP - Telephone Logbooks - Lab Reports	5 Years from analytical report issue*
Administrative Records	Financial and Business Operations	Refer to NDSC Doc. No. CW-L-WI-001
	EH&S Manual, Permits	Indefinitely
	Disposal Records	Indefinitely
	Employee Handbook	Indefinitely
	Personnel files, Employee Signature & Initials, Administrative Training Records (e.g., Ethics)	Refer to HR Manual
	Administrative Policies	Indefinitely
	Technical Training Records	7 years
	Legal Records	Indefinitely
	HR Records	Refer to NDSC Doc. No. CW-L-WI-001
	IT Records	Refer to NDSC Doc. No. CW-L-WI-001
	NDSC Governance Records	Refer to NDSC Doc. No. CW-L-WI-001
	Sales & Marketing	5 years
	Real Estate	Indefinitely

[1] Record Types encompass hardcopy and electronic records.
[2] Examples of Logbook types: Maintenance, Instrument Run, Preparation (standard and samples), Standard and Reagent Receipt, Archiving, Balance Calibration, Temperature (hardcopy or electronic records).
* Exceptions listed in Table 16-2.

16.1.1 All records are stored and retained in such a way that they are secure and readily retrievable at the laboratory facility. All records are protected against fire, theft, loss, environmental deterioration, and vermin. In the case of electronic records, electronic or magnetic sources, storage media are protected from deterioration caused by magnetic fields and/or electronic deterioration.

Access to the data is limited to laboratory and company employees and shall be documented with an access log**.** Records are maintained for a minimum of five years unless otherwise specified by a client or regulatory requirement.

Note: For the Ohio EPA VAP program the laboratory is required to notify Ohio EPA of its intent to dispose of any records.

For raw data and project records, record retention shall be calculated from the date the project report is issued. For other records, such as NDSC Documents, QA, or Administrative Records, the retention time is calculated from the date the record is formally retired. Records related to the programs listed in Table 16-2 have lengthier retention requirements and are subject to the requirements in Section 16.2.

16.2 Programs with Longer Retention Requirements

Some regulatory programs have longer record retention requirements than the standard record retention time. These are detailed in Table 16-2 with their retention requirements. In these cases, the longer retention requirement is enacted. If special instructions exist such that client data cannot be destroyed prior to notification of the client, the container or box containing that data is marked as to who to contact for authorization prior to destroying the data.

Table 16-2. Example: Special Record Retention Requirements

Program	[1]Retention Requirement
Drinking Water – All States	10 years (lab reports and raw data) 10 years - Radiochemistry (project records)
DOE Hanford	5 years, Written approval must be received from DOE Hanford prior to disposal of analytical data
FIFRA – 40 CFR Part 160	Retain for life of research or marketing permit for pesticides regulated by EPA
Housing and Urban Development (HUD) Environmental Lead Testing	10 years
Alaska	10 years
Louisiana – All	10 years
Michigan Department of Environmental Quality – all environmental data	10 years
NY Potable Water NTCRR Part 55-2	10 years
Ohio EPA VAP	10 years and State contacted prior to disposal
OSHA	30 years

[1]Note: Extended retention requirements must be noted with the archive documents or addressed in facility-specific records retention procedures.

16.2.1 The laboratory has procedures to protect and back-up records stored electronically and to prevent unauthorized access to or amendment of these records. All analytical data is maintained as hard copy or in a secure readable electronic format. For analytical reports that are maintained as copies in PDF format, refer to Section 19.15.1 for more information.

16.2.2 The record keeping system allows for historical reconstruction of all laboratory activities that produced the analytical data, as well as rapid recovery of historical data.The history of the sample from when the laboratory took possession of the samples must be readily understood through the documentation. This shall include inter-laboratory transfers of samples and/or extracts.

- The records include the identity of personnel involved in sampling, sample receipt, preparation, or testing. All analytical work contains the initials (at least) of the personnel involved. The laboratory's copy of the COC is stored with the invoice and the work order sheet generated by TALS. The chain of custody would indicate the name of the sampler.
- All information relating to the laboratory facilities' equipment, analytical test methods, and related laboratory activities, such as sample receipt, sample preparation, or data verification are documented.
- The record keeping system facilitates the retrieval of all working files and archived records for inspection and verification purposes (e.g., set format for naming electronic files, set

format for what is included with a given analytical data set. Instrument data is stored sequentially by instrument. A given day's analyses are maintained in the order of the analysis. Run logs are maintained for each instrument or method; a copy of each day's run log or instrument sequence is stored with the data to aid in re-constructing an analytical sequence. Where an analysis is performed without an instrument, bound logbooks or bench sheets are used to record and file data. Standard and reagent information is recorded in logbooks or entered into TALS for each method as required.

- Changes to hardcopy records shall follow the procedures outlined in Section 12 and 19. Changes to electronic records in LIMS or instrument data are recorded in audit trails.
- The reason for a signature or initials on a document is clearly indicated in the records such as "sampled by," "prepared by," "reviewed by", or "analyzed by".
- All generated data except those that are generated by automated data collection systems, are recorded directly, promptly and legibly in permanent dark ink.
- Hard copy data may be scanned into PDF format for record storage as long as the scanning process can be verified in order to ensure that no data is lost and the data files and storage media must be tested to verify the laboratory's ability to retrieve the information prior to the destruction of the hard copy that was scanned. . The procedure for scanning the records is discussed in CW-L-P-001, Records Retention Policy.
- Also refer to Section 19.15.1 'Computer and Electronic Data Related Requirements'.

16.3 Technical and Analytical Records

16.3.1 The laboratory retains records of original observations, derived data and sufficient information to establish an audit trail, calibration records, staff records and a copy of each analytical report issued, for a minimum of five years unless otherwise specified by a client or regulatory requirement. The records for each analysis shall contain sufficient information to enable the analysis to be repeated under conditions as close as possible to the original. The records shall include the identity of laboratory personnel responsible for the sampling, performance of each analysis and reviewing results.

16.3.2 Observations, data and calculations are recorded real-time and are identifiable to the specific task.

16.3.3 Changes to hardcopy records shall follow the procedures outlined in Section 12 and 19. Changes to electronic records in TALS or instrument data are recorded in audit trails.

The essential information to be associated with analysis, such as strip charts, tabular printouts, computer data files, analytical notebooks, and run logs, include:

- Laboratory sample ID code;
- Date of analysis; time of analysis is also required if the holding time is seventy-two (72) hours or less, or when time critical steps are included in the analysis (e.g., drying times, incubations, etc.); instrumental analyses have the date and time of analysis recorded as part of their general operations. Where a time critical step exists in an analysis, location for such a time is included as part of the documentation in a specific logbook or on a benchsheet.
- Instrumentation identification and instrument operating conditions/parameters. Operating conditions/parameters are typically recorded in instrument maintenance logs where available.
- Analysis type;

- All manual calculations and manual integrations;
- Laboratory Analyst's or operator's initials/signature;
- Sample preparation including cleanup, separation protocols, incubation periods or subculture, ID codes, volumes, weights, instrument printouts, meter readings, calculations, reagents;
- Test results;
- Standard and reagent origin, receipt, preparation, and use;
- Calibration criteria, frequency and acceptance criteria;
- Data and statistical calculations, review, confirmation, interpretation, assessment and reporting conventions;
- Quality control protocols and assessment;
- Electronic data security, software documentation and verification, software and hardware audits, backups, and records of any changes to automated data entries; and
- Method performance criteria including expected quality control requirements. These are indicated both in TALS and on specific analytical report formats.

16.3.4 All logbooks used during receipt, preparation, storage, analysis, and reporting of samples or monitoring of support equipment shall undergo a periodic, documented supervisory or peer review on a periodic basis.

16.4 Laboratory Support Activities

In addition to documenting all the above-mentioned activities, the following are retained QA records and project records (previous discussions in this section relate where and how these data are stored):

- All original raw data, whether hard copy or electronic, for calibrations, samples and quality control measures, including analysts' work sheets and data output records (chromatograms, strip charts, and other instrument response readout records);
- A written description or reference to the specific test method used which includes a description of the specific computational steps used to translate parametric observations into a reportable analytical value;
- Copies of final reports;
- Archived SOPs;
- Correspondence relating to laboratory activities for a specific project;
- All corrective action reports, audits and audit responses;
- Proficiency test results and raw data; and
- Results of data review, verification, and crosschecking procedures

16.4.1 Sample Handling Records

Records of all procedures to which a sample is subjected while in the possession of the laboratory are maintained. These include but are not limited to records pertaining to:

- Sample preservation including appropriateness of sample container and compliance with

holding time requirement;

- Sample identification, receipt, acceptance or rejection and login;
- Sample storage and tracking including shipping receipts, sample transmittal / COC forms; and
- Procedures for the receipt and retention of samples, including all provisions necessary to protect the integrity of samples.

16.5 Administrative Records

The laboratory also maintains the administrative records in either electronic or hard copy form. Refer to Table 16-1.

16.6 Records Management, Storage and Disposal

All records (including those pertaining to test equipment), certificates and reports are safely stored, held secure and in confidence to the client. Certification related records are available upon request.

All information necessary for the historical reconstruction of data is maintained by the laboratory. Records that are stored only on electronic media must be supported by the hardware and software necessary for their retrieval.

Records that are stored or generated by computers or personal computers have hard copy, write-protected backup copies, or an electronic audit trail controlling access**.**

The laboratory has a record management system for control of laboratory notebooks, instrument logbooks, standards logbooks, and project records. Laboratory notebooks are sequentially numbered by QA and issued to the analytical group as needed. Bench sheets are filed and archived with the project raw data. Completed logbooks are returned to QA for archive storage. Records are considered archived when moved to the archive server on the local area network or when logbooks are turned in to QA for storage. Access to archived hard-copy information is documented with in/out cards.

16.6.1 Transfer of Ownership

In the event that the laboratory transfers ownership or goes out of business, the laboratory shall ensure that the records are maintained or transferred according to client's instructions. Upon ownership transfer, record retention requirements shall be addressed in the ownership transfer agreement and the responsibility for maintaining archives is clearly established. In addition, in cases of bankruptcy, appropriate regulatory and state legal requirements concerning laboratory records must be followed. In the event of the closure of the laboratory, all records will revert to the control of NDSC. Should the entire company cease to exist, as much notice as possible will be given to clients and the accrediting bodies who have worked with the laboratory during the previous 5 years of such action.

16.6.2 Records Disposal

Records are removed from the archive and destroyed after 5 years unless otherwise specified by a client or regulatory requirement. On a project specific or program basis, clients may need to be notified prior to record destruction. Records are destroyed in a manner that ensures their confidentiality such as shredding, mutilation or incineration. (Refer to Tables 16-1 and 16-2).

Electronic copies of records must be destroyed by erasure or physically damaging off-line storage media so no records can be read.

If a third party records management company is hired to dispose of records, a “Certificate of Destruction” is required. [Refer to Policy No. CW-L-P-001 (Records Retention).]

17.0 AUDITS

17.1 Internal Audits

Internal audits are performed to verify that laboratory operations comply with the requirements of the lab's quality system and with the external quality programs under which the laboratory operates. Audits are planned and organized by the QA staff. Personnel conducting the audits should be independent of the area being evaluated. Auditors will have sufficient authority, access to work areas, and organizational freedom necessary to observe all activities affecting quality and to report the assessments to laboratory management and, when requested, to Executive Management.

Audits are conducted and documented as described in the <NDSC Document No. on performing Internal Auditing, SOP No. CW-Q-S-003. The types and frequency of routine internal audits are described in Table 17-1. Special or ad hoc assessments may be conducted as needed under the direction of the QA staff.

Table 17-1. Types of Internal Audits and Frequency

Description	Performed by	Frequency
Quality Systems Audits	QA Department, QA approved designee, or NDSC QA	All areas of the laboratory annually
Method Audits QA Technical Audits	Joint responsibility: a) QA Manager or designee b) Technical Manager or Designee (Refer to NDSC CW-Q-S-003)	QA Technical Audits Frequency: 50% of methods annually
SOP Method Compliance	Joint responsibility: a) QA Manager or designee b) Technical Manager or Designee (Refer to NDSC CW-Q-S-003)	SOP Compliance Review Frequency: • Every 2 years • Every 1 year (Drinking Water Methods) • 100% of SOPs annually (DoD/DOE Labs)
Special	QA Department or Designee	Surveillance or spot checks performed as needed, e.g., to confirm corrective actions from other audits.
Performance Testing	Analysts with QA oversight	Two successful per year for each TNI-field of testing or as dictated by regulatory requirements

17.1.1 Annual Quality Systems Audit

An annual quality systems audit is required to ensure compliance to analytical methods and SOPs, Eurofins Data Integrity and Ethics Policies (see Sec. 7.2), TNI quality systems, client and

state requirements, and the effectiveness of the internal controls of the analytical process, including but not limited to data review, quality controls, preventive action and corrective action. The completeness of earlier corrective actions is assessed for effectiveness & sustainability. The audit is divided into sections for each operating or support area of the lab, and each section is comprehensive for a given area. The area audits may be performed on a rotating schedule throughout the year to ensure adequate coverage of all areas. This schedule may change as situations in the laboratory warrant.

Note: Part of the quality systems audit relates to regulatory compliance. An assessment of the laboratory's compliance to regulatory requirements is performed by Corporate QA through monthly management reports, review of client and regulatory concerns and also through periodic on-site evaluations.

The annual quality systems audit must include a review of logbooks and benchsheets used in laboratory areas as well as the items listed in the Internal Audit Checklists as described in SOP CW-Q-S-003.

17.1.2 QA Technical Audits

QA technical audits assess data authenticity and analyst integrity. These audits are based on client projects, associated sample delivery groups, and the methods performed. Reported results are compared to raw data to verify the authenticity of results. The validity of calibrations and QC results are compared to data qualifiers, footnotes, and case narratives. Documentation is assessed by examining run logs and records of manual integrations. Manual calculations are checked. Where possible, electronic audit miner programs (e.g., Chrom AuditMiner) are used to identify unusual manipulations of the data deserving closer scrutiny. QA technical audits will include all methods within a two-year period.

The QA Technical Audits must include a review of raw sample data, calibration data and QC data records.

17.1.3 SOP Method Compliance

Compliance of all SOPs with the source methods and compliance of the operational groups with the SOPs will be assessed by the Technical Manager or qualified designee at least every two years. (Annually for methods and administrative SOPs related to DoD/DOE programs and DW compliance methods. - where applicable) It is also recommended that the work of each newly hired analyst is assessed within 3 months of working independently, (e.g., completion of method IDOC). In addition, as analysts add methods to their capabilities, (new IDOC) reviews of the analyst work products will be performed within 3 months of completing the documented training.

17.1.4 Special Audits

Special audits are conducted on an as needed basis, generally as a follow up to specific issues such as client complaints, corrective actions, PT results, data audits, system audits, validation comments, regulatory audits or suspected ethical improprieties. Special audits are focused on a specific issue, and report format, distribution, and timeframes are designed to address the nature of the issue.

17.1.5 Performance Testing

The laboratory participates semi-annually in performance audits conducted through the analysis of PT samples provided by a third party. The laboratory generally participates in the following types of PT studies: Drinking Water, Non-potable Water, Soil, Tissue and Air.

It is Eurofins policy that PT samples be treated as typical samples in the production process. Furthermore, where PT samples present special or unique problems, in the regular production process they may need to be treated differently, as would any special or unique request submitted by any client. The QA Manager must be consulted and in agreement with any decisions made to treat a PT sample differently due to some special circumstance.

Written investigations for unacceptable PT results are required. In some cases it may be necessary for blind QC samples to be submitted to the laboratory to show a return to control.

17.2 External Audits

External audits are performed when certifying agencies or clients conduct on-site inspections or submit performance testing samples for analysis. It is Eurofins TestAmerica policy to cooperate fully with regulatory authorities and clients. The laboratory makes every effort to provide the auditors with access to personnel, documentation, and assistance. Laboratory supervisors are responsible for providing corrective actions to the QA Manager who coordinates the response. Audit responses are due in the time allotted by the client or agency performing the audit.

The laboratory cooperates with clients and their representatives to monitor the laboratory's performance in relation to work performed for the client. The client may only view data and systems related directly to the client's work. All efforts are made to keep other client information confidential.

17.2.1 Confidential Business Information (CBI) Considerations

During on-site audits, auditors may come into possession of information claimed as business confidential. A business confidentiality claim is defined as "a claim or allegation that business information is entitled to confidential treatment for reasons of business confidentiality or a request for a determination that such information is entitled to such treatment." When information is claimed as business confidential, the laboratory must place on (or attach to) the information at the time it is submitted to the auditor, a cover sheet, stamped or typed legend or other suitable form of notice, employing language such as "trade secret", "proprietary" or "company confidential". Confidential portions of documents otherwise non-confidential must be clearly identified. CBI may be purged of references to client identity by the responsible laboratory official at the time of removal from the laboratory. However, sample identifiers may not be obscured from the information. Additional information regarding CBI can be found in within the 2009 and 2016 TNI standards.

17.3 Audit Findings

Audit findings are documented using the corrective action process and database (see Section 14). This database provides a record of the audit findings for each laboratory area and recommended corrective actions to the appropriate Group Leader. The laboratory corrective action is compiled by QA and responses to external audits are signed by the QA Manager and the Laboratory Director

The laboratory's corrective action responses may include action plans that could not be completed within a predefined timeframe. In these instances, a completion date must be set and agreed to by operations management and the QA Manager.

Developing and implementing corrective actions to findings is the responsibility of the Group Leader where the finding originated. Findings that are not corrected by specified due dates are reported monthly to management in the QA monthly report.

If any audit finding casts doubt on the effectiveness of the operations or on the correctness or validity of the laboratory's test results, the laboratory shall take timely corrective action, and shall notify clients in writing if the investigations show that the laboratory results have been affected. Once corrective action is implemented, a follow-up audit is scheduled to ensure that the problem has been corrected.

Clients must be notified promptly in writing of any event such as the identification of defective measuring or test equipment that casts doubt on the validity of results given in any test report or amendment to a test report. The investigation must begin within 24 hours of discovery of the problem and all efforts are made to notify the client within two weeks after the completion of the investigation.

18.0 MANAGEMENT REVIEWS

18.1 Quality Assurance Report

The QA Department is responsible for preparing a comprehensive monthly metrics report to Management to keep them apprised of current quality issues. This report fosters communication, review, and refinement of the QA system to evaluate the suitability of policies and procedures to meet both regulatory and laboratory quality objectives.

The NDSC QA team compiles information from all of the Environment Testing laboratories monthly metrics reports for the Executive Management team. This report includes notable information and concerns regarding the laboratories QA program and a listing of new regulations that may potentially impact the laboratories.

18.2 Annual Management Review

The Laboratory Management team (Laboratory Director, Technical Managers, QA Manager) conducts a review annually of its quality systems to ensure its continuing suitability and effectiveness in meeting client and regulatory requirements and to introduce any necessary changes or improvements. It will also provide a platform for defining goals, objectives and action items that feed into the laboratory planning system. NDSC personnel is be included in this meeting at the discretion of the Laboratory Director. The LIMS review consists of examining any audits, complaints or concerns that have been raised through the year that are related to LIMS. The laboratory will summarize any critical findings that cannot be solved by the lab and report them to Corporate IT.

This management systems review (NDSC Document No. CW-Q-S-004 and Work Instruction No. CW-Q-WI-003) uses information generated during the preceding year to assess the “big picture” by ensuring that routine actions taken and reviewed on a monthly basis are not components of larger systematic concerns. The monthly review should keep the quality systems current and effective, therefore, the annual review is a formal senior management process to review specific existing documentation. Significant issues from the following documentation are compiled or summarized by the QA Manager prior to the review meeting:

- Matters arising from the previous annual review.
- Prior Monthly QA Reports issues.
- Laboratory QA Metrics.
- Review of report reissue requests.
- Review of client feedback and complaints.
- Issues arising from any prior management or staff meetings.
- Minutes from prior senior lab management meetings. Issues that may be raised from these meetings include:
    - Adequacy of staff, equipment and facility resources.
    - Adequacy of policies and procedures.
    - Future plans for resources and testing capability and capacity.

- The annual internal double blind PT program sample performance (if performed),
- Compliance to the Ethics Policy and Data Integrity Plan. Including any evidence/incidents of inappropriate actions or vulnerabilities related to data Integrity.
- For labs analyzing radioactive samples, also include the following:
  - Radiation health and safety
  - Radioactive hazardous waste management
  - Radioactive materials management
- Evaluation of overall risk, including risks to impartiality, confidentiality, reporting statements of conformity, and nonconforming work.

A report is generated by the QA Manager and management. The report is distributed to the Laboratory Manager, President of the Business Unit, Business Unit Manager. The report includes, but is not limited to:

- The date of the review and the names and titles of participants.
- A reference to the existing data quality related documents and topics that were reviewed.
- Quality system or operational changes or improvements that will be made as a result of the review [e.g., an implementation schedule including assigned responsibilities for the changes (Action Table)].

Changes to the quality systems requiring update to the laboratory QA Manual shall be included in the next revision of the QA Manual.

18.3 Potential Integrity Related Managerial Reviews

Potential integrity issues (data or business related) must be handled and reviewed in a confidential manner until such time as a follow-up evaluation, full investigation, or other appropriate actions have been completed and issues clarified. <The NDSC Document No. CW-L-S-002, Internal Investigations, shall be followed. All investigations that result in finding of inappropriate activity are documented and include any disciplinary actions involved, corrective actions taken, and all appropriate notifications of clients.

The Eurofins Environment Testing America Presidents, Business Unit Managers, Laboratory Directors, NDSC Team are informed of any current data integrity or data recall investigations via the monthly metrics report.

19.0 TEST METHODS AND METHOD VALIDATION

19.1 Overview

The laboratory uses methods that are appropriate to meet our clients' requirements and that are within the scope of the laboratory's capabilities. These include sampling, handling, transport, storage and preparation of samples, and, where appropriate, an estimation of the measurement of uncertainty as well as statistical techniques for analysis of environmental data.

Instructions are available in the laboratory for the operation of equipment as well as for the handling and preparation of samples. All instructions, Standard Operating Procedures (SOPs), reference methods and manuals relevant to the working of the laboratory are readily available to all staff. Deviations from published methods are documented (with justification) in the laboratory's approved SOPs. SOPs are submitted to clients for review at their request. Significant deviations from published methods require client approval and regulatory approval where applicable.

19.2 Standard Operating Procedures (SOPs)

The laboratory maintains SOPs that accurately reflect all phases of the laboratory such as assessing data integrity, corrective actions, handling customer complaints as well as all analytical methods and sampling procedures. The method SOPs are derived from the most recently promulgated/approved, published methods and are specifically adapted to the laboratory facility. Modifications or clarifications to published methods are clearly noted in the SOPs. All SOPs are controlled in the laboratory.

- All SOPs contain a revision number, effective date, and appropriate approval signatures. Controlled copies are available to all staff.
- Procedures for writing an SOP are incorporated by reference to NDSC Document No. CW-Q-S-002, Writing a Standard Operating Procedure, or the laboratory's SOP KNOX-QA-0018 "Preparation and Management of Standard Operating Procedures."
- SOPs are reviewed at a minimum of every 2 years (annually for Drinking Water and DoD/DOE SOPs), and where necessary, revised to ensure continuing suitability and compliance with applicable requirements.

  Note: The laboratory must ensure laboratory analysts comply with the procedures in its VAP approved SOPs.

19.3 Laboratory Methods Manual

For each test method, the laboratory shall have available the published referenced method as well as the laboratory developed SOP.

Note: If more stringent standards or requirements are included in a mandated test method or regulation than those specified in this manual, the laboratory shall demonstrate that such requirements are met. If it is not clear which requirements are more stringent, the standard from the method or regulation is to be followed. Any exceptions or deviations from the referenced methods or regulations are noted in the specific analytical SOP.

The laboratory maintains an SOP Index for both technical and non-technical SOPs. Technical SOPs are maintained to describe a specific test method. Non-technical SOPs are maintained to describe functions and processes not related to a specific test method.

19.4 Selection of Methods

Since numerous methods and analytical techniques are available, continued communication between the client and laboratory is imperative to assure the correct methods are utilized. Once client methodology requirements are established, this and other pertinent information is summarized by the PM. These mechanisms ensure that the proper analytical methods are applied when the samples arrive for log-in. For non-routine analytical services (e.g., special matrices, non-routine compound lists), the method of choice is selected based on client needs and available technology. The methods selected should be capable of measuring the specific parameter of interest, in the concentration range of interest, and with the required precision and accuracy.

19.4.1 Sources of Methods

Routine analytical services are performed using standard EPA-approved methodology. In some cases, modification of standard approved methods may be necessary to provide accurate analyses of particularly complex matrices. When the use of specific methods for sample analysis is mandated through project or regulatory requirements, only those methods shall be used.

When clients do not specify the method to be used or methods are not required, the methods used will be clearly validated and documented in an SOP and available to clients and/or the end user of the data.

Refer to Appendix 6 for a list of the currently accepted U.S. EPA analytical method references used by the laboratory.

The laboratory reviews updated versions to all the aforementioned references for adaptation based upon capabilities, instrumentation, etc., and implements them as appropriate. As such, the laboratory strives to perform only the latest versions of each approved method as regulations allow or require.

Other reference procedures for non-routine analyses may include methods established by specific states (e.g., Underground Storage Tank methods), ASTM or equipment manufacturers. Sample type, source, and the governing regulatory agency requiring the analysis will determine the method utilized.

The laboratory informs the client when a method proposed by the client may be inappropriate or out of date. After the client has been informed, and they wish to proceed contrary to the laboratory's recommendation, it will be documented.

19.4.1.1 Client Supplied Methods

Most of the client-supplied method requirements presented to us involve achieving specific quality control criteria, limits of quantitation (LOQ), and/or method detection limits (MDL) using standard EPA methods. These requirements are communicated to the appropriate technical groups prior to the project start up. Each technical group evaluates the scope of work and the requirements to ensure the criteria can be met using the standard EPA method. The data is monitored to ensure the criteria are met throughout the project. The PM notifies the client if

there is a more appropriate method available or if the client's criteria cannot be achieved on a certain sample matrix (i.e., due to matrix or dilutions).

Occasionally, we are asked to transfer a non-standardized method from a client into our lab or to develop a new method, when one is not available. In the case of a method transfer, we set up the client's method and perform some initial evaluation. After the initial evaluation, we may make recommendations on how to improve method performance. If the method appears to be adequate, we determine linearity, specificity, precision, accuracy, MDL, and LOQ by performing calibrations, analyzing method blanks, and carrying out method detection limit and IDOC studies.

In the case of method development, we work with the client and/or data user to determine the level of validation required ensuring that the method meets its intended purpose. In addition to the elements above, we also determine standard and sample stability and robustness depending on the scope of the project. Typically, a standard operating procedure is written and submitted to the client with the results of the validation. These steps are completed prior to analysis of field samples. Data related to the setup of the method are archived.

19.4.2 Demonstration of Capability

Before the laboratory may institute a new method and begin reporting results, the laboratory shall confirm that it can properly operate the method. In general, this demonstration does not test the performance of the method in real world samples, but in an applicable and available clean matrix sample. If the method is for the testing of analytes that are not conducive to spiking, demonstration of capability may be performed on quality control samples.

A demonstration of capability (DOC, Lab SOP # KNOX-QA-0009) is performed whenever there is a change in instrument type (e.g., new instrumentation), matrix, method or personnel (e.g., analyst has not performed the test within the last 12 months).

Note: The laboratory shall have a DOC for all analytes included in the methods that the laboratory performs, and proficiency DOCs for each analyst shall include all analytes that the laboratory routinely performs. Addition of non-routine analytes does not require new DOCs for all analysts if those analysts are already qualified for routine analytes tested using identical chemistry and instrument conditions.

The initial demonstration of capability must be thoroughly documented and approved by the Technical Manager and QA Manager prior to independently analyzing client samples. All associated documentation must be retained in accordance with the laboratory's archiving procedures.

The laboratory must have an approved SOP, demonstrate satisfactory performance, and conduct an MDL study (when applicable). There may be other requirements as stated within the published method or regulations (e.g., retention time window study).

Note: In some instances, a situation may arise where a client requests that an unusual analyte be reported using a method where this analyte is not normally reported. If the analyte is being reported for regulatory purposes, the method must meet all procedures outlined within this QA Manual (SOP, MDL, and Demonstration of Capability). If the client states that the information is not for regulatory purposes, the result may be reported as long as the following criteria are met:

- The instrument is calibrated for the analyte to be reported using the criteria for the method and ICV/CCV criteria are met (unless an ICV/CCV is not required by the method or criteria are per project DQOs).

- The laboratory's nominal or default reporting limit (RL) is equal to the quantitation limit (QL), must be at or above the lowest non-zero standard in the calibration curve and must be reliably determined. Project RLs are client specified reporting levels which may be higher than the QL. Results reported below the QL must be qualified as estimated values. Also, see Section 19.6.1.3, Relationship of Limit of Detection (LOD) to Quantitation Limit (QL).
- The client request is documented and the lab informs the client of its procedure for working with unusual compounds. The final report must be footnoted: *Reporting Limit based on the low standard of the calibration curve.*

19.4.3 Initial Demonstration of Capability (IDOC) Procedures

19.4.3.1 A quality control sample shall be obtained from an outside source. If not available, the QC sample may be prepared by the laboratory using stock standards that are prepared independently from those used in instrument calibration.

19.4.3.2 The analyte(s) shall be diluted in a volume of clean matrix sufficient to prepare four aliquots at the concentration specified by the method or the laboratory SOP.

19.4.3.3 At least four aliquots are prepared (including any applicable routine clean-up procedures) and analyzed according to the test method (either concurrently or over a period of days).

19.4.3.4 Using all of the results, calculate the mean recovery in the appropriate reporting units and the standard deviation for each parameter of interest.

19.4.3.5 When it is not possible to determine the mean and standard deviation, such as for presence, absence and logarithmic values, the laboratory will assess performance against criteria described in the laboratory SOP.

19.4.3.6 Compare the information obtained above to the corresponding acceptance criteria for precision and accuracy in the test method (if applicable) or in laboratory generated acceptance criteria (LCS or interim criteria) if there is no mandatory criteria established. If any one of the parameters do not meet the acceptance criteria, the performance is unacceptable for that parameter.

19.4.3.7 When one or more of the tested parameters fail at least one of the acceptance criteria, the analyst must proceed according to either option listed below:

• Locate and correct the source of the problem and repeat the test for all parameters of interest beginning with 19.4.3.3 above.

• Beginning with 19.4.3.3 above, repeat the test for all parameters that failed to meet criteria. Repeated failure, however, will confirm a general problem with the measurement system. If this occurs, locate and correct the source of the problem and repeat the test for all compounds of interest beginning with 19.4.3.1 above.

Note: Results of successive LCS analyses can be used to fulfill the DOC requirement.

19.4.3.8 Refer to SOP KNOX-QA-0009, Personnel Orientation and Training for further details.

Methods on line prior to the effective date of this Section shall be updated to the procedures outlined above as new analysts perform their demonstration of capability. A copy of the new record will replace that which was used for documentation in the past. At a minimum, the precision and accuracy of four mid-level laboratory control samples must have been compared to the laboratory's quality control acceptance limits.

19.5 Laboratory Developed Methods and Non-Standard Methods

Any new method developed by the laboratory must be fully defined in an SOP and validated by qualified personnel with adequate resources to perform the method. Method specifications and the relation to client requirements must be clearly conveyed to the client if the method is a non-standard method (not a published or routinely accepted method). The client must also be in agreement to the use of the non-standard method.

19.6 Validation of Methods

Validation is the confirmation by examination and the provision of objective evidence that the particular requirements for a specific intended use are fulfilled.

All non-standard methods, laboratory designed/developed methods, standard methods used outside of their scope, and major modifications to published methods must be validated to confirm they are fit for their intended use. The validation will be as extensive as necessary to meet the needs of the given application. The results are documented with the validation procedure used and contain a statement as to the fitness for use.

19.6.1 Method Validation and Verification Activities for All New Methods

While method validation can take various courses, the following activities can be required as part of method validation. Method validation records are designated QC records and are archived accordingly.

When changes are made to any validated methods, the influence of such changes shall be documented and, if appropriate, a new validation shall be performed.

19.6.1.1 **Determination of Method Selectivity** – Method selectivity is the demonstrated ability to discriminate the analyte(s) of interest from other compounds in the specific matrix or matrices from other analytes or interference. In some cases to achieve the required selectivity for an analyte, a confirmation analysis is required as part of the method.

19.6.1.2 **Determination of Method Sensitivity** – Sensitivity can be both estimated and demonstrated. Whether a study is required to estimate sensitivity depends on the level of method development required when applying a particular measurement system to a specific set of samples. Detection limit studies are conducted as described in Section 19.7. Where other protocols for estimations and/or demonstrations of sensitivity are required by regulation or client agreement, these shall be followed.

19.6.1.3 **Relationship of Limit of Detection (LOD) to the Limit of Quantitation (LOQ)** – An important characteristic of expression of sensitivity is the distinction between the LOD and the LOQ. The LOD is the minimum level at which the presence of an analyte can be reliably concluded. The LOQ is the minimum concentration of analyte that can be quantitatively determined with acceptable precision and bias, equivalent to the laboratory's routine reporting limit (RL). For most instrumental measurement systems, there is a region where semi-quantitative data is generated around the LOD (both above and below the estimated MDL or LOD) and below the LOQ. In this region, detection of an analyte may be confirmed but quantification of the analyte is unreliable within the accuracy and precision guidelines of the measurement system. When an analyte is detected below the LOQ, and the presence of the analyte is confirmed by meeting the qualitative identification criteria for the analyte, the analyte can be reliably reported, but the amount of the analyte can only be estimated. If data is to be reported in this region, it must be done so with a qualification that denotes the semi-quantitative nature of the result.

19.6.1.4 **Determination of Interferences** – A determination that the method is free from interferences in a blank matrix is performed.

19.6.1.5 **Determination of Range** – Where appropriate to the method, the quantitation range is determined by comparison of the response of an analyte in a curve to established or targeted criteria. Generally the upper quantitation limit is defined by highest acceptable calibration concentration. The lower quantitation limit or QL cannot be lower than the lowest non-zero calibration level, and can be constrained by required levels of bias and precision.

19.6.1.6 **Determination of Accuracy and Precision** – Accuracy and precision studies are generally performed using replicate analyses, with a resulting percent recovery and measure of reproducibility (standard deviation, relative standard deviation) calculated and measured against a set of target criteria.

19.6.1.7 **Documentation of Method** – The method is formally documented in an SOP. If the method is a minor modification of a standard laboratory method that is already documented in an SOP, an SOP Attachment describing the specific differences in the new method is acceptable in place of a separate SOP.

19.6.1.8 **Continued Demonstration of Method Performance** – Continued demonstration of method performance is addressed in the SOP. Continued demonstration of method performance is generally accomplished by batch specific QC samples such as LCS, method blanks or PT samples.

19.7 Method Detection Limit (MDL) / Limits of Detection (LOD)

The MDL is the minimum measured quantity of a substance that can be reported with 99% confidence that the concentration is distinguishable from method blank results, consistent with 40CFR Part 136 Appendix B, August, 2017. The MDL is equivalent to the TNI LOD or DL, and is also equivalent to the DoD/DOE Quality Systems Manual (QSM) DL. The working or final MDL is the higher of the MDL value determined from spikes (MDLs) and the MDL value determined from blanks (MDLb). An initial MDL study shall be performed during the method validation process and when the method is altered in a way that can reasonably be expected to change its sensitivity. On-going data are collected during each quarter in which samples are being analyzed. At least once every 13 months the MDLs and MDLb are re-calculated and re-evaluated using data collected during the preceding period. Details of the laboratory's procedure for conducting MDL studies are given in NDSC Document No. CA-Q-S-006.

19.8 Verification of Detection Limits

If it is found during the re-evaluation of detection limit results that more than 5% of the spiked samples do not return positive numeric results that meet all method qualitative identification criteria, then then spiking level shall be increased and the initial MDL study pre-performed at the new spiking concentration. For DoD/DOE labs, additional guidance provided in NDSC Document No. No. CA-Q-S-006 - Attachment 2.

19.9 Instrument Detection Limits (IDL)

The IDL is sometimes used to assess the reasonableness of the MDL or in some cases required by the analytical method or program requirements. IDLs are most commonly used in metals analyses but may be useful in demonstration of instrument performance in other areas.

IDLs are calculated to determine an instrument's sensitivity independent of any preparation method. IDLs are calculated either using 7 replicate spike analyses, like MDL but without

sample preparation, or by the analysis of 10 instrument blanks and calculating 3 x the absolute value of the standard deviation.

19.10 Limit of Quantitation

The LOQ shall be at a concentration equivalent to the lowest calibration standard concentration, with the exception of methods using a single-point calibration, and shall be greater than the MDL. The LOQ is verified by preparing and analyzing spikes at concentrations 1-2X the selected LOQ, employing the complete analytical process. For DoD/DOE labs, additional requirements provided in NDSC Document No. CA-Q-S-006.

When the laboratory establishes a quantitation limit, it must be initially verified by the analysis of a low level standard or QC sample at 1-2 times the reporting limit or by a DL check samples at or below the LOQ. The LOQ is verified annually thereafter. The annual requirement is waived for methods that have an annually verified MDL. The laboratory will comply with any regulatory requirements.

19.11 Retention Time Windows

Most organic analyses and some inorganic analyses use chromatography techniques for qualitative and quantitative determinations. For every chromatography analysis or as specified in the reference method, each analyte will have a specific time of elution from the column to the detector. This is known as the analyte's retention time. The variance in the expected time of elution is defined as the retention time window. As the key to analyte identification in chromatography, retention time windows must be established on every column for every analyte used for that method. These records are kept on-file and available for review. Complete details are available in the laboratory SOPs.

19.12 Evaluation of Selectivity

The laboratory evaluates selectivity by following the checks within the applicable analytical methods, which include mass spectral tuning, mass resolution, second column confirmation, ICP interelement interference checks, chromatography retention time windows, and method blanks.

19.13 Estimation of Uncertainty of Measurement

19.13.1 Uncertainty is "a parameter associated with the result of a measurement, that characterizes the dispersion of the values that could reasonably be attributed to the measurand" (as defined by the International Vocabulary of Basic and General Terms in Metrology, ISO Geneva, 1993, ISBN 92-67-10175-1). Knowledge of the uncertainty of a measurement provides additional confidence in a result's validity. Its value accounts for all the factors which could possibly affect the result, such as adequacy of analyte definition, sampling, matrix effects and interferences, climatic conditions, variances in weights, volumes, and standards, analytical procedure, and random variation. Some national accreditation organizations require the use of an "expanded uncertainty" defined as the range within which the value of the measurand is believed to lie within at least a 95% confidence level with the coverage factor k=2.

19.13.2 Uncertainty is not error. Error is a single value (i.e., the difference between the true result and the measured result). On environmental samples, the true result is never known. The measurement is the sum of the unknown true value and the unknown error. Unknown error is a combination of systematic error, or bias, and random error. Bias varies predictably,

constantly, and independently from the number of measurements. Random error is unpredictable, assumed to be Gaussian in distribution, and reducible by increasing the number of measurements.

19.13.3 The minimum uncertainty associated with results generated by the laboratory can be determined by using the Laboratory Control Sample (LCS) accuracy range for a given analyte. The LCS limits are used to assess the performance of the measurement system since they take into consideration all of the laboratory variables associated with a given test over time (except for variability associated with the sampling and the variability due to matrix effects). The percent recovery of the LCS is compared either to the method-required LCS accuracy limits or to the statistical, historical, in-house LCS accuracy limits.

19.13.4 To calculate the uncertainty for the specific result reported, multiply the result by the decimal of the lower end of the LCS range percent value for the lower end of the uncertainty range, and multiply the result by the decimal of the upper end of the LCS range percent value for the upper end of the uncertainty range. These calculated values represent uncertainties at approximately the 99% confidence level with a coverage factor of k = 3. As an example, for a reported result of 1.0 mg/L with an LCS recovery range of 50 to 150%, the estimated uncertainty in the result would be 1.0 +/- 0.5 mg/L.

19.13.5 Refer to SOP KNOX-QA-0019 “Estimation of Measurement Uncertainty” for details regarding calculations that may be used to estimate measurement uncertainty associated with laboratory results.

19.13.6 In the case where a well-recognized test method specifies limits to the values of major sources of uncertainty of measurement (e.g., 524.2, 525, etc.) and specifies the form of presentation of calculated results, no further discussion of uncertainty is required.

19.14 Sample Reanalysis Guidelines

Because there is a certain level of uncertainty with any analytical measurement, a sample re-preparation (where appropriate) and subsequent analysis (hereafter referred to as ‘reanalysis’) may result in either a higher or lower value from an initial sample analysis. There are also variables that may be present (e.g., sample homogeneity, analyte precipitation over time, etc.) that may affect the results of a reanalysis. Based on the above comments, the laboratory will reanalyze samples at a client’s request with the following caveats. Client specific Contractual Terms & Conditions for reanalysis protocols may supersede the following items.

Homogenous samples: If a reanalysis agrees with the original result to within the RPD limits for MS/MSD or Duplicate analyses, or within $\pm$ 1 reporting limit for samples $\leq$ 5x the reporting limit, the original analysis will be reported. At the client’s request, both results may be reported on the same report but not on two separate reports.

- If the reanalysis does not agree (as defined above) with the original result, then the laboratory will investigate the discrepancy and reanalyze the sample a third time for confirmation if sufficient sample is available.
- Any potential charges related to reanalysis are discussed in the contract terms and conditions or discussed at the time of the request. The client will typically be charged for reanalysis unless it is determined that the lab was in error.
- Due to the potential for increased variability, reanalysis may not be applicable to Non-homogenous, Encore, and Sodium Bisulfate preserved samples. See the Area Supervisor or Laboratory Director if unsure.

19.15 Control of Data

The laboratory has policies and procedures in place to ensure the authenticity, integrity, and accuracy of the analytical data generated by the laboratory.

19.15.1 Computer and Electronic Data Related Requirements

The three basic objectives of our computer security procedures and policies are shown below.

The laboratory is currently using the Eurofins LIMS system, which has been highly customized to meet the needs of the laboratory.

19.15.1.1 Maintain the Database Integrity – Assurance that data is reliable and accurate through data verification (review) procedures, password-protecting access, anti-virus protection, data change requirements, as well as an internal LIMS permissions procedure.

- LIMS Database Integrity is achieved through data input validation, internal user controls, documentation of system failures and corrective actions taken, and data change requirements.
- Spreadsheets and other software developed in-house must be verified with documentation through hand calculations prior to use. Cells containing calculations must be lock-protected and controlled.
- Instrument hardware and software adjustments are safeguarded through maintenance logs, audit trails and controlled access.

19.15.1.2 Ensure Information Availability – Protection against loss of information or service is ensured through scheduled back-ups, stable file server network architecture, secure storage of media, line filter, Uninterruptible Power Supply (UPS), and maintaining older versions of software as revisions are implemented.

19.15.1.3 Maintain Confidentiality – Ensure data confidentiality through physical access controls such as password protection or website access approval when electronically transmitting data.

19.15.2 Data Reduction

The complexity of the data reduction depends on the analytical method and the number of discrete operations involved (e.g., extractions, dilutions, instrument readings and concentrations). The analyst calculates the final results from the raw data or uses appropriate computer programs to assist in the calculation of final reportable values.

For manual data entry, e.g., Wet Chemistry, the data is reduced by the analyst and then verified by the Department Manager or alternate analyst prior to updating the data in LIMS. The spreadsheets, or any other type of applicable documents, are signed by both the analyst and alternate reviewer to confirm the accuracy of the manual entry(s).

Manual integration of peaks will be documented and reviewed and the raw data will be flagged using NDSC Document No. CA-Q-S-002, Acceptable Manual Integration Practices.

Analytical results are reduced to appropriate concentration units specified by the analytical method, taking into account factors such as dilution, sample weight or volume, etc. Blank correction will be applied only when required by the method or per manufacturer's indication; otherwise, it should not be performed. Calculations are independently verified by appropriate laboratory staff. Calculations and data reduction steps for various methods are summarized in the respective analytical SOPs or program requirements.

19.15.2.1 All raw data must be retained in the worklist folder, computer file (if appropriate), and/or runlog. All criteria pertinent to the method must be recorded. The documentation is recorded at the time observations or calculations are made and must be signed or initialed/dated (month/day/year). It must be easily identifiable who performed which tasks if multiple people were involved.

(**Note:** This section does not apply to the Ohio VAP as all computations are done by the instrument data system and by the LIMS.)

19.15.2.2 In general, concentration results are reported in milligrams per liter (mg/l) or micrograms per liter (µg/l) for liquids and milligrams per kilogram (mg/kg) or micrograms per kilogram (µg/kg) for solids. For values greater than 10,000 mg/l, results can be reported in percent, i.e., 10,000 mg/l = 1**%.** Units are defined in each lab SOP.

19.15.2.3 In reporting, the analyst or the instrument output records the raw data result using values of known certainty plus one uncertain digit. If final calculations are performed external to LIMS, the results should be entered in LIMS with at least three significant figures. In general, results are reported to 2 significant figures on the final report.

19.15.2.4 For those methods that do not have an instrument printout or an instrumental output compatible with the LIMS, the raw results and dilution factors are entered directly into LIMS by the analyst, and the software calculates the final result for the analytical report. LIMS has a formatter for significant figure criterion for each analyte.

19.15.2.5 The laboratory strives to import data directly from instruments or calculation spreadsheets to ensure that the reported data are free from transcription and calculation errors. For those analyses with an instrumental output compatible with the LIMS, the raw results and dilution factors are transferred into LIMS, electronically after reviewing the quantitation report, and removing unrequested or poor spectrally-matched compounds. The analyst prints a copy of what has been entered to check for errors. This printout and the instrument's printout of calibrations, concentrations, retention times, chromatograms, and mass spectra, if applicable, are retained with the data file. The data file is stored in a monthly folder on the instrument computer; periodically, this file is transferred to the server and, eventually, to a tape file.

19.15.3 Logbook / Worksheet Use Guidelines

Logbooks and worksheets are filled out 'real time' and have enough information on them to trace the events of the applicable analysis/task. (e.g. calibrations, standards, analyst, sample ID, date, time on short holding time tests, temperatures when applicable, calculations are traceable, etc.)

- Corrections are made following the procedures outlined in Section 14.
- Logbooks are controlled by the QA department. A record is maintained of all logbooks in the lab.
- Unused portions of pages must be "Z"'d out, signed and dated.
- Worksheets are created with the approval of the Technical Manager/QA Manager at the facility. The QA Manager controls all worksheets following the procedures in Section 6.

19.15.4 Review / Verification Procedures

A minimum of three levels of review are performed before final sample data is reported to the client.

• Log-In Review.
• Second level technical review.
• Third level project management completeness review.

19.15.4.1 Log-In Review - The data review process starts at the sample receipt stage. Sample control personnel review chain-of-custody forms and project instructions from the project management group. This is the basis of the sample information and analytical instructions entered into the LIMS. The log-in instructions are reviewed by the personnel entering the information, and a second level review is conducted by the project management staff.

19.15.4.2 First Level Data Review - The next level of data review occurs with the analysts. As data are generated, analysts review their work to ensure that the results meet project and SOP requirements. First level reviews include inspection of all raw data (e.g., instrument output for continuous analyzers, chromatograms, spectra, and manual integrations), evaluation of calibration/calibration verification data in the day's analytical run, evaluation of QC data, and reliability of sample results. The analyst transfers data into LIMS, data qualifiers are added as needed. All first level reviews are documented.

19.15.4.3 Second Level Data Review – All analytical data are subject to review by a second qualified analyst or supervisor. Second level reviews include inspection of all raw data (e.g., instrument output, chromatograms, and spectra) including 100% of data associated with any changes made by the primary analyst, such as manual integrations or reassignment of peaks to different analytes, or elimination of false negative analytes. The second review also includes evaluation of initial calibration/calibration verification data in the day's analytical run, evaluation of QC data, reliability of sample results, qualifiers and NCM narratives. Manual calculations are checked in second level review. All second level reviews are documented.

Issues that deem further review include the following:

- QC data are outside the specified control limits for accuracy and precision
- Reviewed sample data does not match with reported results
- Unusual detection limit changes are observed
- Samples having unusually high results
- Samples exceeding a known regulatory limit
- Raw data indicating some type of contamination or poor technique
- Inconsistent peak integration
- Transcription errors
- Results outside of calibration range

19.15.4.4 Unacceptable analytical results may require reanalysis of the samples. Any problems are brought to the attention of the Laboratory Director, Project Manager, Quality Director/Manager, Technical Manager, or Supervisor for further investigation. Corrective action is initiated whenever necessary.

19.15.4.5 The results are then entered or directly transferred into the computer database and a (.pdf) is printed for the client.

19.15.4.6 As a final review prior to the release of the report, the Project Manager reviews the results for appropriateness and completeness. This review and approval ensures that client

requirements have been met and that the final report has been properly completed. The process includes, but is not limited to, verifying that the COC is followed, cover letters / narratives are present, flags are appropriate, and project specific requirements are met. The Project Manager may also evaluate the validity of results for different test methods given expected chemical relationships.

19.15.4.7 Any project that requires a data package is subject to a tertiary data review for transcription errors and acceptable quality control requirements. The Project Manager then signs the final report. The accounting personnel also check the report for any clerical or invoicing errors. When complete, the report is sent out to the client.

19.15.4.8 A brief overview of sample flow~~s~~ and information through the laboratory, as well as data review and validation, is presented in Figure 19-1.

19.15.5 Manual Integrations

Computerized data systems provide the analyst with the ability to re-integrate raw instrument data in order to optimize the interpretation of the data. Though manual integration of data is an invaluable tool for resolving variations in instrument performance and some sample matrix problems, when used improperly, this technique would make unacceptable data appear to meet quality control acceptance limits. Improper re-integrations lead to legally indefensible data, a poor reputation, or possible laboratory decertification. Because guidelines for re-integration of data are not provided in the methods and most methods were written prior to widespread implementation of computerized data systems, the laboratory trains all analytical staff on proper manual integration techniques using NDSC Document No. CA-Q-S-002, Acceptable Manual Integration Practices,

The analyst must adjust baseline or the area of a peak in some situations, for example when two compounds are not adequately resolved or when a peak shoulder needs to be separated from the peak of interest. The analyst must use professional judgment to determine when manual integrating is required. Analysts are encouraged to ask for assistance from a senior analyst or manager when in doubt.

19.15.5.1 Analysts shall not increase or decrease peak areas for the sole purpose of achieving acceptable QC recoveries that would have otherwise been unacceptable. The intentional recording or reporting of incorrect information (or the intentional omission of correct information) is against company principles and policy and is grounds for immediate termination.

19.15.5.2 Client samples, performance evaluation samples, and quality control samples are all treated equally when determining whether or not a peak area or baseline should be manually adjusted.

19.15.5.3 All manual integrations receive a second level review. Manual integrations must be indicated on ~~an~~ expanded scale “before” and “after” chromatograms such that the integration performed can be easily evaluated during data review. Expanded scale “before” chromatograms are also required for all manual integrations on QC parameters (calibrations, calibration verifications, laboratory control samples, internal standards, surrogates, etc.) unless the laboratory has another documented NDSC approved procedure in place that can demonstrate an active process for detection and deterrence of improper integration practices.

Figure 19-1. Example – Work Flow

Action	Personnel Involved
Bottle orders generated upon request - Bottles packed and shipped to the client under CoC	- Client Service Personnel / PM - Bottle Preparation Personnel
Samples received at laboratory - Unpacked and reconciled against client paperwork or CoC - Samples acceptance documented against completed (CoC)[1]	Sample Login Personnel
Samples are entered into LIMS - Job # assigned - Methods/Analytes linked from project - Sample labels generated - LIMS backlogs updated immediately	Sample Login Personnel
Sample Storage - Analysts are notified of short TATs and hold-time samples - Samples stored in assigned locations (refrigerated walk-in coolers, freezers, VOA refrigerators; etc.)	Sample Login Personnel
Sample Receipt Acknowledgement sent to client (when requested)	Sample Login Personnel
Sample Acquisition/Testing - Analysts collect samples from assigned storage. - Remaining sample returned to storage.	Analysts / Login Personnel
Preparation and analysis is performed according to selected analytical method and applicable Project notes - Raw data recorded/reviewed[2] - Data imported to LIMS	Analysts
LIMS performs calculations: - Data reviewed by primary/secondary analyst - Data approved as final	Data Process (LIMS / Analyst)
Generation/review/release of reports (automated through LIMS) - Electronic copy saved in LIMS	Project Management Assistants / Reporting Group
Electronic Data Deliverables (EDDs) generated (as applicable)	PM Group
Hard copy and electronic batch raw data is archived according to data archiving procedures.	Analysts / IT

[1]Refer to Sec. 23.3.
[2]Analyses requiring the analyst's interpretation may involve manual data reduction before entry into LIMS.

20.0 EQUIPMENT and CALIBRATIONS

20.1 Overview

The laboratory purchases the most technically advanced analytical instrumentation for sample analyses. Instrumentation is purchased on the basis of accuracy, dependability, efficiency and sensitivity. Each laboratory is furnished with all items of sampling, preparation, analytical testing and measurement equipment necessary to correctly perform the tests for which the laboratory has capabilities. Each piece of equipment is capable of achieving the required accuracy and complies with specifications relevant to the method being performed. Before being placed into use, the equipment (including sampling equipment) is calibrated and checked to establish that it meets its intended specification. The calibration routines for analytical instruments establish the range of quantitation. Calibration procedures are specified in laboratory SOPs. A list of available laboratory instrumentation is presented in Table 20-1.

Equipment is only operated by authorized and trained personnel. Manufacturer's instructions for equipment use are readily accessible to all appropriate laboratory personnel.

20.2 Preventive Maintenance

The laboratory follows a well-defined maintenance program to ensure proper equipment operation and to prevent the failure of laboratory equipment or instrumentation during use. This program of preventive maintenance helps to avoid delays due to instrument failure.

Routine preventive maintenance procedures and frequency, such as cleaning and replacements, should be performed according to the procedures outlined in the manufacturer's manual. Qualified personnel must also perform maintenance when there is evidence of degradation of peak resolution, a shift in the calibration curve, loss of sensitivity, or failure to continually meet one of the quality control criteria.

Scheduled routine maintenance is defined in each method SOP. It is the responsibility of each Group Leader to ensure that instrument maintenance logs are kept for all equipment in his/her department. Preventative maintenance procedures are also outlined in analytical SOPs or instrument manuals. (Note: for some equipment, the log used to document maintenance is also used to monitor performance.)

Instrument maintenance logs are controlled and are used to document instrument problems, instrument repair and maintenance activities. Maintenance logs shall be kept for all major pieces of equipment. Instrument maintenance logs may also be used to specify instrument parameters.

- Documentation must include all major maintenance activities such as contracted preventive maintenance and service and in-house activities such as the replacement of electrical components, lamps, tubing, valves, columns, detectors, cleaning and adjustments.
- Each entry in the instrument log includes the analyst's initials, the date, a detailed description of the problem (or maintenance needed/scheduled), a detailed explanation of the solution or maintenance performed, and a verification that the equipment is functioning properly (state what was used to determine a return to control. e.g. CCV run on *'date'* was acceptable, or instrument recalibrated on 'date' with acceptable verification, etc.) must also be documented in the instrument records.

- When maintenance or repair is performed by an outside agency, service receipts detailing the service performed are affixed into the maintenance logbook. The analyst signs across the entered item and the logbook page so that it is clear that the insert is missing if only half a signature is found in the logbook.
- In addition, the maintenance records contain:
  - The identification of the instrument/equipment (instrument's Serial Number and Model Number).
  - The date the instrument/equipment was put into use.
  - If available, the condition when the instrument was received (e.g., new, used, reconditioned).
- If an instrument requires repair (subjected to overloading or mishandling), gives suspect results, or otherwise has shown to be defective or outside of specified limits it shall be taken out of operation and tagged as out-of-service or otherwise isolated until such a time as the repairs have been made and the instrument can be demonstrated as operational by calibration and/or verification or other test to demonstrate acceptable performance. The laboratory shall examine the effect of this defect on previous analyses.

In the event of equipment malfunction that cannot be resolved, service shall be obtained from the instrument vendor manufacturer, or qualified service technician, if such a service can be tendered. If on-site service is unavailable, arrangements shall be made to have the instrument shipped back to the manufacturer for repair. Back up instruments, which have been approved, for the analysis shall perform the analysis normally carried out by the malfunctioning instrument. If the back-up is not available and the analysis cannot be carried out within the needed timeframe, the samples shall be subcontracted.

At a minimum, if an instrument is sent out for service or transferred to another facility, it must be recalibrated and the laboratory MDL verified (using an MDLV) prior to return to lab operations.

An inventory of critical spare parts is maintained to minimize delays in the completion of analyses. For most test methods, back-up instrumentation is also maintained to minimize delays.

20.3 Support Equipment

This section applies to all devices that may not be the actual test instrument, but are necessary to support laboratory operations. These include but are not limited to: balances, ovens, refrigerators, freezers, incubators, water baths, temperature measuring devices and volumetric dispensing devices if quantitative results are dependent on their accuracy, as in standard preparation and dispensing or dilution into a specified volume. All raw data records associated with the support equipment are retained to document instrument performance.

20.3.1 Weights and Balances

The accuracy of the balances used in the laboratory is checked every working day, before use. All balances are placed on stable counter tops. Balances must have their calibration checked each day of use with two weights bracketing the working range.

Each balance is checked prior to initial serviceable use with at least two certified ASTM type 1 weights spanning its range of use (weights that have been calibrated to ASTM type 1 weights

may also be used for daily verification). ASTM type 1 weights used only for calibration of other weights (and no other purpose) are inspected for corrosion, damage or nicks at least annually and if no damage is observed, they are calibrated at least every 5 years by an outside calibration laboratory. Any weights (including ASTM Type 1) used for daily balance checks or other purposes are recalibrated/recertified annually to NIST standards (this may be done internally if laboratory maintains "calibration only" ASTM type 1 weights).

All balances are serviced annually by a qualified service representative, who supplies the laboratory with a certificate that identifies traceability of the calibration to the NIST standards.

All of this information is recorded in logs, and the recalibration/recertification certificates are kept on file. Further details on balance calibration may be found in SOP KNOX-QA-0005, Balance Calibration and Weight Verification.

20.3.2 pH, Conductivity, and Turbidity Meters

The pH meters used in the laboratory are accurate to ± 0.1 pH units, and have a scale readability of at least 0.05 pH units. The meters automatically compensate for the temperature, and are calibrated with at least two working range buffer solutions before each use.

Conductivity meters used in the laboratory are capable or measuring conductivity with an error not exceeding 1% or one umhos/cm.

All of this information is documented in logbooks.

Consult the pH and Routine Monitoring of Deionized Water SOPs for further information.

20.3.3 Thermometers

All thermometers are calibrated on an annual basis with a NIST-traceable thermometer. IR thermometers and digital thermometers are calibrated quarterly.

- If the temperature measuring device is used over a range of 10°C or less, then a single point verification within the range of use is acceptable;
- If the temperature measuring device is used over a range of greater than 10°C, then the verification must bracket the range of use.

The NIST mercury reference thermometer(s) are recalibrated every five years (unless thermometer has been exposed to temperature extremes or apparent separation of internal liquid) by an approved outside service and the provided certificate of traceability is kept on file. NIST digital reference thermometers are recalibrated every two years. The NIST thermometer(s) have increments of 1ºC, and have a range applicable to all method and certification requirements. The NIST traceable thermometer is used for no other purpose than to calibrate other thermometers.

Thermometer calibrations are documented as described in SOP KNOX-QA-0010, Temperature Monitoring and Thermometer Calibration.

20.3.4 Refrigerators/Freezer Units, Water baths, Ovens and Incubators

The temperatures of all refrigerator units and freezers used for sample and standard storage are monitored each working day. Monitoring is performed 7 days per week to ensure that samples remain within an acceptable range.

Ovens, water baths and incubators are monitored on days of use.

All of this equipment has a unique identification number, and is assigned a thermometer for monitoring.

Sample storage refrigerator temperatures are kept between > 0ºC and ≤ 6 ºC.

Specific temperature settings/ranges for other refrigerators, ovens, water baths, and incubators can be found in method specific SOPs.

The temperatures are documented in temperature logbooks or in the method-specific logbooks/benchsheets.

20.3.5 Autopipettors, Dilutors, and Syringes

Mechanical volumetric dispensing devices including burettes (except Class A Glassware and glass microliter syringes) are given unique identification numbers and the delivery volumes are verified gravimetrically, at a minimum, on a quarterly basis.

Any device not regularly verified cannot be used for any quantitative measurements

Refer to SOP KNOX-QA-0012, Pipetter and Volumetric Labware Calibration Verification.

Micro-syringes are purchased from Hamilton Company. Each syringe is traceable to NIST. The laboratory keeps on file an “Accuracy and Precision Statement of Conformance” from Hamilton attesting established accuracy.

20.4 Instrument Calibrations

Calibration of analytical instrumentation is essential to the production of quality data. Strict calibration procedures are followed for each method. These procedures are designed to determine and document the method detection limits, the working range of the analytical instrumentation and any fluctuations that may occur from day to day.

Sufficient raw data records are retained to allow an outside party to reconstruct all facets of the initial calibration. Records contain, but are not limited to, the following: calibration date, method, instrument, analyst(s) initials or signatures, analysis date, analytes, concentration, response, and type of calibration (Avg RF, curve, or other calculations that may be used to reduce instrument responses to concentration.)

Sample results must be quantitated from the initial calibration and may not be quantitated from any continuing instrument calibration verification unless otherwise required by regulation, method or program.

If the initial calibration results are outside of the acceptance criteria, corrective action is performed and any affected samples are reanalyzed if possible. If the reanalysis is not possible, any data associated with an unacceptable initial calibration will be reported with appropriate data qualifiers (refer to Section 14).

Note: Instruments are calibrated initially and as needed after that and at least annually. The annual requirement does not apply to Isotope dilution.

20.4.1 Calibration Standards

Calibration standards are prepared using the procedures indicated in the Reagents and Standards section of the determinative method SOP. If a reference method does not specify the number of calibration standards, a minimum of 3 calibration points (exception being ICP and ICP/MS methods) shall be used.

Standards for instrument calibration are obtained from a variety of sources. All standards are traceable to national or international standards of measurement, or to national or international standard reference materials.

The lowest concentration calibration standard that is analyzed during an initial calibration must be at or below the stated reporting limit for the method based on the final volume of extract (or sample).

The other concentrations define the working range of the instrument/method or correspond to the expected range of concentrations found in actual samples that are also within the working range of the instrument/method. Results of samples not bracketed by initial instrument calibration standards (within calibration range to at least the same number of significant figures used to report the data) must be reported as having less certainty, e.g., defined qualifiers or flags (additional information may be included in the case narrative). The exceptions to these rules. ICP and ICPMS methods which define the working range with periodic linear dynamic range studies, rather than through the range of concentrations of daily calibration standards.

All initial calibrations are verified with a standard obtained from a second source and traceable to a national standard, when available (or vendor certified different lot if a second source is not available). For unique situations, such as air analysis where no other source or lot is available, a standard made by a different analyst would be considered a second source. This verification occurs immediately after the calibration curve has been analyzed, and before the analysis of any samples.

20.4.1.1 Calibration Verification

The calibration relationship established during the initial calibration must be verified initially and at least daily as specified in the laboratory method SOPs in accordance with the referenced analytical methods and in the 2009 and 2016 TNI Standard. The process of calibration verification applies to both external standard and internal standard calibration techniques, as well as to linear and non-linear calibration models. Initial calibration verification (ICV) is with a standard source secondary (second source standard) to the calibration standards, but continuing calibration verifications (CCV) may use the same source standards as the calibration curve.

Note: The process of calibration verification referred to here is fundamentally different from the approach called "calibration" in some methods. As described in those methods, the calibration factors or response factors calculated during calibration are used to update the calibration factors or response factors used for sample quantitation. This approach, while employed in other EPA programs, amounts to a daily single-point calibration.

All target analytes and surrogates, including those reported as non-detects, must be included in periodic calibration verifications for purposes of retention time confirmation and to demonstrate that calibration verification criteria are being met, i.e., RPD, per 2009 and 2016 TNI Std. EL-V1M4 Sec. 1.7.2.

Note: For Ohio EPA VAP projects the calibration solution used for calibration verification must be from the same source as that used for the calibration curve unless the method states otherwise.

All samples must be bracketed by periodic analyses of standards that meet the QC acceptance criteria (e.g., calibration and retention time). The frequency is found in the determinative methods or SOPs.

Note: If an internal standard calibration is being used then bracketing calibration verification standards may not be are not required, only daily verifications are needed. The results from

these verification standards must meet the calibration verification criteria and the retention time criteria (if applicable).

Generally, the calibrations must be verified by an ICV analyzed immediately following initial calibration and before sample analysis. The ICV may be used as the first bracketing CCV, if criteria for both are met.

A continuing instrument calibration verification (CCV) is generally analyzed at the beginning of each 12-hour analytical shift during which samples are analyzed. The 12-hour analytical shift begins with the injection of the calibration verification standard (or the MS tuning standard in MS methods). The shift ends after the completion of the analysis of the last sample, QC, or standard that can be injected within 12-hours of the beginning of the shift.

For methods that have quantitation by external calibration models, a CCV is analyzed at the end of each analytical sequence. Some methods have more frequent CCV requirements. See specific SOPs. Most inorganic methods require the CCV to be analyzed after ever 10 samples or injections, including matrix or batch QC samples.

Note: If an internal standard calibration is being used (e.g., GCMS) then bracketing standards are not required, only daily verifications are needed, except as specified by program or method requirements.

If the results of a CCV are outside the established acceptance criteria and analysis of a second consecutive (and immediate) CCV fails to produce results within acceptance criteria, corrective action shall be performed. Once corrective actions have been completed and documented, the laboratory shall demonstrate acceptable instrument / method performance by analyzing two consecutive CCVs, or a new initial instrument calibration shall be performed.

Note: For Ohio EPA VAP projects, if the instrument conditions have been changed, the laboratory must analyze a new calibration curve.

Sample analyses and reporting of data may not occur or continue until the analytical system is calibrated or calibration verified. However, data associated with an unacceptable calibration verification may be fully useable reported based upon discussion and approval of the client under the following special conditions:

a). when the acceptance criteria for the CCV are exceeded high (i.e., high bias) and the associated samples within the batch are non-detects, then those non-detects may be reported case narrative comment explaining the high bias. Otherwise the samples affected by the unacceptable CCV shall be re-analyzed after a new calibration curve has been established, evaluated and accepted; or

b). when the acceptance criteria for the CCV are exceeded low (i.e., low bias), those sample results may be reported if they exceed a maximum regulatory limit/decision level. Otherwise the samples affected by the unacceptable CCV shall be re-analyzed after a new calibration curve has been established, evaluated and accepted.

Samples reported by the 2 conditions identified above will be appropriately flagged.

20.4.1.2 Verification of Linear and Non-Linear Calibrations

Calibration verification for calibrations involves the calculation of the percent drift or the percent difference of the instrument response between the initial calibration and each subsequent analysis of the verification standard. (These calculations are available in the laboratory method SOPs.) Verification standards are evaluated based on the % Difference from the average CF or RF of the initial calibration or based on % Drift or % Recovery if a linear or quadratic curve is used.

Regardless of whether a linear or non-linear calibration model is used, if initial verification criterion is not met, then no sample analyses may take place until the calibration has been verified or a new initial calibration is performed that meets the specifications listed in the method SOPs. If the calibration cannot be verified after the analysis of a single verification standard, then adjust the instrument operating conditions and/or perform instrument maintenance, and analyze another aliquot of the verification standard. If the calibration cannot be verified with the second standard, then a new initial calibration is performed.

- When the acceptance criteria for the calibration verification are exceeded high, i.e., high bias, and there are associated samples that are non-detects, then those non-detects may be reported. Otherwise, the samples affected by the unacceptable calibration verification shall be reanalyzed after a new calibration curve has been established, evaluated and accepted.
- When the acceptance criteria for the calibration verification are exceeded low, i.e., low bias, those sample results may be reported if they exceed a maximum regulatory limit/decision level. Otherwise, the samples affected by the unacceptable verification shall be reanalyzed after a new calibration curve has been established, evaluated and accepted. Alternatively, a reporting limit standard may be analyzed to demonstrate that the laboratory can still support non-detects at their reporting limit.

  Note: This is not permitted for Ohio EPA VAP projects.

20.5 Tentatively Identified Compounds (TICs) – GC/MS Analysis

For samples containing components not associated with the calibration standards, a library search may be made for the purpose of tentative identification. The necessity to perform this type of identification will be determined by the purpose of the analyses being conducted. For example, the RCRA permit or waste delisting requirements may require the reporting of non-target analytes. Only after visual comparison of sample spectra with the nearest library searches may the analyst assign a tentative identification. Data system library search routines should not use normalization routines that would misrepresent the library or unknown spectra when compared to each other. Additional details are provided in NDSC Document No. CA-Q-QM-001, Tentatively Identified Compounds (TICS) - GCMS Analysis and SOP No.. KNOX-MS-0014, Tentatively Identified Compounds.

Note: If the TIC compound is not part of the client target analyte list but is calibrated by the laboratory and is both qualitatively and/or quantitatively identifiable, it should not be reported as a TIC. If the compound is reported on the same form as true TICs, it should be qualified and/or narrated that the reported compound is qualitatively and quantitatively (if verification in control) reported compared to a known standard that is in control (where applicable). Eurofins TestAmerica Knoxville will provide this service upon client request.

Note: For Ohio EPA VAP projects the laboratory may not attest under affidavit that TICs are “certified data.”

20.6 GC/MS Tuning

Prior to any GC/MS analytical sequence, including calibration, the instrument parameters for the tune and subsequent sample analyses within that sequence must be set.

Prior to tuning/auto-tuning the mass spectrometer, the parameters may be adjusted within the specifications set by the manufacturer or the analytical method. These generally do not need any adjustment but it may be required based on the current instrument performance. If the tune

verification does not pass it may be necessary to clean the source or perform additional maintenance. Any maintenance is documented in the maintenance log or run log.

Table 20-1. Example – Instrumentation List

eurofins | Environment Testing TestAmerica

Eurofins TestAmerica Knoxville - Instrument List

Instrument Type	Manufacturer	Model Number	Serial Number	Name	LIMS ID	Year Put into Service	Condition When Received	Software	Firmware
ICP-AES	Thermo Scientific	6500 Duo	20101714	Duo	Duo	2010	NEW	ITeva	ADL1
Mercury Analyzer (CVAAS)	Leeman Labs, Inc.	Hydra AA	6021	HG	HG	2006	NEW	WinHg	CR Rev 0.286
GC/MS Semivolatiles	Hewlett-Packard	5973/6890	US00035127 (GC) US10450996 (MS)	MY	MY	2000	NEW	Chemstation D.01.02.16	5.01.73
GC/MS Semivolatiles	Agilent Technologies	5975/6890	CN10816002 (GC) US81839504 (MS)	MP	MP	2008	NEW	Chemstation E.02.00.493	5.02.04
GC/MS Semivolatiles	Hewlett-Packard	5973/6890	US00026427 (GC) US82321805 (MS)	MA	MA	1999	NEW	Chemstation D.01.02.16	A.03.08
GC/MS Semivolatiles	Agilent Technologies	5973/6890	US10146146 (GC) US10451834 (MS)	MD	MD	2001	NEW	Chemstation D.01.02.16	5.01.73
GC/MS Volatiles	Agilent Technologies	5975C	CN10031137 (GC) US10053001 (MS)	MR	MR	2010	NEW	Chemstation E.02.00.493	5.02.12
GC/MS Volatiles	Agilent Technologies	5973/6890	US10506023 (GC) US44621247 (MS)	MG	MG	2005	NEW	Chemstation D.01.02.SP1	N.05.04
GC/MS Volatiles	Agilent Technologies	5973/6890 N	US10345111 (GC) US33220174 (MS)	MK	MK	2003	NEW	Chemstation D.01.00 Build 75	N.04.13
GC/MS Volatiles	Agilent Technologies	5973/6890 N	CN10111019 (GC) US83120789 (MS)	MH	MH	2018	USED	Chemstation E.02.02.1431	5.02.14
GC/MS Volatiles	Hewlett-Packard	5973/6890	DE00020254 (GC) US80310976 (MS)	MZ	MZ	1998	NEW	Chemstation D.01.02.16	A.03.08
GC/MS Volatiles	Hewlett-Packard	5973/6890	US00021530 (GC) US80210991 (MS)	MX	MX	1998	NEW	Chemstation D.01.02.16	A.03.0
GC/MS Volatiles	Hewlett-Packard	5973/6890	US10452619 (GC) US10214090 (MS)	MS	MS	2020	USED	Chemstation e.02.02	N.06.07
HRGC/HRMS	Finnigan	MAT-95/6890	2631	M1	M1D M1A	1991	NEW	ICIS 8.2.1	A.03.07
HRGC/HRMS	Thermo Scientific	DFS	03097M	DFS 1	D11A	2009	NEW	XCalibur 2.2.0 DFS 2.1.0.707	4.13
HRGC/HRMS	Thermo Scientific	DFS	03190M	DFS 2	D2A	2012	USED	XCalibur 2.2.0 DFS 2.1.0.709	5.00

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Table 20-1. Example – Instrumentation List (Continued)

eurofins | Environment Testing TestAmerica

Eurofins TestAmerica Knoxville - Instrument List

Instrument Type	Manufacturer	Model Number	Serial Number	Name	LIMS ID	Year Put into Service	Condition When Received	Software	Firmware
HRGC/HRMS	Thermo Scientific	DFS	3439M	DFS3	D3A	2019	NEW	XCalibur2.2.0 DFS 2.2.1.720	07.01.00
Ion Chromatograph	Dionex	DX-320	00070167	Lil Mike	320	2000	NEW	Chromeleon 6.5	MW Rev 1.06 BIOS Rev 30.00
Ion Chromatograph	Dionex	DX-600	02080040	Aragon	600	2002	NEW	Chromeleon 6.5	MW Rev 3.46 BIOS Rev 30.01
Ion Chromatograph	Dionex	ICS-1500	03100244	1500	1500	2003	NEW	Chromeleon 6.5	MW Rev 1.0.0 Boot Version 1.10
Ion Chromatograph	Thermofisher Dionex	22153-60315	20023384	IC1	IC1	2020	NEW	Chromeleon 7.2.10	Integrion HPIC System 1.3.0
Ion Chromatograph	Thermofisher Dionex	22176-60004	200340038	IC2	IC2	2020	NEW	Chromeleon 7.2.10	Aquion 1.0.0
Ion Chromatograph	Thermofisher Dionex	22176-60004	200440018	IC3	IC3	2020	NEW	Chromeleon 7.2.10	Aquion 1.0.0
Ion Chromatograph	Thermofisher Dionex	22176-60004	200440017	IC4	IC4	2020	NEW	Chromeleon 7.2.10	Aquion 1.0.0
Calorimeter	Parr	Bomb Calorimeter 1266	A1126DDEA	NA	NA	2003	NEW	NA	Boot FW Ver 1.03
Furnace (Ash)	Barnstead Thermolyne Furnace	30400	1262041236063	NA	NA	2003	NEW	NA	NA
GPC	J2 Scientific	GPC Accuprep	05L-1179-4.0	GPC1	NA	2003	NEW	Accuprep 307D & Accuchrome 2.1.0.9	20.8
GPC	J2 Scientific	GPC Preplinc	1122-1.0	GPC2	NA	2008	USED	PrepLink 1.0.0.25	01060
GPC	J2 Scientific	GPC Preplinc	1171-61	GPC3	NA	2016	USED	Inactive 1.3.0.60	01058
pH Meter	Accumet	Ab15	AB92318635	pH Meter	NA	2004	NEW	NA	NA
Spectrophotometer	Thermo Electron	Spectronic 20D+	3DUK270002	SPEC20	SPEC20	2007	NEW	NA	NA
GC-FID	Varian	3600 FID	0295	DW	DW	1998	NEW	Turbochrom 4.1	ROM03-910354-00 Rev C
GC-FID	Varian	3300 FID	3890	CO	CO	1987	NEW	Turbochrom 4.1	ROM03-910367-00 Rev E
GC-FID	Agilent Technologies	6890 FID	US00011425	GCE	GCE	2019	USED	Chemstation A.10.02.1757	A.03.08
GC-TCD	Varian	3400 TCD	3961	GR	GR	1989	NEW	Turbochrom 4.1	ROM03-910316 Rev C
GC-FID	Varian	3400 FID	4644	GTO	GTO	1987	NEW	Turbochrom 4.1	ROM03-910456 Rev B
GC-FID	Varian	3400 FID	4645	TB	TB	1987	NEW	Turbochrom 4.1	ROM03-910456 Rev B
GC-ECD	Hewlett-Packard	6890 ECD	US00021272	GST	GST	1997	NEW	Turbochrom 4.1	A.03.03
GC-ECD	Hewlett-Packard	5890 ECD	3235A46090	GMN	GMN	1986	NEW	Turbochrom 4.1	NA
GC-ECD	Agilent Technologies	6890 ECD	US10346012	GCD	GCD	2003	NEW	Turbochrom 4.1	N.04.13
LC/MS	SCIEX	5500	EX220471907	LCA	LCA	2020	NEW	Analyst 1.7.2	PIL1802

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21.0 MEASUREMENT TRACEABILITY

21.1 Overview

Traceability of measurements shall be assured using a system of documentation, calibration, and analysis of reference standards. Laboratory equipment that are peripheral to analysis and whose calibration is not necessarily documented in a test method analysis or by analysis of a reference standard shall be subject to ongoing certifications of accuracy. At a minimum, these must include procedures for checking specifications of ancillary equipment: balances, thermometers, temperature, Deionized (DI) and Reverse Osmosis (RO) water systems, automatic pipettes and other volumetric measuring devices. (Refer to Section 20.3). With the exception of Class A Glassware and glass microliter syringes, quarterly accuracy checks are performed for all mechanical volumetric devices. Wherever possible, subsidiary or peripheral equipment is checked against standard equipment or standards that are traceable to national or international standards. Class A Glassware and glass microliter syringes should be routinely inspected for chips, acid etching or deformity (e.g., bent needle). If the Class A glassware or syringe is suspect, the accuracy of the glassware will be assessed prior to use.

21.2 NIST-Traceable Weights and Thermometers

Reference standards of measurement shall be used for calibration only and for no other purpose, unless it can be shown that their performance as reference standards would not be invalidated.

For NIST-traceable weights and thermometers, the laboratory requires that all calibrations be conducted by a calibration laboratory accredited by A2LA, NVLAP (National Voluntary Laboratory Accreditation Program), or another accreditation organization that is a signatory to a MRA (Mutual Recognition Arrangement) of one or more of the following cooperations – ILAC (International Laboratory Accreditation Cooperation) or APLAC (Asia–Pacific Laboratory Accreditation Cooperation). A calibration certificate and scope of accreditation is kept on file at the laboratory.

An external certified service engineer services laboratory balances on an annual basis. This service is documented on each balance with a signed and dated certification sticker. Balance calibrations are checked each day of use. All mercury thermometers are calibrated annually against a traceable reference thermometer. Temperature readings of ovens, refrigerators, and incubators are checked on each day of use.

21.3 Reference Standards / Materials

Reference standards/materials, where commercially available, are traceable to certified reference materials. Commercially prepared reference standards, to the extent available, are purchased from vendors that are accredited to ISO Guide 34 and ISO/IEC Guide 17025. All reference standards from commercial vendors shall be accompanied with a certificate that includes at least the following information:

- Manufacturer
- Analytes or parameters calibrated
- Identification or lot number

- Calibration method
- Concentration with associated uncertainties
- Purity

If a standard cannot be purchased from a vendor that supplies a Certificate of Analysis, the purity of the standard is documented by analysis. The receipt of all reference standards must be documented. Reference standards are labeled with a unique Standard Identification Number and expiration date. All documentation received with the reference standard is retained as a QC record and references the Standard Identification Number.

All reference, primary and working standards/materials, whether commercially purchased or laboratory prepared, must be checked regularly to ensure that the variability of the standard or material from the true value does not exceed method requirements. The accuracy of calibration standards is checked by comparison with a standard from a second source. In cases where a second standard manufacturer is not available, a vendor certified different lot is acceptable for use as a second source. For unique situations, such as air analysis where no other source or lot is available, a standard made by a different analyst would be considered a second source. The appropriate Quality Control (QC) criteria for specific standards are defined in laboratory SOPs. In most cases, the analysis of an Initial Calibration Verification (ICV) or LCS (where there is no sample preparation) is used as the second source confirmation. These checks are generally performed as an integral part of the analysis method (e.g. calibration checks, laboratory control samples).

All standards and materials must be stored and handled according to method or manufacturer's requirements in order to prevent contamination or deterioration. Refer to the Environmental Health & Safety Manual or laboratory SOPs. For safety requirements, please refer to method SOPs and the laboratory's Environmental Health and Safety Manual.

Standards and reference materials shall not be used after their expiration dates unless their reliability is verified by the laboratory and their use is approved by the Quality Assurance Manager. The laboratory must have documented contingency procedures for re-verifying expired standards.

21.4 Documentation and Labeling of Standards, Reagents, and Reference Materials

Reagents must be at a minimum the purity required in the test method. The date of reagent receipt and the expiration date are documented. The lots for most of the common solvents and acids are tested for acceptability prior to company-wide purchase. Refer to NDSC Document No. CA-Q-S-001, Solvent and Acid Lot Testing and Approval.

All manufacturer or vendor supplied Certificate of Analysis or Purity must be retained, stored appropriately, and readily available for use and inspection. These records are maintained in the analytical laboratory. Records must be kept of the date of receipt and date of expiration of standards, reagents and reference materials. In addition, records of preparation of laboratory standards, reagents, and reference materials must be retained, stored appropriately, and be readily available for use and inspection. For detailed information on documentation and labeling, please refer to SOP KNOX-QA-0001, Standard/Reagent Labeling and Documentation.

Commercial materials purchased for preparation of calibration solutions, spike solutions, etc.., are usually accompanied with an assay certificate or the purity is noted on the label. If the assay purity is 96% or better, the weight provided by the vendor may be used without correction. If the assay purity is less than 96% a correction will be made to concentrations applied to solutions prepared from the stock commercial material (For 1613B dioxin/furan analyses the purity must

be 98% or corrections must be made). Blended gas standard cylinders use a nominal concentration if the certified value is within +/-15%, otherwise the certified values is used for the canister concentration.

21.4.1 All standards, reagents, and reference materials must be labeled in an unambiguous manner. Standards are logged into the laboratory's TALS system, and are assigned a unique identification number. The following information is typically recorded in the electronic database within the TALS.

- Standard ID
- Description of Standard
- Department
- Preparer's name
- Final volume and number of vials prepared
- Solvent type and lot number
- Preparation Date
- Expiration Date
- Standard source type (stock or daughter)
- Standard type (spike, surrogate, other)
- Parent standard ID (if applicable)
- Parent Standard Analyte Concentration (if applicable)
- Parent Standard Amount used (if applicable)
- Component Analytes
- Final concentration of each analyte
- Comment box (text field)

Note: All solvents and acids are stored at ambient temperature unless otherwise specified in the SOPs.

Records are maintained electronically for standard and reference material preparation. These records show the traceability to purchased stocks or neat compounds. These records also include method of preparation, date of preparation, expiration date and preparer's name or initials. Preparation procedures are provided in the Method SOPs.

21.4.2 All standards, reagents, and reference materials must be clearly labeled with a minimum of the following information:

- Expiration Date (include prep date for reagents)
- Standard ID
- Special Health/Safety warnings if applicable (Refer to the Health and Safety Manual)

Note: Refer to SOP KNOX-QA-0001 for more details.

21.4.3 In addition, the following information may be helpful:

- Date opened (for multi-use containers, if applicable)

- Description of standard (if different from manufacturer's label or if standard was prepared in the laboratory)
- Recommended Storage Conditions
- Concentration (if applicable)
- Initials of analyst preparing standard or opening container

All containers of prepared reagents must include an expiration date and an ID number to trace back to preparation.

Procedures for preparation of reagents can be found in the Method SOPs.

Standard ID numbers must be traceable through associated logbooks, worksheets and preparation/analytical batch records.

All reagents and standards must be stored in accordance to the following priority: 1) with the manufacturer's recommendations; 2) with requirements in the specific analytical methods as specified in the laboratory SOP.

22.0 SAMPLING

22.1 Overview

Eurofins TestAmerica Knoxville does not provide sampling services. The laboratory's responsibility in the sample collection process lies in supplying the sampler with the necessary coolers, reagent water, sample containers, preservatives, sample labels, custody seals, COC forms, ice, and packing materials required to properly preserve, pack, and ship samples to the laboratory. Eurofins TestAmerica Knoxville also provides pre-cleaned sampling media for sample collection (e.g. source emission testing air sampling media).

22.2 Sampling Containers

The laboratory offers clean sampling containers for use by clients. These containers are obtained from reputable container manufacturers and meet EPA specifications as required. Certificates of cleanliness for bottles and preservatives are provided by the supplier and are maintained at the laboratory. Alternatively, the certificates may be maintained by the supplier and available to the laboratory on-line.

22.2.1 Preservatives

Upon request, preservatives are provided to the client in pre-cleaned sampling containers. In some cases containers may be purchased pre-preserved from the container supplier. Whether prepared by the laboratory or bought pre-preserved, the grades of the preservatives are at a minimum:

- Hydrochloric Acid – Reagent ACS (Certified VOA Free) or equivalent
- Methanol – Purge and Trap grade
- Nitric Acid – Instra-Analyzed or equivalent
- Sodium Bisulfate – ACS Grade or equivalent
- Sodium Hydroxide – Instra-Analyzed or equivalent
- Sulfuric Acid – Instra-Analyzed or equivalent

22.3 Definition of Holding Time

The date and time of sampling documented on the COC form establishes the day and time zero. As a general rule, when the maximum allowable holding time is expressed in days (e.g., 14 days, 28 days), the holding time is based on calendar day measured. Holding times expressed in hours (e.g., 6 hours, 24 hours, etc.) are measured from date and time zero. The first day of holding time ends twenty-four hours after sampling. Holding times for analysis include any necessary reanalysis. However, there are some programs that determine holding time compliance based on the date and specific time of analysis compared to the time of sampling regardless of how long the holding time is.

22.3.1 Semi-Volatile - Holding times for sample preparation for semi-volatile organics are measured from the sampling date until the day solvent contacts the sample. Holding times for

analysis are measured from the date of initiation of extraction to the date of injection into the gas chromatograph.

22.3.2 Volatiles - Holding times for volatile organics are measured from the date (and time where applicable) of sampling to the date of injection into the gas chromatograph. Holding times for Encore preservation of volatiles samples is measured from the date and time of sampling to the date and time of sample preservation.

22.3.3 Inorganics - For inorganic and metals analysis, the preparation/digestion/distillation must be started within the maximum holding time as measured from the sampling date (and time where applicable). TestAmerica Knoxville measures the holding time for inorganics from sampling date (and time where applicable) to analysis date (and time where applicable).

22.4 Sampling Containers, Preservation Requirements, Holding Times

The preservation and holding time criteria specified in the laboratory SOPs are derived from the source documents for the methods. If method required holding times or preservation requirements are not met, the reports will be qualified using a flag, footnote or case narrative. As soon as possible or “ASAP” is an EPA designation for tests for which rapid analysis is advised, but for which neither EPA nor the laboratory have a basis for a holding time.

22.5 Sample Aliquots / Subsampling

Taking a representative sub-sample from a container is necessary to ensure that the analytical results are representative of the sample collected in the field. The size of the sample container, the quantity of sample fitted within the container, and the homogeneity of the sample need consideration when sub-sampling for sample preparation. It is the laboratory’s responsibility to take a representative subsample or aliquot of the sample provided for analysis.

Analysts should handle each sample as if it is potentially dangerous. At a minimum, safety glasses, gloves, and lab coats must be worn when preparing aliquots for analysis.

Refer to SOP KNOX-QA-0006, Subsampling, for details on the procedures to obtain representative subsamples for analysis from the samples submitted by the client.

Tables 22-1 to 22-6 detail holding times, preservation and container requirements, and sample volumes for SDWA and NPDES methods. The sample volumes are intended to be a minimal amount to perform the method, the containers that are used may be of larger size. Please Note: the holding times are program specific and different programs may have different holding times for equivalent methods (e.g., there are differences in Holding times for many Organic analytes between SDWA and NPDES. RCRA methods may also be different).

Table 22-1.
Holding Times, Preservation and Container Requirements: Drinking Water (SDWA)

PARAMETER	CONTAINER	PRESERVATION[1,2] Temp. [6]	Chemical	HOLDING TIME[3]	SAMPLE VOLUME
Nitrate	Plastic/Glass	4ºC	None	48 hours[4]	250 mL
Dioxin	Glass[5]	4ºC	$Na_2S_2O_3$	1 year	1 L

Key to Table

1. Sample preservation should be performed immediately upon sample collection. For composite chemical samples, each aliquot should be preserved at the time of collection. When use of an automated sampler makes it impossible to preserve each aliquot, then chemical samples may be preserved by maintaining at 4ºC until compositing and sample splitting is completed.
2. When any sample is to be shipped by common carrier or sent through the United States mails, it must comply with the Department of Transportation Hazardous Materials Regulations (49 CFR Part 172). The person offering such material for transportation is responsible for ensuring compliance. For the preservation requirements of Table 6-8, the Office of Hazardous Materials, Materials Transportation Bureau, Department of Transportation has determined that the Hazardous Materials Regulations do not apply to the following materials: Hydrochloric acid, (HCl) in water, solutions at concentrations of 0.04% by weight or less (pH about 1.96 or greater); Nitric acid (HNO_3) in water solutions at concentrations of 0.15% by weight or less (pH about 1.62 or greater); Sulfuric acid (H_2SO_4) in water solutions at concentrations of 0.35% by weight or less pH about 1.15 or greater); and Sodium hydroxide (NaOH) in water solutions at concentrations of 0.080% by weight or less (pH about 12.30 or less).
3. Samples should be analyzed as soon as possible after collection. The times listed are the maximum times that samples may be held before analysis and still be considered valid. Holding Times are determined from time of collection to time of analysis.
4. If the sample is chlorinated, the holding time for an un-acidified sample kept at 4ºC is extended to 14 days.
5. With Teflon lined septum.
6. For samples with a temperature requirement of 4ºC, a sample temperature of just above the water freezing temperature to < 6ºC is acceptable.

Table 22-2
Holding Times, Preservation and Container Requirements: NPDES - Inorganic

PARAMETER	CONTAINER [1]	PRESERVATION[2,3] Temp[10].	Chemical	HOLDING TIME[4]	SAMPLE VOLUME
Boron	Plastic[5]	None	HNO_3 to pH<2	6 months	200 mL
Bromide	Plastic/Glass	None	None	28 days	100 mL
Chloride	Plastic/Glass	None	None	28 days	50 mL
Fluoride	Plastic	None	None	28 days	300 mL
Hardness	Plastic/Glass	None	HNO_3 to pH<2[7]	6 months	100 mL
Hydrogen Ion (pH)	Plastic/Glass	None	None	15 min.[6]	200 mL
Mercury	Plastic/Glass	None	HNO_3 to pH<2	28 days	200 mL
Metals[8,9]	Plastic/Glass	None	HNO_3 to pH<2[11]	6 months	200 mL
Nitrate	Plastic/Glass	≤ 6ºC	None	48 hours	100 mL
Nitrate-Nitrite	Plastic/Glass	≤ 6ºC	H_2SO_4 to pH <2	28 days	100 mL
Nitrite	Plastic/Glass	≤ 6ºC	None	48 hours	100 mL
Orthophosphate	Plastic/Glass	≤ 6ºC	Filter within 15 min.	48 hours	250 mL
Sulfate	Plastic/Glass	≤ 6ºC	None	28 days	250 mL

Key to Table

1. Plastic should be Polyethylene.
2. Sample preservation should be performed immediately upon sample collection. For composite chemical samples, each aliquot should be preserved at the time of collection. When use of an automated sampler makes it impossible to preserve each aliquot, then chemical samples may be preserved by maintaining at ≤ 6ºC until compositing and sample splitting is completed.
3. When any sample is to be shipped by common carrier or sent through the United States mails, it must comply with the Department of Transportation Hazardous Materials Regulations (49 CFR Part 172). The person offering such material for transportation is responsible for ensuring compliance. For the preservation requirements of Table 6-8, the Office of Hazardous Materials, Materials Transportation Bureau, Department of Transportation has determined that the Hazardous Materials Regulations do not apply to the following materials: Hydrochloric acid, (HCl) in water, solutions at concentrations of 0.04% by weight or less (pH about 1.96 or greater); Nitric acid (HNO_3) in water solutions at concentrations of 0.15% by weight or less (pH about 1.62 or greater); Sulfuric acid (H_2SO_4) in water solutions at concentrations of 0.35% by weight or less pH about 1.15 or greater); and Sodium hydroxide (NaOH) in water solutions at concentrations of 0.080% by weight or less (pH about 12.30 or less).
4. Samples should be analyzed as soon as possible after collection. The times listed are the maximum times that samples may be held before analysis and still be considered valid. Holding Times are determined from time of collection to time of analysis.
5. May also be collected in quartz or PFTE Plastic.
6. For compliance testing, the analysis must be performed in the field at the time of analysis. If transported to the laboratory for analysis, the analysis will be performed as soon as practical and reported qualified.
7. H_2SO_4 to a pH <2 is also acceptable.
8. Except Mercury and Hexavalent Chromium.
9. For dissolved metals, samples must be filtered on site before adding HNO_3 preservative (or before shipping to laboratory).

Key to Table

10. Aqueous samples must be preserved at ≤6 °C unless otherwise indicated, and should not be frozen unless data demonstrating that sample freezing does not adversely impact sample integrity is maintained on file and accepted as valid by the regulatory authority. Also, for purposes of NPDES monitoring, the specification of ‘‘≤ °C’’ is used in place of the ‘‘4 °C’’ and ‘‘<4 °C’’ sample temperature requirements listed in some methods.

11 Acid preservation may be omitted for shipping and laboratory will acidify at least 24 hours prior to analysis.

Table 22-3
Holding Times, Preservation and Container Requirements: NPDES - Organic

PARAMETER	CONTAINER	PRESERVATION[1,2] Temp.[7]	PRESERVATION[1,2] Chemical	HOLDING TIME[3]	SAMPLE VOLUME
CDD/CDFs[6] – Aqueous: Field/Lab Preservation	Glass	≤ 6ºC	pH <9, 0.008 % $Na_2S_2O_3$[5]	1 year	1 L
CDD/CDFs[6] – Solids/Mixed Phase/ - Field Preservation	Glass	≤ 6ºC	None	7 days[8]	10 grams/1 L
CDD/CDFs[6] – Tissue – Field Preservation	Glass	≤ 6ºC	None	24 hours[8]	10 grams
CDD/CDFs[6] – Solids/Mixed Phase/Tissue - Lab Preservation	Glass	< -10ºC	None	1 year	10 grams/1 L
Polychlorinated Biphenyls (PCB Congeners): Aqueous: Field/Lab Preservation[9]	Glass	≤ 6ºC	0.008 % $Na_2S_2O_3$[5]	1 year	1 L
Polychlorinated Biphenyls (PCB Congeners): Solid/Mixed Phase– Field Preservation[9]	Glass	≤ 6ºC	None	1 year	10 grams/1 L
Polychlorinated Biphenyls (PCB Congeners): Tissue-Field Preservation[9]	Glass	≤ 6ºC	None	24 hours[8]	10 grams
Polychlorinated Biphenyls (PCB Congeners): Solid/Mixed Phase/Tissue – Lab Preservation[9]	Glass	< -10ºC	None	1 year	10 grams/1 L

Key to Table

1. Sample preservation should be performed immediately upon sample collection. For composite chemical samples, each aliquot should be preserved at the time of collection. When use of an automated sampler makes it impossible to preserve each aliquot, then chemical samples may be preserved by maintaining at ≤ 6ºC until compositing and sample splitting is completed.
2. When any sample is to be shipped by common carrier or sent through the United States mails, it must comply with the Department of Transportation Hazardous Materials Regulations (49 CFR Part 172). The person offering such material for transportation is responsible for ensuring compliance. For the preservation requirements of Table 6-8, the Office of Hazardous Materials, Materials Transportation Bureau, Department of Transportation has determined that the Hazardous Materials Regulations do not apply to the following materials: Hydrochloric acid, (HCl) in water, solutions at concentrations of 0.04% by weight or less (pH about 1.96 or greater); Nitric acid (HNO_3) in water solutions at concentrations of 0.15%

Key to Table

by weight or less (pH about 1.62 or greater); Sulfuric acid (H_2SO_4) in water solutions at concentrations of 0.35% by weight or less pH about 1.15 or greater); and Sodium hydroxide (NaOH) in water solutions at concentrations of 0.080% by weight or less (pH about 12.30 or less).

3. Samples should be analyzed as soon as possible after collection. The times listed are the maximum times that samples may be held before analysis and still be considered valid. Holding Times are determined from time of collection to time of analysis (See also Footnote 8).
4. With Teflon lined septum.
5. Should only be used in the presence of residual chlorine. Ascorbic may be used instead.
6. When the extractable analytes of concern fall within a single chemical category, the specified preservative and maximum holding times should be observed for optimum safeguard of sample integrity. When the analytes of concern fall within two or more categories, the sample may be preserved by cooling to $\leq$ 6ºC reducing residual chlorine with 0.0008 % sodium thiosulfate, storing in the dark, and adjusting the pH to 6-9. Samples preserved in this manner may be held for 7 days before extraction and for 40 days after extraction. Exceptions to this optional preservation and holding time procedure are noted in footnote 5 (re the requirement for thiosulfate reduction of residual chlorine) and footnotes 10 and 11(re the analysis of Benzidine).
7. Aqueous samples must be preserved at ≤6 °C unless otherwise indicated, and should not be frozen unless data demonstrating that sample freezing does not adversely impact sample integrity is maintained on file and accepted as valid by the regulatory authority. Also, for purposes of NPDES monitoring, the specification of ‘‘≤ °C’’ is used in place of the ‘‘4 °C’’ and ‘‘<4 °C’’ sample temperature requirements listed in some methods.

8 Holding time from sampling field preservation at <6ºC to time of preservation at <-10ºC.

9 Polychlorinated biphenyls (PCB congeners) are in the proposed 40CFR Part 136 Rule September 23, 2010.

Table 22-4.
Holding Times, Preservation and Container Requirements: RCRA - Aqueous

PARAMETER	CONTAINER [1]	PRESERVATION[2,3] Temp.[9]	Chemical	HOLDING TIME[4]	SAMPLE VOLUME
Chloride	Plastic/Glass	4ºC	None	28 days	100 mL
Hydrogen Ion (pH)	Plastic/Glass	4ºC	None	24 hours[8]	100 mL
Nitrate	Plastic/Glass	4ºC	None	48 hours	28 days
Sulfate	Plastic/Glass	4ºC	None	28 days	400 mL
Mercury	Plastic/Glass	None	HNO_3 to pH<2	28 days	250 mL
Other Metals	Plastic/Glass	None	HNO_3 to pH<2	6 months	250 mL
Acrolein and Acrylonitrile	Glass[7]	4ºC	0.0008 % Na2S2O3[5] Adjust pH to 4-5[10]	14 days	1 L
Benzidines	Glass[7]	4ºC	0.0008 % $Na_2S_2O_3$[5]	7 days[6]	1 L
Chlorinated Hydrocarbons	Glass[7]	4ºC	0.0008 % $Na_2S_2O_3$[5]	7 days[6]	1 L
Dioxins and Furans	Glass[7]	4ºC	0.0008 % $Na_2S_2O_3$[5]	30 days[6]	1 L
Haloethers	Glass[7]	4ºC	0.0008 % $Na_2S_2O_3$[5]	7 days[6]	1 L
Nitroaromatics and cyclic ketones	Glass[7]	4ºC	0.0008 % $Na_2S_2O_3$[5], store in dark	7 days[6]	1 L
Nitrosamines	Glass[7]	4ºC	0.0008 % $Na_2S_2O_3$[5], store in dark	7 days[6]	1 L
PCBs	Glass[7]	4ºC	None	1 year[6]	1 L
Phenols	Glass[7]	4ºC	0.0008 % $Na_2S_2O_3$[5]	7 days[6]	1 L
Phthalate Esters	Glass[7]	4ºC	None	7 days[6]	1 L
Polynuclear Aromatic Hydrocarbons	Glass[7]	4ºC	0.0008 % $Na_2S_2O_3$[5], store in dark	7 days[6]	1 L
Purgeable Hydrocarbons	Glass[7]	4ºC	0.0008 % $Na_2S_2O_3$[5] Adjust pH <2[2]	14 days	40 mL
Purgeable Halocarbons	Glass[7]	4ºC	0.0008 % $Na_2S_2O_3$[5]	14 days	40 mL

Key to Table

1. Plastic should be Polyethylene.
2. Sample preservation should be performed immediately upon sample collection. For composite chemical samples, each aliquot should be preserved at the time of collection. When use of an automated sampler makes it impossible to preserve each aliquot, then chemical samples may be preserved by maintaining at 4ºC until compositing and sample splitting is completed.

Key to Table

3. When any sample is to be shipped by common carrier or sent through the United States mails, it must comply with the Department of Transportation Hazardous Materials Regulations (49 CFR Part 172). The person offering such material for transportation is responsible for ensuring compliance. For the preservation requirements of Table 6-8, the Office of Hazardous Materials, Materials Transportation Bureau, Department of Transportation has determined that the Hazardous Materials Regulations do not apply to the following materials: Hydrochloric acid, (HCl) in water, solutions at concentrations of 0.04% by weight or less (pH about 1.96 or greater); Nitric acid (HNO_3) in water solutions at concentrations of 0.15% by weight or less (pH about 1.62 or greater); Sulfuric acid (H_2SO_4) in water solutions at concentrations of 0.35% by weight or less pH about 1.15 or greater); and Sodium hydroxide (NaOH) in water solutions at concentrations of 0.080% by weight or less (pH about 12.30 or less).
4. Samples should be analyzed as soon as possible after collection. The times listed are the maximum times that samples may be held before analysis and still be considered valid. Holding Times are determined from time of collection to time of preparation (or to time of analysis if there is no separate preparation step).
5. Free Chlorine must be removed by the appropriate addition of $Na_2S_2O_3$.
6. 7 days to extraction. 40 days from extraction to analysis. For Dioxins/Furans: 30 days to extraction 45 days from extraction to analysis. For PCBs, no holding time is specified in SW-846 Chapter 4.
7. With Teflon lined septum.
8. Holding Time is listed as “As Soon as Possible” in SW 846. Per EPA MICE, the recommended maximum holding time for pH in water is 24 hours and pH in soil is 7 days. There are no mandated regulatory requirements.
9. For samples with a temperature requirement of 4°C, a sample temperature of just above the water freezing temperature to $\leq$ 6°C is acceptable.
10 Based on guidance from EPA MICE, if samples are received without pH adjustment, the holding time is 7 days.

Table 22-5.
Holding Times, Preservation and Container Requirements: RCRA – Non-Aqueous

PARAMETER	CONTAINER [1]	PRESERVATION Temp.[7]	PRESERVATION Chemical	HOLDING TIME[2]	SAMPLE WEIGHT
Chloride	Glass	4ºC	None	28 days	50 g
Hydrogen Ion (pH)	Glass	4ºC	None	7 days[6]	50 g
Nitrate	Glass	4ºC	None	N/A	50 g
Mercury	Plastic/Glass	4ºC	None	28 days	50 g
Other Metals	Plastic/Glass	None	None	6 months	50 g
Acrolein and Acrylonitrile	Glass[4]	4ºC	None	14 days	50 g
Benzidines	Glass[4]	4ºC	None	14 days[3]	50 g
Chlorinated Hydrocarbons	Glass[4]	4ºC	None	14 days[3]	50 g
Dioxins and Furans	Glass[4]	4ºC	None	30 days[3]	50 g
Haloethers	Glass[4]	4ºC	None	14 days[3]	50 g
Nitroaromatics and cyclic ketones	Glass[4]	4ºC	None	14 days[3]	50 g
Nitrosamines	Glass[4]	4ºC	None	14 days[3]	50 g
PCBs	Glass[4]	4ºC	None	1 year[3]	50 g
Phenols	Glass[4]	4ºC	None	14 days[3]	50 g
Phthalate Esters	Glass[4]	4ºC	None	14 days[3]	50 g
Polynuclear Aromatic Hydrocarbons	Glass[4]	4ºC	None	14 days[3]	50 g
Purgeable Hydrocarbons	Glass[4]	4ºC	None	14 days[5]	50 g
Purgeable Halocarbons	Glass[4]	4ºC	None	14 days[5]	50 g

Key to Table

1. Plastic should be Polyethylene.
2. Samples should be analyzed as soon as possible after collection. The times listed are the maximum times that samples may be held before analysis and still be considered valid.
3. 14 days to extraction. 40 days from extraction to analysis. For Dioxins/Furans (Method 8290) in Matrices other than tissue: 30 days to extraction 45 days from extraction to analysis. For Dioxins/Furans (Method 8290) in Tissue: 30 days to extraction 45 days from collection to analysis. Holding Times are determined from time of collection to time of preparation (or to time of analysis if there is no separate preparation step). For PCBs, no holding time is specified in SW-846 Chapter 4.
4. With Teflon Lined Septum

5. See Volatile SOP for more detailed preservation requirements.
6. Holding Time is listed as “As Soon as Possible” in SW 846. Per EPA MICE, the recommended maximum holding time for pH in water is 24 hours and pH in soil is 7 days. There are no mandated regulatory requirements.
7. For samples with a temperature requirement of 4°C, a sample temperature of just above the water freezing temperature to ≤ 6°C is acceptable.

Table 22-6.
Holding Times, Preservation and Container Requirements: Air Samples[1]

PARAMETER	CONTAINER	PRESERVATION Temp.	PRESERVATION Chemical	HOLDING TIME[2]	SAMPLE WEIGHT
Volatile Organics (TO-15, TO-14A)	Summa Canister	None	None	30 days	6L or 1L
Volatile Organics (TO-15, TO-14A)	Tedlar Bag	None	None	72 hrs[3,4]	1 L
Polynuclear Aromatic Hydrocarbons (TO-13A)	PUF/XAD	4ºC[5]	None	7 days[6]	N/A
Organochlorine Pesticides/PCBs (TO-4A)	PUF/XAD	4ºC[5]	None	7 days[6]	N/A
Dioxins/Furans (TO-9A)	PUF/XAD	4ºC[5]	None	7 days[6]	N/A

Key to Table

1. For details on Source Air Emission Holding Times, Containers, and Preservation please contact the laboratory for method specific information.
2. Samples should be analyzed as soon as possible after collection. The times listed are the maximum times that samples may be held before analysis and still be considered valid. Holding Times are determined from time of collection to time of preparation (or to time of analysis if there is no separate preparation step).
3. Holding Time is based on SW 846 Method 0040 “SAMPLING OF PRINCIPAL ORGANIC HAZARDOUS CONSTITUENTS FROM COMBUSTION SOURCES USING TEDLAR® BAGS”. Some states specifically enforce this holding time (e.g. Florida, New Jersey) and others have not specified this information in their regulatory requirements.
4. The holding time is 72 hours unless the laboratory has a documented validation study that indicates a longer HT is acceptable for the analytes of interest. Alternatively, the sample may be transferred into a Summa Canister within 72 hours.

5 For samples with a temperature requirement of 4ºC, a sample temperature of just above the water freezing temperature to $\leq$ 6ºC is acceptable.

6 7 days from collection to extraction, 40 days from extraction to analysis.

23.0 HANDLING OF SAMPLES

It is the responsibility of the client to send us representative and/or homogeneous and properly preserved samples of the system from which they are drawn. The laboratory assumes that all multiple sample containers with the same designator/description and bottle type contain a homogeneous, representative sample.

The laboratory may provide the appropriate sample containers, required preservative, chain-of-custody (COC) forms, shipping containers, labels, and custody seals. The laboratory also provides trip blanks and analyte-free water for field blanks. Preparation of methanol containers for field preservation of volatile soil samples is available.

Sample management procedures at the laboratory ensure that sample integrity and custody are maintained and documented from sampling/receipt through disposal.

23.1 Chain of Custody (COC)

The COC form is the written documented history of any sample and is initiated at the time of sampling. This form is completed by the sampling personnel and accompanies the samples to the laboratory where it is received and stored under the laboratory's custody. The purpose of the COC form is to provide a legal written record of the handling of samples from the time of collection until they are received at the laboratory. It also serves as the primary written request for analyses from the client to the laboratory. The COC form acts as a purchase order for analytical services when no other contractual agreement is in effect.

23.1.1 Field Documentation

The information the sampler needs to provide at the time of sampling on the container label is:

- Sample identification
- Date and time
- Preservative

During the sampling process, the COC form is completed and must be legible. This form includes information such as:

- Client name, address, phone number and fax number (if available)
- Project name and/or number
- The sample identification
- Date, time and location of sampling
- Sample collectors name
- The matrix description
- The container description
- The total number of each type of container
- Preservatives used
- Analysis requested

- Requested turnaround time (TAT)
- Any special instructions
- Purchase Order number or billing information (e.g. quote number) if available
- The date and time that each person received or relinquished the sample(s), including their signed name.

When the sampling personnel deliver the samples directly to the laboratory personnel, the samples are stored in a cooler with ice, as applicable, and remain solely in the possession of the client's field technician until the samples are delivered to the laboratory personnel. The sample collector must assure that each container is in his/her physical possession or in his/her view at all times, or stored in such a place and manner to preclude tampering. The field technician relinquishes the samples in writing on the COC form to the sample control personnel at the laboratory or to a laboratory courier. When sampling personnel deliver the samples through a common carrier (Fed-Ex, UPS), the CoC relinquished date/time is completed by the field personnel and samples are released to the carrier. Samples are only considered to be received by the laboratory when personnel at the fixed laboratory facility have physical contact with the samples.

Note: Independent couriers are not required to sign the COC form.

23.1.2 Legal / Evidentiary Chain-of-Custody

TestAmerica Knoxville provides internal sample chain-of-custody tracking. However, it does not provide chain-of-custody evidence for samples identified for legal/evidentiary purposes.

23.2 Sample Receipt

Per State and/or Federal Regulation, the client is responsible to ensure that samples are shipped in accordance with DOT/IATA requirements, and that radioactive materials may only be delivered to licensed facilities. Any samples containing (or suspected to contain) Source, Byproduct, or Special Nuclear Material as defined by 10 CFR should be delivered directly to facilities licensed to handle such radioactive material. Natural material or ores containing naturally occurring radionuclides may be delivered to any Eurofins TestAmerica facility or courier as long as the activity concentration of the material does not exceed 270 pCi/g alpha or 2700 pCi/g beta (49 CFR Part 173).

Samples are received at the laboratory by designated sample receiving personnel and a unique laboratory project identification number is assigned. Each sample container shall be assigned a unique sample identification number that is cross-referenced to the client identification number such that traceability of test samples is unambiguous and documented. Each sample container is affixed with a durable sample identification label. Sample acceptance, receipt, tracking and storage procedures summarized in the following sections.

23.2.1 Laboratory Receipt

When samples arrive at the laboratory, sample receiving personnel inspect the coolers and samples. The integrity of each sample must be determined by comparing sample labels or tags with the COC and by visual checks of the container for possible damage. Any non-conformance, irregularity, or compromised sample receipt must be documented on a Condition Upon Receipt Form (See Example form in Figure 23-2) and brought to the immediate attention of the client. The COC, shipping documents, documentation of any non-conformance, irregularity, or compromised sample receipt, record of client contact, and resulting instructions become part of

the project record. Details on the receipt of samples are found in SOP KNOX-SC-0003, Sample Receipt and Log-in.

Note: North Carolina requires that they be notified when samples are processed that do not meet sample acceptance criteria.

23.3 Sample Acceptance Policy

The laboratory has a written sample acceptance policy (Figure 23-1) that clearly outlines the circumstances under which samples shall be accepted or rejected. These include:

- a COC filled out completely;
- samples must be properly labeled;
- proper sample containers with adequate volume for the analysis (Sampling Guide) and necessary QC;
- samples must be preserved according to the requirements of the requested analytical method (Sampling Guide);
- sample holding times must be adhered to (Sampling Guide);
- the project manager will be notified if any sample is received in damaged condition.

Data from samples which do not meet these criteria are flagged and the nature of the variation from policy is defined.

23.4 Sample Storage

In order to avoid deterioration, contamination or damage to a sample during storage and handling, from the time of receipt until all analyses are complete, samples are stored in refrigerators, freezers or protected locations suitable for the sample matrix. Samples for methods that do not require refrigeration (e.g., aqueous samples for ICP metals analysis) are stored on shelves at ambient temperature. In addition, samples to be analyzed for volatile organic parameters are stored in separate refrigerators designated for volatile organic parameters only. Samples are never to be stored with reagents, standards or materials that may create contamination.

To ensure the integrity of the samples during storage, refrigerator blanks are maintained in the volatile sample refrigerators and analyzed every two weeks.

Laboratory Analysts retrieve the sample container(s) allocated to their analysis from the designated storage location, analyze the sample, and return the remaining sample to the storage location from which it originally came. Residual samples are maintained in the designated storage location until disposal at least thirty days after the report is issued. (Exception: TO-14A or TO-15 canister samples are subject to disposal two days after the report is issued.) Special arrangements are made to store samples for longer periods of time.

Access to the laboratory is controlled such that sample storage need not be locked at all times unless a project specifically demands it. Samples are accessible to laboratory personnel only. Visitors to the laboratory are prohibited from entering the refrigerator and laboratory areas unless accompanied by an employee of Eurofins TestAmerica.

23.5 Hazardous Samples and Foreign Soils

All samples are assumed to be hazardous. All samples are either returned to the client or disposed of appropriately through a hazardous waste disposal firm. All soil samples are sent out for incineration by a USDA-approved waste disposal facility.

23.6 Sample Shipping

In the event that the laboratory needs to ship samples, the samples are placed in a cooler with enough ice to ensure the samples remain just above freezing and at or below 6.0°C during transit. The samples are carefully surrounded by packing material to avoid breakage (yet maintain appropriate temperature). A trip blank is enclosed for those samples requiring water/solid volatile organic analyses. The chain-of-custody form is signed by the sample control technician and attached to the shipping paperwork. Samples are generally shipped overnight express or hand-delivered by a Eurofins TestAmerica courier to maintain sample integrity. All personnel involved with shipping and receiving samples must be trained to maintain the proper chain-of-custody documentation and to keep the samples intact and on ice. The laboratory's Environmental, Health and Safety Manual contains additional shipping requirements.

Note: If a client does not request trip blank analysis on the COC or other paperwork, the laboratory will not analyze the trip blanks that were supplied. However, in the interest of good client service, the laboratory will advise the client at the time of sample receipt that it was noted that they did not request analysis of the trip blank; and that the laboratory is providing the notification to verify that they are not inadvertently omitting a key part of regulatory compliance testing.

23.7 Sample Disposal

Samples should be retained for a minimum of 30 days after the project report is sent, however, provisions may be made for earlier disposal of samples once the holding time is exceeded. Some samples are required to be held for longer periods based on regulatory or client requirements (e.g., 60 days after project report is sent). The laboratory must follow the longer sample retention requirements where required by regulation or client agreement. Several possibilities for sample disposal exist: the sample may be consumed completely during analysis, the sample may be returned to the customer or location of sampling for disposal, or the sample may be disposed of in accordance with the laboratory's waste disposal procedures (SOP: KNOX-SC-0005, Sample Disposal Return and Archive). All procedures in the laboratory's Environmental Health and Safety Manual are followed during disposal. Samples are normally maintained in the laboratory no longer than two months from receipt unless otherwise requested. Unused portions of samples found or suspected to be hazardous according to state or federal guidelines may be returned to the client upon completion of the analytical work. Waste disposal complies with all federal and state laws and regulations.

Figure 23-1. Example: Sample Acceptance Policy

NELAC specifies requirements under which any NELAC accredited laboratory will accept or reject samples. Eurofins TestAmerica Knoxville will review your sample shipment against those requirements listed below, and will communicate any discrepancies to you. Your project manager will assist you in the appropriate resolution of any issues related to sample receipt. Please contact your project manager with any questions.

When completing the chain of custody form, please do not forget to sign your name in the "relinquished by" box.

NELAC requirements are as follows:

- Proper, full and complete documentation, which includes sample identification, the location, date and time of collection, the collector's name, the preservation type, the sample matrix type, the requested testing method, and any special remarks concerning the samples shall be provided. (Chain of Custody filled out properly)

- Each sample shall be labeled with unique, durable and indelible identification, (See note below regarding SUMMA canisters and VOST tubes).

- The samples shall be collected in the appropriate sample containers.

- The samples shall arrive at the laboratory within the specified holding time for the analyses requested.

- Sufficient sample volume must be available to perform the requested analyses and associated quality control.

- All samples submitted for water/solid Volatile Organic analyses must have a Trip Blank submitted at the same time

- The laboratory will notify the client upon sample receipt if the samples exhibit obvious signs of damage, contamination or inadequate preservation. Also, if there is a breach in the chain-of-custody associated with the sample(s), the client will be notified to determine the disposition of the samples.

- Samples shall be rejected if they are received in broken containers or if they exceed the laboratory radiological license limits. The client will be notified to determine the disposition of the samples.

NOTE: Never affix a label directly on a SUMMA canister. A special tag is attached to each canister for this purpose. Never place a label on a VOST tube; use the external container for labeling.

NOTE: Canisters and flow controllers must be returned even if they were not used. Sampling equipment that is lost or not returned will be invoiced to the client at the replacement costs.

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Figure 23-2. Example: Condition Upon Receipt Form

TESTAMERICA KNOXVILLE SAMPLE RECEIPT/CONDITION UPON RECEIPT ANOMALY CHECKLIST Log In Number:

Review Items	Yes	No	NA	If No, what was the problem?	Comments/Actions Taken
1. Are the shipping containers intact?				☐ Containers, Broken	
2. Were ambient air containers received intact?				☐ Checked in lab	
3. The coolers/containers custody seal if present, is it intact?				☐ Yes ☐ NA	
4. Is the cooler temperature within limits? (> freezing temp. of water to 6 °C, VOST: 10°C) Thermometer ID :_____ Correction factor: _____				☐ Cooler Out of Temp, Client Contacted, Proceed/Cancel ☐ Cooler Out of Temp, Same Day Receipt	
5. Were all of the sample containers received intact?				☐ Containers, Broken	
6. Were samples received in appropriate containers?				☐ Containers, Improper; Client Contacted; Proceed/Cancel	
7. Do sample container labels match COC? (IDs, Dates, Times)				☐ COC & Samples Do Not Match ☐ COC Incorrect/Incomplete ☐ COC Not Received	
8. Were all of the samples listed on the COC received?				☐ Sample Received, Not on COC ☐ Sample on COC, Not Received	
9. Is the date/time of sample collection noted?				☐ COC; No Date/Time; Client Contacted	Labeling Verified by: Date:
10. Was the sampler identified on the COC?				☐ Sampler Not Listed on COC	
11. Is the client and project name/# identified?				☐ COC Incorrect/Incomplete	pH test strip lot number:
12. Are tests/parameters listed for each sample?				☐ COC No tests on COC	
13. Is the matrix of the samples noted?				☐ COC Incorrect/Incomplete	
14. Was COC relinquished? (Signed/Dated/Timed)				☐ COC Incorrect/Incomplete	Box 16A: pH Preservation / Box 18A: Residual Chlorine
15. Were samples received within holding time?				☐ Holding Time - Receipt	Preservative: _____
16. Were samples received with correct chemical preservative (excluding Encore)?				☐ pH Adjusted, pH Included (See box 16A) ☐ Incorrect Preservative	Lot Number: _____ Exp Date: _____ Analyst: _____ Date: _____ Time: _____
17. Were VOA samples received without headspace?				☐ Headspace (VOA only)	
18. Did you check for residual chlorine, if necessary? (e.g. 1613B, 1668) Chlorine test strip lot number: _____				☐ Residual Chlorine	
19. For 1613B water samples is pH<9?				☐ If no, notify lab to adjust	
20. For rad samples was sample activity info. Provided?				☐ Project missing info	

Project #: _____ PM Instructions: _____

Sample Receiving Associate: _____ Date: _____

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24.0 ASSURING THE QUALITY OF TEST RESULTS

24.1 Overview

In order to assure our clients of the validity of their data, the laboratory continuously evaluates the quality of the analytical process. The analytical process is controlled not only by instrument calibration as discussed in Section 20, but also by routine process quality control measurements (e.g. Blanks, Laboratory Control Samples (LCS), Matrix Spikes (MS), duplicates (DUP), surrogates, Internal Standards (IS)). These quality control checks are performed as required by the method or regulations to assess precision and accuracy. Quality control samples are to be treated in the exact same manner as the associated field samples being tested. In addition to the routine process quality control samples, Proficiency Testing (PT) Samples (concentrations unknown to laboratory) are analyzed to help ensure laboratory performance.

24.2 Controls

Sample preparation or pre-treatment is commonly required before analysis. Typical preparation steps include homogenization, grinding, solvent extraction, sonication, acid digestion, distillation, reflux, evaporation, drying and ashing. During these pre-treatment steps, samples are arranged into discreet manageable groups referred to as preparation (prep) batches. Prep batches provide a means to control variability in sample treatment. Control samples are added to each prep batch to monitor method performance and are processed through the entire analytical procedure with investigative/field samples.

24.3 Negative Controls

Table 24-1. Example – Negative Controls

Control Type	Details
Method Blank (MB)	Are used to assess preparation and analysis for possible contamination during the preparation and processing steps.
	The specific frequency of use for method blanks during the analytical sequence is defined in the specific standard operating procedure for each analysis. Generally it is 1 for each batch of samples; not to exceed 20 environmental samples.
	The method blank is prepared from a clean matrix similar to that of the associated samples that is free from target analytes (e.g., Reagent water, Ottawa sand, glass beads, etc.) and is processed along with and under the same conditions as the associated samples. The method blank goes through all of the steps of the process (including as necessary: filtration, clean-ups, etc.).
	Reanalyze or qualify associated sample results when the concentration of a targeted analyte in the blank is at or above the reporting limit as established by the method or by regulation, AND is greater than 1/10 of the amount measured in the sample. (Ohio VAP method blanks must be <RL)
Calibration Blanks	Are prepared and analyzed along with calibration standards where applicable or injected at specified frequencies throughout an analytical sequence. They are prepared using the same reagents that are used to prepare the standards. In some analyses the calibration blank may be included in the calibration curve. These blanks are termed Initial Calibration Blanks (ICB) or Continuing Calibration Blanks (CCB)

Table 24-1. Example – Negative Controls

Control Type	Details
Instrument Blanks	Are blank reagents or reagent water that may be processed during an analytical sequence in order to assess contamination in the analytical system. In general, instrument blanks are used to differentiate between contamination caused by the analytical system and that caused by the sample handling or sample prep process. Instrument blanks may also be inserted throughout the analytical sequence to minimize the effect of carryover from samples with high analyte content.
Trip Blank [1]	Are required to be submitted by the client with each shipment of samples requiring aqueous and solid volatiles analyses (or as specified in the client's project plan). Additionally, trip blanks may be prepared and analyzed for volatile analysis of air samples, when required by the client. A trip blank may be purchased (certified clean) or is prepared by the laboratory by filling a clean container with pure deionized water that has been purged to remove any volatile compounds. Appropriate preservatives are also added to the container. The trip blank is sent with the bottle order and is intended to reflect the environment that the containers are subjected to throughout shipping and handling and help identify possible sources if contamination is found. The field sampler returns the trip blank in the cooler with the field samples.
Field Blanks [1]	Are sometimes used for specific projects by the field samplers. A field blank prepared in the field by filling a clean container with pure reagent water and appropriate preservative, if any, for the specific sampling activity being undertaken. (EPA OSWER)
Equipment Blanks [1]	Are also sometimes created in the field for specific projects. An equipment blank is a sample of analyte-free media which has been used to rinse common sampling equipment to check effectiveness of decontamination procedures. (TNI)
Holding Blanks	Also referred to as refrigerator or freezer blanks, are used to monitor the sample storage units for volatile organic compounds during the storage of VOA samples in the laboratory

[1] When known, these field QC samples should not be selected for matrix QC as it does not provide information on the behavior of the target compounds in the field samples. Usually, the client sample ID will provide information to identify the field blanks with labels such as "FB", "EB", or "TB."

Evaluation criteria and corrective action for these controls are defined in the specific standard operating procedure for each analysis.

24.4 Positive Controls

Control samples (e.g., QC indicators) are analyzed with each batch of samples to evaluate data based upon (1) Method Performance (Laboratory Control Sample (LCS) or Blank Spike (BS)), which entails both the preparation and measurement steps; and (2) Matrix Effects (Matrix Spike (MS) (Matrix spikes are not applicable to air) or Sample Duplicate (MD, DUP), which evaluates field sampling accuracy, precision, representativeness, interferences, and the effect of the matrix on the method performed. Each regulatory program and each method within those programs specify the control samples that are prepared and/or analyzed with a specific batch

Note that frequency of control samples vary with specific regulatory, methodology and project specific criteria. Complete details on method control samples are as listed in each analytical SOP.

24.4.1 Method Performance Control - Laboratory Control Sample (LCS)

The LCS measures the accuracy of the method in a blank matrix and assesses method performance independent of potential field sample matrix affects in a laboratory batch.

The LCS is prepared from a clean matrix similar to that of the associated samples that is free from target analytes (for example: Reagent water, Ottawa sand, glass beads, etc.) and is processed along with and under the same conditions as the associated samples. The LCS is spiked with verified known amounts of analytes or is made of a material containing known and verified amounts of analytes, taken through all preparation and analysis steps along with the field samples. Where there is no preparation taken for an analysis (such as in aqueous volatiles), or when all samples and standards undergo the same preparation and analysis process, a calibration verification standard is reported as the LCS. In some instances where there is no practical clean solid matrix available, aqueous LCS's may be processed for solid matrices; final results may be calculated as mg/kg or ug/kg, assuming 100% solids.

The specific frequency of use for LCS during the analytical sequence is defined in the specific standard operating procedure for each analysis. It is generally 1 for each batch of samples; not to exceed 20 environmental samples.

If the mandated or requested test method, or project requirements, do not specify the spiking components, the laboratory shall spike all reportable components to be reported in the Laboratory Control Sample (and Matrix Spike) where applicable (e.g. no spike of pH). However, in cases where the components interfere with accurate assessment (such as simultaneously spiking chlordane, Toxaphene and PCBs in Method 608), the test method has an extremely long list of components or components are incompatible, at a minimum, a representative number of the listed components (see below) shall be used to control the test method. The selected components of each spiking mix shall represent all chemistries, elution patterns and masses, permit specified analytes and other client requested components. However, the laboratory shall ensure that all reported components are used in the spike mixture within a two-year time period.

Note: This section is not applicable to Ohio EPA VAP projects.

- For methods that have 1-10 target analytes, spike all components.
- For methods that include 11-20 target analytes, spike at least 10 or 80%, whichever is greater.
- For methods with more than 20 target analytes, spike at least 16 components.
- Exception: Due to analyte incompatibility in pesticides, Toxaphene and Chlordane are only spiked at client request based on specific project needs.
- Exception: Due to analyte incompatibility between the various PCB aroclors, aroclors 1016 and 1260 are used for spiking as they cover the range of all of the aroclors. Specific aroclors may be used by request on a project specific basis.

24.5 Sample Matrix Controls

Table 24-3. Sample Matrix Control

Control Type	Details	
Matrix Spikes (MS)	Use	Used to assess the effect sample matrix of the spiked sample has on the precision and accuracy of the results generated by the method used;
	Typical Frequency [1]	At a minimum, with each matrix-specific batch of samples processed, an MS is carried through the complete analytical procedure. Unless specified by the client, samples used for spiking are randomly selected and rotated between different client projects. If the mandated or requested test method does not specify the spiking components, the laboratory shall spike all reportable components to be reported in the Laboratory Control Sample and Matrix Spike. Refer to the method SOP for complete details
	Description	Essentially a sample fortified with a known amount of the test analyte(s).
Surrogate	Use	Measures method performance to sample matrix (organics only).
	Typical Frequency [1]	Are added to all samples, standards, and blanks, for all organic chromatography methods except when the matrix precludes its use or when a surrogate is not available. The recovery of the surrogates is compared to the acceptance limits for the specific method. Poor surrogate recovery may indicate a problem with sample composition and shall be reported, with data qualifiers, to the client whose sample produced poor recovery.
	Description	Are similar to matrix spikes except the analytes are compounds with properties that mimic the analyte of interest and are unlikely to be found in environment samples.
Duplicates[2]	Use	For a measure of analytical precision, with each matrix-specific batch of samples processed, a matrix duplicate (MD or DUP) sample, matrix spike duplicate (MSD), or LCS duplicate (LCSD) is carried through the complete analytical procedure.
	Typical Frequency [1]	Duplicate samples are usually analyzed with methods that do not require matrix spike analysis.
	Description	Performed by analyzing two aliquots of the same field sample independently or an additional LCS.
Internal Standards	Use	Are spiked into all environmental and quality control samples (including the initial calibration standards) to monitor the qualitative aspect of organic and some inorganic analytical measurements.
	Typical Frequency [1]	All organic and ICP methods as required by the analytical method.
	Description	Used to correct for matrix effects and to help troubleshoot variability in analytical response and are assessed after data acquisition. Possible sources of poor internal standard response are sample matrix, poor analytical technique or instrument performance.

[1] See the specific analytical SOP for type and frequency of sample matrix control samples.

[2] LCSD's are normally not performed except when regulatory agencies or client specifications require them. The recoveries for the spiked duplicate samples must meet the same laboratory established recovery limits as the accuracy QC samples. If an LCSD is analyzed both the LCS and LCSD must meet the same recovery criteria and be included in the final report. The precision measurement is reported as "Relative Percent Difference" (RPD). Poor precision between duplicates (except LCS/LCSD) may indicate non-homogeneous matrix or sampling.

24.6 Acceptance Criteria (Control Limits)

As mandated by the test method, each individual analyte in the LCS, MS, or Surrogate Spike is evaluated against the control limits published in the test method. Where there are no established acceptance criteria, the laboratory calculates in-house control limits with the use of

control charts or, in some cases, utilizes client project specific control limits. When this occurs, the regulatory or project limits will supersede the laboratory's in-house limits.

Note: For methods, analytes and matrices with very limited data (e.g., unusual matrices not analyzed often), interim limits are established using available data or by analogy to similar methods or matrices.

Once control limits have been established, they are verified, reviewed, and updated if necessary on an annual basis unless the method requires more frequent updating. Control limits are established per method (as opposed to per instrument) regardless of the number of instruments utilized.

Laboratory generated % Recovery acceptance (control) limits are generally established by taking ± 3 Standard Deviations (99% confidence level) from the average recovery of a minimum of 20-30 data points (more points are preferred).

- Regardless of the calculated limit, the limit should be no tighter than the Calibration Verification (ICV/CCV). (Unless the analytical method specifies a tighter limit).
- In-house limits cannot be any wider than those mandated in a regulated analytical method. (e.g., SDWA Dioxin Method 1613B 2,3,7,8-TCDD on-going precision and recovery limits of 73% to 146% recovery). Client or contract required control limits are evaluated against the laboratory's statistically derived control limits to determine if the data quality objectives (DQOs) can be achieved. If laboratory control limits are not consistent with DQOs, then alternatives must be considered, such as method improvements or use of an alternate analytical method.
- The lowest acceptable recovery limit will be 10% (the analyte must be detectable and identifiable). Exception: The lowest acceptable recovery limit for Benzidine will be 5% and the analyte must be detectable and identifiable.

24.6.1 The lab must be able to generate a current listing of their control limits and track when the updates are performed. In addition, the laboratory must be able to recreate historical control limits.

24.6.2 Refer to SOP KNOX-QA-0004, Control Limits and Control Charting for details on the generation of control limits and the use of control charts at Eurofins TestAmerica Knoxville.

24.6.3 A LCS that is within the acceptance criteria establishes that the analytical system is in control and is used to validate the process. Samples that are analyzed with an LCS with recoveries outside of the acceptance limits may be determined as out of control and should be reanalyzed if possible. If reanalysis is not possible, then the results for all affected analytes for samples within the same batch must be qualified when reported. The internal corrective action process (see Section 12) is also initiated if an LCS exceeds the acceptance limits. Sample results may be qualified and reported without reanalysis if:

- The analyte results are below the reporting limit and the LCS is above the upper control limit.
- If the analytical results are above the relevant regulatory limit and the LCS is below the lower control limit. (Note: This is not applicable to Ohio EPA VAP projects.)
- For Department of Defense (DOD) and Department of Energy (DOE) work, there are an allowable number of Marginal Exceedances (ME). The use of allowable marginal exceedances may also be used for evaluation of LCS recoveries for TNI accredited methods.

Note: For Ohio EPA VAP, if an analyte is identified as a contaminant of interest, then the analyte must meet the LCS criteria and marginal exceedance limits are not applicable.

<11 analytes	0 marginal exceedances are allowed.
11 – 30 Analytes	1 marginal exceedance is allowed
31-50 Analytes	2 marginal exceedances are allowed
51-70 Analytes	3 marginal exceedances are allowed
71-90 Analytes	4 marginal exceedances are allowed
> 90 Analytes	5 marginal exceedances are allowed

- Marginal exceedances are recovery exceedances between 3 SD and 4 SD from the mean recovery limit (TNI). (Note: The DOD/DOE QSM has defined limits for marginal exceedances. Refer to the DOD QSM SOP KNOX-QA-0021, DOD/DOE QSM Program Requirements.)
- Marginal exceedances must be random. If the same analyte exceeds the LCS control limit repeatedly, it is an indication of a systematic problem. The source of the error must be located and corrective action taken. The laboratory has a system to monitor marginal exceedances to ensure that they are random. . Refer to the DOD/DOE QSM SOP KNOX-QA-0021, DOD/DOE QSM Program Requirements for details on how the laboratory ensures that the marginal exceedences are random.
- Though marginal exceedences may be allowed, the data must still be qualified to indicate it is outside of the normal limits.

24.6.4 If the MS/MSDs do not meet acceptance limits, the MS/MSD and the associated spiked sample is reported with a qualifier for those analytes that do not meet limits. If obvious preparation errors are suspected, or if requested by the client, unacceptable MS/MSDs are reprocessed and reanalyzed to prove matrix interference. A more detailed discussion of acceptance criteria and corrective action can be found in analytical SOPs and in Section 12.

24.6.5 If a surrogate standard falls outside the acceptance limits, and if there is not obvious chromatographic matrix interference, reanalyze the sample to confirm a possible matrix effect. If the recoveries confirm or there was obvious chromatographic interference, results are reported from the original analysis and a qualifier is added. If the reanalysis meets surrogate recovery criteria, the second run is reported (or both are reported if requested by the client). Under certain circumstances, where all of the samples are from the same location and share similar chromatography, the reanalysis may be performed on a single sample rather than all of the samples and if the surrogate meets the recovery criteria in the reanalysis, all of the affected samples would require reanalysis.

24.7 Additional Procedures to Assure Quality Control

The laboratory has written and approved method SOPs to assure the accuracy of the test method including calibration (see Section 20), use of certified reference materials (see Section 21) and use of PT samples (see Section 17).

A discussion regarding MDLs, Limit of Detection (LOD) and Limit of Quantitation (LOQ) can be found in Section 19.

- Use of formulae to reduce data is discussed in the method SOPs and in Section 20.
- Selection of appropriate reagents and standards is included in Section 11 and 21.
- A discussion on selectivity of the test is included in Section 7.
- Constant and consistent test conditions are discussed in Section 6.
- The laboratories sample acceptance policy is included in Section 23.

25.0 REPORTING RESULTS

25.1 Overview

The results of each test are reported accurately, clearly, unambiguously, and objectively in accordance with State and Federal regulations as well as client requirements. Analytical results are issued in a format that is intended to satisfy customer and laboratory accreditation requirements as well as provide the end user with the information needed to properly evaluate the results. Where there is conflict between client requests and laboratory ethics or regulatory requirements, the laboratory's ethical and legal requirements are paramount, and the laboratory will work with the client during project set up to develop an acceptable solution. Refer to Section 9.

A variety of report formats are available to meet specific needs.

In cases where a client asks for simplified reports, there must be a written request from the client. There still must be enough information that would show any analyses that were out of conformance (QC out of limits) and there should be a reference to a full report that is made available to the client.

Review of reported data is included in Section 19.

25.2 Test Reports

Analytical results are reported in a format that is satisfactory to the client and meets all requirements of applicable accrediting authorities and agencies. A variety of report formats are available to meet specific needs. The report is printed on laboratory letterhead, reviewed, and signed by the appropriate project manager. At a minimum, the standard laboratory report shall contain the following information:

25.2.1 A report title (e.g., Analytical Report) on the cover page with a "Result" column header on the sample results page.

25.2.2 The cover page shall include the laboratory name, address and telephone number.

25.2.3 A unique identification of the report (e.g., Job ID #) and on each page an identification in order to ensure the page is recognized as part of the report and a clear identification of the end.

Note: Page numbers of reports are represented on the upper right hand corner of each page. A table of contents precedes the cover page on each report which includes the page number of each section as well as the total number of pages.

25.2.4 A copy of the chain of custody (COC).

- Any COCs involved with Subcontracting are included.

25.2.5 The name and address of client and a project name/number, if applicable.

25.2.6 Client project manager or other contact.

25.2.7 Description and unambiguous identification of the tested sample(s) including the client identification code.

25.2.8 Date of receipt of sample, date and time of collection, and date(s) of test preparation and performance, and time of preparation or analysis if the required holding time for either activity is less than or equal to 72 hours. Both date and time of preparation and analysis are required to be included in the data report for work done in support of the DoD QSM; see SOP KNOX-QA-0021, “Department of Defense Quality Systems Manual Program Requirements.”

25.2.9 Date reported or date of revision, if applicable.

25.2.10 Method of analysis including method code (EPA, Standard Methods, etc.).

25.2.11 Reporting limit or Minimum Level.

25.2.12 Method detection limits or Estimated Detection Limits (if requested)

25.2.13 Definition of Data qualifiers and reporting acronyms (e.g. ND).

25.2.14 Sample results.

25.2.15 QC data consisting of method blank, surrogate, LCS, and MS/MSD recoveries and control limits.

25.2.16 Condition of samples at receipt including temperature. This may be accomplished in a narrative or by attaching sample login sheets. . This is discussed in the narrative and a copy of the condition upon receipt form is included with the chain of custody.

25.2.17 A statement to the effect that the results relate only to the items tested and the sample as received by the laboratory, except when information is provided by the client. When data is provided by the client there shall be a clear identification of it, and a disclaimer shall be put in the report when the client supplied data can affect the validity of the test.

25.2.18 A statement that the report shall not be reproduced except in full, without prior express written approval by the laboratory coordinator.

25.2.19 A signature and title of the person(s) accepting responsibility for the content of the report and date of issue. Authorized signatories are qualified Project Managers appointed by the Manager of Project Managers.

25.2.20 When TNI accreditation is required, the lab shall certify that the test results meet all requirements of TNI or provide reasons and/or justification if they do not.

25.2.21 Where applicable, a narrative to the report that explains the issue(s) and corrective action(s) taken in the event that a specific accreditation or certification requirement was not met.

25.2.22 When soil samples are analyzed, a specific identification as to whether soils are reported on a “wet weight” or “dry weight” basis.

25.2.23 Appropriate laboratory certification number for the state of origin of the sample, if applicable.

25.2.24 If only part of the report is provided to the client (client requests some results before all of it is complete), it must be clearly indicated on the report (e.g., preliminary report), and that a complete report will follow once all of the work has been completed. A complete report must be sent once all of the work has been completed.

25.2.25 Any non Eurofins TestAmerica subcontracted analysis results are provided as a separate report on the official letterhead of the subcontractor. All Eurofins TestAmerica subcontracting is clearly identified on the report as to which laboratory performed a specific analysis.

25.2.26 For Ohio EPA VAP work, a VAP affidavit must be completed and included with the report. Refer to the current Ohio Rules OAC 3745-300-04 and 3745-300-13(N) for additional details regarding Ohio EPA VAP affidavits.

25.2.27 A Certification Summary Report, where required, will document that, unless otherwise noted, all analytes tested and reported by the laboratory were covered by the noted certifications.

25.3 Reporting Level or Report Type

The laboratory offers four levels of quality control reporting. Each level, in addition to its own specific requirements, contains all the information provided in the preceding level. The packages provide the following information in addition to the information described above:

- Level 1 is a report with all of the elements outlined in Section 25.2 above, excluding 25.2.15 (QC data).
- Level II is a Level I report plus summary information, including results for the method blank reported to the laboratory MDL, percent recovery for laboratory control samples and matrix spike samples, and the RPD values for all MSD and sample duplicate analyses.
- Level III contains all the information supplied in Level II, but presented on the CLP-like summary forms, and relevant calibration information. A Level II report is not included, unless specifically requested. No raw data is provided.
- Level IV is the same as Level III with the addition of all raw supporting data.

In addition to the various levels of QC packaging, the laboratory also provides reports in EDD (see below). Initial reports may be provided to clients by facsimile. Procedures used to ensure client confidentiality are outlined in Section 25.6.

25.4 Electronic Data Deliverables (EDDs)

EDDs are routinely offered as part of Eurofins TestAmerica's services in addition to the test report as described in Section 25.2. When NELAP accreditation is required and both a test report and EDD are provided to the client, the official version of the test report will be the combined information of the report and the EDD. Eurofins TestAmerica Knoxville offers a variety of EDD formats including industry standard and client specific electronic deliverables.

EDDs shall be subject to a review to ensure their accuracy and completeness. If EDD generation is automated, review may be reduced to periodic screening if the laboratory can demonstrate that it can routinely generate that EDD without errors. Any revisions to the EDD format must be reviewed until it is demonstrated that it can routinely be generated without errors. If the EDD can be reproduced accurately and if all subsequent EDDs can be produced error-free, each EDD does not necessarily require a review.

25.5 Supplemental Information for Test

The lab identifies any unacceptable QC analyses or any other unusual circumstances or observations such as environmental conditions and any non-standard conditions that may have

affected the quality of a result. This is typically in the form of a footnote or a qualifier and/or a narrative explaining the discrepancy in the front of the report. See Appendix 4 for example data qualifiers.

Numeric results with values outside of the calibration range, either high or low are qualified as estimated.

Where quality system requirements are not met, a statement of compliance/non-compliance with requirements and/or specifications is required, including identification of test results derived from any sample that did not meet TNI sample acceptance requirements such as improper container, holding time, or temperature.

Where applicable, a statement on the estimated uncertainty of measurements; information on uncertainty is needed when a client's instructions so require.

When, as requested by the client and agreed to by Eurofins TestAmerica, the report includes a statement of conformity to specification or standard (see Special Services, Section 7.4), the report shall clearly identify:

- to which results the statement applies,
- which specifications, standard or parts thereof are met or not, and
- the decision rule that was applied (unless the decision rule is inherent in the requested specification or standard, taking into account the level of risk (such as false accept and false reject and statistical assumptions) associated with the decision rule.

Opinions and Interpretations - The test report contains objective information, and generally does not contain subjective information such as opinions and interpretations. If such information is required by the client, the Laboratory Director will determine if a response can be prepared. If so, the Laboratory Director will designate the appropriate member of the management team to prepare a response. The response will be fully documented, and reviewed by the Laboratory Director, before release to the client. There may be additional fees charged to the client at this time, as this is a non-routine function of the laboratory.

Note: Review of data deliverable packages for submittal to regulatory authorities requires responses to non-conforming data concerning potential impact on data quality. This necessitates a limited scope of interpretation, and this work is performed by the QA Department. This is the only form of "interpretation" of data that is routinely performed by the laboratory.

When opinions or interpretations are included in the report, the laboratory provides an explanation as to the basis upon which the opinions and interpretations have been made. Opinions and interpretations are clearly noted as such and where applicable, a comment should be added suggesting that the client verify the opinion or interpretation with their regulator.

25.6 Environmental Testing Obtained From Subcontractors

If the laboratory is not able to provide the client the requested analysis, the samples would be subcontracted following the procedures outlined in the NDSC Document No. CW-L-S-004, Subcontracting,

Data reported from analyses performed by a subcontractor laboratory are clearly identified as such on the analytical report provided to the client. Results from a subcontract laboratory outside of Eurofins TestAmerica Knoxville are reported to the client on the subcontract laboratory's original report stationary and the report includes any accompanying documentation.

Note: For Ohio EPA VAP projects Eurofins TestAmerica's affidavit must not attest that the data from the subcontract laboratory are certified. The subcontractor's affidavit must be included and the report must clearly state the test were subcontracted.

25.7 Client Confidentiality

Eurofins TestAmerica Knoxville is responsible for maintaining in confidence all client information obtained or created. In situations involving the transmission of environmental test results by telephone, facsimile or other electronic means, client confidentiality must be maintained.

The laboratory will not intentionally divulge to any person (other than the client or any other person designated by the client in writing) any information regarding the services provided by the laboratory or any information disclosed to the laboratory by the client. Furthermore, information known to be potentially endangering to national security or an entity's proprietary rights will not be released.

Information about the client obtained from sources other than the client (e.g., complainant, regulators) shall be confidential between client and the laboratory. The source of this information shall be confidential to the laboratory and shall not be shared with the client, unless agreed by the source.

Note: This shall not apply to the extent that the information is required to be disclosed by the laboratory under the compulsion of legal process. The laboratory will, to the extent feasible, provide reasonable notice to the client before disclosing the information.

Note: Authorized representatives of an accrediting authority are permitted to make copies of any analyses or records relevant to the accreditation process, and copies may be removed from the laboratory for purposes of assessment.

25.7.1 Report deliverable formats are discussed with each new client. If a client requests that reports be mailed, the reports are to meet all requirements of this document.

25.8 Format of Reports

The format of reports is designed to accommodate each type of environmental test carried out and to minimize the possibility of misunderstanding or misuse.

25.9 Amendments to Test Reports

Corrections, additions, or deletions to reports are only made when justification arises through supplemental documentation. Justification is documented using the laboratory's corrective action system (refer to Section 12).

The revised report is retained on the Archive data server, as is the original report. The revised report is stored in the Archive data server under the job number followed by "rev" The revised report will have the word "revised" on the cover page.

When the report is re-issued, a notation of "Reissued Report" is placed on the cover/signature page of the report or at the top of the narrative page with a brief explanation of reason for the re-issue.

25.10 Policies on Client Requests for Amendments

25.10.1 Policy on Data Omissions or Reporting Limit Increases

Fundamentally, our policy is simply to not omit previously reported results (including data qualifiers) or to not raise reporting limits and report sample results as ND. This policy has few exceptions. Exceptions are:

- Laboratory error.
- Sample identification is indeterminate (confusion between COC and sample labels).
- An incorrect analysis (not analyte) was requested (e.g., COC lists 8315 but client wanted 8310). A written request for the change is required.
- Incorrect limits reported based on regulatory requirements.
- The requested change has absolutely ***no possible*** impact on the interpretation of the analytical results and there is ***no possibility*** of the change being interpreted as misrepresentation by anyone inside or outside of our company.

25.10.2 Multiple Reports

The laboratory does not issue multiple reports for the same work order where there is different information on each report (this does not refer to copies of the same report) unless required to meet regulatory needs and approved by QA.

Appendix 1. List of Governing Documents applicable to the QA Manual

(NDSC and Laboratory SOPs and Policies)

NDSC Doc. No.	Title
CA-C-S-001	Work Sharing Process
CA-I-P-002	Electronic Reporting and Signature Policy
CA-L-P-002	Contract Compliance Policy
CA-Q-M-002	Quality Management Plan
CA-Q-QM-001	Guidance on Tentatively Identified Compounds (TICs) – GC/MS Analysis
CA-Q-S-001	Acid and Solvent-Lot Testing and Approval Program
CA-Q-S-002	Manual Integrations
CA-Q-S-006	Detection and Quantitation Limits
CA-Q-S-009	Root Cause Analysis
CA-T-P-001	Qualified Products List
CW-E-M-001	Environmental Health & Safety Manual
CW-F-P-002	Company-Wide Authorization Matrix
CW-F-P-004	Procurement and Contracts Policy
CW-F-S-007	Fixed Asset Acquisition, Retention and Safeguarding
CW-I-M-001	IT Change Control Procedure Manual
CW-L-P-001	Records Retention Policy
CW-L-P-004	Ethics Policy
CW-L-S-002	Internal Investigation
CW-L-S-004	Subcontracting
CW-Q-S-001	Document Control and Archiving
CW-Q-S-002	Writing a Standard Operating Procedure (SOPs)
CW-Q-S-003	Internal Auditing
CW-Q-S-004	Management Systems Review
CW-Q-S-005	Data Recall Process
CW-Q-S-001	Document Control and Archiving

Local Laboratory Document No.	Title
KNOX-QA-0001	Standard/Reagent Labeling and Documentation
KNOX-QA-0004	Control Limits and Control Charting
KNOX-QA-0005	Balance Calibration and Weight Verification
KNOX-QA-0006	Subsampling
KNOX-QA-0008	Nonconformance and Corrective Action
KNOX-QA-0009	Personnel Orientation and Training
KNOX-QA-0010	Temperature Monitoring and Thermometer Calibration
KNOX-QA-0011	Document Control & Distribution
KNOX-QA-0012	Pipette and Volumetric Labware Calibration Verification
KNOX-QA-0018	Preparation and Management of Standard Operating Procedures
KNOX-QA-0019	Estimation of Measurement Uncertainty
KNOX-QA-0021	Department of Defense Quality Systems Manual Program Requirements
KNOX-AD-0003	Documenting Project Requirements
KNOX-MS-0014	Tentatively Identified Compounds (TICS)
KNOX-SC-0003	Sample Receipt and Login
KNOX-SC-0005	Sample Disposal, Return and Archive

Appendix 2. List of Laboratory Certifications, Accreditations, Validations

The laboratory maintains accreditations, certifications, and approvals with numerous state and national entities. Programs vary but may include on-site audits, reciprocal agreements with another entity, performance testing evaluations, review of the QA Manual, Standard Operating Procedures, Method Detection Limits, training records, etc. At the time of this QA Manual revision, the laboratory has accreditation/ certification/licensing with the following organizations:

eurofins | Environment Testing TestAmerica

Certification List

Laboratory	Program	Authority	Identification	Expiration Date
Knoxville	Dept. of Defense ELAP	ANAB	L2311	02/13/2022
Knoxville	Dept. of Energy	ANAB	L2311.01	02/13/2022
Knoxville	ISO/IEC 17025	ANAB	L2311	02/14/2022
Knoxville	NELAP	Florida	E87177	07/01/2021
Knoxville	NELAP	Kansas	E-10349	10/31/2021
Knoxville	NELAP	Louisiana	83979	06/30/2021
Knoxville	NELAP	New Hampshire	299919	01/17/2022
Knoxville	NELAP	New Jersey	TN001	07/01/2021
Knoxville	NELAP	New York	10781	04/01/2021
Knoxville	NELAP	Oregon	TNI0189	01/01/2022
Knoxville	NELAP	Pennsylvania	68-00576	12/31/2021
Knoxville	NELAP	Texas	T104704380-18-12	08/31/2021
Knoxville	NELAP	Utah	TN00009	07/31/2021
Knoxville	NELAP	Virginia	460176	09/14/2021
Knoxville	State	Arkansas DEQ	88-0688	06/17/2021
Knoxville	State	California	2423	06/30/2021
Knoxville	State	Colorado	TN00009	02/28/2021
Knoxville	State	Connecticut	PH-0223	09/30/2021
Knoxville	State	Georgia (DW)	906	12/11/2022
Knoxville	State	Hawaii	NA	12/11/2021
Knoxville	State	Kentucky (DW)	90101	01/01/2021 *
Knoxville	State	Louisiana (DW)	LA019	12/31/2021
Knoxville	State	Maryland	277	03/31/2021
Knoxville	State	Michigan	9933	12/11/2022
Knoxville	State	Nevada	TN00009	07/31/2021
Knoxville	State	North Carolina (DW)	21705	07/31/2021
Knoxville	State	North Carolina (WW/SW)	64	12/31/2021
Knoxville	State	Ohio VAP	CL0059	06/02/2023
Knoxville	State	Oklahoma	9415	08/31/2021
Knoxville	State	Tennessee	02014	12/11/2022
Knoxville	State	Washington	C593	01/19/2022
Knoxville	State	West Virginia (DW)	9955C	01/02/2022
Knoxville	State	West Virginia DEP	345	05/01/2021
Knoxville	State	Wisconsin	998044300	08/31/2021
Knoxville	US Federal Programs	US Fish & Wildlife	058448	07/31/2021
Knoxville	US Federal Programs	USDA	P330-19-00236	08/20/2022

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* Certification Valid - Laboratory is Pending Renewal with the Program Authority

For more information, or to contact a local Eurofins representative nearest you, please visit our website at www.testamericainc.com

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Appendix 3. References used to prepare the QA Manual

The QAM has been prepared to be consistent with the requirements of the following documents:

- ANSI/ASQC, E4-1994, "Specifications and Guidelines for Quality Management Systems for Environmental Data Collection and Environmental Technology Programs" (American National Standard, January 5, 1995, or most recent version)
- "EPA Requirements for Quality Management Programs" (QA/R-2) (EPA/240/B-01/002, May 31, 2006).
- EPA 600/4-79-019, *Handbook for Analytical Quality Control in Water and Wastewater Laboratories,* EPA, March 1979.
- *Test Methods for Evaluating Solid Waste Physical/Chemical Methods (SW846), Third Edition, September 1986, Final Update I, July 1992, Final Update IIA, August 1993, Final Update II, September 1994; Final Update IIB, January 1995; Final Update III, December 1996; Final Update IV, January 2008; Final Update V, August 2015.*
- Federal Register, 40 CFR Parts 136, 141, 172, 173, 178, 179 and 261.
- *Manual for the Certification of Laboratories Analyzing Drinking Water (EPA 815-R-05-004, January 2005) (DW labs only)*
- APHA, *Standard Methods for* the Examination of Water and Wastewater, 18th Edition, 19th, 20th, 21st, 22nd and on-line Editions.
- U.S. Department of Energy Order 414.1B, Quality Assurance, Approved April 29, 2004.
- U.S. Department of Energy Order 414.1C, Quality Assurance, June 17, 2005.
- U.S. Department of Energy Order 414.1D, Quality Assurance, April, 25, 2011.
- U.S. Department of Defense (DoD)/Department of Energy (DOE) Consolidated Quality Systems Manual (QSM) for Environmental Laboratories, Version 5.1.1, dated 2017

Appendix 4. QA Manual Crosswalk with TNI and ISO/IEC 17025 Standards

Sec. No.	Title	2009 and 2016 TNI Standard Reference	ISO/IEC 17025:2005(E) Reference	ISO/IEC 17025:2017(E) Reference
-	QUALITY ASSURANCE MANUAL COVER PAGE	V1M2 Sec. 4.2.8.3		
1.0	Title Page			
2.0	Table of Contents	V1M2 Secs. 4.2.8.3-4.2.8.4		8.1.2, 8.2.1
3.0	Introduction, Scope and Applicability	V1M2 Sec. 4.2.8.4		
3.1	Introduction and Compliance References	V1M2 Secs. 1.1; 1.2; 2.0; 3.2; 4.1.2; 4.2.4	4.1.2; 4.2.4	5.3; 5.4; 8.2.4; 8.3.1
3.2	Terms and Definitions	V1M2 Secs. 3.0; 4.2.4	4.2.4	
3.3	Scope /Field of Testing	V1M2 Secs. 1.2; 4.2.4	4.1.2; 4.2.4	5.3; 5.4; 8.2.1; 8.2.4
3.4	Management of The Manual	V1M2 Secs. 4.2.1; 4.2.7; 4.3.3.2; 4.3.3.3	4.2.1; 4.2.7; 4.3.3.2; 4.3.3.3	5.3
4.0	Management and Responsibilities	V1M2 Sec. 4		8.2.4; 8.2.5
4.1	Overview	V1M2 Secs. 4.1.1, 4.1.3; 4.1.5	4.1.1; 4.1.3; 4.1.5; 4.2.6	5.1; 5.2; 5.5; 5.6 ; 6.2.1; 6.2.4
4.2	Roles and Responsibilities	V1M2 Secs. 4.1.4; 4.1.5; 4.1.6; 4.2.1; 4.2.6; 5.2.4	4.1.3; 4.1.5; 4.1.6; 4.2.1; 4.2.6; 5.2.4	4.1.1 to 4.1.3; 4.1.5; 5.5; 5.6; 6.2.1; 6.2.4; 6.2.6 8.2.2;
4.3	Business Continuity and Contingency Plan *(Prev. Deputies)*	V1M2 Secs. 4.1.5; 4.1.7.2; 4.2.7	4.1.5; 4.2.7	
5.0	**Error! Reference source not found.**	V1M2 Secs. 5.2; 5.2.1	5.2.1	6.1; 6.2.3
5.1	Overview	V1M2 Secs. 5.2.2; 5.2.3; 5.2.5	5.2.2; 5.2.3; 5.2.5	6.2.2
5.2	Education and Experience Requirements for Technical Staff	V1M2 Secs. 5.2.1; 5.2.3; 5.2.4	5.2.1; 5.2.3; 5.2.4	6.2.2 to 6.2.4
5.3	Training	V1M2 Sec. 5.2.5	5.2.5	4.2.1; 6.2.2; 6.2.4; 6.2.5
5.4	Data Integrity and Ethics Training Program	V1M2 Sec. 4.2.8.1; 5.2.7		4.1.1
6.0	ACCOMMODATIONS AND ENVIRONMENTAL CONDITIONS	V1M2 Sec. 5.3		6.1; 6.3.1
6.1	Overview	V1M2 Secs. 5.3.1; 5.3.3; 5.3.4; 5.3.5	5.3.1; 5.3.3; 5.3.4; 5.3.5	6.3.1
6.2	Environment	V1M2 Secs. 5.3.1; 5.3.2; 5.3.3; 5.3.4; 5.3.5	5.3.1; 5.3.2; 5.3.3; 5.3.4; 5.3.5	6.3.1 to 6.3.5
6.3	Work Area	V1M2 Secs. 5.3.3; 5.3.4; 5.3.5	5.3.3; 5.3.4; 5.3.5	6.3.1
6.4	Responding to Emergencies			
6.5	Building Security	V1M2 Sec. 5.3.4	5.3.4	6.3.4
7.0	QUALITY SYSTEM			6.1; 8.2.4
7.1	Quality Policy	V1M2 Secs. 4.1.5; 4.2.2; 4.2.3; 4.2.8.3	4.1.5; 4.2.2; 4.2.3	8.2.3; 8.6.1
7.2	Ethics and Data Integrity	V1M2 Secs. 4.1.5; 4.16; 4.2.2; 4.2.8.1; 5.2.7	4.1.5; 4.2.2	4.1.1 to 4.1.3; 4.2.1; 6.2.1; 8.2.2; 8.2.3
7.3	Quality System Documentation	V1M2 Secs. 4.1.5; 4.2.2; 4.2.5	4.2.2; 4.2.5	8.2.4

Sec. No.	Title	2009 and 2016 TNI Standard Reference	ISO/IEC 17025:2005(E) Reference	ISO/IEC 17025:2017(E) Reference
7.4	QA/QC Objectives for the Measurement of Data	V1M2 Sec. 4.2.2	4.1.5; 4.2.2	6.2.4
7.5	Criteria for Quality Indicators			
7.6	Statistical Quality Control			
7.7	Quality System Metrics			
8.0	Document Control	V1M2 Secs. 4.2.7; 4.3.1; 4.3.2.2 ; 4.3.3.3; 4.3.3.4	4.2.7; 4.3.1; 4.3.2.2; 4.3.3.3; 4.3.3.4	8.2.4; 8.3.1
8.1	Overview			8.2.5; 8.3.1; 8.3.2
8.2	Document Approval and Issue	V1M2 Secs. 4.3.2; 4.3.2.1-4.3.2.3; 4.3.3.1	4.3.2.1; 4.3.2.2; 4.3.2.3; 4.3.3.1	8.2.5; 8.3.2
8.3	Procedures for Document Control Policy	V1M2 Secs. 4.3.2.1–4.3.2.2; 4.3.3.1	4.3.2.1; 4.3.2.2; 4.3.3.1	8.2.5; 8.3.2
8.4	Obsolete Documents	V1M2 Secs. 4.3.2.1–4.3.2.2	4.3.2.1; 4.3.2.2	8.2.5; 8.3.2
9.0	SERVICE TO THE CLIENT	V1M2 Secs. 4.4.1 - 4.4.4	4.4.1; 4.4.2; 4.4.3; 4.4.4	7.1.1; 7.1.1.4; 7.1.1.5; 7.1.1.8; 7.1.2.1
9.1	Overview	V1M2 Secs. 4.4.5; 4.5.5; 5.7.1	4.4.5; 5.7.1	
9.2	Review Sequence and Key Personnel	V1M2 Sec. 4.4.5	4.4.5	7.1.1.6
9.3	Balancing Laboratory Work Load and Capacity			
9.4	Documentation	V1M2 Sec. 5.7.1	5.7.1	
9.5	Special Services	V1M2 Secs. 4.7.1-4.7.2	4.7.1; 4.7.2	7.1.1.3; 7.1.1.7
9.6	Client Communication	V1M2 Secs. 4.7.1-4.7.2	4.7.1; 4.7.2	7.1.1.7
9.7	Reporting	V1M2 Secs. 4.7.1-4.7.2	4.7.1; 4.7.2	7.1.1.7
9.8	Client Feedback and Surveys	V1M2 Secs. 4.7.1-4.7.2	4.7.1; 4.7.2	7.1.1.7; 8.6.2
10.0	SUBCONTRACTING OF TESTS	V1M2 Secs. 4.4.3; 4.5.4	4.4.3; 4.5.4	
10.1	Overview	V1M2 Secs. 4.5.1 - 4.5.3; 4.5.5; 5.3.1	4.5.1; 4.5.2; 4.5.3; 5.3.1	6.6.1; 7.1.2.1; 7.1.2.2
10.2	Qualifying and Monitoring Subcontractors	V1M2 Secs. 4.5.1; 4.5.2; 4.5.3; 4.5.5	4.5.1; 4.5.2; 4.5.3	6.6.1; 7.1.2.1; 7.1.2.2
10.3	Oversight and Reporting	V1M2 Sec. 4.5.5		
10.4	Contingency Planning			
10.5	Use of NELAP and A2LA Logo			
11.0	PURCHASING SERVICES AND SUPPLIES	V1M2 Sec. 4.6.1	4.6.1	
11.1	Overview	V1M2 Secs. 4.6.2; 4.6.3; 4.6.4	4.6.2; 4.6.3; 4.6.4	6.6.1; 6.6.2
11.2	Glassware	V1M2 Sec. 5.5.13.1		
11.3	Reagents, Standards & Supplies	V1M2 Secs. 4.6.2; 4.6.3; 4.6.4 2016 V1M4 1.7.2.5	4.6.2; 4.6.3; 4.6.4	6.6.1 to 6.6.3
11.4	Purchase of Equipment / Instruments / Software			
11.5	Services			
11.6	Suppliers			
12.0	COMPLAINTS	V1M2 Sec. 4.8	4.8	8.6.1; 8.6.2
12.1	Overview			7.9.1 to 7.9.3 8.6.1; 8.6.2;
12.2	External Complaints			7.9.2 to 7.9.7 8.6.1; 8.6.2;
12.3	Internal Complaints			8.6.1; 8.6.2

Sec. No.	Title	2009 and 2016 TNI Standard Reference	ISO/IEC 17025:2005(E) Reference	ISO/IEC 17025:2017(E) Reference
12.4	Management Review			8.6.1; 8.6.2
13.0	CONTROL OF NON-CONFORMING WORK	V1M2 Secs. 4.9.1; 5.10.5	4.9.1; 5.10.5	7.10.1
13.1	Overview	V1M2 Secs. 4.9.1; 4.11.3; 4.11.5	4.9.1; 4.11.3; 4.11.5	7.10.1
13.2	Responsibilities and Authorities	V1M2 Secs. 4.9.1; 4.11.3; 4.11.5; 5.2.7	4.9.1; 4.11.3; 4.11.5	7.10.1
13.3	Evaluation of Significance and Action Taken	V1M2 Secs. 4.9.1; 4.11.3; 4.11.5	4.9.1; 4.11.3; 4.11.5	4.1.5; 7.10.1; 7.10.2; 8.5.3
13.4	Prevention of Non-Conforming Work	V1M2 Secs. 4.9.4; 4.11.2	4.9.2; 4.11.2	7.10.2; 7.10.3; 8.5.3
13.5	Method Suspension / Restriction (Stop Work Procedures)	V1M2 Secs. 4.9.1; 4.9.2; 4.11.5	4.9.1; 4.9.2; 4.11.5	7.10.1; 7.10.2
14.0	CORRECTIVE ACTION	V1M2 Sec. 4.11		4.1.4; 4.1.5
14.1	Overview	V1M2 Secs. 4.9.2; 4.11.1; 4.11.2	4.9.2; 4.11.1; 4.11.2	7.10.2 8.7.1; 8.7.3;
14.2	General	V1M2 Sec. 4.11.2; 4.11.3	4.11.2; 4.11.3	7.7.2; 8.5.3; 8.7.1;
14.3	Closed Loop Correction Action Process	V1M2 Sec. 4.11.2; 4.11.3; 4.11.4; 4.11.6; 4.11.7; 4.12.2	4.11.2; 4.11.3; 4.11.4; 4.12.2	8.5.3; 8.6.1; 8.7.2
14.4	Technical Corrective Actions	V1M2 Sec. 4.11.6		8.7.1
14.5	Basic Corrections	V1M2 Secs. 4.11.1; 4.13.2.3	4.11.1; 4.13.2.3	7.5.2; 8.7.1
15.0	PREVENTIVE ACTION / IMPROVEMENT	V1M2 Secs. 4.10; 4.12.1; 4.12.2	4.10; 4.12.1; 4.12.2	4.1.4
15.1	Overview	V1M2 Secs. 4.15.1; 4.15.2	4.15.1; 4.15.2	8.6.2
15.2	Management of Change			
16.0	CONTROL OF RECORDS	V1M2 Secs. 4.2.7; 4.13.1.1; 4.13.3	4.2.7; 4.13.1.1	8.4.2
16.1	Overview	V1M2 Secs. 4.13.1.1 - 4.13.1.4; 4.13.2.1-4.13.2.3; 4.13.3	4.13.1.1 - 4.13.1.4; 4.13.2.1-4.13.2.3	8.4.1; 8.4.2
16.2	Programs with Longer Retention Requirements			
16.3	Technical and Analytical Records	V1M2 Sec. 4.13.2.2 - 4.13.2.3	4.13.2.2; 4.13.2.3	7.5.1; 8.4.2
16.4	Laboratory Support Activities			7.5.2; 8.4.2
16.5	Administrative Records			8.4.2
16.6	Records Management, Storage and Disposal	V1M2 Sec. 4.13.3		4.2.1; 8.4.2
17.0	AUDITS			
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19.8	Verification of Detection Limits	V1M2 Sec. 5.9.3		
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Appendix 5. Terms/Glossary and Acronyms (EL-V1M2 Sec. 3.1)

Terms/Glossary:

Acceptance Criteria: Specified limits placed on characteristics of an item, process, or service defined in requirement documents. (ASQC)

Accreditation: The process by which an agency or organization evaluates and recognizes a laboratory as meeting certain predetermined qualifications or standards, thereby accrediting the laboratory.

Accuracy: The degree of agreement between an observed value and an accepted reference value. Accuracy includes a combination of random error (precision) and systematic error (bias) components which are due to sampling and analytical operations; a data quality indicator. (QAMS)

Analyst: The designated individual who performs the "hands-on" analytical methods and associated techniques and who is the one responsible for applying required laboratory practices and other pertinent quality controls to meet the required level of quality.

Analytical Uncertainty: A subset of Measurement Uncertainty that includes all laboratory activities performed as part of the analysis. (TNI)

Anomaly: A condition or event, other than a deficiency, that may affect the quality of the data, whether in the laboratory's control or not.

Assessment: The evaluation process used to measure or establish the performance, effectiveness, and conformance of an organization and/or its systems to defined criteria (to the standards and requirements of laboratory accreditation). (TNI)

Audit: A systematic and independent examination of facilities, equipment, personnel, training, procedures, record-keeping, data validation, data management, and reporting aspects of a system to determine whether QA/QC and technical activities are being conducted as planned and whether these activities will effectively achieve quality objectives. (TNI)

Batch: Environmental samples that are prepared and/or analyzed together with the same process and personnel, using the same lot(s) of reagents. A **preparation batch** is composed of one (1) to twenty (20) environmental samples of the same quality systems matrix, meeting the above mentioned criteria and with a maximum time between the start of processing of the first and last sample in the batch to be twenty-four (24) hours. An **analytical batch** is composed of prepared environmental samples (extracts, digestates or concentrates) which are analyzed together as a group. An analytical batch can include prepared samples originating from various quality system matrices and can exceed twenty (20) samples. (TNI)

Bias: The systematic or persistent distortion of a measurement process, which causes errors in one direction (i.e., the expected sample measurement is different from the sample's true value). (TNI)

Blank: A sample that has not been exposed to the analyzed sample stream in order to monitor contamination during sampling, transport, storage or analysis. The blank is subjected to the usual analytical and measurement process to establish a zero baseline or background value and is sometimes used to adjust or correct routine analytical results. (ASQC)

Calibration: A set of operations that establish, under specified conditions, the relationship between values of quantities indicated by a measuring instrument or measuring system, or values represented by a material measure or a reference material, and the corresponding values realized by standards. (TNI)

1) In calibration of support equipment the values realized by standards are established through the use of reference standards that are traceable to the International System of Units (SI).

2) In calibration according to methods, the values realized by standards are typically established through the use of Reference Materials that are either purchased by the laboratory with a certificate of analysis or purity, or prepared by the laboratory using support equipment that has been calibrated or verified to meet specifications.

Calibration Curve: The mathematical relationship between the known values, such as concentrations, of a series of calibration standards and their instrument response. (TNI)

Calibration Standard: A substance or reference material used to calibrate an instrument (QAMS)

Certified Reference Material (CRM): A reference material accompanied by a certificate, having a value, measurement uncertainty, and stated metrological traceability chain to a national metrology institute. (TNI)

Chain of Custody (COC) Form: Record that documents the possession of the samples from the time of collection to receipt in the laboratory. This record generally includes: the number and types of containers; the mode of collection; the collector; time of collection; preservation; and requested analyses. (TNI)

Compromised Samples: Those samples which are improperly sampled, insufficiently documented (chain of custody and other sample records and/or labels), improperly preserved, collected in improper containers, or exceeding holding times when delivered to a laboratory. Under normal conditions, compromised samples are not analyzed. If emergency situation require analysis, the results must be appropriately qualified.

Confidential Business Information (CBI): Information that an organization designates as having the potential of providing a competitor with inappropriate insight into its management, operation or products. TNI and its representatives agree to safeguard identified CBI and to maintain all information identified as such in full confidentiality.

Confirmation: Verification of the identity of a component through the use of an approach with a different scientific principle from the original method. These may include, but are not limited to Second Column Confirmation; Alternate wavelength; Derivatization; Mass spectral interpretation; Alternative detectors or Additional Cleanup procedures. (TNI)

Conformance: An affirmative indication or judgment that a product or service has met the requirements of the relevant specifications, contract, or regulation; also the state of meeting the requirements. (ANSI/ASQC E4-1994)

Correction: Actions necessary to correct or repair analysis specific non-conformances. The acceptance criteria for method specific QC and protocols as well as the associated corrective actions. The analyst will most frequently be the one to identify the need for this action as a result of calibration checks and QC sample analysis**.** No significant action is taken to change behavior, process or procedure.

Corrective Action: The action taken to eliminate the causes of an existing nonconformity, defect or other undesirable situation in order to prevent recurrence. (ISO 8402)

Data Integrity: The condition that exists when data are sound, correct, and complete, and accurately reflect activities and requirements. (TNI)

Data Audit: A qualitative and quantitative evaluation of the documentation and procedures associated with environmental measurements to verify that the resulting data re of acceptable quality (i.e., that they meet specified acceptance criteria).

Data Reduction: The process of transforming the number of data items by arithmetic or statistical calculations, standard curves, and concentration factors, and collation into a more useable form. (TNI)

Deficiency: An unauthorized deviation from acceptable procedures or practices, or a defect in an item (ASQC), whether in the laboratory's control or not.

Demonstration of Capability: A procedure to establish the ability of the analyst to generate analytical results of acceptable accuracy and precision. (TNI)

Document Control: The act of ensuring that documents (and revisions thereto) are proposed, reviewed for accuracy, approved for release by authorized personnel, distributed properly, and controlled to ensure use of the correct version at the location where the prescribed activity if performed. (ASQC)

Duplicate Analyses: The analyses or measurements of the variable of interest performed identically on two subsamples of the same sample. The results from duplicate analyses are used to evaluate analytical or measurement precision but not the precision of sampling, preservation or storage internal to the laboratory. (EPA-QAD)

Equipment Blank: Sample of analyte-free media which has been used to rinse common sampling equipment to check effectiveness of decontamination procedures.

External Standard Calibration: Calibrations for methods that do not utilize internal standards to compensate for changes in instrument conditions.

Field Blank: Blank prepared in the field by filing a clean container with pure de-ionized water and appropriate preservative, if any, for the specific sampling activity being undertaken (EPA OSWER)

Field of Accreditation: Those matrix, technology/method, and analyte combinations for which the accreditation body offers accreditation.

Holding Blank: A blank matrix stored with field samples of a similar matrix (Volatiles only) that measures storage contribution to any source of contamination.

Holding Times: The maximum time that samples may be held prior to analyses and still be considered valid or not compromised. (40 CFR Part 136)

Internal Standard: A known amount of standard added to a test portion of a sample as a reference for evaluating and controlling the precision and bias of the applied analytical test method. (TNI)

Internal Standard Calibration: Calibrations for methods that utilize internal standards to compensate for changes in instrument conditions.

Instrument Blank: A clean sample (e.g., distilled water) processed through the instrumental steps of the measurement process; used to determine instrument contamination. (EPA-QAD)

Instrument Detection Limit (IDL): The minimum amount of a substance that can be measured with a specified degree of confidence that the amount is greater than zero using a specific instrument. The IDL is associated with the instrumental portion of a specific method only, and sample preparation steps are not considered in its derivation. The IDL is a statistical estimation at a specified confidence interval of the concentration at which the relative uncertainty is ± 100%. The IDL represents a range where qualitative detection occurs on a specific instrument. Quantitative results are not produced in this range.

Laboratory Control Sample (however named, such as laboratory fortified blank, spiked blank, or QC check sample): A sample matrix, free from the analytes of interest, spiked with verified known amounts of analytes or a material containing known and verified amounts of analytes, taken through all preparation and analysis steps of the procedure unless otherwise noted in a reference method. It is generally used to establish intra-laboratory or analyst specific precision and bias or to assess the performance of all or a portion of the measurement system.

An LCS shall be prepared at a minimum of 1 per batch of 20 or less samples per matrix type per sample extraction or preparation method except for analytes for which spiking solutions are not available such as total suspended solids, total dissolved solids, total volatile solids, total solids, pH, color, odor, temperature, dissolved oxygen or turbidity. The results of these samples shall be used to determine batch acceptance.

Least Squares Regression (1st Order Curve): The least squares regression is a mathematical calculation of a straight line over two axes. The y axis represents the instrument response (or Response ratio) of a standard or sample and the x axis represents the concentration. The regression calculation will generate a correlation coefficient (r) that is a measure of the "goodness of fit" of the regression line to the data. A value of 1.00 indicates a perfect fit. In order to be used for quantitative purposes, r must be greater than or equal to 0.99 for organics and 0.995 for inorganics.

Limit(s) of Detection (LOD) [a.k.a., Method Detection Limit (MDL)]: The MDL is the minimum measured quantity of a substance that can be reported with 99% confidence that the concentration is distinguishable from method blank results, consistent with 40CFR Part 136 Appendix B, August, 2017.

Limit(s) of Quantitation (LOQ) [a.k.a., Reporting Limit]: The minimum levels, concentrations, or quantities of a target variable (e.g., target analyte) that can be reported with a specified degree of confidence.

(QS) Matrix: The component or substrate that contains the analyte of interest. For purposes of batch and QC requirement determinations, the following matrix distinctions shall be used:

Aqueous: Any aqueous sample excluded from the definition of Drinking Water or Saline/Estuarine. Includes surface water, groundwater effluents, and TCLP or other extracts.

Drinking Water: Any aqueous sample that has been designated as a potable or potential potable water source.

Saline/Estuarine: Any aqueous sample from an ocean or estuary, or other salt water source such as the Great Salt Lake.

Non-Aqueous Liquid: Any organic liquid with <15% settleable solids.

Biological Tissue: Any sample of a biological origin such as fish tissue, shellfish, or plant material. Such samples shall be grouped according to origin.

Solids: Includes soils, sediments, sludges, and other matrices with >15% settleable solids.

Chemical Waste: A product or by-product of an industrial process that results in a matrix not previously defined.

Air & Emissions: Whole gas or vapor samples including those contained in flexible or rigid wall containers and the extracted concentrated analytes of interest from a gas or vapor that are collected with a sorbant tube, impinger solution, filter, or other device. (TNI)

Matrix Spike (spiked sample or fortified sample): A sample prepared, taken through all sample preparation and analytical steps of the procedure unless otherwise noted in a referenced method, by adding a known amount of target analyte to a specified amount of sample for which an independent test result of target analyte concentration is available. Matrix spikes are used, for example, to determine the effect of the matrix on a method's recovery efficiency.

Matrix Spike Duplicate (spiked sample or fortified sample duplicate): A replicate matrix spike prepared and analyzed to obtain a measure of the precision of the recovery for each analyte.

Method Blank: A sample of a matrix similar to the batch of associated samples (when available) that is free from the analytes of interest and is processed simultaneously with and under the same conditions as samples through all steps of the analytical procedures, and in which no target analytes or interferences are present at concentrations that impact the analytical results for sample analyses.

Method Detection Limit: See Limit of Detection (LOD)

Negative Control: Measures taken to ensure that a test, its components, or the environment do not cause undesired effects, or produce incorrect test results.

Non-conformance: An indication, judgment, or state of not having met the requirements of the relevant specifications, contract, or regulation.

Observation: A record of phenomena that (1) may assist in evaluation of the sample data; (2) may be of importance to the project manager and/or the client, and yet not at the time of the observation have any known effect on quality.

Performance Audit: The routine comparison of independently obtained qualitative and quantitative measurement system data with routinely obtained data in order to evaluate the proficiency of an analyst or laboratory.

Positive Control: Measures taken to ensure that a test and/or its components are working properly and producing correct or expected results from positive test subjects.

Precision: The degree to which a set of observations or measurements of the same property, obtained under similar conditions, conform to themselves; a data quality indicator. Precision is usually expressed as standard deviation, variance or range, in either absolute or relative terms. (TNI)

Preservation: Any conditions under which a sample must be kept in order to maintain chemical and/or biological integrity prior to analysis. (TNI)

Proficiency Testing: A means of evaluating a laboratory's performance under controlled conditions relative to a given set of criteria through analysis of unknown samples provided by an external source. (TNI)

Proficiency Testing Program: The aggregate of providing rigorously controlled and standardized environmental samples to a laboratory for analysis, reporting of results, statistical evaluation of the results and the collective demographics and results summary of all participating laboratories. (TNI)

Proficiency Test Sample (PT): A sample, the composition of which is unknown to the laboratory and is provided to test whether the laboratory can produce analytical results within specified acceptance criteria. (TNI)

Quality Assurance: An integrated system of management activities involving planning, implementation, assessment, reporting and quality improvement to ensure that a process, item or service is of the type of quality needed and expected by the client. (TNI)

Quality Assurance [Project] Plan (QAPP): A formal document describing the detailed quality control procedures by which the quality requirements defined for the data and decisions pertaining to a specific project are to be achieved. (EAP-QAD)

Quality Control: The overall system of technical activities that measures the attributes and performance of a process, item, or service against defined standards to verify that they meet the stated requirements established by the customer; operational techniques and activities that are used to fulfill requirements for quality; also the system of activities and checks used to ensure that measurement systems are maintained within prescribed limits, providing protection against “out of control” conditions and ensuring that the results are of acceptable quality. (TNI)

Quality Control Sample: A sample used to assess the performance of all or a portion of the measurement system. One of any number of samples, such as Certified Reference Materials, a quality system matrix fortified by spiking, or actual samples fortified by spiking, intended to demonstrate that a measurement system or activity is in control. (TNI)

Quality Manual: A document stating the management policies, objectives, principles, organizational structure and authority, responsibilities, accountability, and implementation of an agency, organization, or laboratory, to ensure the quality of its product and the utility of its product to its users. (TNI)

Quality System: A structured and documented management system describing the policies, objectives, principles, organizational authority, responsibilities, accountability, and implementation plan of an organization for ensuring quality in its work processes, products (items), and services. The quality system provides the framework for planning, implementing, and assessing work performed by the organization and for carrying out required QA and QC activities. (TNI)

Raw Data: The documentation generated during sampling and analysis. This documentation includes, but is not limited to, field notes, electronic data, magnetic tapes, untabulated sample results, QC sample results, print outs of chromatograms, instrument outputs, and handwritten records. (TNI)

Record Retention: The systematic collection, indexing and storing of documented information under secure conditions.

Reference Material: Material or substance one or more properties of which are sufficiently homogeneous and well established to be used for the calibration of an apparatus, the assessment of a measurement method, or for assigning values to materials. (TNI)

Reference Standard: Standard used for the calibration of working measurement standards in a given organization or a given location. (TNI)

Sampling: Activity related to obtaining a representative sample of the object of conformity assessment, according to a procedure.

Second Order Polynomial Curve (Quadratic): The 2nd order curves are a mathematical calculation of a slightly curved line over two axis. The y axis represents the instrument response (or Response ratio) of a standard or sample and the x axis represents the concentration. The 2nd order regression will generate a coefficient of determination (COD or r^2) that is a measure of the "goodness of fit" of the quadratic curvature the data. A value of 1.00 indicates a perfect fit. In order to be used for quantitative purposes, r^2 must be greater than or equal to 0.99.

Selectivity: The ability to analyze, distinguish, and determine a specific analyte or parameter from another component that may be a potential interferent or that may behave similarly to the target analyte or parameter within the measurement system. (TNI)

Sensitivity: The capability of a method or instrument to discriminate between measurement responses representing different levels (e.g., concentrations) of a variable of interest. (TNI)

Spike: A known mass of target analyte added to a blank, sample or sub-sample; used to determine recovery efficiency or for other quality control purposes.

Standard: The document describing the elements of laboratory accreditation that has been developed and established within the consensus principles of standard setting and meets the approval requirements of standard adoption organizations procedures and policies. (TNI)

Standard Operating Procedures (SOPs): A written document which details the method for an operation, analysis, or action, with thoroughly prescribed techniques and steps. SOPs are officially approved as the methods for performing certain routine or repetitive tasks. (TNI)

Storage Blank: A blank matrix stored with field samples of a similar matrix (volatiles only) that measures storage contribution to any source of contamination.

Surrogate: A substance with properties that mimic the analyte of interest. It is unlikely to be found in environment samples and is added to them for quality control purposes.

Surrogate compounds must be added to all samples, standards, and blanks, for all organic chromatography methods except when the matrix precludes its use or when a surrogate is not available. Poor surrogate recovery may indicate a problem with sample composition and shall be reported to the client whose sample produced poor recovery. (QAMS)

Systems Audit (also Technical Systems Audit): A thorough, systematic, qualitative on-site assessment of the facilities, equipment, personnel, training, procedures, record keeping, data validation, data management, and reporting aspects of a total measurement system. (EPA-QAD)

Technical Manager: A member of the staff of an environmental laboratory who exercises actual day-to-day supervision of laboratory operations for the appropriate fields of accreditation and reporting of results

Technology: A specific arrangement of analytical instruments, detection systems, and/or preparation techniques.

Traceability: The ability to trace the history, application, or location of an entity by means of recorded identifications. In a calibration sense, traceability relates measuring equipment to national or international standards, primary standards, basic physical constants or properties, or reference materials. In a data collection sense, it relates calculations and data generated throughout the project back to the requirements for the quality of the project. (TNI)

Trip Blank: A blank matrix placed in a sealed container at the laboratory that is shipped, held unopened in the field, and returned to the laboratory in the shipping container with the field samples.

Uncertainty: A parameter associated with the result of a measurement that characterizes the dispersion of the value that could reasonably be attributed to the measured value.

Acronyms:

CAR – Corrective Action Report

CCV – Continuing Calibration Verification

CF – Calibration Factor

CFR – Code of Federal Regulations

COC – Chain of Custody

DOC – Demonstration of Capability

DQO – Data Quality Objectives

DUP - Duplicate

EHS – Environment, Health and Safety

EPA – Environmental Protection Agency

GC - Gas Chromatography

GC/MS - Gas Chromatography/Mass Spectrometry

HPLC - High Performance Liquid Chromatography

ICP - Inductively Coupled Plasma Atomic Emission Spectroscopy

ICP/MS – ICP/Mass Spectrometry

ICV – Initial Calibration Verification

IDL – Instrument Detection Limit
IH – Industrial Hygiene
IS – Internal Standard
LCS – Laboratory Control Sample
LCSD – Laboratory Control Sample Duplicate
LIMS – Laboratory Information Management System
LOD – Limit of Detection
LOQ – Limit of Quantitation
MDL – Method Detection Limit
MDLCK – MDL Check Standard
MDLV – MDL Verification Check Standard
MRL – Method Reporting Limit Check Standard
MS – Matrix Spike
MSD – Matrix Spike Duplicate
SDS - Safety Data Sheet
NELAP - National Environmental Laboratory Accreditation Program
PT – Performance Testing
TNI – The NELAC Institute
QAM – Quality Assurance Manual
QA/QC – Quality Assurance / Quality Control
QAPP – Quality Assurance Project Plan
RF – Response Factor
RPD – Relative Percent Difference
RSD – Relative Standard Deviation
SD – Standard Deviation
SOP – Standard Operating Procedure
TAT – Turn-Around-Time
VOA – Volatiles
VOC – Volatile Organic Compound

Appendix 6. Analytical Methods and Matrices

Analytical Parameters	Fields of Testing[1]				
	Drinking Water (SDWA)	**Non-Potable Water (CWA/RCRA)**	**Solid and Chemical Materials (RCRA-SW846)**	**Air and Emissions (CAA)**	**Biological Tissue**
Anions (Br^-, Cl^-, F^-, NO_2, NO_3, o-PO_4, SO_4	**EPA 300.0**	**EPA 300.0 (CWA) EPA 9056 (RCRA)**	**EPA 9056**	Mod Method 26A/0050 (9057 Mod)	---
Ash	---	---	**ASTM D482**	---	---
Density	---	---	ASTM D1963, D854	---	---
Halogens	---	---	EPA 5050/9056 (KNOX-WC-0016)	---	---
Heat of Combustion (Btu)	---	---	**ASTM D5865, D240**	---	---
Hexavalent Chromium	---	---	---	Method 0061/7199	---
pH	---	EPA 9040C (RCRA) SM4500 H+B (CWA)	EPA 9040C EPA 9045D	---	---
Hardness	---	**SM 2340B (CWA)**	---	---	---
Particulates	---	---	---	PM-10, **0050/5, 5B, 5J**, 201A, **202**, **TCEQ-23**, OTM-27, OTM-28	---
Mercury	---	**EPA 7470A (RCRA)**	**EPA 7471A&B**	**EPA 29/0060/7470A** ASTM D6784-02/7470A EPA IO-3.1/7470A	**EPA3051/3052/ 7470A**
Metals	---	**EPA 200.7 (CWA) EPA 3005A/ 6010B&C EPA 3010A/ 6010B&C (RCRA)**	**EPA 3050B/ 6010B&C EPA 3051/3052/6010B&C**	**EPA 29/ 0060/6010B&C EPA IO-3.1/6010B&C 40CFR60-12 (Pb)**	**EPA 3051/3052/ 6010B&C**
Sulfur	---	---	ASTM D3177, ASTM D129	---	---
Viscosity	---	---	**ASTM D445**	---	---

(continued) Analytical Methods and Matrices

Analytical Parameters	Fields of Testing[1]				
	Drinking Water (SDWA)	**Non-Potable Water (CWA/RCRA)**	**Solid and Chemical Materials (RCRA-SW846)**	**Air and Emissions (CAA)**	**Biological Tissue**
Volatiles by GC/MS	---	**EPA 5030B EPA 8260B (RCRA)**	**EPA 5035 EPA 8260B**	**TO-14A, TO-15 EPA 0031/5041A (VOST)**	---
Volatiles by GC	---	---	---	EPA 0040 Condensates **ASTM D1946**	---
Semivolatiles by GC/MS	---	**EPA 3520C EPA 8270C&D (RCRA)**	**EPA3550B&C EPA 3640A EPA 8270C&D**	**EPA 3542/8270C&D TO-13A**	---
Semivolatiles by GC	---	**EPA 3520C PCBs EPA 8082A (RCRA)**	**EPA 3550B&C EPA 3640A EPA 8082A**	**EPA 3542**/8082A **EPA 3542**/8081A **TO-4A, TO-10A** TCO/Grav	**EPA 3550B&C EPA 3640A** PCBs EPA 8082A
Dioxins/Furans by HRGC/HRMS	**EPA 1613B**	**EPA 1613B (CWA) EPA 8290 EPA 8290A (RCRA)**	**EPA 3640A EPA 8290 EPA 8290A**	**TO-9A, Method 23/8290/8290A EPA 0023A/8290/8290A**	**EPA 3640A EPA 8290 EPA 8290A EPA 1613B**
Lipids	---	---	---	---	EPA 8290, Micro-Lipids (KNOX-OP-0019)
PCBs by HRGC/HRMS	---	**EPA 1668A&B&C CWA**	**EPA3640A EPA 1668A&B&C**	**EPA 1668A&B&C**	**EPA 3640A EPA 1668A&B&C**
Polycyclic Aromatic Hydrocarbons (PAH)	---	**EPA 3520C EPA 8270C&D** ID0016	**EPA 3550B&C/3640A/ 8270C&D EPA 3540C/3640A/ ID0016**	**EPA 3542/8270C&D EPA 3542/ID0016**	**EPA 3550B&C/ 3640A/** 8270C&D EPA **3540C/3640A/ ID0016**

(1) **NELAC accredited analytical methods and accepted prep methods are highlighted in bold fon**

Appendix 7. Example Data Qualifiers (Standard Flagging Suite)[1]

Qualifier	Footnote
!	Laboratory is not accredited for this parameter.
*	RPD of the LCS and LCSD exceeds the control limits
*	LCS or LCSD is outside acceptance limits
>	The analyte exceeded the indicated concentration
^	Instrument related QC is outside acceptance limits
4	MS,MSD: The analyte present in the original sample is greater than 4 times the matrix spike concentration; therefore control limits are not applicable
AP	Altered Pattern
B	Compound was found in the blank and sample
B7	Target analyte detected in the method blank at or above method reporting limit. Concentration found in sample was at 10X the concentration found in the blank
c	Compound falls outside routine control limits, but is within acceptable client specific calibration criteria
cn	Refer to Case Narrative for further detail
D	Sample results are obtained from a dilution; the surrogate or matrix spike recoveries reported are calculated from diluted samples
D	Surrogate or matrix spike recoveries were not obtained because the extract was diluted for analysis; also compounds analyzed at a dilution may be flagged with a D
D	Sample results are obtained from a dilution; the surrogate or matrix spike recoveries reported are calculated from diluted samples.
DW	Result has been dry weight corrected
E	Result exceeded calibration range.
F1	MS and/or MSD Recovery is outside acceptance limits.
F2	MS/MSD RPD exceeds control limits
F3	Duplicate RPD exceeds the control limit
F4	MS/MSD RPD exceeds control limits due to sample size difference.
F5	Duplicate RPD exceeds limit, and one or both sample results are less than 5 times RL. The data are considered valid because the absolute difference is less than the RL.
H	Sample was prepped or analyzed beyond the specified holding time
h	Alternate peak selection upon analytical review.
I	Indicates the presence of an interference, recovery is not calculated.
J	Result is less than the RL but greater than or equal to the MDL and the concentration is an approximate value.
NC	Not Confirmed
p	The %RPD between the primary and confirmation column/detector is >40%. The lower value has been reported.
P	The %RPD between the primary and confirmation column/detector is >40%. The higher value has been reported
Q	Result was qualitatively confirmed, but not quantitated.
q	The reported result is the estimated maximum possible concentration of this analyte, quantitated using the theoretical ion ratio. The measured ion ratio does not meet qualitative identification criteria and indicates a possible interference.
S	Ion suppression
U	Indicates the analyte was analyzed for but not detected.

V	Serial Dilution exceeds the control limits
W	PS: Post-digestion spike was outside control limits
X	Surrogate is outside control limits

1 – This is a standard list of qualifiers commonly used. Other qualifiers are available in LIMS.

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EUROFINS/TESTAMERICA KNOXVILLE
SOP CHANGE FORM

SOP NUMBER: KNOX-ID-0013 Rev 19

SOP TITLE: Analysis of Polychlorinated Biphenyl (PCB) Isomers by Isotope Dilution HRGC/HRMS

SOP SECTIONS AFFECTED BY CHANGE: 10.3.6

REASON FOR ADDITION OR CHANGE: FL DOH NELAP audit finding based on TNI 2016 V1M4 1.7.1.1.k.i and ii

CHANGE EFFECTIVE FROM: 08/13/21

CHANGE OR ADDITION (SPECIFY SECTION; USE ADDITIONAL SHEETS IF NECESSARY)

10.2.11. A readback of the low calibration point used in the calibration curve should have a read-back (%D) of no more than 50% for any calibration model. The readback of the non low level standards of the initial calibration should have a read-back (%D) of no more than 30% for any calibration model.

SUBMITTED BY/DATE: KRM 08/10/21

APPROVED BY:

Technical Reviewer Signature	Date 8/11/21
Environmental Health & Safety Signature	Date 8.11.21
QA Signature	Date 8/11/2021
Management Signature	Date 8/11/2021

STANDARD OPERATING PROCEDURE
TITLE: Analysis of Polychlorinated Biphenyl (PCB) Isomers by Isotope Dilution HRGC/HRMS

(SUPERSEDES: KNOX-ID-0013, Revision 18)

Approvals (Signature/Date):

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1 Scope and Application

1.1 This procedure is designed to meet analytical program requirements where HRGC/HRMS analysis of polychlorinated biphenyl (PCB) isomers is specified. The procedure is used by Eurofins TestAmerica Knoxville for the qualitative and quantitative measurement of all 209 PCB congeners in a variety of environmental matrices at part-per-trillion (ppt) to part-per-quadrillion (ppq) concentrations. This procedure is based on EPA method 1668A, 1668B and 1668C.

1.2 The compounds listed in Table 1 may be determined by this procedure. The detection limits and quantitation levels in this method are usually dependent on the level of interferences rather than instrumental limitations. The estimated minimum levels (EMLs) in Table 4 are the levels at which the PCBs can be determined with only common laboratory interferences present. The actual limits of detection and quantitation will vary depending on the complexity of the matrix.

1.3 The Low Calibration Levels (LCLs) of the method are listed in Table 3 for individual congeners. Analysis of a one-tenth aliquot of the sample permits measurements of concentrations up to 10 times the upper calibration range. Samples containing concentrations of PCBs that are greater than ten times the upper calibration are analyzed by protocols designed for such concentration levels.

1.4 The GC/MS portions of this method are for use only by analysts experienced with HRGC/HRMS or under the close supervision of such qualified persons. Each laboratory that uses this method must demonstrate the ability to generate acceptable results using the procedure in section 9.1.

1.5 This procedure is a "performance-based" method. These reference methods allow modifications to overcome interferences or lower the cost of measurements, if all performance criteria in the methods are met and method equivalency is established. Deviations from the referenced methods have been incorporated into this procedure and are listed in section 17.1. Deviations to this procedure are only allowed as specified in section 11.1.

1.6 Because of the extreme toxicity of many of these compounds, the analyst must take the necessary precautions to prevent exposure to materials known or believed to contain PCBs. It is the responsibility of the laboratory personnel to ensure that safe handling procedures are employed. Section 5 of this procedure discusses safety procedures.

2 Summary of Method

2.1 All solid and semi-solid samples are screened by GC/ECD prior to extraction. Other sample matrices may be screened if the potential for high congener concentrations exists. Variations in sample size, spiking levels and final volume are established based on the screening result.

2.2 After sample extraction, cleanup, and concentration, recovery standards are added to each extract, and an aliquot of the extract is injected into the gas chromatograph. The analytes are separated by the GC and detected by a high-resolution (≥10,000) mass spectrometer. Two exact masses are monitored for each analyte.

2.3 An individual PCB congener is identified by comparing the GC retention time and ion-abundance ratio of two exact masses with the corresponding retention time of an authentic

standard and the theoretical or acquired ion-abundance ratio of the two exact masses.

2.4 Quantitative analysis is performed using selected ion current profile areas using the internal standard technique.

2.5 The quality of the analysis is assured through reproducible calibration and verification of the extraction, cleanup, and GC/MS systems.

3 Definitions

These definitions and purposes are specific to this method but conform to common usage as much as possible.

Note: Terminology differences existing in some isotope dilution reference methods regarding the functionality of the labeled analogs may lead to confusion. For example, EPA's Office of Solid Waste methods (8280, 8290) use the term "Internal Standards" to describe the labeled analogs which are added to the sample prior to extraction and used to quantitate the native targets. EPA's Office of Water methods (1613B, 1668) use the term "Labeled Analogs" to describe these same compounds while using the term "Internal Standards" to describe the labeled analogs which are added to the extract just prior to analysis and used to quantitate the recovery of the labeled analogs added before extraction. EPA's Office of Solid Waste methods (8280, 8290) uses the term "Recovery Standards" to describe these later labeled analogs.

The terminology conventions established by the EPA's Office of Solid Waste methods (8280, 8290) are used in the laboratory for all Standard Operating Procedures and internal communications as defined in this section.Analyte – A PCB tested for by this method. The analytes are listed in Table 1.

3.1 Calibration verification standard (VER) – The mid-point calibration standard (CS3) that is used to verify calibration. See Table 6a.

3.2 CB – Chlorinated biphenyl congener. One of the 209 individual chlorinated biphenyl congeners determined using this method. The 209 CBs are listed in Table 1.

3.3 Congener – Any member of a particular homologous series, for example, 2,2’-DiCB.

3.4 CS0.5, CS1, CS2, CS3, CS4, CS5 – See Calibration standards and Table 6a.

3.5 Estimated Detection Limit (EDL) – The sample specific estimated detection limit (EDL) is the concentration of a given analyte required to produce a signal with a peak height of at least 2.5 times the background signal level (noise level).

3.6 Estimated Maximum Possible Concentration (EMPC) – The calculated concentration of a signal having the same retention time as a PCB congener but which does not meet all of the other qualitative identification criteria defined in the method.

3.7 Estimated Minimum Level (EML) – The lowest concentration at which an analyte can be measured reliably with common laboratory interferences present. This is the reporting limit (RL).

3.8 FC-43 (PFTBA) - Perfluorotributylamine; used to calibrate the exact mass scale in the HRMS.

3.9 Field blank – An aliquot of reagent water or other reference matrix that is placed in a sample container in the laboratory or the field, and treated as a sample in all respects, including exposure to sampling site conditions, storage, preservation, and all analytical procedures. The purpose of the field blank is to determine if the field or sample transporting procedures and environments have contaminated the sample.

3.10 GC – Gas chromatograph or gas chromatography.

3.11 Homologous Series – A series of compounds in which each member contains the same number of chlorine atoms. The members of the series are called homologs.

3.12 HRGC – High resolution GC.

3.13 HRMS – High resolution MS.

3.14 ICV – Initial Calibration Verification Standard. A calibration standard from a second source, traceable to a national standard if possible. The ICV is analyzed after the Initial calibration to verify the concentration of the initial calibration standards.

3.15 Isotope Dilution Analytes (IDAs) – Isotopically labeled analogs of the target analytes that are added to every sample, blank, quality control spike sample, and calibration solution. They are added to the sample before extraction and are used to calculate the concentration of the target analytes or detection limits.

3.16 IPR (also known as IDOC) – Initial precision and recovery; four aliquots of the PAR standard analyzed to establish the ability to generate acceptable precision and accuracy. An IPR is performed prior to the first time this method is used and any time the method or instrumentation undergoes significant modification.

3.17 Isomer – PCB congeners that contain the same number of chlorine atoms, but differ in the structural arrangement of the chlorine atoms. For example, PCB-4 and PCB-9 are isomers.

3.18 Laboratory blank – See Method blank.

3.19 Laboratory control sample (LCS) – See ongoing precision and recovery standard (OPR).

3.20 Laboratory reagent blank – See Method blank.

3.21 Level of Chlorination (LOC) Congeners – The first and last eluting congeners in each homolog (or level of chlorination). (For the SPB-Octyl Column the LOC Congeners are 1, 3; 4, 15; 19, 37; 54, 77; 104, 126; 155, 169; 188, 189; 202, 205; 208, 206; 209)

3.22 Method blank – An aliquot of a clean test matrix that is treated exactly as a sample including exposure to all glassware, equipment, solvents, reagents, internal standards, and surrogates that are used with samples. The method blank is used to determine if analytes or interferences are present in the laboratory environment, the reagents, or the apparatus.

3.23 Minimum Level (ML) – The level at which the entire analytical system must give a recognizable signal and acceptable calibration point for the analyte. It is equivalent to the concentration of the lowest calibration standard, assuming that all method-specified sample weights, volumes, and cleanup procedures have been employed.

Note: EML is the lowest concentration at which an analyte can be measured reliably with common laboratory interferences present

3.24 MS – Mass spectrometer or mass spectrometry.

3.25 OPR (also know as ODOC) – Ongoing precision and recovery standard (OPR); a laboratory blank spiked with known quantities of analytes. The OPR is analyzed exactly like a sample. Its purpose is to assure that the results produced by the laboratory remain within the limits specified in this method for precision and recovery.

3.26 PAR – Precision and recovery standard; secondary standard that is diluted and spiked to form the IPR and OPR.

3.27 PFK – Perfluorokerosene; the mixture of compounds used to calibrate the exact mass scale of the HRMS.

3.28 Primary dilution standard – A solution containing the specified analytes that is purchased or prepared from stock solutions and diluted as needed to prepare calibration solutions and other solutions.

3.29 Quality control check sample (QCS) – A sample containing all or a subset of the analytes at known concentrations. The QCS is obtained from a source external to the laboratory or is prepared from a source of standards different from the source of calibration standards. It is used to check laboratory performance with test materials prepared external to the normal preparation process.

3.30 PCB – Polychlorinated biphenyl.

3.31 Reagent water – Water demonstrated to be free from the analytes of interest and potentially interfering substances at the method minimum level for the analyte.

3.32 Recovery Standard (RS) – Isotopically labeled compounds which are added to every sample, blank, and quality control spike sample extract prior to analysis. They are used to measure the recovery of the internal standards and the cleanup standards.

3.33 Relative Percent Difference (RPD) – A measure of the difference between two values normalized to one of the values. It is used to determine the accuracy of the concentration measurements of second source verification standards.

3.34 Relative standard deviation (RSD) – The standard deviation times 100 divided by the mean. Also termed "coefficient of variation."

3.35 RF or RRF (Relative Response Factor) - The ratio of the response of the mass spectrometer to a known amount of a compound relative to that of a known amount of a reference standard as measured in the initial and continuing calibrations. It is used to determine instrument performance and it is used to calculate the concentration of target analytes, internal standard recoveries, or detection limits in samples, blanks, and quality control samples. See Section 10.3.4.2.

3.36 SPE – Solid-phase extraction; an extraction technique in which an analyte is extracted from an

aqueous sample by passage over or through a material capable of reversibly adsorbing the analyte. Also termed liquid-solid extraction.

3.37 Specificity – The ability to measure an analyte of interest in the presence of interferences and other analytes of interest encountered in a sample.

3.38 Stock solution - A solution containing an analyte that is prepared using a reference material traceable to EPA, the National Institute of Science and Technology (NIST), or a source that will attest to the purity and authenticity of the reference material.

3.39 Field Surrogate Standards (SU) - Isotopically labeled compounds that are added to XAD samples and calibration solution. They are added to XAD sampling tubes before sampling and are used to measure sampling and recovery efficiency.

3.40 Surrogate Standard (SU) – Isotopically labeled compounds that are added to samples, blanks, quality control samples, and calibration solutions. They are added to the samples after extraction but prior to extract cleanup, and are used to assess the efficiency of the cleanup procedures.

3.41 Toxic Congeners (or Toxic Isomers) – PCBs determined by the World Health Organization and USEPA to have dioxin-like toxicity. (PCBs 77, 81, 105, 114, 118, 123, 126, 156, 157, 167, 169, 189)

3.42 Toxic/LOC Congeners – PCBs belonging to either the Toxic Congeners list or the LOC Congeners list.

3.43 VER – See Calibration verification standard.

3.44 Additional definitions can be found in the Eurofins TestAmerica Knoxville Quality Assurance Manual (QAM), current revision.

4 Interferences

4.1 Solvents, reagents, glassware, and other sample processing hardware may yield artifacts and/or elevated baselines causing misinterpretation of chromatograms. Where possible, glassware is cleaned by extraction or solvent rinse. The non-coplanar PCB congeners 105, 114, 118, 123, 156, 157, 167, and 180 have been shown to be very difficult to completely eliminate from the laboratory at the minimum levels in this method, and baking of glassware in a kiln or furnace at 450 - 500°C may be necessary to remove these and other contaminants.

4.2 All materials used in the analysis shall be demonstrated to be free from interferences by running laboratory method blanks (section 9.5) initially and with each sample batch.

4.3 Interferences coextracted from samples will vary considerably from source to source, depending on the diversity of the site being sampled. Interfering compounds may be present at concentrations several orders of magnitude higher than the PCBs. The most frequently encountered interferences are chlorinated dioxins and dibenzofurans, methoxy biphenyls, hydroxy-diphenyl ethers, benzylphenyl ethers, polynuclear aromatics, and pesticides. Because very low levels of PCBs are measured by this method, the elimination of interferences is essential. Cleanup steps can be used to reduce or eliminate these interferences and thereby

permit reliable determination of the PCBs at the levels shown in Table 3.

5 Safety

5.1 Employees must abide by the policies and procedures in the Corporate Safety Manual, Radiation Safety Manual and this document.

5.2 Eye protection that satisfies ANSI Z87.1 (as per the Corporate Safety Manual), laboratory coat and appropriate gloves must be worn while samples, standards, solvents and reagents are being handled. Disposable gloves that have become contaminated will be removed and discarded, other gloves will be cleaned immediately.

5.2.1 Latex and vinyl gloves provide no protection against most of the organic solvents used in this method. For the operations described herein, Nitrile gloves are to be worn. For operations using solvents that may splash, SilverShield® gloves are recommended. SilverShield® gloves protect against breakthrough for most of the solvents used in this procedure.

5.3 The effluents of sample splitters for the gas chromatograph and roughing pumps on the mass spectrometer must be vented to the laboratory hood exhaust system or must pass through an activated charcoal filter.

5.4 The gas chromatograph and mass spectrometer contain zones that have elevated temperatures. The analyst needs to be aware of the locations of those zones, and must cool them to room temperature prior to working on them or use thermal protection when working on them while they are above room temperature.

5.5 The mass spectrometer is under high vacuum. The mass spectrometer must be brought to atmospheric pressure prior to working on the source. Alternatively, the source may be removed from the vacuum manifold through a vacuum interlock.

5.6 There are areas of high voltage in both the gas chromatograph and the mass spectrometer. Depending on the type of work involved, either turn the power to the instrument off, or disconnect it from its source of power. If the work involved requires measurement of voltage supplies, the instrument may be left on.

5.7 Primary Materials Used: The following is a list of the materials used in this method, which have a serious or significant hazard rating. **NOTE: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the SDS for each of the materials listed in the table.** A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the SDS for each material before using it for the first time or when there are major changes to the SDS.

Material	Hazards	Exposure Limit (1)	Signs and symptoms of exposure
Methylene Chloride	Carcinogen, Irritant	25 ppm-TWA, 125 ppm-STEL	Causes irritation to respiratory tract. Has a strong narcotic effect with symptoms of mental confusion, light-headedness, fatigue, nausea, vomiting and headache. Causes irritation, redness and pain to the skin and eyes. Prolonged contact can cause burns. Liquid degreases the skin. May be absorbed through skin.
Hexane	Flammable, Irritant	500 ppm-TWA	Inhalation of vapors irritates the respiratory tract. Overexposure may cause lightheadedness, nausea, headache, and blurred vision. Vapors may cause irritation to the skin and eyes.
Methanol	Flammable, Poison, Irritant	200 ppm-TWA	A slight irritant to the mucous membranes. Toxic effects exerted upon nervous system, particularly the optic nerve. Symptoms of overexposure may include headache, drowsiness and dizziness. Methyl alcohol is a defatting agent and may cause skin to become dry and cracked. Skin absorption can occur; symptoms may parallel inhalation exposure. Irritant to the eyes.
Toluene	Flammable, Poison, Irritant	200 ppm-TWA 300 ppm-Ceiling	Inhalation may cause irritation of the upper respiratory tract. Symptoms of overexposure may include fatigue, confusion, headache, dizziness and drowsiness. Peculiar skin sensations (e. g. pins and needles) or numbness may be produced. Causes severe eye and skin irritation with redness and pain. May be absorbed through the skin.
Acetone	Flammable	1000 ppm-TWA	Inhalation of vapors irritates the respiratory tract. May cause coughing, dizziness, dullness, and headache.
Nonane	Flammable	None established	Harmful if inhaled/swallowed. Vapor/mist is irritating to eyes, mucous memebranes and upper respiratory tract. Causes skin irritiation.
1 – Exposure limit refers to the OSHA regulatory exposure limit.			

5.8 Chemicals that have been classified as **carcinogens**, **potential carcinogens**, or **mutagens include**: methylene chloride, polychlorinated biphenyls, and toluene. The toxicity or carcinogenicity of each reagent used in this method is not precisely defined; however, each chemical compound should be treated as a potential health hazard. From this viewpoint, exposure to these chemicals must be kept to a minimum.)

5.9 Chemicals known to be **flammable** are: acetone, hexane, nonane and toluene.

5.10 Exposure to chemicals will be maintained as low as reasonably achievable; therefore, unless they are known to be non-hazardous, all samples will be opened, transferred and prepared in a fume hood, or under other means of mechanical ventilation. Solvent and waste containers will be kept closed unless transfers are being made.

5.11 The preparation of all standards will be conducted in a fume hood with the sash closed as far as the operations will permit.

5.12 Confinement: Work areas should be isolated and posted with signs. Glassware and tools should be segregated. Bench tops should be covered with plastic backed absorbent paper.

5.13 Waste: Good technique includes minimizing contaminated waste. Plastic bag liners should be used in waste cans.

5.14 Accidents: Remove contaminated clothing immediately, taking precautions not to contaminate skin or other articles. Wash exposed skin vigorously and repeatedly until medical attention is obtained.

5.15 All work must be stopped in the event of a known or potential compromise to the health or safety of laboratory personnel. The situation must be reported immediately to a laboratory supervisor.

6 Equipment and Supplies

6.1 Gas chromatograph - Shall have splitless or on-column injection port for capillary column, temperature program with isothermal hold, and shall meet all of the performance specifications in Section 10.

6.1.1 Column #1 – 30±5-m long x 0.25±0.02-mm ID; 0.25-µm film SPB-Octyl (Supelco 2-4218, or equivalent).

6.2 Mass spectrometer - Electron impact ionization, shall be capable of repetitively selectively monitoring 20 exact masses minimum at high resolution (≥10,000) during a period less than 1.0 second, and shall meet all of the performance specifications in Section 10.

6.3 GC/MS interface - The mass spectrometer (MS) shall be interfaced to the GC such that the end of the capillary column terminates within 1 cm of the ion source but does not intercept the electron or ion beams.

6.4 Data system - Capable of collecting, recording, and storing MS data.

7 Reagents and Standards

CAUTION: Refer to Safety Data Sheets (SDS) for specific safety information on chemicals and reagents prior to use or as needed.

CAUTION: During preparation of reagents, associates shall wear lab coat, gloves, safety glasses with side shields, and laboratory approved shoes as a minimum. Reagents shall be prepared in a fume hood.

7.1 Solvents - Acetone, toluene, n-hexane, methanol, methylene chloride, and nonane; pesticide quality.

7.2 Perfluorokerosene (PFK) high boiling mass spectroscopy grade; bp 210-260°C; d^{20}_4 1.94; n^{20}_D 1.330; Fluka (Catalog No. - 77275).

7.3 FC-43 (PFTBA) is used in neat form to tune and calibrate the mass spectrometer. (Scientific Instrument Services Catalog No. FC-43-35)

7.4 $^{13}C_{12}$ Labeled PCB Congener Isotope Dilution Analytes (IDA): Obtained as individual Certified Reference Standards from Cambridge Isotope Laboratories (CIL, Andover Massachusetts) and

Wellington Laboratories (Guelph, Ontario, Canada). Refer to Table 5b for a list of individual standards. These standards are purchased at 40 µg/mL or 50 µg/mL in nonane. If the chemical purity is 98% or greater, the stated manufacturer concentration may be used without correction to compute the concentration of the standard. Once a standard ampoule has been vortexed and opened, the solution is transferred to an amber glass vial with a Teflon®-lined screw cap. When not being used, standards are stored in a dark box at room temperature. These purchased standards are used to prepare the following mixed stock solutions and spiking solutions:

7.4.1 IDA Stock Solution: Prepared by diluting the individual $^{13}C_{12}$ labeled IDAs listed in Table 5b, to a concentration of 1000 ng/mL in nonane. The concentration is verified by GC/MS before use.

7.4.2 IDA Spiking Solution: Prepared by diluting the 1000 ng/mL IDA stock solution to a concentration of 10 ng/mL in acetone. 1.0 to 4.0 mL of this solution is added to each solid/tissue sample prior to extraction. Refer to Table 12, "Assignment of Sample Preparation Protocols" to determine the exact volume to add. Prior to extraction, aqueous samples are spiked with IDA spiking solution at 1/5 the levels of solid/tissue samples (typically 20 uL).

7.4.3 Internal Standard Stock Solution (IS): Prepared by diluting the individual $^{13}C_{12}$ labeled ISTDslisted in Table 5b to a concentration of 1000 ng/mL in nonane. The concentration is verified by GC/MS before use.

7.4.4 Internal Standard Spiking Solution: Prepared by diluting the 1000 ng/mL internal standard stock solution to a concentration of 100 ng/mL in nonane. Fifty to 100 µL of this spiking solution is added to each solid/tissue sample extract prior to analysis (refer to Table 12), whereas, 20 µL is added to each aqueous sample extract.

7.4.5 Surrogate Stock Solution: Prepared by diluting the individual $^{13}C_{12}$ labeled cleanup standards listed in Table 5b, to a concentration of 5000 ng/mL in nonane. The concentration is verified by GC/MS before use.

7.4.6 Surrogate Standard Spiking Solution: Prepared by diluting the 5000 ng/mL surrogate standard stock solution to a concentration of 10 ng/mL in hexane. One half to 1.0 mL of this solution is added to each solid/tissue sample extract prior to cleanup (refer to Table 12), whereas, 0.20 mL is added to each aqueous sample extract.

7.4.7 Air Surrogate Standard Stock Solution: Prepared by diluting the individual $^{13}C_{12}$ labeled sampling air standards listed in Table 5b to a concentration of 5000 ng/mL in nonane. The concentration is verified by GC/MS before use.

7.4.8 Air Surrogate Spiking Solution: Prepared by diluting the 5000 ng/mL air surrogate stock solution to a concentration of 50 ng/mL in nonane.

7.5 Target PCB Congener Standard Mix: Obtained as a Certified Reference Standard from Accustandard (New Haven, CT). This standard contains all 209 PCB congeners at 4000 ng/mL in nonane. If the chemical purity is 98% or greater, the weight may be used without correction to compute the concentration of the standard. Once a standard ampoule has been vortexed and opened, the solution is transferred to an amber glass vial with a Teflon®-lined screw cap. When

not being used, the standard is stored in a dark box at room temperature. This purchased standard is used to prepare the following target stock solution and spiking solution:

7.5.1 Target PCB Congener Stock Solution: Prepared by diluting the 4000 ng/mL target PCB congener standard mix to a concentration of 40 ng/mL in nonane. The concentration is verified by GC/MS before use.

7.5.2 LCS Target Spiking Solution: Prepared by diluting the 4000 ng/mL target PCB congener standard to a concentration of 5.0 ng/mL in acetone. One mL of this solution is added to each solid/tissue LCS prior to extraction, whereas, 0.20 mL is added to each aqueous LCS.

7.6 Calibration Standard Solutions (CS 0.5 through CS 5) are prepared by dilution of the target PCB congener standards in section 7.5 and 7.5.1 and the IDA standards in section 7.4 in nonane. Table 6a shows the calibration solution analytes and final concentrations. Table 6b provides details for preparation of these calibration solutions.

7.6.1 This series of solutions is used to establish linearity and relative response factors for all compounds in the initial calibration solutions. These RRFs are used to quantify PCB congeners in the calibration verification (VER) and all samples. The CS3 standard is used for calibration verification. The VER solution is also used to verify chromatographic performance.

7.7 PCB Congener Mix 1 through 5 standard solutions containing all 209 isomers are Certified Reference Standards (Accustandard Product No's. M-1668A-1, M-1668A-2, M-1668A-3, M-1668A-4, M-1668A-5). Stock solutions are purchased at 250-750 µg/mL in isooctane. Once the ampoule has been sonicated and opened, the solution is transferred to an amber glass vial with Teflon®-lined cap and is used as received. These five mixes are run to determine the retention times for each of the congeners and which congeners will co-elute for each new SPB Octyl column used.

7.7.1 Initial Calibration Verification Standard. This is a single solution containing all 209 individual PCBs as well as IDA and Istd at the following concentrations:

- mono, di, and tri CBs at 50 ng/mL
- tetra, penta, hexa and hepta CBs at 100 ng/mL
- octa, nona, and deca CBs at 150 ng/mL
- IDAs and Istds are at the same concentration as the calibration standards (CS 0.5 – CS 5).

This solution is always analyzed immediately after the initial calibration.

7.7.1.1 Combine 20 uL of each mix from Section 7.7 (PCB Congener Mixes 1 through 5) with 100 uL of the 1000 ng/mL $^{13}C_{12}$ labeled IDA stock solution, 20 uL of the 5000 ng/mL $^{13}C_{12}$ labeled surrogate standard stock solution, 100 uL of the 1000 ng/mL $^{13}C_{12}$ labeled istd standard stock solution, 20 uL of the of the 5000 ng/mL $^{13}C_{12}$ labeled field spike and 660 uL of nonane to produce the

concentrations listed in section 7.7.1.

7.7.2 Retention Time Calibration Mixes: These 5 solutions are injected to establish the retention time data referenced in section 10.2.3. Combine 10 uL of the Accustandard PCB Congener Mix 1 (Section 7.7) with 50 uL of the 1000 ng/mL 13C12 IDA stock solution, 10 uL of the 5000 ng/mL 13C12 labeled surrogate standard stock solution, 50 uL of the 1000 ng/mL 13C12 labeled istd stock solution, 10 uL of the 13C12 of the 5000 ng/mL labeled air surrogate spike and 370 uL of nonane to produce the concentrations listed in section 7.7.1. Repeat the process using the Accustandard PCB Congener Mixes 2 through 5.

7.8 QC Check Sample - A QC Check Sample should be obtained from a source independent of the calibration standards. This check sample is a certified standard reference material (SRM) containing the PCBs in known concentrations in a sample matrix similar to the matrix under test. The National Institute of Standards and Technology (NIST) in Gaithersburg, Maryland has an SRM 1944 – New York/New Jersey Waterway Sediment that the NYSDEC recommends for use.

8 Sample Collection, Preservation and Storage

8.1 Sampling is not performed for this method by Eurofins TestAmerica Knoxville. For information regarding sample shipping, refer to SOP KNOX-SC-0003, “Sample Receipt and Login”, current revision.

8.2 Holding Times

8.2.1 Store sample extracts in a dark box at room temperature until analyzed. If stored in the dark at room temperature, sample extracts may be stored for up to one year.

9 Quality Control

9.1 Initial precision and recovery (IPR) or initial demonstration of capabilty (IDOC) samples are analyzed to demonstrate the ability to generate acceptable precision and accuracy.

9.1.1 For aqueous samples, extract, clean, concentrate, and analyze four 1-L aliquots of reagent water spiked with IDAs, surrogate standard, internal standard and the LCS spiking solution, according to the procedures in section 11. For solid/tissue samples, extract, clean, concentrate, and analyze four aliquots of sodium sulfate/corn oil spiked with IDAs, surrogate standard, internal standard and the LCS spiking solution, according to the procedures in section 11. All steps that are to be used for processing samples, including preparation, extraction and cleanup, shall be included in this test.

9.1.2 Using the results of the set of four analyses, compute the average percent recovery (%R) of the extracts and the relative standard deviation (%RSD) of the concentration in ng/L (aqueous) and ng/g (solid) for each compound.

9.1.3 For each PCB and labeled compound, compare the %RSD and %R with the corresponding limits for initial precision and recovery in the approprirate Table 10A or 10B. If the RSD and %R for all compounds meet the acceptance criteria, system performance is acceptable and analysis of samples may begin. If, however, any individual

%RSD exceeds the precision limit or any individual %R falls outside the range for accuracy, system performance is unacceptable for that compound. Correct the problem and repeat the test.

9.2 Isotope Dilution Analytes (IDAs)

9.2.1 Every sample, blank, and QC sample is spiked with isotope dilution analytes. IDA recoveries in samples, blanks, and QC samples must be assessed to ensure that recoveries are within established limits. When properly applied, results from isotope dilution techniques are independent of recovery. The recovery of each IDA should be within the limits in Table 10A or 10B depending on the method revision being used. If the recovery is outside these limits the following corrective action should be taken:

- Check all calculations for error.
- Ensure that instrument performance is acceptable; if applicable, a daily CCV will be analyzed to confirm instrument performance.
- Recalculate the data and/or reanalyze if either of the above checks reveal a problem.
- If the recovery of any IDA is less than the limits indicated in Tables 10A and 10B, calculate the S/N ratio of the IDA. If the S/N is > 10 and the estimated detection limits (EDLs) are less than the estimated minimum levels (EMLs), report the data as is with qualifiers in the report and a discussion in the case narrative. If the S/N is < 10 or the estimated detection limits (EDLs) are greater than the estimated minimum levels (EMLs), re-extract and reanalyze the sample. If the ion chromatogram of the PFK lock mass indicates ion suppression in the region where the IDA elutes, reanalyzing the extract at up to a 1/10 dilution may improve the IDA recovery. If the poor IDA recovery is judged to be a result of sample matrix, a reduced portion of the sample may be re-extracted or additional cleanups may be employed. The decision to reanalyze or flag the data should be made in consultation with the client.

9.2.2 Refer to the QC Program document (QA-003) for further details of the corrective actions.

9.3 Surrogate Standards: Every sample, blank, and QC sample extract is spiked with $^{13}C_{12}$ labeled surrogate standards after extraction but prior to extract cleanup. They are used to assess the efficiency of the cleanup procedures.

9.4 ISTDs: Every sample, blank, and QC sample extract is spiked with $^{13}C_{12}$ labeled istds prior to analysis. They are used to measure the recovery of the istds and the surrogate standards.

9.5 Method Blanks

9.5.1 A laboratory method blank must be run along with each analytical batch of 20 or fewer samples. The method blank consists of reagent water for aqueous samples, sodium sulfate for solid and tissue samples, processed in the same manner and at the same time as the associated samples. The method blank is used to identify any background interference or contamination of the analytical system that may lead to the reporting of elevated concentration levels or false positive data. Analyze the method blank immediately after analysis of the LCS to demonstrate freedom from contamination. If CBs will be carried

over from the LCS into the method blank, analyze one or more aliquots of solvent between the LCS and the method blank. The method blank should not contain any of the compounds of interest at a concentration above the estimated minimum level (EML) shown in Table 4.

9.5.2 Corrective action is required when compounds of interest are detected in the method blank above the EML. Corrective action may include reanalysis of the method blank. Contact the Project Manager to determine further corrective action. At a minimum, all associated results are qualified with a B flag. Re-extraction and reanalysis of all samples associated with a contaminated method blank is required if requested by the client or Project Manager. Investigation of the source of the method blank contamination will be initiated before further samples are extracted.

9.5.3 The method blank must have acceptable IDA recoveries. If IDA recoveries are not acceptable, the data must be evaluated to determine if the method blank has served the purpose of demonstrating that the analysis is free of contamination. If IDA recoveries are low and the following criteria are met, the MB is considered acceptable: S/N > 10/1 for all IDA masses, EDL<ML for all target analytes, all positive detections are below the ML.

9.6 Instrument Blank

9.6.1 Instruments must be evaluated for contamination during each 12-hour analytical run. This may be accomplished by analysis of a method blank. If a method blank is not available, an instrument blank must be analyzed.

9.6.2 An instrument blank consists of solvent with the IDAs and internal standards added. It is evaluated in the same way as the method blank.

9.7 Laboratory Control Sample – A laboratory control sample (LCS) is prepared and analyzed with every batch of 20 or fewer samples. All target analytes must be within established control limits specified in the appropriate Table 10A or 10B. The LCS is spiked with the compounds listed in Table 5a.

9.7.1 If any target in the LCS is outside the control limits, corrective action must occur. Corrective action may include re-extraction and reanalysis of the batch.

- If the batch is not re-extracted and reanalyzed, the reasons for accepting the batch must be clearly presented in the project records and the report.
- If re-extraction and reanalysis of the batch is not possible due to limited sample volume or other constraints, the LCS is reported and the failure is documented in the project narrative.

9.8 QC Check Sample - Analyze the QC Check Sample (section 7.8) periodically to assure the accuracy of calibration standards and the overall reliability of the analytical process. It is suggested that the QC Check Sample be analyzed at least annually.

10 Calibration and Standardization

10.1 Three types of calibration procedures are required. The first type establishes retention times, relative retention times and relative retention time windows to be used during the subsequent calibrations and analyses. The second type, initial calibration, is required to establish response factors and is required before any samples are analyzed. It may be required intermittently throughout sample analyses as dictated by the results of continuing calibration procedures described below. The third type, continuing calibration, consists of analyzing the continuing calibration verification solution (VER). No samples are to be analyzed until acceptable calibration as described in sections 10.2, 10.3 and 10.4 is demonstrated and documented.

10.2 Retention Time Calibration

Retention time calibration is required if the retention time criteria cannot be met.

10.2.1 The absolute retention time of CB 209 must exceed 55 minutes. Otherwise the GC temperature program must be adjusted and the test repeated until the requirement is met.

10.2.2 **NOTE**: When adjusting chromatographic conditions, the resolution requirements of sections 10.4.5.7 to 10.4.5.8 must be maintained.

10.2.3 Tune the instrument to meet the mass resolution and mass accuracy requirements of section 10.3.2. Document the resolution and accuracy.

10.2.4 Analyze 1 µL (2uL for MATs) of each of the five individual PCB mixtures (section 7.7.3). Set the switch-points for the MID descriptors. The switch-points must be set to insure that the first and last eluting isomer of each homolog group and the labeled IDAs are acquired properly. Determine the average retention time of each PCB congener using the elution order information in Table 11.

NOTE 1: PCB Mixture 5 (M-1668A-5) contains the first and last eluting isomer in each homolog group for the SPB-Octyl column (see Table 7).

NOTE 2: Laboratory data has indicated that the SPB-Octyl column can exhibit significant differences in performance from column to column. It has also been indicated that the column's performance can change significantly due to oxidation with subsequent changes in congener retention times and elution order. The individual PCB mixtures should be analyzed whenever the column's performance or specific congeners retention times are in doubt.

10.2.5 Calculate the relative retention times for all target and labeled congeners, using their retention time references from Table 2 (RT Ref). Calculate the relative retention time for each run in which the congener and its retention time reference are present Five RRTs will be calculated for each IDA. Use the generated retention times for all target and labeled congeners as the RT calibration source in the calculation software.

10.2.6 Calculate the relative retention time window using the absolute retention time windows (RT Window) from Table 2.

$$\text{RRT Limit Low} = \frac{RT_A - (RT_{WIN}/2)}{RT_{IS}}$$

$$\text{RRT Limit High} = \frac{RT_{A} + (RT_{WIN}/2)}{RT_{IS}}$$

Where:

RT_A = Average retention time of analyte.

RT_{IS} = Average retention time of RT reference.

RT_{WIN} = Absolute RT window in seconds from Table 2.

10.2.7 A single pair of RRT limits is used for all congeners in coeluting set. Use the RRT Limit Low that was calculated for the first eluting congener, and the RRT Limit High calculated for the last eluting congener (in the coelution set).

10.3 Initial Calibration

Initial calibration is required before any samples are analyzed for PCBs. Initial calibration is also required if any continuing calibration (section 10.4) does not meet the required criteria in section 10.4.5 after routine maintenance.

10.3.1 Prepare multi-level calibration standards containing the compounds and concentrations as specified in Table 6a. Calibration standards should be stored at room temperature and preferably in amber vials. Calibration standard solutions have an expiration date of ten (10) years from date of receipt based on stability of PCBs and history of vendor's recertification of standard lots.

10.3.2 Establish operating parameters for the GC/MS system (suggested operating conditions are displayed in Figure 1 and Figure 2). By using a PFK or FC43 molecular leak, tune the instrument (see the appropriate instrument manufacturer's operating manual for tuning instructions) to meet the minimum resolving power of 10,000 (10 percent valley) at mass 342.9792 (PFK) or 313.9834 (FC43). For each MID descriptor group, monitor and record the mass resolution and exact masses of three reference peaks covering the mass range of the descriptor (see below). By using peak matching techniques, verify that the deviation between the exact mass and the theoretical mass for each mass monitored is less than 5 ppm. Iteratively adjust operating parameters and tuning values until the mass resolution and mass accuracy criteria are met for each ion. Document the mass resolution and mass accuracy for each of MID group ion sets. Because of the extensive mass range covered in each MID group, it may not be possible to maintain 10,000 resolution throughout the mass range of the MID group. Therefore, resolution must be greater than 8,000 throughout the mass range and must be greater than 10,000 in the center of the mass range for each MID group. The minimum resolution of 10,000 must be met for mass 342.9792 or 313.9834.

MID Group 1 PFK ions - 192.9888, 230.98563, 280.98243
MID Group 2 PFK ions - 268.98243, 292.98243, 380.97605

MID Group 3 PFK ions - 342.97924, 380.97605, 430.97285
MID Group 4 PFK ions - 404.97604, 442.97285, 530.96646

MID Group 1 FC43 ions - 168.9883, 213.9897, 263.9866
MID Group 2 FC43 ions - 263.9866, 313.9834, 375.9802
MID Group 3 FC43 ions - 313.9834, 375.9802, 413.9770
MID Group 4 FC43 ions - 413.9770, 463.9738, 501.9706

10.3.3 Inject a 1 µL (2uL for MATs) aliquot of the CS 0.5 calibration solution.

10.3.3.1 Ion abundance ratios, minimum levels, and signal-to-noise ratios.

10.3.3.1.1 Measure the selected ion current profile areas for each congener or congener group, and compute the ion abundance ratios at the exact masses specified in Table 8. Compare the computed ratio to the theoretical ratio given in Table 9.

10.3.3.1.2 All Toxic/LOC and labeled compounds in the CS-0.5 standard must be within the QC limits in Table 9 for their respective ion abundance ratios; otherwise, the mass spectrometer must be adjusted and this test repeated until the mass ratios fall within the limits specified. If the adjustment alters the resolution of the mass spectrometer, resolution must be verified (Section 10.3.2) prior to repeat of the test.

10.3.3.1.3 The peaks representing the CBs and labeled compounds in the CS-0.5 calibration standard must have signal-to-noise ratios (S/N) $\geq$ 10; otherwise, the mass spectrometer must be adjusted and this test repeated until the signal to noise criteria are met.

10.3.3.1.4 When using PFK an exception to the ion abundance ratio and signal to noise ratio requirements is the secondary ion for dichlorinated biphenyls (mass 223.9974). High background from PFK fragments at 223.9974 results in noise levels which exceed 10% of the signal height at levels that are reliably quantifiable. FC-43 does not interfer with dichlorinated biphenyls.

10.3.4 Analyze 1 µL (2uL for MATs) of each of the other calibration standards.

10.3.4.1 Isomer specificity

10.3.4.1.1 Use the CS-3 calibration standard to evaluate column performance. The toxic isomers must be uniquely resolved from all other congeners. Isomers may be unresolved so long as they have the same toxicity equivalency factor (TEF) and response factor and so long as these unresolved isomers are uniquely resolved from all

other congeners. For example, the SPB-Octyl column achieves unique GC resolution of all Toxics except congeners with IUPAC numbers 156 and 157. This isomeric pair is uniquely resolved from all other congeners and these congeners have the same TEF and response factor.

10.3.4.1.2 Evaluate and document the percent valley between PCBs 34 and 23. The valley height must be less than 40 percent of the height of the shorter of the two peaks.

10.3.4.1.3 Evaluate and document the percent valley between PCBs 187 and 182. The valley height must be less than 40 percent of the height of the shorter of the two peaks.

10.3.4.1.4 Classify each congener as resolved or as a member of a coelution set. To be documented as resolved, the valleys between any two isomers must be less than 40 percent of the height of the shorter of the two adjacent peaks. Each member of a coelution set is designated with a qualifier in the format of CXXX, where XXX = the lowest numbered congener in the set. For example, if PCB 156 and PCB 157 coelute, qualify PCB 157 with "C156".

10.3.4.2 Calculate the RRF of each compound of interest (target analytes, coelution sets, IDAs, and surrogate standards) vs. the appropriate reference standard (as specified in Table 2) using the following equation:

$$RRF = \frac{As \times Cis}{Ais \times Cs}$$

Where:

As = sum of the areas of the quantitation ions of the compound of interest.
Ais = sum of the areas of the quantitation ions of the appropriate reference standard.
Cis = concentration of the appropriate reference standard.
Cs = concentration of the compound of interest.

NOTE: When calculating the RRF for a coelution set, sum the areas of all isomers in the set. Use the resulting RRF for all congeners within the set.

10.3.4.3 Calculate the mean relative response factor (mean RRF) and the percent relative standard deviation (%RSD) of the response factors for each compound of interest in the six calibration standard solutions using the following equations:

$$\overline{RRF}_{n=6} = \frac{1}{n} \times \sum_{i=1}^{n} RF_i$$

$$\%RSD_{n=6} = \sqrt{\frac{\sum_{i=1}^{n}\left(RF_i - \overline{RRF}\right)^2}{n-1}} \times \frac{100}{\overline{RRF}}$$

10.3.4.4 Criteria for Acceptable Calibration - The criteria listed below for acceptable calibration must be met before sample analyses are performed. If acceptable initial calibration is not achieved, identify the root cause, perform corrective action, and repeat the initial calibration. If the root cause can be traced to problems with an individual analysis within the calibration series, repeat the individual analysis and recalculate the percent relative standard deviation. If the calibration is acceptable, document the problem and proceed, otherwise repeat the initial calibration.

10.3.4.5 The percent relative standard deviation (%RSD) for the mean relative response factors for the unlabeled native analytes calculated by isotope dilution must not exceed 20 percent. The percent RSD for the mean relative response factors for the unlabeled target analytes calculated by IDA must not exceed 35 percent. The percent RSD for the mean relative response factors for the labeled IDAs must not exceed 35 percent.

10.3.5 Analyze 1 µL (2uL for MATs) of the Initial Calibration Verification (ICV) Standard in section 7.6. 1 after completion of the ICAL prior to sample analysis. Calculate the concentration of the ICV using the RRFs from the CS-3 standard analyzed in section 10.3.4. Calculate the percent difference (%D) between the expected and the calculated ICV concentration using the following formula:

$$\%D = \frac{\left(C_{Exp} - C_{Calc}\right)}{C_{Exp}} \times 100$$

Where:
C_{Exp} = The expected concentration of the ICV Standard.
C_{Calc} = The calculated concentration of the ICV Standard.

10.3.5.1 The criteria for acceptance of the ICV Standard are as follows:

10.3.5.2 The %D may not exceed ±35% for more than 4 of the native and labeled compounds.

- The %D may not exceed ±50% for any native or labeled compound.

10.3.5.3 All data associated with compounds with percent differences exceeding ±35% must be reviewed before acceptance and shall be documented as a NCM. In addition, all data associated with these compounds require corrective action that may include the following:

- Reanalyze the ICV Standard.
- Replace and reanalyze the ICV Standard.
- Evaluate the instrument performance.

- Evaluate the Initial Calibration Standards.

10.4 Continuing Calibration

10.4.1 Continuing calibration is performed at the beginning of a 12-hour period after successful mass resolution check.

10.4.2 Document the mass resolution performance as specified in section 10.3.2 at both the beginning and end of the 12-hour period.

10.4.3 Analyze 1 µL (2uL for MATs) of the Continuing Calibration Verification Standard (VER/CS3). Calculate the concentration (C) of the compounds of interest (target analytes, IDAs, and surrogate standards) vs. the appropriate quantitation reference (as specified in Table 2) using the following equation:

$$C = \frac{As \times Cis}{Ais \times RRF}$$

Where:

As = sum of the areas of the quantitation ions of the compound of interest.
Ais = sum of the areas of the quantitation ions of the appropriate reference standard.
Cis = concentration of the appropriate reference standard.
RRF = mean relative response factor from section 10.3.4.2.

10.4.4 Calculate the concentrations as percentages of the test concentrations and compare them to the limits specified in the appropriate Table 10A or 10B using the following equation:

$$C_{ver}\% = \frac{C_{ver}}{C_{test}} \times 100$$

Where:

C_{ver} = the concentration of the VER standard calculated in section 10.4.3
C_{test} = the test concentration of the VER standard listed in Table 6a.

10.4.5 Criteria for Acceptable Calibration - The criteria listed below for acceptable calibration must be met before sample analyses are performed. If the acceptance criteria are met, the calibration is deemed to be in control and the RRFs generated from the initial calibration are used to quantify samples. If acceptable calibration is not achieved, identify the root cause, perform corrective action, and repeat the continuing calibration. If a second consecutive attempt at a continuing calibration fails, two consecutive calibrations must meet the criteria, or an initial calibration must be run before proceeding with client samples.

10.4.5.1 The ion abundance ratios of the peaks representing the Toxics/LOCs and labeled standards must be within the control limits specified in Table 9.

10.4.5.2 The S/N for the GC signals present in every selected ion current profile (including those for labeled standards) must be ≥ 10.

10.4.5.3 For Toxic and LOC congeners, as listed in the first section of Table 10A or 10B, the percent of the calculated concentration relative to the test concentration must be within 70-130% for 1668A and 1668B and 75-125% for 1668C.

10.4.5.4 For non-Toxic congeners the calculated concentrations must be within 70-130% of the test concentrations for 1668A, 1668B and 1668C.

10.4.5.5 The absolute retention times (RT) of the labeled IDAs must be within ±15 seconds of the retention times obtained during initial calibration.

10.4.5.6 The relative retention times (RRT) of the Toxics/LOC congeners must be within their respective RRT limits generated in the retention time calibration in section 10.2.

10.4.5.6.1 If the RRTs or RTs are not within the limits above, the GC may not be performing properly. However, routine column maintenance may include removing short amounts of the beginning of the column when active sites or non-volatile compounds in sample extracts cause poor chromatography and loss of specificity. Shortening of the column can cause the RRTs or RTs to fall outside the above limits.

10.4.5.6.2 When the RRT of any compound or the RT of any IDA is not within the above limits, corrective action must be taken. If the GC is not performing properly, correct the problem and repeat the test. If the GC is performing properly but the RRTs or RTs have changed due to routine column maintenance, adjust the GC or replace the GC column, then repeat the test or repeat the retention time calibration.

10.4.5.7 Evaluate and document the percent valley between PCBs 34 and 23. The valley height must be less than 40 percent of the height of the shorter of the two peaks.

10.4.5.8 Evaluate and document the percent valley between PCBs 187 and 182. The valley height must be less than 40 percent of the height of the shorter of the two peaks.

10.4.6 Daily calibration must be performed every 12 hours of instrument operation. The 12-hour shift begins with the documentation of the mass resolution followed by the injection of the Continuing Calibration Standard (VER).

11 Procedure

11.1 One time procedural variations are allowed only if deemed necessary in the professional judgement of supervision to accommodate variations in sample matrix, radioactivity, chemistry, sample size, or other parameters. Any variations in the procedure, except those specified by project specific instructions, shall be completely documented using a Nonconformance Memo

and approved by a Technical Specialist, Project Manager, and QA Manager. If contractually required, the client shall be notified.

Any unauthorized deviations from this procedure must also be documented as a nonconformance, with a cause and corrective action described.

11.2 Sample Extraction and Cleanup

The extraction and cleanup procedures are described in SOP KNOX-OP-0021, "Extraction of Polychlorinated Biphenyl (PCB) Isomers for Analysis by Isotope Dilution HRGC/HRMS", current revision.

11.3 Sample Analysis

11.3.1 Analyze the sample extracts under the same instrument operating conditions used to perform the instrument calibrations. Inject 2 µL into the GC/MS and acquire data beginning at 11 minutes and ending after decachlorobiphenyl has eluted from the column.

11.3.2 The RTs of the unambiguous labeled congeners (RT Markers) are used to calculate a least squares best fit regression for retention times compared to those of the retention time calibration.

11.3.3 The resulting regression is used to calculate predicted retention times for target analytes. These predicted retention times are used by the software to identify candidate peaks for targets.

11.3.4 The analyst reviews the peaks identified as targets and determines whether to accept the identification. This determination is made by evaluating the retention time values against the shift from the predicted retention times

11.3.5 A RRT window is calculated by multiplying the RRT Limit High and the RRT Limit Low by the retention time of the designated RT reference.

11.4 HRGC/HRMS Troubleshooting Guide

11.4.1 Perform the instrument's leak check: Evaluate the air spectrum. Mass 28 should be less than 5 times mass 69. If the air spectrum is not acceptable, replace transfer line ferrule and/or pump out PFK reservoir.

11.4.2 Check the voltage on the 5V power supply: If voltage is not reading 5V, adjust voltage to read 5V.

11.4.3 Check daily calibration standard: Evaluate the signal to noise (S/N), examine peak shape/chromatography, and evaluate response factors. Refer to specific SOP's requirements and acceptance criteria for all natives and internal standards. If daily calibration is not acceptable , perform the following troubleshooting options as needed and reanalyze a daily CS-3 calibration standard:

- Replace the inlet seal and clean the injection port body with methanol to improve response factor.

- Perform column/injector port maintenance and/or retune the instrument to resolve chromatography/resolution, signal to noise issues.
- Perform a gain test on the electron multiplier.
- Adjust the tuning parameters of the instrumentation to achieve optimum sensitivity and peak shape.
- Evaluate carryover from sample analyses. If carryover or contamination is suspected, run solvent rinses (nonane) under MID to evaluate the contamination.
- When troubleshooting cannot resolve an instrument problem, a manufacture's service engineer may be consulted for possible solution, or called onsite for diagnosis/repair.

11.5 Refer to the Eurofins TestAmerica Knoxville Quality Assurance Manual, KX-QAM, current revision for the HRGC/HRMS instrument equipment maintenance table.

11.6 Refer to Eurofins TestAmerica SOP CA-I-S-0006, current revision for requirements for computer hardware and software.

12 Data Analysis and Calculations

12.1 Qualitative Identification Criteria for PCBs

For a gas chromatographic peak to be identified as a PCB, it must meet all of the following criteria:

12.1.1 The signals for the two exact masses in Table 8 must be present and must maximize within ±2 seconds.

12.1.2 The signal to noise ratio (S/N) for each GC peak at each exact mass must be greater than or equal to (≥) 2.5. (This requirement does not apply to the secondary ion for dichlorinated biphenyls [mass 223.9974]). High background from PFK fragments at 223.9974 results in noise levels which exceed 10% of the signal height at levels that are reliably quantifiable.

12.1.3 The ratio of the integrated areas of the two exact masses specified in Table 8 must be within the limits in Table 9 . Alternately, the ratios may be within ±15% of the ratio in the midpoint (CS-3) calibration or calibration verification (VER), whichever is most recent.

12.1.4 The relative retention time of the peak for a CB must be within the RRT QC limits calculated in section 10.2.5.

NOTE: For target CBs determined by IDA quantitation, a given CB congener may fall within more than one RT window and be misidentified unless the RRT windows are made very narrow, as in Table 2. Therefore, consistency of the RT and RRT with other congeners and the labeled compounds may be required for rigorous congener identification. Retention time regression analysis may be employed for this purpose.

12.1.5 If identification is ambiguous, (i.e., some, but not all of the identification criteria are met for a congener) an experienced analyst must determine the presence or absence of the congener.

12.2 Quantitation for PCBs

12.2.1 Calculate the IDA Recoveries (Ris) relative to the istd according to the following equation:

$$Ris = \frac{Ais \times Qrs}{Ars \times RRFis \times Qis} \times 100\%$$

Where:

Ais = sum of the areas of the quantitation ions of the appropriate IDA
Ars = sum of the areas of the quantitation ions of the istd
Qrs = ng of istd added to extract
Qis = ng of IDA added to sample
RRFis = mean relative response factor of IDA obtained during initial calibration

NOTE: In some situations, such as source testing, the extract is split for multiple analyses. In this case, Qrs must be correctly calculated to account for the splitting of extracts before the istd was added.

$$Qrs = Qrss \times Split$$

Where:

Qrs = ng of istd added to extract
Qrss= ng of istd added to the split portion of the extract
Split= split ratio of the extract

12.2.2 Calculate the concentration of individual PCBs according to the following equation:

$$Concentration = \frac{As \times Qis}{Ais \times RRF \times W \times S}$$

Where:

As = sum of the areas of the quantitation ions of the compound of interest
Ais = sum of the areas of the quantitation ions of the appropriate IDA
Qis = ng of IDA added to sample
RRF = mean relative response factor of compound obtained during initial calibration
W = amount of sample extracted (grams or liters)
S = decimal expression of solids (optional, if results are requested to be reported on dry weight basis)

12.2.3 If reporting results for Total Homolog Groups, calculate the total concentration of all isomers within each homolog group by summing the concentrations of the individual PCB isomers within that homolog group.

12.2.4 If no peaks are present in the region of the ion chromatogram where the compounds of

interest are expected to elute, calculate the estimated detection limit (EDL) for that compound according to the following equation:

$$EDL = \frac{N \times 2.5 \times Qis}{His \times RRF \times W}$$

Where:

N = sum of peak to peak noise of quantitation ion signals in the region of the ion chromatogram where the compound of interest is expected to elute
His = sum of peak heights of quantitation ions for appropriate IDA
Qis = ng of IDA added to sample
RRF = mean relative response factor of compound obtained during initial calibration
W = amount of sample extracted (grams or liters)

12.2.5 If peaks are present in the region of the ion chromatogram which do not meet the qualitative criteria listed in section 12.1, calculate an Estimated Maximum Possible Concentration (EMPC). Use the equation in section 12.2.2, except that "As" should represent the sum of the area under each mass peak calculated using the theoretical chlorine isotope ratio. The peak selected to calculate the theoretical area should be the one which gives the lower of the two possible results (i.e., the EMPC will always be lower than the result calculated from the uncorrected areas).

12.2.6 If the concentration in the final extract of any PCB isomer exceeds the upper method calibration limits, a dilution of the extract must be performed. If compound concentrations exceeding the calibration range cannot be brought within the calibration range by a dilution, a post dilution spike can be performed. The lab may report the measured concentration and indicate that the value exceeds the calibration limit by flagging the results with "E". Consultation with the client should occur before compounds are reported which exceed the calibration range.

12.3 The estimated minimum level (EML) is defined as the lowest concentration at which an analyte can be measured reliably with common laboratory interferences present assuming a sample is extracted at the recommended weight or volume and is carried through all normal extraction and analysis procedures. The EMLs for different matrices and extract volumes are listed in Table 4. Deviations from the extraction amounts or final volumes listed will result in corresponding changes in the actual sample EMLs.

12.4 Flag all compound results in the sample which are below the estimated minimum level with a "J" qualifier.

12.5 Flag all compound results in the sample which were detected in the method blank with a "B" qualifier.

12.6 Flag all compound results in the sample which are above the upper calibration limit with an "E" qualifier.

12.7 Flag all compound results in the sample which are "Estimated Maximum Possible Concentrations" with a "Q" qualifier or appropriate program qualifier.

12.8 Flag compound results in the sample that may contain co-eluting compounds with a "C" qualifier.

12.8.1 Flag congeners known to coelute with a higher numbered congener with a "C" qualifier.

12.8.2 Flag congeners that coelute with a lower numbered congener with a "Cx" qualifier where x is the CAS PCB number of the lowest numbered congener in the coeluting group.

12.9 If DOD QSM requirements are used, refer to SOP KNOX-QA-0021.

12.10 Flag compound results in the sample that may be affected by ion suppression with a "S" qualifier. When ion suppression of a PFK trace occurs at greater than or equal to 20% of full scale on both the lock mass and QC mass traces, and when the suppression is sustained for greater than 4 seconds, the suppression must be evaluated to determine which, if any, PCB congeners co-elute with the suppression. Samples may be diluted to decrease the effects of the ion suppression and rerun. Congeners that are determined to co-elute with the suppression are flagged with an "S" qualifier.

12.11 Data Review

12.11.1 Refer to Figure 3 for an example data review checklists used to perform and document the review of the data. Using the data review checklist, the analyst also creates a narrative which includes any qualifications of the sample data.

12.11.2 The analyst who performs the initial data calculations must first level review the raw data to document that they have performed the qualitative and quantitative analysis on the sample data.

12.11.3 A second analyst must verify all qualitative peak identifications and second level review the raw data. If discrepancies are found, the data must be reviewed by the first analyst before a change is made.

12.11.4 All items listed on the data review checklist must be checked by both the analyst who performed the initial qualitative and quantitative analysis and the analyst who performed the second level review. An example data review checklist is shown in Figure 3.

13 Method Performance

13.1 Method Detection Limit (MDL) - An MDL must be determined for each analyte in each routine matrix prior to the analysis of any samples. The procedure for determination of the method detection limit is given in the SOP CA-Q-S006, current revision, based on 40 CFR Part 136 Appendix B. The result of the MDL determination must support the reporting limit (ML). Current method detection limits are available in the TestAmerica Laboratory Information Management System (TALS).

13.2 Initial Demonstration of Capability – Each analyst must perform an initial demonstration of capability (IDOC) for each target analyte prior to performing the analysis independently. The IDOC is determined by analyzing four replicate spikes (e.g., LCSs) as detailed in Eurofins TestAmerica Knoxville SOP KNOX-QA-0009.

13.3 Training Qualification: The group/team leader has the responsibility to ensure that this procedure is performed by an associate who has been properly trained in its use and has the required experience. Refer to SOP KNOX-QA-0009 current revision for further requirements for performing and documenting initial and on-going demonstrations of capability**.**

14 Pollution Prevention

14.1 All attempts will be made to minimize the use of solvents and standard materials.

15 Waste Management

15.1 All waste will be disposed of in accordance with Federal, State and Local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this method and the policies in section 13 of the Corporate Safety Manual for "Waste Management and Pollution Prevention."

15.2 Waste Streams Produced by the Procedure: The following waste streams are produced when this method is carried out.

15.2.1 Miscellaneous disposable glassware, chemical resistant gloves, bench paper and similar materials shall be placed in the incinerable laboratory waste stream, contained in a steel or poly satellite accumulation container.

16 References

16.1 Knoxville Laboratory Quality Assurance Manual (QAM), current revision.

16.2 Method 1668, Revision A, B and C: Chlorinated Biphenyl Congeners in Water, Soil, Sediment and Tissue by HRGC/HRMS, EPA-821-R-00-002, December 1999.

16.3 Method 1613: Tetra- through Octa-Chlorinated Dioxins and Furans by Isotope Dilution HRGC/HRMS [Revision B], EPA#: 821/B-94-005a YEAR: 1994

16.4 Ballschmiter, K. and M. Zell, "Analysis of Polychlorinated Biphenyls (PCB) by Glass Capillary Gas Chromatography", *Fresenius Z. Anal. Chem*., 302:20-31 (1980).

16.5 Schulte, E. and R. Malisch, "Berechnung der Wahren PCB-Gehalte in Umweltproben I. Ermittlung der Zusammensetzung Zweier Technischer PCB-Gemische," *Fresenius Z. Anal. Chem*., 314:545-551 (1983).

16.6 Guitart, R., P. Puig and J. Gómez-Catalán, "Requirement for a Standardized Nomenclature Criterion for PCBs: Computer-Assisted Assignment of Correct Congener Denomination and Numbering," *Chemosphere*, 27(8):1451-1459 (1993).

16.7 Rigaudy, J. and Klesney, S.P., Nomenclature of Organic Chemistry, Pergamon, 1979.

16.8 Pretsch, Clerc, Seibl, Simon, Tables of Spectral Data for Structure Determination of Organic Compounds, Second Edition, Springer-Verlag, 1989.

16.9 CRC Handbook of Chemistry and Physics, 71st edition, CRC Press, 1990-1991.

16.10 Email dated 03/02/10 from Richard Reding Ph.D., Chief U.S. EPA, Engineering & Analytical Support Branch. Engineering & Analysis Division Office of Science and Technology, Office of Water – Subject: Use of QC Acceptance Limits."

17 Miscellaneous

17.1 Deviations from EPA Method 1668, Revisions A, B and C.

17.1.1 Additional internal standards are used in this procedure. The additional standards are listed in Table 1.

17.1.2 Additional labeled standards are used in this procedure as field sampling surrogates. The additional standards are listed in Table 1

17.1.3 This procedure uses IDAs which are within the same MID group as the target congener that is being calculated. This improvement is used to reduce the effects of full verses reduced accelerating voltage tuning differences which can be exaggerated when crossing MID groups.

17.1.4 The calibration procedure in the method 1668A, revision B, and C call for a single point standard for the non-Toxic/LOC congeners. This procedure uses a multi-point calibration for all 209 congeners.

17.1.5 This procedure uses MID groups that differ from the method. The procedure uses 4 groups, rather than 6, to improve instrument stability, by holding the magnet current steady for longer periods. Therefore alternate PFK lock masses are monitored, to reflect the mass ranges of the procedures MID groups. The SOP allows the use of FC-43 as the instrument reference compound based on the manufacturer's recommendation. Section 10.2.1 of Methods 1668A, 1668B and 1668C allows the use of other reference material than PFK.

17.1.6 This procedure uses average retention times (and average relative retention times) produced by analysis of the 5 mixes specified, rather than single analyses of the diluted 209 standard.

17.1.7 The absolute retention times, relative retention times, and relative retention time limits used by the laboratory differ from those listed in all versions of Method 1668. Each SPB-Octyl column lot used by the laboratory has exhibited slightly different retention time characteristics resulting in different absolute retention times than those listed in the method or those observed with another SPB-Octyl column lot. To ensure that the correct peak assignments are made, a retention time study is performed for each new column lot to verify the original study. This study includes the analysis of the five retention time mixes listed in Method 1668A as described in Section 10.2. If the retention time study is found to be different than the original elution pattern, a new ICAL will be performed. This procedure requires a minimum elution time of 55 minutes for PCB 209.

17.1.8 The calibration verification procedures in the method call for updating the retention times, relative retention times and response factors for non-Toxic compounds during daily calibration and use the retention times, relative retention times and response factors

from the initial calibration for Toxic and LOC compounds. This laboratory uses the retention times and relative retention times from analysis of the 5-mix series, which contains all congeners, and uses response factors from the initial calibration for all 209 compounds. The practice of updating the relative retention times of only a subset of compounds causes significant error in the linear regression prediction formulas used by targeting software to identify the compounds. This procedure has provisions for updating all RTs and RRTs by analyzing a new retention time calibration series.

17.1.9 The EMLs listed in Table 4 differ from those listed in the reference method. The EMLs are set above the mean plus 2 standard deviations for the higher of detections or EDLs for method blanks. In no case is the EML lower than the low calibration limit. The survey period was approximately 14 months.

17.1.10 This procedure uses a 100uL final volume for solid samples, or a variation depending on the protocol assigned to the sample based on screen results prior to extraction.

17.1.11 Method 1668C requires istd standards be added immediately prior to analysis. In this procedure istd standards are added to sample extracts at the conclusion of the extraction / clean up process prior to analysis.

17.1.12 Method 1668C implies that all qualitative criteria must be met in order for a positive hit to be reported. This procedure allows positive hits to be reported with "Q" qualifiers or appropriate program qualifier.

17.1.13 This procedure uses calculated concentrations for determining if a dilution is required not area counts as suggested by Method 1668C, section 17.5.

17.1.14 This procedure reports positive hits below the estimated minimum level (EML) which are determined to be greater than 2.5 signal to noise. These hits are qualified with a "J" or appropriate program qualifier.

17.1.15 This procedure does not report blank corrected values.

17.1.16 When samples are diluted to bring a native analyte within the upper calibration range all 209 congeners are reported from this analysis. This procedure reports all congeners from one analytical analysis.

17.1.17 This procedure uses a daily calibration CCV consisting of all 209 congeners at the beginning of each 12 hour analytical shift.

17.1.18 Extracts are stored in a dark box at room temperature in the same manner as standards.

17.1.19 1668C EML limits are listed in Table 4. However, this laboratory currently uses the 1668A/B limits

17.1.20 The limits established in 1668B, Table 6 for the IPR and OPR performance evaluations have been observed by EPA reviewers to penalize an analyst who achieves better performance than those observed in the inter-laboratory study. Based on EPA's recommendations (section 16.10), Eurofins TestAmerica Knoxville performs analysis by Method 1668B with the QC acceptance criteria in Table 6 of EPA Method 1668A.

Table 10 "Acceptance Criteria for Performance Tests" of this SOP will be used for the IPR and OPR limits.

17.2 List of tables and figures referenced in the body of the SOP.

17.2.1 Table 1 – Polychlorinated Biphenyls Determined by High Resolution Gas Chromatography (HRGC)/High Resolution Mass Spectrometry (HRMS)

17.2.2 Table 2 – RT References, Quantitation References, Retention Times (RT), and Relative Retention Times (RRTs) for the 209 CB congeners on SPB-Octyl

17.2.3 Table 3 – Low Calibration Levels Based on Various Final Extract Volumes

17.2.4 Table 4 – Estimated Minimum Levels EPA 1668A, B & C– Matrix and Concentration

17.2.5 Table 5a – Concentration of Native PCB Congener Stock and Spiking Solutions

17.2.6 Table 5b – Concentration of $^{13}C_{12}$ Labeled PCB Congener Stock and Spiking Solutions

17.2.7 Table 6a – Concentration of PCBs in Calibration Solutions

17.2.8 Table 6b – Preparation of Calibration Solutions

17.2.9 Table 7 – Window Defining Mixture and SPB-Octyl Resolution Test Compounds

17.2.10 Table 8 – Ions Monitored for HRGC/HRMS Analysis of PCBs

17.2.11 Table 9 – Theoretical Ion Abundance Ratios and Control Limits for PCBs

17.2.12 Table 10A – Acceptance Criteria for Performance Tests (1668A & B)

17.2.13 Table 10B – Acceptance Criteria for Performance Tests (1668C)

17.2.14 Table 11 – Retention Times of Isomers on SPB-Octyl Column for PCB Standard Mixes

17.2.15 Table 12 – Assignment of Sample Preparation Protocols

17.2.16 Figure 1 – Recommended GC Operating Conditions

17.2.17 Figure 2 – Recommended MID Descriptors

17.2.18 Figure 3 – Example Data Review Checklist

17.2.19 Appendix I SPB-Octyl Guard Column Installation and Conditioning

Table 1 - Polychlorinated Biphenyls Determined by HRGC/HRMS

PCB Number[1]	BZ/IUPAC Number[2].	PCB Chemical Structure Name[3]	CAS Registry[4] Number	Labeled Analog	CAS Registry[4] Number	Usage
1	1	2-monochlorobiphenyl	2051-60-7	$^{13}C_{12}$-2-monochlorobiphenyl	234432-85-0	IDA
2	2	3-monochlorobiphenyl	2051-61-8			
3	3	4-monochlorobiphenyl	2051-62-9	$^{13}C_{12}$-4-monochlorobiphenyl	208263-77-8	IDA
4	4	2,2'-dichlorobiphenyl	13029-08-8	$^{13}C_{12}$-2,2'-dichlorobiphenyl	234432-86-1	IDA
5	5	2,3-dichlorobiphenyl	16605-91-7			
6	6	2,3'-dichlorobiphenyl	25569-80-6			
7	7	2,4-dichlorobiphenyl	33284-50-3			
8	8	2,4'-dichlorobiphenyl	34883-43-7	$^{13}C_{12}$-2,4'-dichlorobiphenyl		Air Surrogate
9	9	2,5-dichlorobiphenyl	34883-39-1	$^{13}C_{12}$-2,5-dichlorobiphenyl	250694-89-4	Istd
10	10	2,6-dichlorobiphenyl	33146-45-1			
11	11	3,3'-dichlorobiphenyl	2050-67-1			
12	12	3,4-dichlorobiphenyl	2974-92-7			
13	13	3,4'-dichlorobiphenyl	2974-90-5			
14	14	3,5-dichlorobiphenyl	34883-41-5			
15	15	4,4'-dichlorobiphenyl	2050-68-2	$^{13}C_{12}$-4,4'-dichlorobiphenyl	208263-67-6	IDA
16	16	2,2',3-trichlorobiphenyl	38444-78-9			
17	17	2,2',4-trichlorobiphenyl	37680-66-3			
18	18	2,2',5-trichlorobiphenyl	37680-65-2			
19	19	2,2',6-trichlorobiphenyl	38444-73-4	$^{13}C_{12}$-2,2',6-trichlorobiphenyl	234432-87-2	IDA
20	20	2,3,3'-trichlorobiphenyl	38444-84-7			
21	21	2,3,4-trichlorobiphenyl	55702-46-0			
22	22	2,3,4'-trichlorobiphenyl	38444-85-8			
23	23	2,3,5-trichlorobiphenyl	55720-44-0			
24	24	2,3,6-trichlorobiphenyl	55702-45-9			
25	25	2,3',4-trichlorobiphenyl	55712-37-3			
26	26	2,3',5-trichlorobiphenyl	38444-81-4			
27	27	2,3',6-trichlorobiphenyl	38444-76-7			
28	28	2,4,4'-trichlorobiphenyl	7012-37-5	$^{13}C_{12}$-2,4,4'-trichlorobiphenyl	208263-76-7	Surrogate Std
29	29	2,4,5-trichlorobiphenyl	15862-07-4			
30	30	2,4,6-trichlorobiphenyl	35693-92-6			
31	31	2,4',5-trichlorobiphenyl	16606-02-3	$^{13}C_{12}$-2,4',5-trichlorobiphenyl		Istd
32	32	2,4',6-trichlorobiphenyl	38444-77-8	$^{13}C_{12}$-2,4',6-trichlorobiphenyl		Istd
33	33	2',3,4-trichlorobiphenyl (2,3',4'-trichlorobiphenyl)	38444-86-9			
34	34	2',3,5-trichlorobiphenyl (2,3',5'-trichlorobiphenyl)	37680-68-5			
35	35	3,3',4-trichlorobiphenyl	37680-69-6			
36	36	3,3',5-trichlorobiphenyl	38444-87-0			
37	37	3,4,4'-trichlorobiphenyl	38444-90-5	$^{13}C_{12}$-3,4,4'-trichlorobiphenyl	208263-79-0	IDA
38	38	3,4,5-trichlorobiphenyl	53555-66-1			
39	39	3,4',5-trichlorobiphenyl	38444-88-1			
40	40	2,2',3,3'-tetrachlorobiphenyl	38444-93-8			
41	41	2,2',3,4-tetrachlorobiphenyl	52663-59-9			
42	42	2,2',3,4'-tetrachlorobiphenyl	36559-22-5			
43	43	2,2',3,5-tetrachlorobiphenyl	70362-46-8			
44	44	2,2',3,5'-tetrachlorobiphenyl	41464-39-5			
45	45	2,2',3,6-tetrachlorobiphenyl	70362-45-7			
46	46	2,2',3,6'-tetrachlorobiphenyl	41464-47-5			
47	47	2,2',4,4'-tetrachlorobiphenyl	2437-79-8			
48	48	2,2',4,5-tetrachlorobiphenyl	70362-47-9			
49	49	2,2',4,5'-tetrachlorobiphenyl	41464-40-8			
50	50	2,2',4,6-tetrachlorobiphenyl	62796-65-0			
51	51	2,2',4,6'-tetrachlorobiphenyl	68194-04-7			
52	52	2,2',5,5'-tetrachlorobiphenyl	35693-99-3	$^{13}C_{12}$-2,2',5,5'-tetrachlorobiphenyl	160901-66-6	Istd
53	53	2,2',5,6'-tetrachlorobiphenyl	41464-41-9			
54	54	2,2',6,6'-tetrachlorobiphenyl	15968-05-5	$^{13}C_{12}$-2,2',6,6'-tetrachlorobiphenyl	234432-88-3	IDA
55	55	2,3,3',4-tetrachlorobiphenyl	74338-24-2			
56	56	2,3,3',4'-tetrachlorobiphenyl	41464-43-1			
57	57	2,3,3',5-tetrachlorobiphenyl	70424-67-8			
58	58	2,3,3',5'-tetrachlorobiphenyl	41464-49-7			

PCB Number[1]	BZ/IUPAC Number[2]	PCB Chemical Structure Name[3]	CAS Registry[4] Number	Labeled Analog	CAS Registry[4] Number	Usage
59	59	2,3,3',6-tetrachlorobiphenyl	74472-33-6			
60	60	2,3,4,4'-tetrachlorobiphenyl	33025-41-1			
61	61	2,3,4,5-tetrachlorobiphenyl	33284-53-6			
62	62	2,3,4,6-tetrachlorobiphenyl	54230-22-7			
63	63	2,3,4',5-tetrachlorobiphenyl	74472-34-7			
64	64	2,3,4',6-tetrachlorobiphenyl	52663-58-8			
65	65	2,3,5,6-tetrachlorobiphenyl	33284-54-7			
66	66	2,3',4,4'-tetrachlorobiphenyl	32598-10-0			
67	67	2,3',4,5-tetrachlorobiphenyl	73575-53-8			
68	68	2,3',4,5'-tetrachlorobiphenyl	73575-52-7			
69	69	2,3',4,6-tetrachlorobiphenyl	60233-24-1			
70	70	2,3',4',5-tetrachlorobiphenyl	32598-11-1			
71	71	2,3',4',6-tetrachlorobiphenyl	41464-46-4			
72	72	2,3',5,5'-tetrachlorobiphenyl	41464-42-0			
73	73	2,3',5',6-tetrachlorobiphenyl	74338-23-1			
74	74	2,4,4',5-tetrachlorobiphenyl	32690-93-0			
75	75	2,4,4',6-tetrachlorobiphenyl	32598-12-2			
76	76	2',3,4,5-tetrachlorobiphenyl (2,3',4',5'-tetrachlorobiphenyl)	70362-48-0			
77	77	3,3',4,4'-tetrachlorobiphenyl	32598-13-3	$^{13}C_{12}$-3,3',4,4'-tetrachlorobiphenyl	160901-67-7	IDA
78	78	3,3',4,5-tetrachlorobiphenyl	70362-49-1			
79	79	3,3',4,5'-tetrachlorobiphenyl	41464-48-6	$^{13}C_{12}$-3,3',4,5'-tetrachlorobiphenyl		Air Surrogate
80	80	3,3',5,5'-tetrachlorobiphenyl	33284-52-5			
81	81	3,4,4',5-tetrachlorobiphenyl	70362-50-4	$^{13}C_{12}$-3,4,4',5-tetrachlorobiphenyl	160901-68-8	IDA
82	82	2,2',3,3',4-pentachlorobiphenyl	52663-62-4			
83	83	2,2',3,3',5-pentachlorobiphenyl	60145-20-2			
84	84	2,2',3,3',6-pentachlorobiphenyl	52663-60-2			
85	85	2,2',3,4,4'-pentachlorobiphenyl	65510-45-4			
86	86	2,2',3,4,5-pentachlorobiphenyl	55312-69-1			
87	87	2,2',3,4,5'-pentachlorobiphenyl	38380-02-8			
88	88	2,2',3,4,6-pentachlorobiphenyl	55215-17-3			
89	89	2,2',3,4,6'-pentachlorobiphenyl	73575-57-2			
90	90	2,2',3,4',5-pentachlorobiphenyl	68194-07-0			
91	91	2,2',3,4',6-pentachlorobiphenyl	68194-05-8			
92	92	2,2',3,5,5'-pentachlorobiphenyl	52663-61-3			
93	93	2,2',3,5,6-pentachlorobiphenyl	73575-56-1			
94	94	2,2',3,5,6'-pentachlorobiphenyl	73575-55-0			
95	95	2,2',3,5',6-pentachlorobiphenyl	38379-99-6	$^{13}C_{12}$-2,2',3,5',6-pentachlorobiphenyl		Air Surrogate
96	96	2,2',3,6,6'-pentachlorobiphenyl	73575-54-9			
97	97	2,2',3',4,5-pentachlorobiphenyl (2,2',3,4',5'-pentachlorobiphenyl)	41464-51-1			
98	98	2,2',3',4,6-pentachlorobiphenyl (2,2',3,4',6'-pentachlorobiphenyl)	60233-25-2			
99	99	2,2',4,4',5-pentachlorobiphenyl	38380-01-7			
100	100	2,2',4,4',6-pentachlorobiphenyl	39485-83-1			
101	101	2,2',4,5,5'-pentachlorobiphenyl	37680-73-2	$^{13}C_{12}$-2,2',4,5,5'-pentachlorobiphenyl	160901-69-9	istd
102	102	2,2',4,5,6'-pentachlorobiphenyl	68194-06-9			
103	103	2,2',4,5',6-pentachlorobiphenyl	60145-21-3			
104	104	2,2',4,6,6'-pentachlorobiphenyl	56558-16-8	$^{13}C_{12}$-2,2',4,6,6'-pentachlorobiphenyl	234432-89-4	IDA
105	105	2,3,3',4,4'-pentachlorobiphenyl	32598-14-4	$^{13}C_{12}$-2,3,3',4,4'-pentachlorobiphenyl	160901-70-2	IDA
106	106	2,3,3',4,5-pentachlorobiphenyl	70424-69-0			
107	107/109	2,3,3',4',5-pentachlorobiphenyl	70424-68-9			
108	108/107	2,3,3',4,5'-pentachlorobiphenyl	70362-41-3			
109	109/108	2,3,3',4,6-pentachlorobiphenyl	74472-35-8			
110	110	2,3,3',4',6-pentachlorobiphenyl	38380-03-9			
111	111	2,3,3',5,5'-pentachlorobiphenyl	39635-32-0	$^{13}C_{12}$-2,3,3',5,5'-pentachlorobiphenyl	160901-71-3	Surrogate Std
112	112	2,3,3',5,6-pentachlorobiphenyl	74472-36-9			
113	113	2,3,3',5',6-pentachlorobiphenyl	68194-10-5			
114	114	2,3,4,4',5-pentachlorobiphenyl	74472-37-0	$^{13}C_{12}$-2,3,4,4',5-pentachlorobiphenyl	160901-72-4	IDA
115	115	2,3,4,4',6-pentachlorobiphenyl	74472-38-1			
116	116	2,3,4,5,6-pentachlorobiphenyl	18259-05-7			
117	117	2,3,4',5,6-pentachlorobiphenyl	68194-11-6			

PCB Number[1]	BZ/IUPAC Number[2]	PCB Chemical Structure Name[3]	CAS Registry[4] Number	Labeled Analog	CAS Registry[4] Number	Usage
118	118	2,3',4,4',5-pentachlorobiphenyl	31508-00-6	$^{13}C_{12}$-2,3',4,4',5-pentachlorobiphenyl	160901-73-5	IDA
119	119	2,3',4,4',6-pentachlorobiphenyl	56558-17-9			
120	120	2,3',4,5,5'-pentachlorobiphenyl	68194-12-7			
121	121	2,3',4,5',6-pentachlorobiphenyl	56558-18-0			
122	122	2',3,3',4,5-pentachlorobiphenyl (2,3,3',4',5'-pentachlorobiphenyl)	76842-07-4			
123	123	2',3,4,4',5-pentachlorobiphenyl (2,3',4,4',5'-pentachlorobiphenyl)	65510-44-3	$^{13}C_{12}$-2',3,4,4',5-pentachlorobiphenyl	160901-74-6	IDA
124	124	2',3,4,5,5'-pentachlorobiphenyl (2,3',4',5',5-pentachlorobiphenyl)	70424-70-3			
125	125	2',3,4,5,6'-pentachlorobiphenyl (2,3',4',5',6-pentachlorobiphenyl)	74472-39-2			
126	126	3,3',4,4',5-pentachlorobiphenyl	57465-28-8	$^{13}C_{12}$-3,3',4,4',5-pentachlorobiphenyl	160901-75-7	IDA
127	127	3,3',4,5,5'-pentachlorobiphenyl	39635-33-1	$^{13}C_{12}$-3,3',4,5,5'-pentachlorobiphenyl		Istd
128	128	2,2',3,3',4,4'-hexachlorobiphenyl	38380-07-3			
129	129	2,2',3,3',4,5-hexachlorobiphenyl	55215-18-4			
130	130	2,2',3,3',4,5'-hexachlorobiphenyl	52663-66-8			
131	131	2,2',3,3',4,6-hexachlorobiphenyl	61798-70-7			
132	132	2,2',3,3',4,6'-hexachlorobiphenyl	38380-05-1			
133	133	2,2',3,3',5,5'-hexachlorobiphenyl	35694-04-3			
134	134	2,2',3,3',5,6-hexachlorobiphenyl	52704-70-8			
135	135	2,2',3,3',5,6'-hexachlorobiphenyl	52744-13-5			
136	136	2,2',3,3',6,6'-hexachlorobiphenyl	38411-22-2			
137	137	2,2',3,4,4',5-hexachlorobiphenyl	35694-06-5			
138	138	2,2',3,4,4',5'-hexachlorobiphenyl	35065-28-2	$^{13}C_{12}$-2,2',3,4,4',5'-hexachlorobiphenyl	160901-76-8	Istd
139	139	2,2',3,4,4',6-hexachlorobiphenyl	56030-56-9			
140	140	2,2',3,4,4',6'-hexachlorobiphenyl	59291-64-4			
141	141	2,2',3,4,5,5'-hexachlorobiphenyl	52712-04-6			
142	142	2,2',3,4,5,6-hexachlorobiphenyl	41411-61-4			
143	143	2,2',3,4,5,6'-hexachlorobiphenyl	68194-15-0			
144	144	2,2',3,4,5',6-hexachlorobiphenyl	68194-14-9			
145	145	2,2',3,4,6,6'-hexachlorobiphenyl	74472-40-5			
146	146	2,2',3,4',5,5'-hexachlorobiphenyl	51908-16-8			
147	147	2,2',3,4',5,6-hexachlorobiphenyl	68194-13-8			
148	148	2,2',3,4',5,6'-hexachlorobiphenyl	74472-41-6			
149	149	2,2',3,4',5',6-hexachlorobiphenyl	38380-04-0			
150	150	2,2',3,4',6,6'-hexachlorobiphenyl	68194-08-1			
151	151	2,2',3,5,5',6-hexachlorobiphenyl	52663-63-5			
152	152	2,2',3,5,6,6'-hexachlorobiphenyl	68194-09-2			
153	153	2,2',4,4',5,5'-hexachlorobiphenyl	35065-27-1	$^{13}C_{12}$-2,2',4,4',5,5'-hexachlorobiphenyl		Air Surrogate
154	154	2,2',4,4',5,6'-hexachlorobiphenyl	60145-22-4			
155	155	2,2',4,4',6,6'-hexachlorobiphenyl	33979-03-2	$^{13}C_{12}$-2,2',4,4',6,6'-hexachlorobiphenyl	234432-90-7	IDA
156	156	2,3,3',4,4',5-hexachlorobiphenyl	38380-08-4	$^{13}C_{12}$-2,3,3',4,4',5-hexachlorobiphenyl	160901-77-9	IDA
157	157	2,3,3',4,4',5'-hexachlorobiphenyl	69782-90-7	$^{13}C_{12}$-2,3,3',4,4',5'-hexachlorobiphenyl	160901-78-0	IDA
158	158	2,3,3',4,4',6-hexachlorobiphenyl	74472-42-7			
159	159	2,3,3',4,5,5'-hexachlorobiphenyl	39635-35-3			
160	160	2,3,3',4,5,6-hexachlorobiphenyl	41411-62-5			
161	161	2,3,3',4,5',6-hexachlorobiphenyl	74472-43-8			
162	162	2,3,3',4',5,5'-hexachlorobiphenyl	39635-34-2			
163	163	2,3,3',4',5,6-hexachlorobiphenyl	74472-44-9			
164	164	2,3,3',4',5',6-hexachlorobiphenyl	74472-45-0			
165	165	2,3,3',5,5',6-hexachlorobiphenyl	74472-46-1			
166	166	2,3,4,4',5,6-hexachlorobiphenyl	41411-63-6			
167	167	2,3',4,4',5,5'-hexachlorobiphenyl	52663-72-6	$^{13}C_{12}$-2,3',4,4',5,5'-hexachlorobiphenyl	161627-18-5	IDA
168	168	2,3',4,4',5',6-hexachlorobiphenyl	59291-65-5			
169	169	3,3',4,4',5,5'-hexachlorobiphenyl	32774-16-6	$^{13}C_{12}$-3,3',4,4',5,5'-hexachlorobiphenyl	160901-79-1	IDA
170	170	2,2',3,3',4,4',5-heptachlorobiphenyl	35065-30-6	$^{13}C_{12}$-2,2',3,3',4,4',5-heptachlorobiphenyl	160901-80-4	IDA
171	171	2,2',3,3',4,4',6-heptachlorobiphenyl	52663-71-5			
172	172	2,2',3,3',4,5,5'-heptachlorobiphenyl	52663-74-8			
173	173	2,2',3,3',4,5,6-heptachlorobiphenyl	68194-16-1			
174	174	2,2',3,3',4,5,6'-heptachlorobiphenyl	38411-25-5			
175	175	2,2',3,3',4,5',6-heptachlorobiphenyl	40186-70-7			

PCB Number[1]	BZ/IUPAC Number[2]	PCB Chemical Structure Name[3]	CAS Registry[4] Number	Labeled Analog	CAS Registry[4] Number	Usage
176	176	2,2',3,3',4,6,6'-heptachlorobiphenyl	52663-65-7			
177	177	2,2',3,3',4',5,6-heptachlorobiphenyl (2,2',3,3',4,5',6'-heptachlorobiphenyl)	52663-70-4			
178	178	2,2',3,3',5,5',6-heptachlorobiphenyl	52663-67-9	$^{13}C_{12}$-2,2',3,3',5,5',6-heptachlorobiphenyl	160901-81-5	Surrogate Std
179	179	2,2',3,3',5,6,6'-heptachlorobiphenyl	52663-64-6			
180	180	2,2',3,4,4',5,5'-heptachlorobiphenyl	35065-29-3	$^{13}C_{12}$-2,2',3,4,4',5,5'-heptachlorobiphenyl	160901-82-6	Istd
181	181	2,2',3,4,4',5,6-heptachlorobiphenyl	74472-47-2			
182	182	2,2',3,4,4',5,6'-heptachlorobiphenyl	60145-23-5			
183	183	2,2',3,4,4',5',6-heptachlorobiphenyl	52663-69-1			
184	184	2,2',3,4,4',6,6'-heptachlorobiphenyl	74472-48-3			
185	185	2,2',3,4,5,5',6-heptachlorobiphenyl	52712-05-7			
186	186	2,2',3,4,5,6,6'-heptachlorobiphenyl	74472-49-4			
187	187	2,2',3,4',5,5',6-heptachlorobiphenyl	52663-68-0			
188	188	2,2',3,4',5,6,6'-heptachlorobiphenyl	74487-85-7	$^{13}C_{12}$-2,2',3,4',5,6,6'-heptachlorobiphenyl	234432-91-8	IDA
189	189	2,3,3',4,4',5,5'-heptachlorobiphenyl	39635-31-9	$^{13}C_{12}$-2,3,3',4,4',5,5'-heptachlorobiphenyl	160901-83-7	IDA
190	190	2,3,3',4,4',5,6-heptachlorobiphenyl	41411-64-7			
191	191	2,3,3',4,4',5',6-heptachlorobiphenyl	74472-50-7			
192	192	2,3,3',4,5,5',6-heptachlorobiphenyl	74472-51-8			
193	193	2,3,3',4',5,5',6-heptachlorobiphenyl	69782-91-8			
194	194	2,2',3,3',4,4',5,5'-octachlorobiphenyl	35694-08-7	$^{13}C_{12}$-2,2',3,3',4,4',5,5'-octachlorobiphenyl	208263-74-5	Istd
195	195	2,2',3,3',4,4',5,6-octachlorobiphenyl	52663-78-2			
196	196	2,2',3,3',4,4',5,6'-octachlorobiphenyl	42740-50-1			
197	197	2,2',3,3',4,4',6,6'-octachlorobiphenyl	33091-17-7			
198	198	2,2',3,3',4,5,5',6-octachlorobiphenyl	68194-17-2			
199	201/199	2,2',3,3',4,5,5',6'-octachlorobiphenyl	52663-75-9			
200	199/200	2,2',3,3',4,5,6,6'-octachlorobiphenyl	52663-73-7			
201	200/201	2,2',3,3',4,5',6,6'-octachlorobiphenyl	40186-71-8			
202	202	2,2',3,3',5,5',6,6'-octachlorobiphenyl	2136-99-4	$^{13}C_{12}$-2,2',3,3',5,5',6,6'-octachlorobiphenyl	105600-26-8	IDA
203	203	2,2',3,4,4',5,5',6-octachlorobiphenyl	52663-76-0			
204	204	2,2',3,4,4',5,6,6'-octachlorobiphenyl	74472-52-9			
205	205	2,3,3',4,4',5,5',6-octachlorobiphenyl	74472-53-0	$^{13}C_{12}$-2,3,3',4,4',5,5',6-octachlorobiphenyl	234446-64-1	IDA
206	206	2,2',3,3',4,4',5,5',6-nonachlorobiphenyl	40186-72-9	$^{13}C_{12}$-2,2',3,3',4,4',5,5',6-nonachlorobiphenyl	208263-75-6	IDA
207	207	2,2',3,3',4,4',5,6,6'-nonachlorobiphenyl	52663-79-3			
208	208	2,2',3,3',4,5,5',6,6'-nonachlorobiphenyl	52663-77-1	$^{13}C_{12}$-2,2',3,3',4,5,5',6,6'-nonachlorobiphenyl	234432-92-9	IDA
209	209	2,2',3,3',4,4',5,5',6,6'-decachlorobiphenyl	2051-24-3	$^{13}C_{12}$-decachlorobiphenyl	160901-84-8	IDA

1. The PCB congener number is from Method 1668C and Chemical Abstract Services.
2. The BZ number is from Ballschmiter and Zell (1980). The IUPAC number, when different from the BZ, follows the recommended changes to the BZ number per Schulte and Malisch (1983) and Guitart et al. (1993).
3. The chemical structure names are from Ballschmiter and Zell (1980). IUPAC nomenclature structure names are listed in parenthesis when different from the BZ name (source CAS Registry).
4. Chemical Abstract Service Registry number (source CAS Registry and 1668A Table 1).

Table 2 - RT References, Quantitation References, Retention Times (RTs), and Relative Retention Times (RRTs) for the 209 CB congeners on SPB-Octyl

Cl No[1]	IUPAC No[2,3]	RT Ref[4]	Quantitation Reference[5]	RT Win[6] (sec)
1	1L	9L	9L	30
1	3L	9L	9L	30
1	1	1L	1L	10
1	2	3L	1L/3L	6
1	3	3L	3L	6
2	4L	9L	9L	30
2	9L	9L	9L	25
2	8L	4L	4L/15L	6
2	15L	9L	9L	20
2	4	4L	4L	10
2	10	4L	4L/15L	6
2	9	4L	4L/15L	6
2	7	4L	4L/15L	6
2	6	4L	4L/15L	6
2	5	4L	4L/15L	6
2	8	4L	4L/15L	6
2	14	15L	4L/15L	6
2	11	15L	4L/15L	6
2	13	15L	4L/15L	6
2	12	15L	4L/15L	6
2	15	15L	15L	10
3	19L	32L	32L	30
3	32L	32L	32L	6
3	31L	31L	31L	6
3	28L	31L	31L	20
3	37L	31L	31L	30
3	19	19L	19L	10
3	30	19L	19L	6
3	18	19L	19L	6
3	17	19L	19L	6
3	27	19L	19L	6
3	24	19L	19L	6
3	16	19L	19L	6
3	32	19L	19L	6
3	34	19L	37L	6
3	23	19L	37L	6
3	29	19L	37L	10
3	26	19L	37L	10
3	25	37L	37L	6
3	31	37L	37L	6
3	28	37L	37L	10
3	20	37L	37L	10
3	21	37L	37L	10
3	33	37L	37L	10
3	22	37L	37L	6
3	36	37L	37L	6
3	39	37L	37L	6
3	38	37L	37L	6
3	35	37L	37L	6
3	37	37L	37L	6
4	54L	52L	32L	20
4	52L	52L	52L	25
4	79L	81L	81L/77L	6
4	81L	52L	52L	20
4	77L	52L	52L	20
4	54	54L	54L	10
4	50	54L	81L/77L	10
4	53	54L	81L/77L	10
4	45	54L	81L/77L	10
4	51	54L	81L/77L	10
4	46	54L	81L/77L	6
4	52	54L	81L/77L	6
4	73	54L	81L/77L	6
4	43	54L	81L/77L	6
4	69	54L	81L/77L	10
4	49	54L	81L/77L	10
4	48	54L	81L/77L	6
4	65	54L	81L/77L	10
4	47	54L	81L/77L	10
4	44	54L	81L/77L	10
4	62	54L	81L/77L	10
4	75	54L	81L/77L	10

Cl No[1]	IUPAC No[2,3]	RT Ref[4]	Quantitation Reference[5]	RT Win[6] (sec)
4	59	54L	81L/77L	10
4	42	54L	81L/77L	6
4	41	54L	81L/77L	10
4	71	54L	81L/77L	10
4	40	54L	81L/77L	10
4	64	54L	81L/77L	6
4	72	81L	81L/77L	6
4	68	81L	81L/77L	6
4	57	81L	81L/77L	6
4	58	81L	81L/77L	6
4	67	81L	81L/77L	6
4	63	81L	81L/77L	6
4	61	81L	81L/77L	12
4	70	81L	81L/77L	12
4	76	81L	81L/77L	12
4	74	81L	81L/77L	10
4	66	81L	81L/77L	6
4	55	81L	81L/77L	6
4	56	81L	81L/77L	6
4	60	81L	81L/77L	6
4	80	81L	81L/77L	6
4	79	81L	81L/77L	6
4	78	81L	81L/77L	6
4	81	81L	81L	6
4	77	77L	77L	6
5	104L	101L	101L	20
5	95L	104L	104L	10
5	101L	101L	101L	25
5	111L	101L	101L	20
5	123L	101L	127L	20
5	118L	101L	127L	20
5	114L	101L	127L	20
5	105L	101L	127L	20
5	127L	127L	127L	25
5	126L	101L	127L	20
5	104	104L	104L	10
5	96	104L	104L	10
5	103	104L	104L	6
5	94	104L	104L	6
5	95	104L	104L	10
5	100	104L	104L	10
5	93	104L	104L	10
5	102	104L	104L	10
5	98	104L	104L	10
5	88	104L	104L	12
5	91	104L	104L	10
5	84	104L	104L	6
5	89	104L	104L	6
5	121	104L	104L	6
5	92	123L	104L	6
5	113	104L	104L	10
5	90	104L	104L	10
5	101	104L	104L	10
5	83	104L	104L	12
5	99	104L	104L	10
5	112	104L	104L	6
5	119	104L	104L	16
5	108 (109)	104L	104L	16
5	86	104L	104L	16
5	97	104L	104L	16
5	125	104L	104L	16
5	87	104L	104L	10
5	117	104L	104L	12
5	116	104L	104L	12
5	85	104L	104L	10
5	110	104L	104L	10
5	115	104L	104L	10
5	82	104L	104L	6
5	111	104L	104L	6
5	120	104L	104L	6
5	107 (108)	104L	123L/114L/118L/105L/126L	10
5	124	104L	123L/114L/118L/105L/126L	10
5	109 (107)	104L	123L/114L/118L/105L/126L	6
5	123	123L	123L	6
5	106	123L	123L/114L/118L/105L/126L	6
5	118	118L	118L	6
5	122	118L	123L/114L/118L/105L/126L	6

Cl No[1]	IUPAC No[2,3]	RT Ref[4]	Quantitation Reference[5]	RT Win[6] (sec)
5	114	114L	114L	6
5	105	105L	105L	6
5	127	105L	123L/114L/118L/105L/126L	6
5	126	126L	126L	6
6	155L	138L	101L	20
6	153L	167L	156L/157L/167L/169L	10
6	138L	138L	138L	100
6	167L	138L	138L	20
6	156L	138L	138L	6
6	157L	138L	138L	20
6	169L	138L	138L	6
6	155	155L	155L	10
6	152	155L	155L	6
6	150	155L	155L	6
6	136	155L	155L	6
6	145	155L	155L	6
6	148	155L	155L	6
6	151	155L	155L	10
6	135	155L	155L	10
6	154	155L	155L	10
6	144	155L	155L	6
6	147	155L	156L/157L/167L/169L	10
6	149	155L	156L/157L/167L/169L	10
6	134	155L	156L/157L/167L/169L	10
6	143	155L	156L/157L/167L/169L	10
6	139	155L	156L/157L/167L/169L	10
6	140	155L	156L/157L/167L/169L	10
6	131	155L	156L/157L/167L/169L	6
6	142	155L	156L/157L/167L/169L	6
6	132	155L	156L/157L/167L/169L	10
6	133	155L	156L/157L/167L/169L	6
6	165	167L	156L/157L/167L/169L	6
6	146	167L	156L/157L/167L/169L	6
6	161	167L	156L/157L/167L/169L	6
6	153	167L	156L/157L/167L/169L	10
6	168	167L	156L/157L/167L/169L	10
6	141	167L	156L/157L/167L/169L	6
6	130	167L	156L/157L/167L/169L	6
6	137	167L	156L/157L/167L/169L	6
6	164	167L	156L/157L/167L/169L	6
6	138	167L	156L/157L/167L/169L	14
6	163	167L	156L/157L/167L/169L	14
6	129	167L	156L/157L/167L/169L	14
6	160	167L	156L/157L/167L/169L	10
6	158	167L	156L/157L/167L/169L	6
6	166	167L	156L/157L/167L/169L	10
6	128	167L	156L/157L/167L/169L	10
6	159	167L	156L/157L/167L/169L	6
6	162	167L	156L/157L/167L/169L	6
6	167	167L	167L	6
6	156	156L	156L/157L	6
6	157	157L	156L/157L	10
6	169	169L	169L	6
7	188L	180L	180L	20
7	178L	180L	180L	20
7	180L	180L	180L	100
7	170L	180L	180L	20
7	189L	180L	194L	20
7	188	188L	188L	6
7	179	188L	188L/170L	6
7	184	188L	188L/170L	6
7	176	188L	188L/170L	6
7	186	188L	188L/170L	6
7	178	188L	188L/170L	6
7	175	188L	188L/170L	6
7	187	188L	188L/170L	6
7	182	188L	188L/170L	6
7	183	188L	188L/170L	6
7	185	188L	188L/170L	6
7	174	188L	188L/170L	6
7	177	188L	188L/170L	6
7	181	188L	188L/170L	6
7	171	188L	188L/170L	10
7	173	188L	188L/170L	6
7	172	189L	188L/170L	6
7	192	189L	188L/170L	6
7	193	189L	188L/170L	6

Cl No[1]	IUPAC No[2,3]	RT Ref[4]	Quantitation Reference[5]	RT Win[6] (sec)
7	180	189L	188L/170L	6
7	191	189L	188L/170L	6
7	170	189L	170L	6
7	190	189L	188L/170L	6
7	189	189L	189L	6
8	202L	194L	180L	20
8	194L	194L	194L	25
8	205L	194L	194L	30
8	202	202L	202L	10
8	201	202L	202L	6
8	204	202L	202L	6
8	197	202L	202L	6
8	200	202L	202L	6
8	198	202L	202L	10
8	199	202L	202L	6
8	196	205L	202L	6
8	203	205L	202L	6
8	195	205L	205L	6
8	194	205L	205L	6
8	205	205L	205L	6
9	208L	194L	194L	20
9	206L	194L	194L	30
9	208	208L	208L	6
9	207	208L	208L/206L	6
9	206	206L	206L	6
10	209L	194L	194L	30
10	209	209L	209L	6

1. Number of chlorines on congener.
2. Suffix "L" indicates labeled compound.
3. IUPAC Number per Table 2 of Method 1668A. (Numbers in parentheses are PCB Numbers from 1668C)
4. Retention time reference that is used to locate target congener.
5. Quantitation reference that is used to calculate the concentration of the target congener or labeled standard.
6. RT window width for congener or group of two or more congeners.

Table 3 - Low Calibration Levels (LCLs) Based on Final Extract Volumes

		20 µL Extract Volume	100µL Extract Volume
Analyte	Minimum Cal. Level CS 0.5 (ng/mL)	Water 1L (ng/L)	Solids and Tissues 10g[2] (ng/g)
Monochlorobiphenyls	0.5	0.01[1]	0.005
Dichlorobiphenyls	0.5	0.01[1]	0.005
Trichlorobiphenyls	0.5	0.01	0.005
Tetrachlorobiphenyls	0.5	0.01	0.005
Pentachlorobiphenyls	0.5	0.01	0.005
Hexachlorobiphenyls	0.5	0.01	0.005
Heptachlorobiphenyls	0.5	0.01	0.005
Octachlorobiphenyls	0.5	0.01	0.005
Nonachlorobiphenyls	0.5	0.01	0.005
Decachlorobiphenyl	0.5	0.01	0.005

1. This value reflects the LCL. Reliable detection at this level may not be attained due to evaporative loss in adjusting the extract volume to 20 µL for these homolog groups.

2. The values for solids and tissues reflect the LCLs for Protocol 1 as described in Table 12. If the sample is prepared by another protocol described in that table, the LCLs shown in this table must be adjusted appropriately.

Table 4 - Estimated Minimum Levels EPA 1668A, B & C- Matrix and Concentration

	TestAmerica Knoxville EMLs [1] (1668A, B & C)			1668C Reference Method EMLs [2]		
Parameter	**Water ng/L**	**Other [3] ng/g**	**Extract ng/mL**	**Water ng/L**	**Other [3] ng/g**	**Extract ng/mL**
PCB 1	0.040	0.01	1.0	0.02	0.002	1
PCB 2	0.040	0.01	1.0	0.02	0.002	1
PCB 3	0.040	0.01	1.0	0.05	0.005	2.5
PCB 4	0.060	0.02	2.0	0.05	0.005	2.5
PCB 5	0.040	0.01	1.0	0.02	0.002	1
PCB 6	0.040	0.01	1.0	0.02	0.002	1
PCB 7	0.040	0.01	1.0	0.02	0.002	1
PCB 8	0.060	0.02	2.0	0.05	0.005	2.5
PCB 9	0.040	0.01	1.0	0.02	0.002	1
PCB 10	0.040	0.01	1.0	0.05	0.005	2.5
PCB 11	0.060	0.02	2.0	0.10	0.01	5
PCB 12	0.080	0.02	2.0	0.05	0.005	2.5
PCB 14	0.040	0.01	1.0	0.02	0.002	1
PCB 13	0.080	0.02	2.0	0.05	0.005	2.5
PCB 15	0.040	0.01	1.0	0.05	0.005	2.5
PCB 16	0.040	0.01	1.0	0.02	0.002	1
PCB 17	0.040	0.01	1.0	0.02	0.002	1
PCB 18	0.080	0.02	2.0	0.05	0.005	2.5
PCB 30	0.080	0.02	2.0	0.05	0.005	2.5
PCB 19	0.040	0.01	1.0	0.02	0.002	1
PCB 20	0.080	0.02	2.0	0.05	0.005	2.5
PCB 28	0.080	0.02	2.0	0.05	0.005	2.5
PCB 21	0.080	0.02	2.0	0.05	0.005	2.5
PCB 33	0.080	0.02	2.0	0.05	0.005	2.5
PCB 22	0.040	0.01	1.0	0.02	0.002	1
PCB 23	0.040	0.01	1.0	0.02	0.002	1
PCB 24	0.040	0.01	1.0	0.02	0.002	1
PCB 25	0.040	0.01	1.0	0.02	0.002	1
PCB 26	0.080	0.02	2.0	0.05	0.005	2.5
PCB 29	0.080	0.02	2.0	0.05	0.005	2.5
PCB 27	0.040	0.01	1.0	0.02	0.002	1
PCB 31	0.040	0.02	2.0	0.05	0.005	2.5
PCB 32	0.040	0.01	1.0	0.02	0.002	1
PCB 34	0.040	0.01	1.0	0.02	0.002	1
PCB 35	0.040	0.01	1.0	0.02	0.002	1
PCB 36	0.040	0.01	1.0	0.02	0.002	1
PCB 37	0.040	0.01	1.0	0.02	0.002	1
PCB 38	0.040	0.01	1.0	0.02	0.002	1
PCB 39	0.040	0.01	1.0	0.02	0.002	1
PCB 40	0.120	0.03	3.0	0.1	0.01	5
PCB 41	0.120	0.03	3.0	0.1	0.01	5
PCB 71	0.120	0.03	3.0	0.1	0.01	5
PCB 42	0.040	0.01	1.0	0.05	0.005	2.5
PCB 43	0.080	0.02	2.0	0.05	0.005	2.5
PCB 73	0.080	0.02	2.0	0.05	0.005	2.5
PCB 44	0.120	0.03	3.0	0.1	0.01	5
PCB 47	0.120	0.03	3.0	0.1	0.01	5
PCB 65	0.120	0.03	3.0	0.1	0.01	5
PCB 45	0.080	0.02	2.0	0.05	0.005	2.5
PCB 51	0.080	0.02	2.0	0.05	0.005	2.5
PCB 46	0.040	0.01	1.0	0.02	0.002	1
PCB 48	0.040	0.01	1.0	0.05	0.005	2.5
PCB 49	0.080	0.02	2.0	0.1	0.01	5
PCB 69	0.080	0.02	2.0	0.1	0.01	5
PCB 50	0.080	0.02	2.0	0.1	0.01	5
PCB 53	0.080	0.02	2.0	0.1	0.01	5
PCB 52	0.040	0.01	1.0	0.05	0.005	2.5

Parameter	TestAmerica Knoxville EMLs [1] (1668A, B & C)			1668C Reference Method EMLs [2]		
	Water ng/L	Other [3] ng/g	Extract ng/mL	Water ng/L	Other [3] ng/g	Extract ng/mL
PCB 54	0.040	0.01	1.0	0.05	0.005	2.5
PCB 55	0.040	0.01	1.0	0.05	0.005	2.5
PCB 56	0.040	0.01	1.0	0.05	0.005	2.5
PCB 57	0.040	0.01	1.0	0.05	0.005	2.5
PCB 58	0.040	0.01	1.0	0.05	0.005	2.5
PCB 59	0.120	0.03	3.0	0.1	0.01	5
PCB 62	0.120	0.03	3.0	0.1	0.01	5
PCB 75	0.120	0.03	3.0	0.1	0.01	5
PCB 60	0.040	0.01	1.0	0.05	0.005	2.5
PCB 61	0.160	0.04	4.0	0.2	0.02	10
PCB 70	0.160	0.04	4.0	0.2	0.02	10
PCB 74	0.160	0.04	4.0	0.2	0.02	10
PCB 76	0.160	0.04	4.0	0.2	0.02	10
PCB 63	0.040	0.01	1.0	0.005	0.05	2.5
PCB 64	0.040	0.01	1.0	0.05	0.005	2.5
PCB 66	0.040	0.01	1.0	0.05	0.005	2.5
PCB 67	0.040	0.01	1.0	0.05	0.005	2.5
PCB 68	0.040	0.01	1.0	0.05	0.005	2.5
PCB 72	0.040	0.01	1.0	0.05	0.005	2.5
PCB 77	0.040	0.01	1.0	0.05	0.005	2.5
PCB 78	0.040	0.01	1.0	0.05	0.005	2.5
PCB 79	0.040	0.01	1.0	0.05	0.005	2.5
PCB 80	0.040	0.01	1.0	0.05	0.005	2.5
PCB 81	0.040	0.01	1.0	0.05	0.005	2.5
PCB 82	0.040	0.01	1.0	0.05	0.005	2.5
PCB 83	0.080	0.02	2.0	0.1	0.01	5
PCB 84	0.040	0.01	1.0	0.02	0.002	1
PCB 85	0.120	0.03	3.0	0.1	0.01	5
PCB 116	0.120	0.03	3.0	0.1	0.01	5
PCB 117	0.120	0.03	3.0	0.1	0.01	5
PCB 86	0.240	0.06	6.0	0.2	0.02	10
PCB 87	0.240	0.06	6.0	0.2	0.02	10
PCB 97	0.240	0.06	6.0	0.2	0.02	10
PCB 109	0.240	0.06	6.0	0.2	0.02	10
PCB 119	0.240	0.06	6.0	0.2	0.02	10
PCB 125	0.240	0.06	6.0	0.2	0.02	10
PCB 88	0.080	0.02	2.0	0.05	0.005	2.5
PCB 91	0.080	0.02	2.0	0.05	0.005	2.5
PCB 89	0.040	0.01	1.0	0.05	0.005	2.5
PCB 90	0.120	0.03	3.0	0.2	0.02	10
PCB 101	0.120	0.03	3.0	0.2	0.02	10
PCB 113	0.120	0.03	3.0	0.2	0.02	10
PCB 92	0.040	0.01	1.0	0.05	0.005	2.5
PCB 93	0.080	0.02	2.0	0.2	0.02	10
PCB 100	0.080	0.02	2.0	0.2	0.02	10
PCB 94	0.040	0.01	1.0	0.05	0.005	2.5
PCB 95	0.040	0.01	1.0	0.2	0.02	10
PCB 96	0.040	0.01	1.0	0.05	0.005	2.5
PCB 98	0.080	0.02	2.0	0.2	0.02	10
PCB 102	0.080	0.02	2.0	0.2	0.02	10
PCB 99	0.080	0.02	2.0	0.1	0.01	5
PCB 112	0.040	0.01	1.0	0.05	0.005	2.5
PCB 103	0.040	0.01	1.0	0.05	0.005	2.5
PCB 104	0.040	0.01	1.0	0.05	0.005	2.5
PCB 105	0.040	0.01	1.0	0.05	0.005	2.5
PCB 106	0.040	0.01	1.0	0.05	0.005	2.5
PCB 107	0.040	0.01	1.0	0.05	0.005	2.5
PCB 108	0.080	0.02	2.0	0.1	0.01	5
PCB 124	0.080	0.02	2.0	0.1	0.01	5
PCB 110	0.080	0.02	2.0	0.1	0.01	5

Parameter	TestAmerica Knoxville EMLs [1] (1668A, B & C)			1668C Reference Method EMLs [2]		
	Water ng/L	Other [3] ng/g	Extract ng/mL	Water ng/L	Other [3] ng/g	Extract ng/mL
PCB 115	0.080	0.02	2.0	0.1	0.01	5
PCB 111	0.040	0.01	1.0	0.05	0.005	2.5
PCB 114	0.040	0.01	1.0	0.05	0.005	2.5
PCB 118	0.040	0.01	1.0	0.1	0.01	5
PCB 120	0.040	0.01	1.0	0.05	0.005	2.5
PCB 121	0.040	0.01	1.0	0.05	0.005	2.5
PCB 122	0.040	0.01	1.0	0.05	0.005	2.5
PCB 123	0.040	0.01	1.0	0.05	0.005	2.5
PCB 126	0.040	0.01	1.0	0.05	0.005	2.5
PCB 127	0.040	0.01	1.0	0.05	0.005	2.5
PCB 128	0.080	0.02	2.0	0.1	0.01	5
PCB 166	0.080	0.02	2.0	0.1	0.01	5
PCB 129	0.160	0.04	4.0	0.2	0.02	10
PCB 138	0.160	0.04	4.0	0.2	0.02	10
PCB 163	0.160	0.04	4.0	0.2	0.02	10
PCB 130	0.040	0.01	1.0	0.05	0.005	2.5
PCB 131	0.040	0.01	1.0	0.05	0.005	2.5
PCB 132	0.040	0.01	1.0	0.05	0.005	2.5
PCB 133	0.040	0.01	1.0	0.05	0.005	2.5
PCB 134	0.080	0.02	2.0	0.1	0.01	5
PCB 143	0.080	0.02	2.0	0.1	0.01	5
PCB 135	0.080	0.02	2.0	0.1	0.01	5
PCB 151	0.080	0.02	2.0	0.1	0.01	5
PCB 136	0.040	0.01	1.0	0.05	0.005	2.5
PCB 137	0.040	0.01	1.0	0.05	0.005	2.5
PCB 164	0.040	0.01	1.0	0.05	0.005	2.5
PCB 139	0.080	0.02	2.0	0.1	0.01	5
PCB 140	0.080	0.02	2.0	0.1	0.01	5
PCB 141	0.040	0.01	1.0	0.05	0.005	2.5
PCB 142	0.040	0.01	1.0	0.05	0.005	2.5
PCB 144	0.040	0.01	1.0	0.05	0.005	2.5
PCB 145	0.040	0.01	1.0	0.05	0.005	2.5
PCB 146	0.040	0.01	1.0	0.05	0.005	2.5
PCB 147	0.080	0.02	2.0	0.1	0.01	5
PCB 149	0.080	0.02	2.0	0.1	0.01	5
PCB 148	0.040	0.01	1.0	0.05	0.005	2.5
PCB 150	0.040	0.01	1.0	0.05	0.005	2.5
PCB 152	0.040	0.01	1.0	0.05	0.005	2.5
PCB 153	0.080	0.02	2.0	0.1	0.01	5
PCB 168	0.080	0.02	2.0	0.1	0.01	5
PCB 154	0.040	0.01	1.0	0.1	0.01	5
PCB 155	0.040	0.01	1.0	0.05	0.005	2.5
PCB 156	0.080	0.02	2.0	0.1	0.01	5
PCB 157	0.080	0.02	2.0	0.1	0.01	5
PCB 158	0.040	0.01	1.0	0.05	0.005	2.5
PCB 159	0.040	0.01	1.0	0.05	0.005	2.5
PCB 160	0.160	0.04	4.0	0.2	0.02	10
PCB 161	0.040	0.01	1.0	0.05	0.005	2.5
PCB 162	0.040	0.01	1.0	0.05	0.005	2.5
PCB 165	0.040	0.01	1.0	0.05	0.005	2.5
PCB 167	0.040	0.01	1.0	0.05	0.005	2.5
PCB 169	0.040	0.01	1.0	0.05	0.005	2.5
PCB 170	0.040	0.01	1.0	0.05	0.005	2.5
PCB 171	0.080	0.02	2.0	0.1	0.01	5
PCB 173	0.080	0.02	2.0	0.1	0.01	5
PCB 172	0.040	0.01	1.0	0.05	0.005	2.5
PCB 174	0.040	0.01	1.0	0.05	0.005	2.5
PCB 175	0.040	0.01	1.0	0.05	0.005	2.5
PCB 176	0.040	0.01	1.0	0.05	0.005	2.5
PCB 177	0.040	0.01	1.0	0.05	0.005	2.5

Parameter	TestAmerica Knoxville EMLs [1] (1668A, B & C)			1668C Reference Method EMLs [2]		
	Water ng/L	Other [3] ng/g	Extract ng/mL	Water ng/L	Other [3] ng/g	Extract ng/mL
PCB 178	0.040	0.01	1.0	0.05	0.005	2.5
PCB 179	0.040	0.01	1.0	0.05	0.005	2.5
PCB 180	0.080	0.02	2.0	0.1	0.01	5
PCB 193	0.080	0.02	2.0	0.1	0.01	5
PCB 181	0.040	0.01	1.0	0.05	0.005	2.5
PCB 182	0.040	0.01	1.0	0.05	0.005	2.5
PCB 183	0.080	0.02	2.0	0.1	0.01	5
PCB 185	0.080	0.02	2.0	0.1	0.01	5
PCB 184	0.040	0.01	1.0	0.05	0.005	2.5
PCB 186	0.040	0.01	1.0	0.05	0.005	2.5
PCB 187	0.040	0.01	1.0	0.05	0.005	2.5
PCB 188	0.040	0.01	1.0	0.05	0.005	2.5
PCB 189	0.040	0.01	1.0	0.05	0.005	2.5
PCB 190	0.040	0.01	1.0	0.05	0.005	2.5
PCB 191	0.040	0.01	1.0	0.05	0.005	2.5
PCB 192	0.040	0.01	1.0	0.05	0.005	2.5
PCB 194	0.040	0.01	1.0	0.05	0.005	2.5
PCB 195	0.040	0.01	1.0	0.05	0.005	2.5
PCB 196	0.040	0.01	1.0	0.05	0.005	2.5
PCB 197	0.040	0.01	1.0	0.1	0.01	5
PCB 200	0.040	0.01	1.0	0.1	0.01	5
PCB 198	0.080	0.02	2.0	0.1	0.01	5
PCB 199	0.080	0.02	2.0	0.1	0.01	5
PCB 201	0.040	0.01	1.0	0.05	0.005	2.5
PCB 202	0.040	0.01	1.0	0.1	0.01	5
PCB 203	0.040	0.01	1.0	0.05	0.005	2.5
PCB 204	0.040	0.01	1.0	0.05	0.005	2.5
PCB 205	0.040	0.01	1.0	0.05	0.005	2.5
PCB 206	0.040	0.01	1.0	0.05	0.005	2.5
PCB 207	0.040	0.01	1.0	0.05	0.005	2.5
PCB 208	0.040	0.01	1.0	0.05	0.005	2.5
PCB 209	0.040	0.01	1.0	0.05	0.005	2.5

[1] The estimated minimum level (EML) is defined as the lowest concentration at which an analyte can be measured reliably with common laboratory interferences present assuming a sample is extracted at the recommended weight or volume and is carried through all normal extraction and analysis procedures The values for other matrices (solids, tissues and solid wastes) reflect the EMLs for Protocol 1 as described in Table 12. If the sample is prepared by another protocol described in that section, the EMLs shown in this table must be adjusted appropriately. The EMLs are based on the mean plus 2 standard deviations for matrix-pooled historical blank data and calibration data obtained while performing EPA 1668A. The survey period was fourteen months, ending in February 2004. Individual EMLs may be adjusted to reflect more recent data.

[2] For reference purposes only, not currently used by TestAmerica Knoxville

[3] Other matrices include solids, tissues and solid wastes

Table 5a- Concentration of Target PCB Congener Stock and Spiking Solutions

Target PCB Congener	PCB	Standard Source	Catalog Number	Vendor Conc (ng/mL)	LCS Spiking Solution Conc (ng/mL)
2-MoCB	1	AccuStd	S-99994-4x	4000	5.0
4-MoCB	3	AccuStd	S-99994-4x	4000	5.0
2,2'-DiCB	4	AccuStd	S-99994-4x	4000	5.0
4,4'-DiCB	15	AccuStd	S-99994-4x	4000	5.0
2,2',6-TrCB	19	AccuStd	S-99994-4x	4000	5.0
3,4,4'-TrCB	37	AccuStd	S-99994-4x	4000	5.0
2,2',6,6'-TeCB	54	AccuStd	S-99994-4x	4000	5.0
3,3',4,4'-TeCB	77	AccuStd	S-99994-4x	4000	5.0
3,4,4',5-TeCB	81	AccuStd	S-99994-4x	4000	5.0
2,2',4,6,6'-PeCB	104	AccuStd	S-99994-4x	4000	5.0
2,3,3',4,4'-PeCB	105	AccuStd	S-99994-4x	4000	5.0
2,3,4,4',5-PeCB	114	AccuStd	S-99994-4x	4000	5.0
2,3',4,4',5-PeCB	118	AccuStd	S-99994-4x	4000	5.0
2',3,4,4',5-PeCB	123	AccuStd	S-99994-4x	4000	5.0
3,3',4,4',5-PeCB	126	AccuStd	S-99994-4x	4000	5.0
2,2',4,4',6,6'-HxCB	155	AccuStd	S-99994-4x	4000	5.0
2,3,3',4,4',5-HxCB	156	AccuStd	S-99994-4x	4000	5.0
2,3,3',4,4',5'-HxCB	157	AccuStd	S-99994-4x	4000	5.0
2,3',4,4',5,5'-HxCB	167	AccuStd	S-99994-4x	4000	5.0
3,3',4,4',5,5'-HxCB	169	AccuStd	S-99994-4x	4000	5.0
2,2',3,4',5,6,6'-HpCB	188	AccuStd	S-99994-4x	4000	5.0
2,3,3',4,4',5,5'-HpCB	189	AccuStd	S-99994-4x	4000	5.0
2,2',3,3',5,5',6,6'-OcCB	202	AccuStd	S-99994-4x	4000	5.0
2,3,3',4,4',5,5',6-OcCB	205	AccuStd	S-99994-4x	4000	5.0
2,2',3,3',4,4',5,5',6-NoCB	206	AccuStd	S-99994-4x	4000	5.0
2,2',3,3',4',5,5',6,6'-NoCB	208	AccuStd	S-99994-4x	4000	5.0
DeCB	209	AccuStd	S-99994-4x	4000	5.0
All other CB congeners	NA	AccuStd	S-99994-4x	4000	5.0

Table 5b: Concentration of $^{13}C_{12}$ Labeled PCB Congener Stock and Spiking Solutions

Labeled PCB Congener	**PCB**	**Standard Source**	**Catalog Number**	**Vendor Conc (ng/mL)**	Stock Conc (ng/mL)	Spiking Solution Conc (ng/mL)
IDA						
$^{13}C_{12}$-2-chlorobiphenyl	1L	Cambridge	EC-4908	40,000	1000	10
$^{13}C_{12}$-4-chlorobiphenyl	3L	Cambridge	EC-4990	40,000	1000	10
$^{13}C_{12}$-2,2'-dichlorobiphenyl	4L	Cambridge	EC-4911	40,000	1000	10
$^{13}C_{12}$-4,4'-dichlorobiphenyl	15L	Cambridge	EC-1402	40,000	1000	10
$^{13}C_{12}$-2,2',6-trichlorobiphenyl	19L	Cambridge	EC-4909	40,000	1000	10
$^{13}C_{12}$-3,4,4'-trichlorobiphenyl	37L	Cambridge	EC-4901	40,000	1000	10
$^{13}C_{12}$-2,2',6,6'-tetrachlorobiphenyl	54L	Cambridge	EC-4912	40,000	1000	10
$^{13}C_{12}$-3,3',4,4'-tetrachlorobiphenyl	77L	Cambridge	EC-1404	40,000	1000	10
$^{13}C_{12}$-3,4,4',5-tetrachlorobiphenyl	81L	Cambridge	EC-1412	40,000	1000	10
$^{13}C_{12}$-2,2',4,6,6'-pentachlorobiphenyl	104L	Cambridge	EC-4910	40,000	1000	10
$^{13}C_{12}$-2,3,3',4,4'-pentachlorobiphenyl	105L	Cambridge	EC-1420	40,000	1000	10
$^{13}C_{12}$2,3,4,4',5-pentachlorobiphenyl -	114L	Cambridge	EC-4902	40,000	1000	10
$^{13}C_{12}$-2,3',4,4',5-pentachlorobiphenyl	118L	Cambridge	EC-1435	40,000	1000	10
$^{13}C_{12}$-2',3,4,4',5-pentachlorobiphenyl	123L	Cambridge	EC-4904	40,000	1000	10
$^{13}C_{12}$-3,3',4,4',5-pentachlorobiphenyl	126L	Cambridge	EC-1425	40,000	1000	10
$^{13}C_{12}$-2,2',4,4',6,6'-hexachlorobiphenyl	155L	Cambridge	EC-4167	40,000	1000	10
$^{13}C_{12}$-2,3,3',4,4',5-hexachlorobiphenyl	156L	Cambridge	EC-1422	40,000	1000	10
$^{13}C_{12}$-2,3,3',4,4',5'-hexachlorobiphenyl	157L	Cambridge	EC-4051	40,000	1000	10
$^{13}C_{12}$-2,3',4,4',5,5'-hexachlorobiphenyl	167L	Cambridge	EC-4050	40,000	1000	10
$^{13}C_{12}$-3,3',4,4',5,5'-hexachlorobiphenyl	169L	Cambridge	EC-1416	40,000	1000	10
$^{13}C_{12}$-2,2',3,3',4,4',5-heptachlorobiphenyl	170L	Cambridge	EC-4905	40,000	1000	10
$^{13}C_{12}$-2,2',3,4',5,6,6'-heptachlorobiphenyl	188L	Cambridge	EC-4913	40,000	1000	10
$^{13}C_{12}$-2,3,3',4,4',5,5'-heptachlorobiphenyl	189L	Cambridge	EC-1409	40,000	1000	10
$^{13}C_{12}$-2,2',3,3',5,5',6,6'-octachlorobiphenyl	202L	Cambridge	EC-1408	40,000	1000	10
$^{13}C_{12}$-2,3,3',4,4',5,5',6-octachlorobiphenyl	205L	Cambridge	EC-4199	40,000	1000	10
$^{13}C_{12}$-2,2',3,3',4,4',5,5',6-nonachlorobiphenyl	206L	Cambridge	EC-4900	40,000	1000	10
$^{13}C_{12}$-2,2',3,3',4,5,5',6,6'-nonachlorobiphenyl	208L	Cambridge	EC-1419	40,000	1000	10
$^{13}C_{12}$-2,2',3,3',4,4',5,5',6,6'-decachlorobiphenyl	209L	Cambridge	EC-1410	40,000	1000	10
Istd						
$^{13}C_{12}$-2,5-dichlorobiphenyl	9L	Cambridge	EC-4165	40,000	1000	100
$^{13}C_{12}$-2,4',5-trichlorobiphenyl	31L	Wellington	MBP-31	50,000	1000	100
$^{13}C_{12}$-2,4',6-trichlorobiphenyl	32L	Cambridge	EC-4163	40,000	1000	100
$^{13}C_{12}$-2,2',5,5'-tetrachlorobiphenyl	52L	Cambridge	EC-1424	40,000	1000	100
$^{13}C_{12}$-2,2',4,5,5'-pentachlorobiphenyl	101L	Cambridge	EC-1405	40,000	1000	100
$^{13}C_{12}$-3,3',4,5,5'-pentachlorobiphenyl	127L	Cambridge	EC-1421	40,000	1000	100
$^{13}C_{12}$-2,2',3,4,4',5'-hexachlorobiphenyl	138L	Cambridge	EC-1436	40,000	1000	100
$^{13}C_{12}$-2,2',3,4,4',5,5'-heptachlorobiphenyl	180L	Cambridge	EC-1407	40,000	1000	100
$^{13}C_{12}$-2,2',3,3',4,4',5,5'-octachlorobiphenyl	194L	Cambridge	EC-1418	40,000	1000	100
Surrogates						
$^{13}C_{12}$-2,4,4'-trichlorobiphenyl	28L	Cambridge	EC-1413	40,000	5000	10
$^{13}C_{12}$-2,3,3',5,5'-pentachlorobiphenyl	111L	Cambridge	EC-1415	40,000	5000	10
$^{13}C_{12}$-2,2',3,3',5,5',6-heptachlorobiphenyl	178L	Cambridge	EC-1417	40,000	5000	10
Air Surrogate Standards						
$^{13}C_{12}$-2,4'-dichlorobiphenyl	8L	Cambridge	EC-5095	40,000	5000	50
$^{13}C_{12}$-3,3',4,5'-tetrachlorobiphenyl	79L	Cambridge	EC-5048	40,000	5000	50
$^{13}C_{12}$-2,2',3,5',6-pentachlorobiphenyl	95L	Wellington	MBP-95	50,000	5000	50
$^{13}C_{12}$-2,2',4,4',5,5'-hexachlorobiphenyl	153L	Cambridge	EC-1406	40,000	5000	50

Table 6a - Concentration of PCBs in Calibration Solutions

Analyte Type	PCB[1]	CS 0.5 ng/mL	CS 1 ng/mL	CS 2 ng/mL	CS 3[2] ng/mL	CS 4 ng/mL	CS 5 ng/mL
Congeners							
2-MoCB	1	0.5	1.0	5.0	50	400	2000
4-MoCB	3	0.5	1.0	5.0	50	400	2000
2,2'-DiCB	4	0.5	1.0	5.0	50	400	2000
4,4'-DiCB	15	0.5	1.0	5.0	50	400	2000
2,2',6'-TrCB	19	0.5	1.0	5.0	50	400	2000
3,4,4'-TrCB	37	0.5	1.0	5.0	50	400	2000
2,2',6,6'-TeCB	54	0.5	1.0	5.0	50	400	2000
3,3',4,4'-TeCB	77	0.5	1.0	5.0	50	400	2000
3,4,4',5-TeCB	81	0.5	1.0	5.0	50	400	2000
2,2',4,6,6'-PeCB	104	0.5	1.0	5.0	50	400	2000
2,3,3',4,4'-PeCB	105	0.5	1.0	5.0	50	400	2000
2,3,4,4',5-PeCB	114	0.5	1.0	5.0	50	400	2000
2,3',4,4',5-PeCB	118	0.5	1.0	5.0	50	400	2000
2',3,4,4',5-PeCB	123	0.5	1.0	5.0	50	400	2000
3,3',4,4',5-PeCB	126	0.5	1.0	5.0	50	400	2000
2,2',4,4',6,6'-HxCB	155	0.5	1.0	5.0	50	400	2000
2,3,3',4,4',5-HxCB	156	0.5	1.0	5.0	50	400	2000
2,3,3',4,4',5'-HxCB	157	0.5	1.0	5.0	50	400	2000
2,3',4,4',5,5'-HxCB	167	0.5	1.0	5.0	50	400	2000
3,3',4,4',5,5'-HxCB	169	0.5	1.0	5.0	50	400	2000
2,2',3,4',5,6,6'-HpCB	188	0.5	1.0	5.0	50	400	2000
2,3,3',4,4',5,5'-HpCB	189	0.5	1.0	5.0	50	400	2000
2,2',3,3',5,5',6,6'-OcCB	202	0.5	1.0	5.0	50	400	2000
2,3,3',4,4',5,5',6-OcCB	205	0.5	1.0	5.0	50	400	2000
2,2',3,3',4,4',5,5',6-NoCB	206	0.5	1.0	5.0	50	400	2000
2,2',3,3',4',5,5',6,6'-NoCB	208	0.5	1.0	5.0	50	400	2000
DeCB	209	0.5	1.0	5.0	50	400	2000
All other CB congeners		0.5	1.0	5.0	50	400	2000
Labeled Congeners							
$^{13}C_{12}$-2-MoCB	1L	100	100	100	100	100	100
$^{13}C_{12}$-4-MoCB	3L	100	100	100	100	100	100
$^{13}C_{12}$-2,2'-DiCB	4L	100	100	100	100	100	100
$^{13}C_{12}$-4,4'-DiCB	15L	100	100	100	100	100	100
$^{13}C_{12}$-2,2',6-TrCB	19L	100	100	100	100	100	100
$^{13}C_{12}$-3,4,4'-TrCB	37L	100	100	100	100	100	100
$^{13}C_{12}$-2,2',6,6'-TeCB	54L	100	100	100	100	100	100
$^{13}C_{12}$-3,3',4,4'-TeCB	77L	100	100	100	100	100	100
$^{13}C_{12}$-3,4,4',5-TeCB	81L	100	100	100	100	100	100
$^{13}C_{12}$-2,2',4,6,6'-PeCB	104L	100	100	100	100	100	100
$^{13}C_{12}$-2,3,3',4,4'-PeCB	105L	100	100	100	100	100	100
$^{13}C_{12}$-2,3,4,4',5-PeCB	114L	100	100	100	100	100	100
$^{13}C_{12}$-2,3',4,4',5-PeCB	118L	100	100	100	100	100	100
$^{13}C_{12}$-2',3,4,4',5-PeCB	123L	100	100	100	100	100	100
$^{13}C_{12}$-3,3',4,4',5-PeCB	126L	100	100	100	100	100	100
$^{13}C_{12}$-2,2',4,4',6,6'-HxCB	155L	100	100	100	100	100	100
$^{13}C_{12}$-2,3,3',4,4',5-HxCB	156L	100	100	100	100	100	100
$^{13}C_{12}$-2,3,3',4,4',5'-HxCB	157L	100	100	100	100	100	100
$^{13}C_{12}$-2,3',4,4',5,5'-HxCB	167L	100	100	100	100	100	100
$^{13}C_{12}$-3,3',4,4',5,5'-HxCB	169L	100	100	100	100	100	100
$^{13}C_{12}$-2,2',3,3',4,4',5-HpCB	170L	100	100	100	100	100	100
$^{13}C_{12}$-2,2',3,4',5,6,6'-HpCB	188L	100	100	100	100	100	100
$^{13}C_{12}$-2,3,3',4,4',5,5'-HpCB	189L	100	100	100	100	100	100
$^{13}C_{12}$-2,2',3,3',5,5',6,6'-OcCB	202L	100	100	100	100	100	100

Analyte Type	PCB[1]	CS 0.5 ng/mL	CS 1 ng/mL	CS 2 ng/mL	CS 3[2] ng/mL	CS 4 ng/mL	CS 5 ng/mL
$^{13}C_{12}$-2,3,3',4,4',5,5',6-OcCB	205L	100	100	100	100	100	100
$^{13}C_{12}$-2,2',3,3',4,4',5,5',6-NoCB	206L	100	100	100	100	100	100
$^{13}C_{12}$-2,2',3,3',4',5,5',6,6'-NoCB	208L	100	100	100	100	100	100
$^{13}C_{12}$-DeCB	209L	100	100	100	100	100	100
Surrogate Standards							
$^{13}C_{12}$-2,4,4'-TriCB	28L			5.0	50	400	--
$^{13}C_{12}$-2,3,3',5,5'-PeCB	111L			5.0	50	400	--
$^{13}C_{12}$-2,2',3,3',5,5',6-HpCB	178L			5.0	50	400	--
Istd							
$^{13}C_{12}$-2,5-DiCB	9L	100	100	100	100	100	100
$^{13}C_{12}$-2,4',5-TriCB	31L	100	100	100	100	100	100
$^{13}C_{12}$-2,4',6-TriCB	32L	100	100	100	100	100	100
$^{13}C_{12}$-2,2',5,5'-TeCB	52L	100	100	100	100	100	100
$^{13}C_{12}$-2,2',4',5,5'-PeCB	101L	100	100	100	100	100	100
$^{13}C_{12}$-3,3',4,5,5'-PeCB	127L	100	100	100	100	100	100
$^{13}C_{12}$-2,2',3',4,4',5'-HxCB	138L	100	100	100	100	100	100
$^{13}C_{12}$-2,2',3,4,4',5,5'-HpCB	180L	100	100	100	100	100	100
$^{13}C_{12}$-2,2',3,3',4,4',5,5'-OcCB	194L	100	100	100	100	100	100
Labeled Air Surrogates							
$^{13}C_{12}$-2,4'-DiCB	8L			5.0	50	400	--
$^{13}C_{12}$-3,3',4,5'-TeCB	79L			5.0	50	400	--
$^{13}C_{12}$-2,2',3,5',6-PeCB	95L			5.0	50	400	--
$^{13}C_{12}$-2,2',4,4',5,5'-HxCB	153L			5.0	50	400	--

Notes:

1. Suffix “L” indicates labeled compound.

2. The CS 3 standard is also used as the calibration verification solution.

Table 6b – Preparation of Calibration Solutions

Calibration Standard ID	Parent Standards	Parent Conc. (ng/mL)	Volume Added (mL)	Final Volume (mL)	Final Conc (ng/mL)
CS 0.5	Target PCB Congener Stock Solution	40	0.0125	1.0	0.50
	IDA Stock Solution	1000	0.1000		100
	Istd Stock Solution	1000	0.1000		100
CS 1	Target PCB Congener Stock Solution	40	0.0250	1.0	1.0
	IDA Stock Solution	1000	0.1000		100
	Istd Stock Solution	1000	0.1000		100
CS 2	Target PCB Congener Stock Solution	40	0.1250	1.0	5.0
	IDA Stock Solution	1000	0.1000		100
	Istd Stock Solution	1000	0.1000		100
	Surrogate Stock Solution	5000	0.0010		5.0
	Air Surrogate Stock Solution	5000	0.0010		5.0
CS 3	Target PCB Congener Standard Mix	4000	0.0375	3.0	50
	IDA Stock Solution	1000	0.3000		100
	Istd Stock Solution	1000	0.3000		100
	Surrogate Stock Solution	5000	0.0300		50
	Air Surrogate Stock Solution	5000	0.0300		50
CS 4	Target PCB Congener Standard Mix	4000	0.1000	1.0	400
	IDA Stock Solution	1000	0.1000		100
	Istd Stock Solution	1000	0.1000		100
	Surrogate Stock Solution	5000	0.0800		400
	Air Surrogate Stock Solution	5000	0.0800		400
CS 5	Target PCB Congener Standard Mix	4000	0.5000	1.0	2000
	IDA Stock Solution	1000	0.1000		100
	Istd Stock Solution	1000	0.1000		100

Table 7 - GC Window Defining Mixture and SPB –Octyl Resolution Test Compounds

SPB-Octyl

Congener Group	First Eluted		Last Eluted	
Mono	1	2-	3	4-
Di	4	2,2'-	15	4,4'-
Tri	19	2,2',6-	37	3,4,4'-
Tetra	54	2,2',6,6'-	77	3,3',4,4'-
Penta	104	2,2',4,6,6'-	126	3,3',4,4',5-
Hexa	155	2,2',4,4',6,6'-	169	3,3',4,4',5,5'-
Hepta	188	2,2',3,4',5,6,6'-	189	2,3,3',4,4',5,5'-
Octa	202	2,2',3,3',5,5',6,6'-	205	2,3,3',4,4',5,5',6-
Nona	208	2,2',3,3',4,5,5',6,6'-	206	2,2',3,3',4,4',5,5',6-
Deca	209	2,2',3,3',4,4',5,5',6,6'-	209	2,2',3,3',4,4',5,5',6,6'-

SPB-Octyl Resolution Test Compounds

23	2,3,5-trichlorobiphenyl
34	2',3,5-trichlorobiphenyl (2,3',5'-trichlorobiphenyl)
182	2,2',3,4,4',5,6'-heptachlorobiphenyl
187	2,2',3,4',5,5',6-heptachlorobiphenyl

Table 8 - Ions Monitored for HRGC/HRMS Analysis of PCBs

Descriptor	Accurate Mass	Ion ID	Elemental Composition	Analyte
1	180.9888 (168.9833)	Lock	C_4F_7 ($C_3F_7^+$)	PFK (FC43)
	188.0393	M	$C_{12}H_9{}^{35}Cl$	Mono
	190.0363	M+2	$C_{12}H_9{}^{37}Cl$	Mono
	200.0795	M	$^{13}C_{12}H_9{}^{35}Cl$	Mono-13C12
	202.0766	M+2	$^{13}C_{12}H_9{}^{37}Cl$	Mono-13C12
	222.0003	M	$C_{12}H_8{}^{35}Cl_2$	Di
	223.9974	M+2	$C_{12}H_8{}^{35}Cl^{37}Cl$	Di
	234.0406	M	$^{13}C_{12}H_8{}^{35}Cl_2$	Di-13C
	236.0376	M+2	$^{13}C_{12}H_8{}^{35}Cl^{37}Cl$	Di-13C
	255.9613	M	$C_{12}H_7{}^{35}Cl_3$	Tri
	257.9584	M+2	$C_{12}H_7{}^{35}Cl_2{}^{37}Cl$	Tri
	268.0016	M	$^{13}C_{12}H_7{}^{35}Cl_3$	Tri-13C
	269.9986	M+2	$^{13}C_{12}H_7{}^{35}Cl_2{}^{37}Cl$	Tri-13C
	280.9824 (263.9866)	QC	C_6F_{11} ($C_5NF_{10}^+$)	PFK (FC43)
	289.9224	M	$C_{12}H_6{}^{35}Cl_4$	Tetra
	291.9194	M+2	$C_{12}H_6{}^{35}Cl_3{}^{37}Cl$	Tetra
	301.9626	M	$^{13}C_{12}H_6{}^{35}Cl_4$	Tetra-13C
	303.9597	M+2	$^{13}C_{12}H_6{}^{35}Cl_3{}^{37}Cl$	Tetra-13C
2	255.9613	M	$C_{12}H_7{}^{35}Cl_3$	Tri
	257.9584	M+2	$C_{12}H_7{}^{35}Cl_2{}^{37}Cl$	Tri
	268.0016	M	$^{13}C_{12}H_7{}^{35}Cl_3$	Tri-13C
	268.9824 (263.9866)	Lock	C_5F_{11}($C_5NF_{10}^+$)	PFK (FC43)
	269.9986	M+2	$^{13}C_{12}H_7{}^{35}Cl_2{}^{37}Cl$	Tri-13C
	289.9224	M	$C_{12}H_6{}^{35}Cl_4$	Tetra
	291.9194	M+2	$C_{12}H_6{}^{35}Cl_3{}^{37}Cl$	Tetra
	301.9626	M	$^{13}C_{12}H_6{}^{35}Cl_4$	Tetra-13C
	303.9597	M+2	$^{13}C_{12}H_6{}^{35}Cl_3{}^{37}Cl$	Tetra-13C
	325.8804	M+2	$C_{12}H_5{}^{35}Cl_4{}^{37}Cl$	Penta
	327.8775	M+4	$C_{12}H_5{}^{35}Cl_3{}^{37}Cl_2$	Penta
	337.9207	M+2	$^{13}C_{12}H_5{}^{35}Cl_4{}^{37}Cl$	Penta-13C
	339.9178	M+4	$^{13}C_{12}H_5{}^{35}Cl_3{}^{37}Cl_2$	Penta-13C
	359.8415	M+2	$C_{12}H_4{}^{35}Cl_5{}^{37}Cl$	Hexa
	361.8385	M+4	$C_{12}H_4{}^{35}Cl_4{}^{37}Cl_2$	Hexa
	371.8817	M+2	$^{13}C_{12}H_4{}^{35}Cl_5{}^{37}Cl$	Hexa-13C
	373.8788	M+4	$^{13}C_{12}H_4{}^{35}Cl_4{}^{37}Cl_2$	Hexa-13C
	380.9760 (375.9802)	QC	$C_{10}F_{14}$ ($C_8NF_{14}^+$)	PFK (FC43)

Table 8 - Ions Monitored for HRGC/HRMS Analysis of PCBs (continued)

Descriptor	Accurate Mass	Ion ID	Elemental Composition	Analyte
3	325.8804	M+2	$C_{12}H_5{}^{35}Cl_4{}^{37}Cl$	Penta
	327.8775	M+4	$C_{12}H_5{}^{35}Cl_3{}^{37}Cl_2$	Penta
	337.9207	M+2	$^{13}C_{12}H_5{}^{35}Cl_4{}^{37}Cl$	Penta-13C
	339.9178	M+4	$^{13}C_{12}H_5{}^{35}Cl_3{}^{37}Cl_2$	Penta-13C
	342.9792 (313.9834)	Lock	C_8F_{13} ($C_6NF_{12}^+$)	PFK (FC43)
	359.8415	M+2	$C_{12}H_4{}^{35}Cl_5{}^{37}Cl$	Hexa
	361.8385	M+4	$C_{12}H_4{}^{35}Cl_4{}^{37}Cl_2$	Hexa
	371.8817	M+2	$^{13}C_{12}H_4{}^{35}Cl_5{}^{37}Cl$	Hexa-13C
	373.8788	M+4	$^{13}C_{12}H_4{}^{35}Cl_4{}^{37}Cl_2$	Hexa-13C
	393.8025	M+2	$C_{12}H_3{}^{35}Cl_6{}^{37}Cl$	Hepta
	395.7995	M+4	$C_{12}H_3{}^{35}Cl_5{}^{37}Cl_2$	Hepta
	405.8428	M+2	$^{13}C_{12}H_3{}^{35}Cl_6{}^{37}Cl$	Hepta-13C
	407.8398	M+4	$^{13}C_{12}H_3{}^{35}Cl_5{}^{37}Cl_2$	Hepta-13C
	427.7635	M+2	$C_{12}H_2{}^{35}Cl_7{}^{37}Cl$	Octa
	429.7606	M+4	$C_{12}H_2{}^{35}Cl_6{}^{37}Cl_2$	Octa
	430.9728 (413.9770)	QC	C_9F_{17} ($C_8NF_{16}^+$)	PFK (FC43)
	439.8038	M+2	$^{13}C_{12}H_2{}^{35}Cl_7{}^{37}Cl$	Octa-13C
	441.8008	M+4	$^{13}C_{12}H_2{}^{35}Cl_6{}^{37}Cl_2$	Octa-13C
4	393.8025	M+2	$C_{12}H_3{}^{35}Cl_6{}^{37}Cl$	Hepta
	395.7995	M+4	$C_{12}H_3{}^{35}Cl_5{}^{37}Cl_2$	Hepta
	404.9760 (413.9770)	Lock	$C_{10}F_{15}$ ($C_8NF_{16}^+$)	PFK (FC43)
	405.8428	M+2	$^{13}C_{12}H_3{}^{35}Cl_6{}^{37}Cl$	Hepta-13C
	407.8398	M+4	$^{13}C_{12}H_3{}^{35}Cl_5{}^{37}Cl_2$	Hepta-13C
	427.7635	M+2	$C_{12}H_2{}^{35}Cl_7{}^{37}Cl$	Octa
	429.7606	M+4	$C_{12}H_2{}^{35}Cl_6{}^{37}Cl_2$	Octa
	439.8038	M+2	$^{13}C_{12}H_2{}^{35}Cl_7{}^{37}Cl$	Octa-13C
	441.8008	M+4	$^{13}C_{12}H_2{}^{35}Cl_6{}^{37}Cl_2$	Octa-13C
	461.7246	M+2	$C_{12}H^{35}Cl_7{}^{37}Cl_2$	Nona
	463.7216	M+4	$C_{12}H^{35}Cl_6{}^{37}Cl_3$	Nona
	473.7648	M+2	$^{13}C_{12}H^{35}Cl_7{}^{37}Cl_2$	Nona-13C
	475.7619	M+4	$^{13}C_{12}H^{35}Cl_6{}^{37}Cl_3$	Nona-13C
	495.6856	M+2	$C_{12}{}^{35}Cl_8{}^{37}Cl_2$	Deca
	497.6826	M+4	$C_{12}{}^{35}Cl_7{}^{37}Cl_3$	Deca
	504.9697 (501.9706)	QC	$C_{12}F_{19}$ ($C_9NF_{20}^+$)	PFK (FC43)
	507.7258	M+2	$^{13}C_{12}{}^{35}Cl_8{}^{37}Cl_2$	Deca-13C
	509.7229	M+4	$^{13}C_{12}{}^{35}Cl_7{}^{37}Cl_3$	Deca-13C

1. Nuclidic masses used: H = 1.007825 C = 12.00000 ^{13}C = 13.003355 F = 18.9984
O = 15.994915 ^{35}Cl = 34.968853 ^{37}Cl = 36.965903

Table 9 - Theoretical Ion Abundance Ratios and Control Limits for PCBs

Chlorine Atoms	mass's Forming Ratios	Theoretical Ratio	Lower QC Limit	Upper QC Limit
1	m/m+2	3.13	2.66	3.60
2	m/m+2	1.56	1.33	1.79
3	m/m+2	1.04	0.88	1.20
4	m/m+2	0.77	0.65	0.89
5	m+2/m+4	1.55	1.32	1.78
6	m+2/m+4	1.24	1.05	1.43
7	m+2/m+4	1.05	0.89	1.21
8	m+2/m+4	0.89	0.76	1.02
9	m+2/m+4	0.77	0.65	0.89
10	m+2/m+4	0.69	0.59	0.79

Table 10A - Acceptance Criteria for Performance Tests (1668A & B)

			IPR (IDOC)			
Toxic & LOC Congeners	**PCB**	**Test Conc (ng/mL) [1]**	**%RSD**	**%R**	**VER %R**	**LCS %R**
2-chlorobiphenyl	1	50	40	60-140	70-130	50-150
4-chlorobiphenyl	3	50	40	60-140	70-130	50-150
2,2'-dichlorobiphenyl	4	50	40	60-140	70-130	50-150
4,4'-dichlorobiphenyl	15	50	40	60-140	70-130	50-150
2,2',6-trichlorobiphenyl	19	50	40	60-140	70-130	50-150
3,4,4'-trichlorobiphenyl	37	50	40	60-140	70-130	50-150
2,2',6,6'-tetrachlorobiphenyl	54	50	40	60-140	70-130	50-150
3,3',4,4'-tetrachlorobiphenyl	77	50	40	60-140	70-130	50-150
3,4,4',5-tetrachlorobiphenyl	81	50	40	60-140	70-130	50-150
2,2'4,6,6'-pentachlorobiphenyl	104	50	40	60-140	70-130	50-150
2,3,3',4,4'-pentachlorobiphenyl	105	50	40	60-140	70-130	50-150
2,3,4,4',5-pentachlorobiphenyl	114	50	40	60-140	70-130	50-150
2,3',4,4',5-pentachlorobiphenyl	118	50	40	60-140	70-130	50-150
2',3',4,4',5-pentachlorobiphenyl	123	50	40	60-140	70-130	50-150
3,3',4,4',5-pentachlorobiphenyl	126	50	40	60-140	70-130	50-150
2,2',4,4',6,6'-hexachlorobiphenyl	155	50	40	60-140	70-130	50-150
2,3,3',4,4',5-hexachlorobiphenyl	156	50	40	60-140	70-130	50-150
2,3',4,4',5,5'-hexachlorobiphenyl	157	50	40	60-140	70-130	50-150
2,3,3',4,4',5'-hexachlorobiphenyl	167	50	40	60-140	70-130	50-150
3,3',4,4',5,5'-hexachlorobiphenyl	169	50	40	60-140	70-130	50-150
2,2',3,4'5,6,6'-heptachlorobiphenyl	188	50	40	60-140	70-130	50-150
2,3,3',4,4',5,5'-heptachlorobiphenyl	189	50	40	60-140	70-130	50-150
2,2',3,3',5,5',6,6'-octachlorobiphenyl	202	50	40	60-140	70-130	50-150
2,3,3',4,4',5,5',6-octachlorobiphenyl	205	50	40	60-140	70-130	50-150
2,2',3,3',4,4',5,5',6-nonachlorobiphenyl	206	50	40	60-140	70-130	50-150
2,2',3,3',4,5,5',6,6'-nonachlorobiphenyl	208	50	40	60-140	70-130	50-150
2,2',3,3',4,4',5,5',6,6'-decachlorobiphenyl	209	50	40	60-140	70-130	50-150
IDAs						
$^{13}C_{12}$-2-chlorobiphenyl	1L	100	50	35-135	50-150	30-140
$^{13}C_{12}$-4-chlorobiphenyl	3L	100	50	35-135	50-150	30-140
$^{13}C_{12}$-2,2'-dichlorobiphenyl	4L	100	50	35-135	50-150	30-140
$^{13}C_{12}$-4,4'-dichlorobiphenyl	15L	100	50	35-135	50-150	30-140
$^{13}C_{12}$-2,2',6-trichlorobiphenyl	19L	100	50	35-135	50-150	30-140
$^{13}C_{12}$-3,4,4'-trichlorobiphenyl	37L	100	50	35-135	50-150	30-140
$^{13}C_{12}$-2,2',6,6'-tetrachlorobiphenyl	54L	100	50	35-135	50-150	30-140
$^{13}C_{12}$-3,3',4,4'-tetrachlorobiphenyl	77L	100	50	35-135	50-150	30-140
$^{13}C_{12}$-3,4,4',5-tetrachlorobiphenyl	81L	100	50	35-135	50-150	30-140
$^{13}C_{12}$-2,2',4,6,6'-pentachlorobiphenyl	104L	100	50	35-135	50-150	30-140
$^{13}C_{12}$-2,3,3',4,4'-pentachlorobiphenyl	105L	100	50	35-135	50-150	30-140
$^{13}C_{12}$2,3,4,4',5-pentachlorobiphenyl -	114L	100	50	35-135	50-150	30-140
$^{13}C_{12}$-2,3',4,4',5-pentachlorobiphenyl	118L	100	50	35-135	50-150	30-140
$^{13}C_{12}$-2',3,4,4',5-pentachlorobiphenyl	123L	100	50	35-135	50-150	30-140
$^{13}C_{12}$-3,3',4,4',5-pentachlorobiphenyl	126L	100	50	35-135	50-150	30-140
$^{13}C_{12}$-2,2',4,4',6,6'-hexachlorobiphenyl	155L	100	50	35-135	50-150	30-140
$^{13}C_{12}$-2,3,3',4,4',5-hexachlorobiphenyl	156L	100	50	35-135	50-150	30-140
$^{13}C_{12}$-2,3,3',4,4',5'-hexachlorobiphenyl	157L	100	50	35-135	50-150	30-140
$^{13}C_{12}$-2,3',4,4',5,5'-hexachlorobiphenyl	167L	100	50	35-135	50-150	30-140
$^{13}C_{12}$-3,3',4,4',5,5'-hexachlorobiphenyl	169L	100	50	35-135	50-150	30-140
$^{13}C_{12}$-2,2',3,3',4,4',5-heptachlorobiphenyl	170L	100	50	35-135	50-150	30-140
$^{13}C_{12}$-2,2',3,4',5,6,6'-heptachlorobiphenyl	188L	100	50	35-135	50-150	30-140
$^{13}C_{12}$-2,3,3',4,4',5,5'-heptachlorobiphenyl	189L	100	50	35-135	50-150	30-140
$^{13}C_{12}$-2,2',3,3',5,5',6,6'-octachlorobiphenyl	202L	100	50	35-135	50-150	30-140
$^{13}C_{12}$-2,3,3',4,4',5,5',6-octachlorobiphenyl	205L	100	50	35-135	50-150	30-140

			IPR (IDOC)			
Toxic & LOC Congeners	**PCB**	**Test Conc (ng/mL) [1]**	**%RSD**	**%R**	**VER %R**	**LCS %R**
$^{13}C_{12}$-2,2',3,3',4,4',5,5',6-nonachlorobiphenyl	206L	100	50	35-135	50-150	30-140
$^{13}C_{12}$-2,2',3,3',4,5,5',6,6'-nonachlorobiphenyl	208L	100	50	35-135	50-150	30-140
$^{13}C_{12}$-2,2',3,3',4,4',5,5',6,6'-decachlorobiphenyl	209L	100	50	35-135	50-150	30-140
Surrogate Standards						
$^{13}C_{12}$-2,4,4'-trichlorobiphenyl	28L	50	45	45-120	60-130	40-125
$^{13}C_{12}$-2,3,3',5,5'-pentachlorobiphenyl	111L	50	45	45-120	60-130	40-125
$^{13}C_{12}$-2,2',3,3',5,5',6-heptachlorobiphenyl	178L	50	45	45-120	60-130	40-125
Air Surrogate Standards						
$^{13}C_{12}$-2,4'-dichlorobiphenyl	8L	50			50-150	50-150
$^{13}C_{12}$-3,3',4,5'-tetrachlorobiphenyl	79L	50			50-150	50-150
$^{13}C_{12}$-2,2',3,5',6-pentachlorobiphenyl	95L	50			50-150	50-150
$^{13}C_{12}$-2,2',4,4',5,5'-hexachlorobiphenyl	153L	50			50-150	50-150

[1] - Test concentrations are based on ng/mL in the sample extract or standard solution.

Table 10B - Acceptance Criteria for Performance Tests (1668C)

			IPR (IDOC)				
Toxic & LOC Congeners	**PCB**	**Test Conc (ng/mL) [1]**	**%RSD**	**Mean %R**	**VER %R**	**OPR (LCS) %R**	**Labeled Compound %R in Samples**
2-chlorobiphenyl	1	50	25	70-130	75-125	60-135	
4-chlorobiphenyl	3	50	25	70-130	75-125	60-135	
2,2'-dichlorobiphenyl	4	50	25	70-130	75-125	60-135	
4,4'-dichlorobiphenyl	15	50	25	70-130	75-125	60-135	
2,2',6-trichlorobiphenyl	19	50	25	70-130	75-125	60-135	
3,4,4'-trichlorobiphenyl	37	50	25	70-130	75-125	60-135	
2,2',6,6'-tetrachlorobiphenyl	54	50	25	70-130	75-125	60-135	
3,3',4,4'-tetrachlorobiphenyl	77	50	25	70-130	75-125	60-135	
3,4,4',5-tetrachlorobiphenyl	81	50	25	70-130	75-125	60-135	
2,2'4,6,6'-pentachlorobiphenyl	104	50	25	70-130	75-125	60-135	
2,3,3',4,4'-pentachlorobiphenyl	105	50	25	70-130	75-125	60-135	
2,3,4,4',5-pentachlorobiphenyl	114	50	25	70-130	75-125	60-135	
2,3',4,4',5-pentachlorobiphenyl	118	50	25	70-130	75-125	60-135	
2',3',4,4',5-pentachlorobiphenyl	123	50	25	70-130	75-125	60-135	
3,3',4,4',5-pentachlorobiphenyl	126	50	25	70-130	75-125	60-135	
2,2',4,4',6,6'-hexachlorobiphenyl	155	50	25	70-130	75-125	60-135	
2,3,3',4,4',5-hexachlorobiphenyl	156	50	25	70-130	75-125	60-135	
2,3',4,4',5,5'-hexachlorobiphenyl	157	50	25	70-130	75-125	60-135	
2,3,3',4,4',5'-hexachlorobiphenyl	167	50	25	70-130	75-125	60-135	
3,3',4,4',5,5'-hexachlorobiphenyl	169	50	25	70-130	75-125	60-135	
2,2',3,4'5,6,6'-heptachlorobiphenyl	188	50	25	70-130	75-125	60-135	
2,3,3',4,4',5,5'-heptachlorobiphenyl	189	50	25	70-130	75-125	60-135	
2,2',3,3',5,5',6,6'-octachlorobiphenyl	202	50	25	70-130	75-125	60-135	
2,3,3',4,4',5,5',6-octachlorobiphenyl	205	50	25	70-130	75-125	60-135	
2,2',3,3',4,4',5,5',6-nonachlorobiphenyl	206	50	25	70-130	75-125	60-135	
2,2',3,3',4,5,5',6,6'-nonachlorobiphenyl	208	50	25	70-130	75-125	60-135	
2,2',3,3',4,4',5,5',6,6'-decachlorobiphenyl	209	50	25	70-130	75-125	60-135	
IDAs							
$^{13}C_{12}$-2-chlorobiphenyl	1L	100	70	20-135	50-145	15-145	5-145
$^{13}C_{12}$-4-chlorobiphenyl	3L	100	70	20-135	50-145	15-145	5-145
$^{13}C_{12}$-2,2'-dichlorobiphenyl	4L	100	70	20-135	50-145	15-145	5-145
$^{13}C_{12}$-4,4'-dichlorobiphenyl	15L	100	70	20-135	50-145	15-145	5-145
$^{13}C_{12}$-2,2',6-trichlorobiphenyl	19L	100	70	20-135	50-145	15-145	5-145
$^{13}C_{12}$-3,4,4'-trichlorobiphenyl	37L	100	70	20-135	50-145	15-145	5-145
$^{13}C_{12}$-2,2',6,6'-tetrachlorobiphenyl	54L	100	70	20-135	50-145	15-145	5-145
$^{13}C_{12}$-3,3',4,4'-tetrachlorobiphenyl	77L	100	50	45-135	50-145	40-145	10-145
$^{13}C_{12}$-3,4,4',5-tetrachlorobiphenyl	81L	100	50	45-135	50-145	40-145	10-145
$^{13}C_{12}$-2,2',4,6,6'-pentachlorobiphenyl	104L	100	50	45-135	50-145	40-145	10-145
$^{13}C_{12}$-2,3,3',4,4'-pentachlorobiphenyl	105L	100	50	45-135	50-145	40-145	10-145
$^{13}C_{12}$2,3,4,4',5-pentachlorobiphenyl -	114L	100	50	45-135	50-145	40-145	10-145
$^{13}C_{12}$-2,3',4,4',5-pentachlorobiphenyl	118L	100	50	45-135	50-145	40-145	10-145
$^{13}C_{12}$-2',3,4,4',5-pentachlorobiphenyl	123L	100	50	45-135	50-145	40-145	10-145
$^{13}C_{12}$-3,3',4,4',5-pentachlorobiphenyl	126L	100	50	45-135	50-145	40-145	10-145
$^{13}C_{12}$-2,2',4,4',6,6'-hexachlorobiphenyl	155L	100	50	45-135	50-145	40-145	10-145
$^{13}C_{12}$-2,3,3',4,4',5-hexachlorobiphenyl	156L	100	50	45-135	50-145	40-145	10-145
$^{13}C_{12}$-2,3,3',4,4',5'-hexachlorobiphenyl	157L	100	50	45-135	50-145	40-145	10-145
$^{13}C_{12}$-2,3',4,4',5,5'-hexachlorobiphenyl	167L	100	50	45-135	50-145	40-145	10-145
$^{13}C_{12}$-3,3',4,4',5,5'-hexachlorobiphenyl	169L	100	50	45-135	50-145	40-145	10-145
$^{13}C_{12}$-2,2',3,3',4,4',5-heptachlorobiphenyl	170L	100	50	45-135	50-145	40-145	10-145
$^{13}C_{12}$-2,2',3,4',5,6,6'-heptachlorobiphenyl	188L	100	50	45-135	50-145	40-145	10-145
$^{13}C_{12}$-2,3,3',4,4',5,5'-heptachlorobiphenyl	189L	100	50	45-135	50-145	40-145	10-145
$^{13}C_{12}$-2,2',3,3',5,5',6,6'-octachlorobiphenyl	202L	100	50	45-135	50-145	40-145	10-145
$^{13}C_{12}$-2,3,3',4,4',5,5',6-octachlorobiphenyl	205L	100	50	45-135	50-145	40-145	10-145

			IPR (IDOC)				
Toxic & LOC Congeners	**PCB**	**Test Conc (ng/mL) [1]**	**%RSD**	**Mean %R**	**VER %R**	**OPR (LCS) %R**	**Labeled Compound %R in Samples**
$^{13}C_{12}$-2,2',3,3',4,4',5,5',6-nonachlorobiphenyl	206L	100	50	45-135	50-145	40-145	10-145
$^{13}C_{12}$-2,2',3,3',4,5,5',6,6'-nonachlorobiphenyl	208L	100	50	45-135	50-145	40-145	10-145
$^{13}C_{12}$-2,2',3,3',4,4',5,5',6,6'-decachlorobiphenyl	209L	100	50	45-135	50-145	40-145	10-145
Surrogate Standards							
$^{13}C_{12}$-2,4,4'-trichlorobiphenyl	28L	50	70	20-135	65-135	15-145	5-145
$^{13}C_{12}$-2,3,3',5,5'-pentachlorobiphenyl	111L	50	50	45-135	75-125	40-145	10-145
$^{13}C_{12}$-2,2',3,3',5,5',6-heptachlorobiphenyl	178L	50	50	45-135	75-125	40-145	10-145
Air Surrogate Standards							
$^{13}C_{12}$-2,4'-dichlorobiphenyl	8L	50			50-150	50-150	50-150
$^{13}C_{12}$-3,3',4,5'-tetrachlorobiphenyl	79L	50			50-150	50-150	50-150
$^{13}C_{12}$-2,2',3,5',6-pentachlorobiphenyl	95L	50			50-150	50-150	50-150
$^{13}C_{12}$-2,2',4,4',5,5'-hexachlorobiphenyl	153L	50			50-150	50-150	50-150

[1] - Test concentrations are based on ng/mL in the sample extract or standard solution.

Table 11 - Retention Times of Isomers on the SPB-Octyl Column for the PCB Standard Mixes[1]

PCB Congener Mix 1 (AccustandardM-1668-1)

Cl Level	BZ No.[2]	Cl Level	BZ No.[2]	Cl Level	BZ No.[2]
1	2	4	78	6	161
2	10	4	81	6	153
2	9	5	96	6	130
2	6	5	103	6	129
2	8	5	95	6	166
2	14	5	88	6	159
2	11	5	89	6	167
3	30	5	92	6	156
3	27	5	113	7	179
3	32	5	83	7	176
3	34	5	119	7	178
3	26	5	87	7	175
3	31	5	85	7	183
3	33	5	82	7	177
3	36	5	120	7	171
3	38	5	124	7	172
3	35	5	106	7	191
4	50	5	122	7	170
4	45	5	105	7	190
4	52	5	127	8	200/201
4	49	6	152	8	204
4	75	6	136	8	199/200
4	41	6	148	8	198
4	72	6	151	8	196
4	57	6	144	8	195
4	63	6	143	8	194
4	66	6	142	9	207
4	79	6	133		

PCB Congener Mix 2 (Accustandard M-1668-2)

Cl Level	BZ No.[2]	Cl Level	BZ No.[2]	Cl Level	BZ No.[2]
2	7	4	55	6	139
2	5	4	60	6	132
2	12	5	94	6	165
3	18	5	100	6	168
3	24	5	91	6	137
3	23	5	121	6	160
3	28	5	90	6	128
3	22	5	99	6	162
3	39	5	109/108	6	157
4	53	5	117	7	184
4	51	5	111	7	186
4	73	5	108/107	7	187
4	48	5	118	7	185
4	62	5	114	7	181
4	71	6	150	7	192
4	68	6	145	8	197
4	58	6	135	8	201/199
4	61	6	149	8	203

Table 11 - Retention Times of Isomers on the SPB-Octyl Column for the PCB Standard Mixes[1] (Continued)

PCB Congener Mix 3 (Accustandard M-1668-3)

Cl Level	BZ No.[2]	Cl Level	BZ No.[2]	Cl Level	BZ No.[2]
2	13	4	80	6	140
3	17	5	93	6	146
3	29	5	84	6	141
3	20	5	101	6	164
4	46	5	112	6	158
4	65	5	86	7	182
4	59	5	116	7	174
4	40	5	107/109	7	173
4	67	6	154	7	193
4	76	6	147		

PCB Congener Mix 4 (Accustandard M-1668-4)

Cl Level	BZ No.[2]	Cl Level	BZ No.[2]	Cl Level	BZ No.[2]
3	25	4	64	5	123
3	21	4	70	6	134
4	69	5	102	6	131
4	47	5	97	6	163
4	42	5	115	7	180

PCB Congener Mix 5 (AccustandardM-1668-5)

Cl Level	BZ No.[2]	Cl Level	BZ No.[2]	Cl Level	BZ No.[2]
1	1	4	74	6	169
1	3	4	56	7	188
2	4	4	77	7	189
2	15	5	104	8	202
3	19	5	98	8	205
3	16	5	125	9	208
3	37	5	110	9	206
4	54	5	126	10	209
4	43	6	155		
4	44	6	138		

Notes:

[1] Each congener mix is analyzed in triplicate to establish the retention times of the PCB isomers in the absence of co-eluting isomers. The elution order listed here is used to assign peak identifications in the separate mixture analysis. The average retention time established in the analysis of the separate mixtures is then used to establish relative retention times. (See sections 10.2.3)

[2] BZ/IUPAC Number, if different.

Table 12: Assignment of Sample Preparation Protocols

1668 Protocol	Protocol Name	Sample Amount Extracted (g)	Fraction of Extract Cleaned	IS Added (mL)	Cleanup Std Added (mL)	Recovery Std Added (µL)	Extract Delivery Volume (µL)	Prep Split Factor	Prep Dilution Factor	QuantIMS Final Volume
P1	Clean	10	1/2	1	0.5 (added aftersplit)	50	50	2	1	100
P2	Low	2	1	1	1	100	100	1	1	100
P3	Medium	1.25	1/2	2	1 (added after split)	100	250	2	2.5	200
P4	High	1	1/4	4	1 (added after split)	100	500	4	5	400

Figure 1 - Recommended GC Operating Conditions

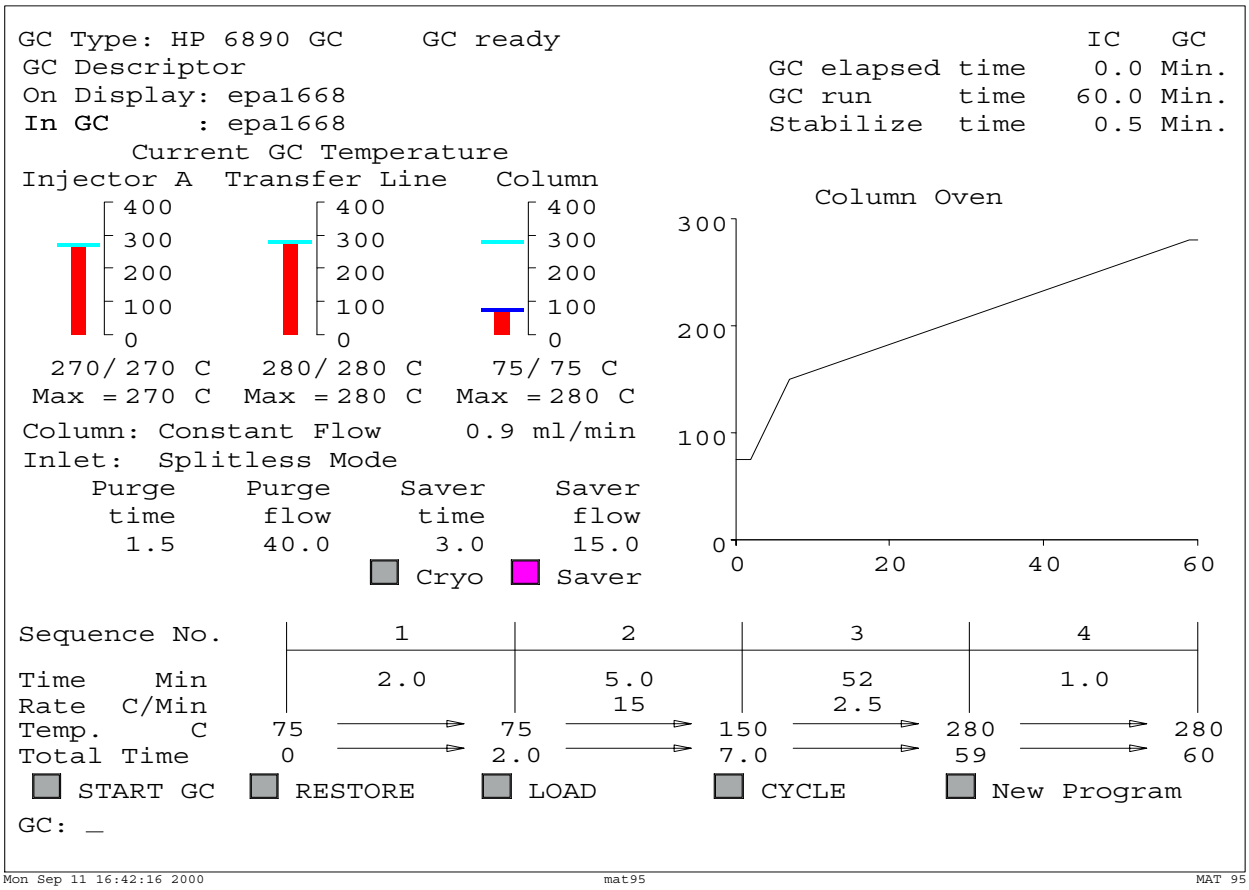
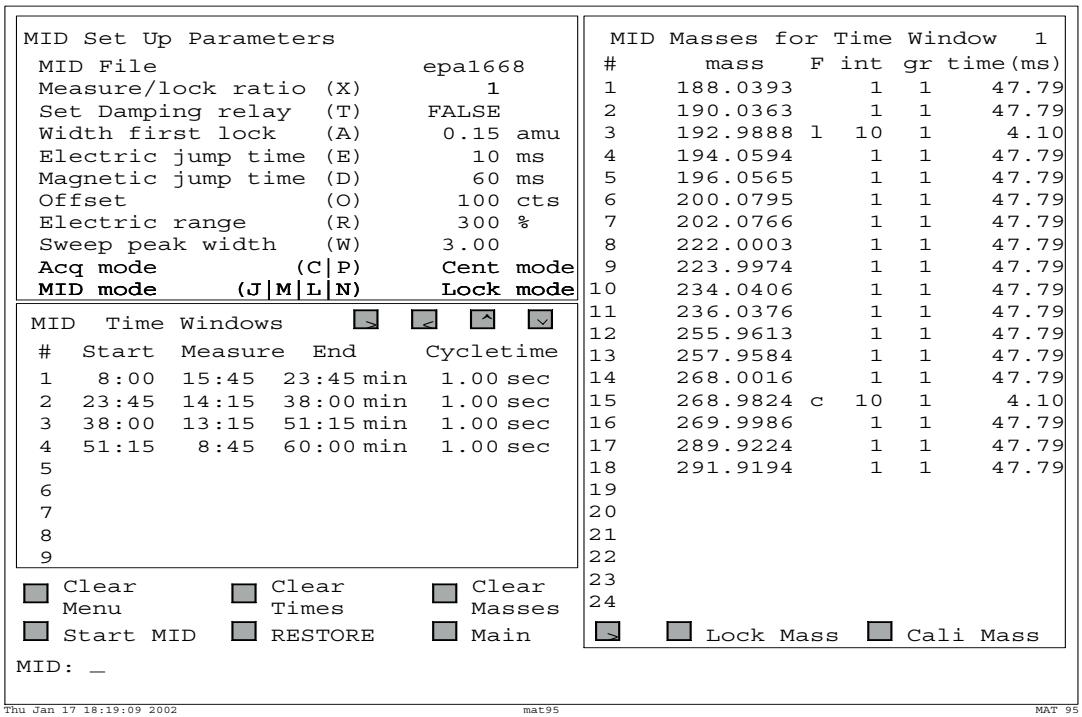


Figure 2 - Recommended MID Descriptors



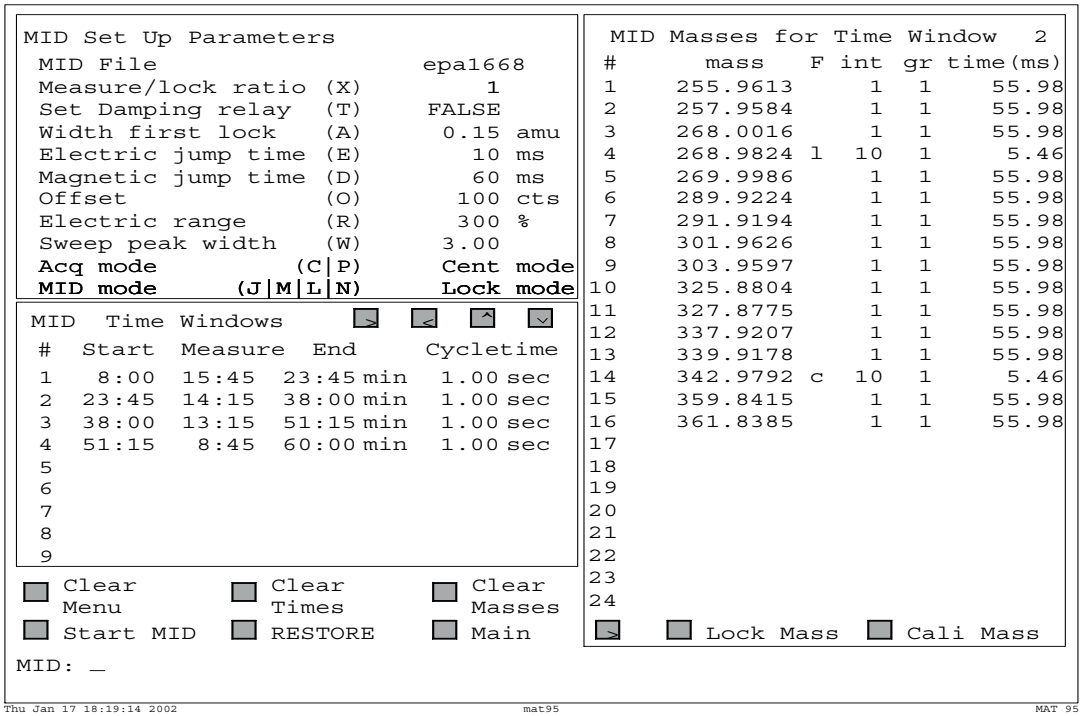
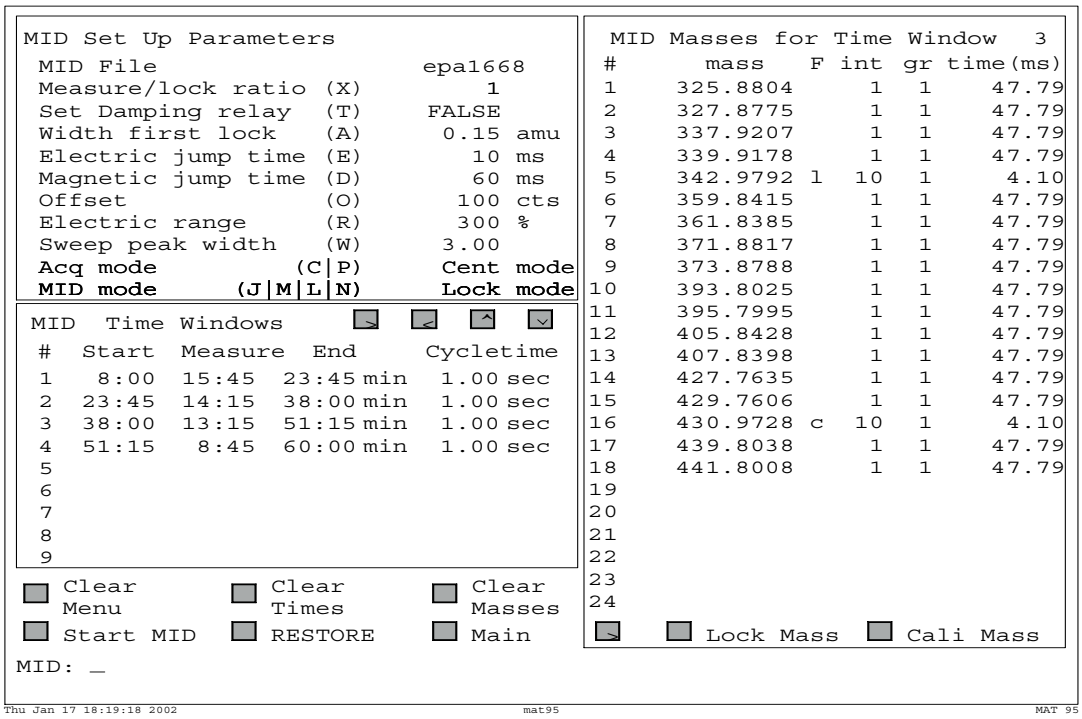


Figure 2 - Recommended MID Descriptors (Continued)



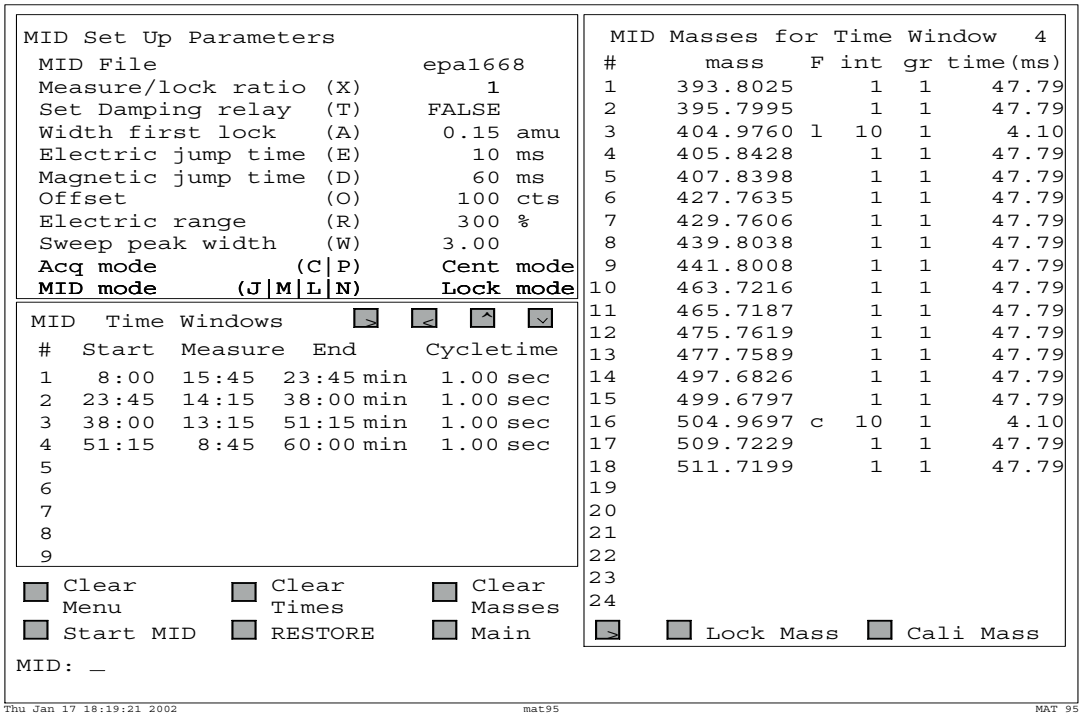


Figure 3 - Example Data Review Checklist

Eurofins/TestAmerica Knoxville Specialty Organics Group GC/MS Initial Calibration Data Review Checklist
Method 1668 or SOP Number: KNOX-ID-0013 Revision 19

Mass Res Date/Time:		Inst:		2nd Source Filename:	
ADII Batch #(s):		WL #:		Job #:	

CS0.5 Filename	CS1 Filename	CS2 Filename	CS3 Filename	CS4 Filename	CS5 Filename

Review Items	N/A	Yes	No	If No, why is data reportable?	2nd √
1. Was the mass resolution documented before beginning the initial calibration?					
2. Was the instrument resolution ≥8,000 throughout (≥ 10,000 for m/z 342.9792, PFK) and ≥10,000 in the center of each m/z range for the PFK masses or FC43 masses?					
3. Were the measured exact masses listed above within 5 ppm at reduced accelerating voltage?					
4. Have PCB Mixes 1 - 5 been analyzed using the installed column to assign congener retention times, method retention times, and MID switch points?					
5. Were the calibration standard solutions, at the number and concentrations specified in the SOP, analyzed?					
6. Was date/time of analysis verified as correct?					
7. Was the valley height less than 40% of the height of the shorter of the two peaks for the pair PCB 23 and PCB 34, and the pair PCB 182 and PCB 187 in the CS3 standard?					
8. Did the PCB co-elution 156/157 co-max within 2 seconds at peak maximum on the SPB-octyl?					
9. Was the absolute retention time of PCB 209 greater than 55 minutes in the CS3 standard?					
10. Were the response factors calculated for each labeled standard and unlabeled native analyte using the SOP specified reference compound (Table 2), quantitation ions (Table 8), and formula (10.3.4.2)?					
11. Is the %RSD acceptable for all native analytes (within ± 20% calculated by IDAs, and within ± 35% when not calculated by IDAs)?					
12. Is the %RSD acceptable (within ± 35%) for all labeled standards?					
13. Are all S/N ratios ≥10 for the GC signals in each EICP (extracted ion chromatographic profile) including IDAs? (Exception: Secondary native dichloro biphenyl channel m/z 223.9974, PFK)					
14. Are the ion abundance ratios for all native Toxics/LOCs and all labeled compounds within the control limits specified? (Exception: Native dichlorobiphenyls, PFK) (Table 9)					
15. Were all toxic congeners uniquely resolved from non-toxic congeners?					
16. Was an ICV analyzed and calculated according to Section 10.3.5 of the SOP?				< 5 outliers, none more than ±50% D.	
17. If manual integrations were performed, are the analyst's name, reason and date noted in AD II?					
18. If criteria were not met, was a NCM generated?					
19. Do the ICAL AD II batches contain a completed checklist for this work list?					

Analyst:	Date:
Comments:	
2nd Level Reviewer :	Date:
Comments:	

Figure 3- Example Data Review Checklist (Continued)

Eurofins/TestAmerica Knoxville Specialty Organics Group GC/MS Continuing Calibration Data Review Checklist
Method 1668 or SOP Number: KNOX-ID-0013 Revision 19

Start Mass Res:		WL #:		CS3 Filename:		Inst/ Date:	Job #'s:
End Mass Res:		AD II Batches:				ICAL Event#	

Review Items	N/A	Yes	No	If No, why is data reportable?	2nd Level
1. Was the mass resolution documented at both the beginning and end of the 12 hour shift and is data verified as within the 12 hour clock?					
2. Were all graphics uploaded to AD II?					
3. Was the mass resolution scanned and attached to the corresponding WDMCCV?					
4. Was the instrument resolution ≥8,000 throughout (≥ 10,000 for m/z 342.9792) and ≥10,000 in the center of each m/z range for the PFK masses as listed in the SOP or ≥10,000 in the center of each m/z range for the FC43 masses as listed in the SOP.					
5. Were the measured exact masses listed above within 5 ppm at reduced accelerating voltage?					
6. Were the date and time of analysis verified as correct?					
7. Were the MID switch points set to encompass the retention time windows of each congener group?					
8. Was the valley height less than 40% of the height of the shorter of the two peaks for the pair PCB 23 and PCB 34, and the pair PCB 182 and PCB 187?					
9. Did the PCB co-elution 156/157 co-max within 2 sec at peak max on the SPB-octyl?					
10. Was the continuing calibration performed at the beginning of the 12 hour period after successful mass resolution and GC resolution performance check?					
11. Was the %D for all Toxic analytes within ± 30% for 1668A/B and ± 25% for 1668C? (PCB 81, 77, 123, 118, 114, 105, 126, 167, 156, 157, 169, 189) Was the %D for all LOC analytes within ± 30% for 1668A/B and ± 25% for 1668C? (PCB 1, 3, 4, 15, 19, 37, 54, 104, 155, 188, 202, 205, 206, 208, 209)					
12. Was the %D for all non-toxic/non-LOC analytes within ± 30% (for all versions of 1668)?					
13. Were the response factors calculated for each labeled standard and unlabeled target analyte using the SOP specified reference compound (Table 2), quantitation ions (Table 8), and formula (10.3.4.2)?					
14. Were the absolute retention times of all labeled IDAs within ± 15 seconds of the retention times obtained during initial calibration?					
15. Are %D within ± 50% for all labeled IDAs (for 1668A/B) or -50/+45% (for 1668C) in the calibration?					
16. Are the %D within ± 50% for all labeled field surrogates (for all versions of 1668) in the calibration?					
17. Are the %D within -40/+30% (for 1668A/B) or ± 25% (for 1668C) for all labeled surrogates in the calibration? **Note: for 1668C, PCB28L's lower limit can extend to -35%D.**					
18. Are all S/N ratios ≥10 for the GC signals in each EICP (extracted ion chromatographic profile) including internal standards?					
19. Are RRTs of all unlabled toxic/LOC analytes within their respective RRT limits?					
20. If manual integrations were performed, are they clearly identified in the AD II batch with the analyst, date and reason?					
21. If criteria were not met, was a NCM generated?					
22. Do the AD II batches contain a completed checklist for this work list?					

Analyst:	**Date:**
Comments:	
2nd Level Reviewer :	**Date:**
Comments:	

Figure 3 - Example Data Review Checklist (Continued)

Eurofins/TestAmerica Knoxville PCB Batch Data Review Checklist
Method 1668 - KNOX-ID-0013-R19

WL #:_____
ADII Batch #(s): _____
Job #(s):_____
_____

Review Items	N/A	Yes	No	Why is data reportable?	2nd √
1. Was the correct ICAL used for quantitation? (Check the ICAL event number in every sample and CCV.)				**ICAL Event #**_____	
2. Have the appropriate checklists been completed for the Work List?					
3. Were all special project requirements met (checked in backlog report and in AD II)?					
4. DoD requirements met?				☐ NCM#140-11724: Add to Case Narrative if Manual Integrations Performed ☐ Narrate reasons for multiple analyses of samples	
5. Were the prep factors and dilution factors verified in AD II?				☐ **Dilution-Respike IDA** (NCM#_____)	
6. Sample analyses done within preparation and analytical holding time (Check for H-flag in sample result in AD II)?				☐ **Holding Time-Initial Analysis** (NCM#_____) ☐ **Holding Time-Reanalysis** (NCM#_____)	
7. Are IDAs, surrogates and field surrogates (if applicable) within QC limits?				☐ **IDA-Low-S/N 10:1** (NCM#_____) ☐ **IDA-High-Isotope Dilution** (NCM#_____)	
8. Are IDAs, surrogates and field surrogate (if applicable) ion abundance ratios within limits?				☐ (NCM#_____)	
9. Were peaks ≥2.5 S/N, which did not meet one or more of the criteria listed in section 12.1 of the SOP calculated and reported as EMPCs?					
10. Are positive results within calibration range?				☐ **ICAL-Range Exceed;No Sat.** (NCM#_____)	
11. Are all non-detects that are G-qualified narrated?				☐ (NCM#_____)	
12. Are all manual integrations documented with analyst ID, reason and date in AD II?					
13. Are all graphics uploaded to AD II?					
14. Final report acceptable (1. Job Data Review was checked and all CCV's, QC, and samples are turned to 2nd level, 2. The narrative was checked in Supervisor Desktop for all deviations and grammar errors, and 3. All QC links were verified and at least one sample from every job is linked to the ICAL)?					
15. LCS done per prep batch and all LCS/LCSD recoveries and RPDs within QC limits?				☐ **LCS/LCSD-%R High** (NCM#_____) ☐ **LCS/LCSD-Insuff. Sample** (NCM#_____)	
16. Method blank done per prep batch and method blank or instrument blank analyzed with each sequence?					
17. Are all analytes present in the method blank ≤ EML or within the specific program requirements?				☐ **Method Blank-Report, 10X** (NCM#_____) ☐ **Method Blank-Report ND** (NCM#_____) ☐ **Method Blank-Insuff. Sample** NCM#_____)	

1st Level Reviewed by:	**Date:**
Comments:	
2nd Level Reviewed by:	**Date:**
Comments:	

Appendix I - SPB-Octyl Guard Column Installation and Conditioning

1. Guard column installation on SPB-Octyl

- SPB-Octyl is 30M x 0.25mm ID x 0.25 uM film thickness (Supelco # 24218-U)

- Guard column is 10M non-polar fused silica, 0.25mm ID. This can be purchased in a 60M length and cut down to 10M as needed (Supelco # 25783). The measuring/cutting process is a chore and usually requires an assistant. Supelco does not offer 0.25mm ID non polar fused silica in a 10M length. Restek also does not offer a 10M pre-cut length.

- Butt connector with ferrule – Supelco # 23796
- Butt connector ferrules – Supelco # 23797 (pack of 2)

- The attachment of the guard column to the column via the butt connector and ferrule is a bit delicate. It can be learned by reading the instructions or it can be demonstrated. Upon GC installation, the butt connector threading usually loosens up a bit. This generally should not cause the ferrule to leak, but it is a good idea to snug up the threads a couple times until the threads no longer come loose.

2. SPB-Octyl column conditioning

- As purchased, the SPB-Octyl is unsuitable for installation into an ion source. The column requires an extensive conditioning before use. Even with the conditioning, at our upper run temperature of 270°C, a new column will bleed extensively on the back end of the mass 181 tuning peak, but this bleed will decrease over time.

- To condition an SPB-Octyl column, it should be installed in a GC at ambient temperature with gas flowing to allow sufficient time (about 1 hour) for oxygen to purge from the system. It is then heated to 270°C for at least 18 hours with the column flowing into the oven to avoid trashing the source. **24 hours is preferable.** Set the GC mode to splitless, constant flow. Enter the column dimensions as follows: Length: 60m, ID=250nm, film thickness=0.250um. Flow rate should be 1.5mL/min (in an HP6890 GC, with a 10m guard column and a 30m spb-octyl, this should give a head pressure reading of ~ 47psi when the oven has reached 270 C). Check that you have flow through the column by immersing the source end into a beaker of methanol and watch for bubbles. They should come out of the source end very rapidly if you have installed everything correctly. This conditioning time will bring the SPB-Octyl column to a state where the operator can install it into a source and starting making test runs to establish the GC length configuration that will elute decachlorobiphenyl at 56:00 minutes.

STANDARD OPERATING PROCEDURE

TITLE: Extraction of Polychlorinated Biphenyl (PCB) Isomers for Analysis by Isotope Dilution HRGC/HRMS (EPA Methods 1668A, 1668B and 1668C)

(SUPERSEDES: KNOX-OP-0021, Revision 9)

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1. Scope and Application

1.1 This procedure is used by Eurofins TestAmerica Knoxville for the extraction of PCB isomers from a variety of environmental matrices. This procedure is based on EPA methods 1668A, 1668B and 1668C, and is designed to meet analytical program requirements where isotope dilution HRGC/HRMS analysis of polychlorinated biphenyl (PCB) isomers is specified.

1.2 This procedure is based on "performance-based" methods. These reference methods allow modifications to overcome interferences or lower the cost of measurements if all performance criteria in the methods are met and method equivalency is established. Modifications and enhancements to the referenced methods have been incorporated into this procedure and are listed in section 17.1.

1.3 Because of the extreme toxicity of many of these compounds, the analyst must take the necessary precautions to prevent exposure to materials known or believed to contain PCBs. It is the responsibility of all laboratory personnel to ensure that safe handling procedures are employed. Section 5 of this procedure discusses safety procedures.

2. Summary of Method

2.1 Screening and protocol assignment

2.1.1 All solid, sediment, waste and tissue samples are screened by GC/ECD prior to extraction. Aqueous samples may be screened if the potential for congener levels is suspected to be above 40 ng/L. Variations to sample size, spiking levels and final volume are established based on the screening result. Refer to Table 5.

2.2 Extraction

2.2.1 Aqueous samples (samples containing less than one percent solids): Stable isotopically labeled analogs of the toxic PCBs plus additional labeled PCBs (i.e., $^{13}C_{12}$ labeled isotope dilution analytes) are typically spiked into a 1-L sample, and the sample is extracted with methylene chloride using separatory funnel techniques.

2.2.2 Solid and sediment samples: A 1.0 g sub-sample is screened to determine the greatest concentration for individual congeners. Based on the screen results, the sample is prepared by one of four protocols (refer to Table 5). Each protocol defines the sample amount to be extracted, the fraction of the extract to be used, and the final extract volume. Protocol 1 is for samples which can be processed without adjustments for high levels of PCB congeners. In this protocol, the isotope dilution analytes are spiked into a sample containing 10 g of solids. The sample is extracted for 18-24 hours with 1:1 hexane/acetone using a Soxhlet extractor. The extract is concentrated for cleanup.

NOTE: Sample sizes may be adjusted for dry weight or processed as received. The laboratory default for soil samples is to process as received. The laboratory default for sediment samples is to adjust the sample amount extracted to a minimum dry weight.

2.2.3 Multi-phase samples: Samples containing multiple phases are separated and the phases are extracted following the procedures for the appropriate matrix. The extracts may be combined for cleanup and analysis or processed separately. Specific handling of multi-phase samples should be discussed and documented with the project manager prior to extraction of samples.

2.2.4 Tissue samples: A 1.0 g sub-sample may be screened to determine the greatest concentration for individual congeners. Based on the screen results, the sample is prepared by one of four protocols (refer to Table 5). Each protocol defines the sample amount to be extracted, the fraction of the extract to be used, and the final extract volume. Protocol 1 is for samples which can be processed without adjustments for high levels of PCB congeners. In this protocol, a 10 g aliquot of homogenized tissue is blended with sodium sulfate, spiked with the isotope dilution analytes, and extracted for 18-24 hours in a Soxhlet extractor using 1:1 hexane/acetone. If required, a portion of the extract is used to determine the lipid content. (Refer to SOP KNOX-OP-0020, “Gravimetric Percent Lipids Determination” current revision.) The remaining extract is concentrated for cleanup.

2.2.5 Waste samples: Non-aqueous liquids such as oils and organic solvents are diluted or solvent exchanged in hexane.

2.2.6 Refer to SOP KNOX-OP-0010, current revision, for extraction of source air and ambient air samples.

2.3 After extraction, samples may be cleaned up using back-extraction with sulfuric acid, gel-permeation chromatography and silica gel chromatography. Cleanup surrogates are added to each extract prior to initiating any cleanup steps.

2.4 After cleanup, the extract is concentrated to the appropriate final volume (refer to Table 5). Internal standards are added to each extract and extracts are delivered to the HRMS lab for analysis. Refer to SOP KNOX-ID-0013, current revision.

3. Definitions

NOTE: Terminology differences existing in some isotope dilution reference methods regarding the functionality of the labeled analogs may lead to confusion. For example, EPA's Office of Solid Waste methods (8280, 8290) use the term "Internal Standards" to describe the labeled analogs which are added to the sample prior to extraction and used to quantitate the native targets. EPA's Office of Water methods (1613B, 1668, Revision A) use the term "Labeled Analogs" to describe these same compounds while using the term "Internal Standards" to describe the labeled analogs which are added to the extract just prior to analysis and used to quantitate the recovery of the labeled analogs added before extraction. EPA's Office of Solid Waste methods (8280, 8290) uses the term "Recovery

Standards" to describe these later labeled analogs.

The terminology listed below is used in the laboratory for all Standard Operating Procedures and internal communications.

3.1 Isotope dilution analytes (IDAs): Isotopically labeled analogs of the target analytes that are added to every sample, blank, quality control spike sample, and calibration solution. They are added to the sample before extraction and are used to calculate the concentration of the target analytes or detection limits.

3.2 Cleanup surrogates (SU): Isotopically labeled compounds that are added to samples, blanks, quality control samples, and calibration solutions. They are added to the samples after extraction but prior to extract cleanup, and are used to judge the efficiency of the cleanup procedures.

3.3 Internal standards (IS): Isotopically labeled compounds which are added to every sample, blank, and quality control spike sample extract prior to analysis. They are used to measure the recovery of the IDAs and the cleanup surrogates.

3.4 Additional definitions can be found in the Eurofins Knoxville Quality Assurance Manual.

4. Interferences

4.1 Solvents, reagents, glassware, and other sample processing hardware may yield artifacts and/or elevated baselines causing misinterpretation of chromatograms. Specific selection of reagents and solvents may be required. Where possible, reagents are cleaned by extraction or solvent rinse. The non-coplanar PCB congeners 105, 114, 118, 123, 156, 157, 167, and 180 have been shown to be very difficult to completely eliminate from the laboratory at the minimum levels in this method, and baking of glassware in a kiln or furnace at 450 - 500°C may be necessary to remove these and other contaminants.

4.2 Proper cleaning of glassware is extremely important because glassware may not only contaminate the samples but may also remove the analytes of interest by adsorption on the glass surface. For specific glassware cleaning procedures, see SOP KNOX-QA-0002, “Glassware Cleaning”, current revision.

4.3 All materials used in the analysis shall be demonstrated to be free from interferences by preparing laboratory method blanks (section 9.5) with each sample batch.

4.4 Interferences co-extracted from samples will vary considerably from source to source, depending on the diversity of the site being sampled. Interfering compounds may be present at concentrations several orders of magnitude higher than the PCBs. The most frequently encountered interferences are chlorinated dioxins and dibenzofurans, methoxy biphenyls, hydroxy-diphenyl ethers, benzylphenyl ethers, polynuclear aromatics and pesticides. Because very low levels of PCBs are measured by this method, the elimination of interferences is essential. The cleanup steps given in section 11.9 can be used to reduce or eliminate these interferences and

thereby permit reliable determination of the PCBs at the levels required by the method.

4.5 Cleanup of tissue - The natural lipid content of tissue can interfere in the analysis of tissue samples for PCBs. The lipid contents of different species and portions of tissue can vary widely. Lipids are soluble to varying degrees in organic solvents and may be present in sufficient quantity to overwhelm the column chromatographic cleanup procedures used for sample extracts. Lipids must be removed by the sulfuric acid cleanup procedure (section 11.9.5) followed by GPC cleanup (section 11.9.6).

5. Safety

5.1 Employees must abide by the policies and procedures in the Corporate Safety Manual, Radiation Safety Manual and this document.

5.2 Eye protection that satisfies ANSI Z87.1 (as per the Corporate Safety Manual), laboratory coat and appropriate gloves must be worn while samples, standards, solvents and reagents are being handled. Disposable gloves that have become contaminated will be removed and discarded; other gloves will be cleaned immediately.

5.3 Latex and vinyl gloves provide no protection against most of the organic solvents used in this method. For the operations described herein, Nitrile gloves are to be worn. For operations using solvents that may splash, SilverShield® gloves are recommended. SilverShield® gloves protect against breakthrough for most of the solvents used in this procedure.

5.4 When using a scalpel, wear cut-resistant gloves and cut away from your hand.

5.5 Primary Materials Used: The following is a list of the materials used in this method which have a serious or significant hazard rating. **NOTE: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the SDS for each of the materials listed in the table.** A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the SDS for each material before using it for the first time or when there are major changes to the SDS.

Material	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Sulfuric Acid (1)	Corrosive, Oxidizer, Dehydrator	1 mg/m^3	This material will cause burns if comes into contact with the skin or eyes. Inhalation of vapors will cause irritation of the nasal and respiratory system.

Material	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Methylene Chloride	Carcinogen, Irritant	25 ppm-TWA, 125 ppm-STEL	Causes irritation to respiratory tract. Has a strong narcotic effect with symptoms of mental confusion, light-headedness, fatigue, nausea, vomiting and headache. Causes irritation, redness and pain to the skin and eyes. Prolonged contact can cause burns. Liquid degreases the skin. May be absorbed through skin.
Hexane	Flammable, Irritant	500 ppm-TWA	Inhalation of vapors irritates the respiratory tract. Overexposure may cause lightheadedness, nausea, headache, and blurred vision. Vapors may cause irritation to the skin and eyes.
Methanol	Flammable, Poison, Irritant	200 ppm-TWA	A slight irritant to the mucous membranes. Toxic effects exerted upon nervous system, particularly the optic nerve. Symptoms of overexposure may include headache, drowsiness and dizziness. Methyl alcohol is a defatting agent and may cause skin to become dry and cracked. Skin absorption can occur; symptoms may parallel inhalation exposure. Irritant to the eyes.
Toluene	Flammable, Poison, Irritant	200 ppm-TWA, 300 ppm-Ceiling	Inhalation may cause irritation of the upper respiratory tract. Symptoms of overexposure may include fatigue, confusion, headache, dizziness and drowsiness. Peculiar skin sensations (e. g. pins and needles) or numbness may be produced. Causes severe eye and skin irritation with redness and pain. May be absorbed through the skin.
Acetone	Flammable	1000 ppm-TWA	Inhalation of vapors irritates the respiratory tract. May cause coughing, dizziness, dullness, and headache.
Nonane	Flammable	None established	Harmful if inhaled/swallowed. Vapor/mist is irritating to eyes, mucous membranes and upper respiratory tract. Causes skin irritation.
Benzene	Flammable, Toxic, Carcinogen	PEL: 1 ppm TWA ; 5 ppm, 15 min. STEL	Causes skin irritation. Toxic if absorbed through skin. Causes severe eye irritation. Toxic if inhaled. Vapor or mist causes irritation to mucous membranes and upper respiratory tract. Exposure can cause narcotic effect. Inhalation at high concentrations may have an initial stimulatory effect on the central nervous system characterized by exhilaration, nervous excitation and/or giddiness, depression, drowsiness or fatigue. Victim may experience tightness in the chest, breathlessness, and loss of consciousness.
1 – Always add acid to water to prevent violent reactions. 2 – Exposure limit refers to the OSHA regulatory exposure limit.			

5.6 Chemicals that have been classified as **carcinogens**, **potential carcinogens**, or **mutagens include** benzene, methylene chloride, polychlorinated biphenyls, and toluene. The toxicity or carcinogenicity of each reagent used in this method is not precisely defined; however, each chemical compound should be treated as a potential health hazard. From this viewpoint, exposure to these chemicals must be kept to a minimum.

5.7 Chemicals known to be **flammable** include acetone, benzene, hexane, nonane, and toluene.

5.8 The following material is known to be **corrosive**: Sulfuric acid.

5.9 Exposure to chemicals will be maintained as low as reasonably achievable; therefore, unless they are known to be non-hazardous, all samples will be opened, transferred and prepared in a fume hood or under other means of mechanical ventilation. Solvent and waste containers will be kept closed unless transfers are being made.

5.10 All procedures that involve solvents such as acetone, toluene, methylene chloride and hexane (e.g., glassware cleaning and the preparation of standards and reagents) must be conducted in a fume hood with the sash closed as far as the operations permit.

5.11 All work must be stopped in the event of a known or potential compromise to the health or safety of laboratory personnel. The situation must be reported immediately to a laboratory supervisor.

6. Equipment and Supplies

NOTE: All glassware used in extraction and cleanup procedures is pre-cleaned as described in SOP KNOX-QA-0002, “Glassware Cleaning”, current revision. When extracting PCB samples, all of the reusable glassware used in the extraction and concentration process is identified with a "P" (for aqueous sample glassware), “L” or “LL” (for solid sample glassware) etched mark. All glassware used must be low level glassware.

6.1 Miscellaneous Laboratory Equipment

6.1.1 Laboratory fume hood of sufficient size to contain the equipment used for sample preparation

6.1.2 Oven, capable of maintaining a temperature of 125 to 135°C

6.1.3 Balance, >100 g capacity, accurate to $\pm$ 0.01 g

6.1.4 Balance, >2000 g capacity, accurate to $\pm$ 0.1 g

6.1.5 Syringes, various sizes

6.1.6 Borosilicate 5.75 inch and 9.0 inch disposable pipettes with latex bulbs

6.1.7 Glass pipets, 1 mL, Class A

6.1.8 Graduated cylinders, 100 mL and 1000 mL capacity

6.1.9 PTFE squirt bottles, 500 mL

6.1.10 250 mL and 500 mL flasks

6.1.11 Ultra-Pure PTFE boiling stones

6.1.12 Glass wool, solvent rinsed

6.2 Tissue Homogenization Equipment:

6.2.1 Laboratory blender with glass body and stainless steel blades

6.2.2 Industrial meat grinder, Intedge Industries, Model C2H, or equivalent

6.2.3 Laboratory homogenizer, OMNI GLH-01, Model LR060902, or equivalent

6.2.4 Scalpels or knives

6.2.5 Cut-resistant gloves

6.3 Aqueous Sample Extraction Equipment

6.3.1 Separatory funnels, 250, 500, and 2000 mL, with PTFE stopcocks

6.3.2 Separatory funnel rotator

6.4 Solid/Tissue Sample Extraction Equipment

6.4.1 Soxhlet extractors, 50-mm ID, 200-mL capacity with 500 mL flasks

6.4.2 Glass condensers, capable of fitting top of Soxhlet apparatus

6.4.3 Whatman high purity glass fiber extraction thimbles

6.4.4 Heating mantles with temperature control

6.4.5 Flasks, Erlenmeyer, 500 mL

6.4.6 Beakers, 500 mL

6.4.7 Spatulas, stainless steel

6.5 Filtration Equipment

6.5.1 Glass or stainless steel funnels

6.5.2 Phase separation paper, Glass fiber filters

6.5.3 Buchner funnel, 15 cm

6.5.4 Glass-fiber filters for Buchner funnel

6.5.5 Filtration flasks, 1.5 to 2.0 L, with side arm

6.6 Cleanup Equipment

6.6.1 Disposable pipets, 150 mm long x 5 mm ID

6.6.2 Disposable pipets, 230 mm long x 5 mm ID

6.6.3 20 mm x 240 mm glass columns with support ring and tapered tip, for silica gel cleanup

6.6.4 Vortex mixer

6.6.5 Gel Permeation chromatography system

6.7 Concentration Equipment

6.7.1 Nitrogen blowdown apparatus, N-EVAP (Organomation Associates, Inc., South Berlin, MA), installed in a fume hood

6.7.2 Kuderna-Danish (KD) Apparatus - 500 mL

6.7.3 Concentrator Tube, 10 mL, attached to KD with clips

6.7.4 Snyder Column, three-ball macro

6.7.5 Water Bath, heated, with concentric ring cover, capable of temperature control (± 5°C) up to 95°C. The bath must be used in a hood or with a solvent recovery system.

6.7.6 Heating mantles with temperature control

6.8 Sample Vials

6.8.1 Borosilicate glass, 12 mL and 40 mL disposable with PTFE caps

6.8.2 Mini-vials, 1.1 mL capacity with a tapered bottom, with PTFE, rubber septa and screw caps

6.8.3 Amber glass vials with PTFE-lined screw caps

6.9 Screening Equipment

6.9.1 Shaker table

6.9.2 An analytical system complete with a gas chromatograph and a ^{63}Ni electron capture detector and a data system capable of measuring peak height.

7. Reagents and Standards

CAUTION: Refer to Safety Data Sheets (SDS) for specific safety information on chemicals and reagents prior to use or as needed

CAUTION: During preparation of reagents, associates shall wear a lab coat, gloves, safety glasses with side shields, and laboratory approved shoes as a minimum. Reagents shall be prepared in a fume hood.

7.1 Pre-cleaned glass wool - Pre-clean the glass wool with methylene chloride in a Soxhlet extractor for a minimum of 4 hours.

7.2 Sulfuric acid, concentrated, reagent grade (specific gravity 1.84)

7.3 Sodium chloride, reagent grade - Prepare a 5% (w/v) solution in reagent water.

7.4 Sodium sulfate, reagent grade, anhydrous, J.T Baker 3375, or equivalent

7.4.1 Sodium sulfate may be cleaned by putting approximately 600 g of sodium sulfate in large amber-colored glass jars and completely covering with

methylene chloride and letting the sodium sulfate soak for 5 minutes. The methylene chloride is drained and this step is repeated. After the methylene chloride is drained, the sodium sulfate is transferred to a Buchner funnel fitted onto a vacuum flask and rinsed 2 times with methylene chloride while a vacuum is being applied. The sodium sulfate is then placed into shallow borosilicate glass dishes where it is allowed to dry. It is placed in an oven at 125-130°C for 1 hour to complete the drying process. After drying, the sodium sulfate is transferred into pre-cleaned glass jars with PTFE lined screw caps.

7.5 Purified nitrogen

7.6 Solvents - Acetone, toluene, n-hexane, methanol, methylene chloride, benzene and nonane; pesticide quality

7.7 Reagent water must be produced by a Millipore DI system, or equivalent, that is capable of producing water with ≥18 megohm-cm (MΩ-cm) resistivity. Reagent water must be free of the analytes of interest as demonstrated through the analysis of method blanks.

7.8 Silica gel, S679-212, Fisher Chromatographic Silica Gel, 100-200 mesh or equivalent - Prepare by Soxhlet extraction with methylene chloride for at least 6 hours. Transfer to a shallow, borosilicate glass dish and air dry. After drying, cover with aluminum foil and activate in an oven at 125-135°C for a minimum of 4 hours. Store in labeled glass jars in a desiccator until use.

7.9 3.3% Deactivated silica gel – To prepare, add 6.6 mL of reagent water to 200 g of pre-cleaned silica gel (section 7.8) in a 500 mL amber glass jar with a PTFE lined screw cap. Mix thoroughly by shaking until no lumps are visible, and the silica gel is free flowing and no longer sticks to the side of the jar. The Tornado II portable paint shaker may be used to aid in mixing these reagents. Store in a desiccator. Shake well again before each use.

7.10 Acidic silica gel – To prepare, add 57 mL of concentrated sulfuric acid to 180 g of pre-cleaned silica gel (section 7.8) in a 500 mL amber glass jar with a PTFE lined screw cap. Mix thoroughly by shaking until no lumps are visible and the silica gel is free flowing and no longer sticks to the side of the jar. The Tornado II portable paint shaker may be used to aid in mixing these reagents. Store in a desiccator. Shake well again before each use.

7.11 8082 Surrogate Mix: A surrogate spiking solution which contains 0.2 mg/L tetrachloro-m-xylene and decachlorobiphenyl. This mix is spiked into samples that are being prepared for PCB screening.

7.12 $^{13}C_{12}$ Labeled PCB Congener Standards: Obtained from Cambridge Isotope Laboratories (CIL, Andover Massachusetts) and Wellington Laboratories (Guelph, Ontario, Canada). Refer to Tables 2 and 3 for a list of individual standards. If the chemical purity is 98% or greater, the weight may be used without correction to compute the concentration of the standard. Once a standard ampoule has been

vortexed and opened, the solution is transferred to an amber glass vial with a PTFE lined screw cap. When not being used, standards are stored in the dark in a refrigerator. These purchased individual standards are used to prepare the following mixed stock solutions and spiking solutions:

7.12.1 Isotope Dilution Analyte (IDA) Stock Solution: Prepared by adding 0.625 mL of the 40,000 ng/mL $^{13}C_{12}$ PCB 170 standard to 25 mL of the 1000 ng/mL 1668 $^{13}C_{12}$ PCB mix from Cambridge Isotope Laboratories. Refer to Table 2. The concentration is verified by GC/MS before use.

NOTE: Blow down the 625 µL of PCB 170 to approximately 150-200 µL on NEVAP before adding to the 25 mL volumetric flask.

7.12.2 IDA Spiking Solutions: They are prepared by diluting the 1000 ng/mL IDA stock solution to a concentration of 10 ng/mL in acetone in a 200 mL volumetric. 0.200 to 4.0 mL of the 10 ng/mL solution is added to each sample prior to extraction. 0.200 mL is added to each aqueous sample extract. Refer to Table 5, "Assignment of Sample Preparation Protocols" to determine the exact volume to add to solid samples.

7.12.3 Internal standard Stock Solution: Cambridge Part# EC-5595 labeled internal standards listed in Table 3 at a concentration of 1000 ng/mL in nonane. The concentration is verified by GC/MS before use.

7.12.4 Internal standard Spiking Solution: Prepared by diluting the 1000 ng/mL internal standard stock solution to a concentration of 100 ng/mL in nonane in a 25 mL volumetric. 100 µL of this spiking solution is added to each solid, tissue or waste sample extract prior to analysis, whereas, 20 µL is added to each aqueous sample extract.

7.12.5 Cleanup Surrogate Stock Solution: Cambridge Part# EC-4978-5X labeled cleanup surrogates listed in Table 3, at a concentration of 5000 ng/mL in nonane. The concentration is verified by GC/MS before use.

7.12.6 Cleanup Surrogate Spiking Solution: Prepared by diluting the 5000 ng/mL cleanup surrogate stock solution to a concentration of 10 ng/mL in hexane. 0.5 mL or 1.0 mL of this solution is added to each solid/tissue sample extract prior to cleanup (refer to Table 5), whereas, 0.20 mL is added to each aqueous sample extract.

7.12.7 Field Surrogate Stock Solution: Prepare by adding 0.3 mL of ($^{13}C_{12}$ PCB 95) 50,000 ng/mL of Wellington MBP-95 to 3.0 mL of 5000 ng/mL Cambridge EC-5596 to a concentration of 5000 ng/mL in nonane.

7.12.8 Field Surrogate Spiking Solution: Prepare by diluting 5000 ng/mL stock solution to a concentration of 10 ng/mL in hexane.

7.13 Target Analyte PCB Congener Standard Mix - Obtained as a Certified Reference Standard from Accustandard (New Haven, CT). This standard contains all 209 PCB congeners at 4000 ng/mL in nonane. If the chemical purity is 98% or greater, the

weight may be used without correction to compute the concentration of the standard. Once a standard ampoule has been vortexed and opened, the solution is transferred to an amber glass vial with a PTFE-lined screw cap. When not being used, the standard is stored in the dark in a refrigerator at 4 ± 2°C. This purchased standard is used to prepare the LCS spiking solution:

7.13.1 LCS Spiking Solutions: They are prepared by diluting the 4000 ng/mL target analyte PCB congener standard to a concentration of 5.0 ng/mL in acetone. The concentration is verified by GC/MS before use. 1.0 mL of the 5.0 ng/mL solution is added to each solid LCS prior to extraction, whereas 0.200 mL is added to each aqueous LCS prior to extraction.

7.14 QC Check Sample: A QC Check Sample should be obtained from a source independent of the calibration standards. This check sample is a certified standard reference material (SRM) containing the PCBs in known concentrations in a sample matrix similar to the matrix under test.

8. Sample Collection, Preservation and Storage

8.1 Sampling is not performed for this method by Eurofins TestAmerica Knoxville. For information regarding sample shipping, refer to SOP KNOX-SC-0003, "Sample Receipt and Login", current revision.

8.2 Sample Storage

8.2.1 Store aqueous samples in the dark at ≥0°C to ≤6°C.

8.2.2 Store solid and tissue samples in the dark at ≤-10°C.

8.3 Holding Times

8.3.1 If stored according to the conditions specified in section 8.2, samples may be stored for up to one year.

8.3.2 Store sample extracts in the dark at room temperature until analyzed. If stored in the dark at room temperature, sample extracts may be stored for up to one year.

9. Quality Control

9.1 An initial demonstration of capability (IDOC) is performed to demonstrate the ability to generate acceptable precision and accuracy.

9.1.1 Extract, cleanup, concentrate, and analyze four 1-L aliquots of reagent water (for aqueous samples) or four aliquots of sodium sulfate (for solid/tissue samples) spiked with IDAs, cleanup surrogates, internal standards and the LCS target analyte spiking solution, according to the procedures in section 11. All sample processing steps that are to be used for processing samples, including preparation, extraction and cleanup, shall be included in this test.

9.1.2 Using the results of the set of four analyses, compute the average percent recovery (%R) of the extracts and the relative standard deviation (RSD) of the concentration in ng/mL for each compound.

9.1.3 For each PCB and labeled compound listed in Table 4, compare the RSD and %R with the corresponding limits for initial precision and recovery. If the RSD and %R for all compounds meet the acceptance criteria, system performance is acceptable and analysis of samples may begin. If, however, any individual RSD exceeds the precision limit or any individual %R falls outside the range for accuracy, system performance is unacceptable for that compound. Correct the problem and repeat the test.

9.2 Isotope Dilution Analytes (IDAs): Every sample, blank, and QC sample is spiked with $^{13}C_{12}$ labeled IDAs prior to extraction. IDAs in samples, blanks, and QC samples are used to calculate the concentration of the target analytes or detection limits.

9.3 Cleanup Surrogates: Every sample, blank, and QC sample extract is spiked with $^{13}C_{12}$ labeled cleanup surrogates after extraction but prior to extract cleanup. They are used to assess the efficiency of the cleanup procedures.

9.4 Internal standards: Every sample, blank, and QC sample extract is spiked with $^{13}C_{12}$ labeled internal standards prior to analysis. They are used to measure the recovery of the IDAs and the cleanup surrogates.

9.5 Method Blank: A laboratory method blank must be run along with each batch of 20 or fewer samples. The method blank is processed in the same manner and at the same time as the associated samples. The method blank is used to identify any background interference or contamination of the analytical system that may lead to the reporting of elevated concentration levels or false positive data. Refer to the table below for a cross-reference between the sample matrix and method blank matrix.

Sample Matrix	**Method Blank Matrix**
Aqueous	Reagent water
Solid	Sodium sulfate
Tissue	Sodium sulfate
Waste	Hexane

9.6 Laboratory Control Sample: A laboratory control sample (LCS) is prepared and analyzed with every batch of 20 or fewer samples. The LCS extract must be subject to the same cleanup procedures as the associated sample extracts. LCS spike components and concentrations are listed in Table 1. Refer to the table below for a cross-reference between the sample matrix and LCS matrix

Sample Matrix	**LCS Matrix**
Aqueous	Reagent water

Solid	Sodium sulfate
Tissue	Sodium sulfate
Waste	Hexane

9.7 QC Check Sample: Analyze the QC Check Sample (section7.14) periodically to assure the accuracy of calibration standards and the overall reliability of the analytical process. It is suggested that the QC Check Sample be analyzed at least annually.

9.8 Quality Control Batch: The batch is a set of up to 20 client samples that are of the same matrix and are processed together using the same procedures and reagents. The batch must contain a method blank and a laboratory control sample.

10. Calibration and Standardization

10.1 Not applicable.

11. Procedure

11.1 One time procedural variations are allowed only if deemed necessary in the professional judgment of supervision to accommodate variations in sample matrix, radioactivity, chemistry, sample size, or other parameters. Any variations in the procedure, except those specified by project specific instructions, shall be completely documented using a Nonconformance Memo and approved by a Technical Specialist, Project Manager, and QA Manager. If contractually required, the client shall be notified.

11.2 Any unauthorized deviations from this procedure must also be documented as a nonconformance, with a cause and corrective action described.

11.3 Samples are extracted by the following procedures depending upon sample matrix. Water samples are prepared by separatory funnel liquid/liquid extraction. Solid samples including soils, sediments, tissues and solid waste materials are prepared by Soxhlet extraction. Non-aqueous liquid wastes and organic solvents are prepared by waste dilution techniques.

NOTE: Samples should be removed from the refrigerator several hours before extraction and allowed to come to room temperature before measuring the volume or performing the extraction.

11.4 Aqueous Samples (samples containing 1% solids or less)

11.4.1 Remove samples from the refrigerator and allow them to come to room temperature before measuring the volume or performing the extraction. Inspect the samples carefully for biphasic sample characteristics. If this condition exists, document the observation and contact the project manager for instructions before proceeding with the extraction.

11.4.2 For aqueous samples that potentially contain >1% visible solids (as determined visually by an experienced analyst), a percent solid determination must be performed using the following procedure.

11.4.2.1 Add 10 mL of the well shaken sample to a tared aluminum weighing dish. Weigh the dish to three significant figures. Dry the dish overnight in an oven at 105 °C. Reweigh the dish and calculate the percent solids using the following equation.

$$\%\ Solids = \frac{\text{weight of dish + sample after } d\text{rying} - \text{weight of dish}}{\text{weight of dish + sample before } d\text{rying} - \text{weight of dish}} \times 100$$

11.4.3 For samples with > 1% visible solids, consult the project manager for further instructions before proceeding with extraction using one of the options listed below:

11.4.3.1 Samples can be filtered, extracted separately as solid and aqueous fractions, and analyzed separately.

11.4.3.2 Samples can be filtered, extracted separately as solid and aqueous fractions, and the extracts combined for a single analysis.

11.4.3.3 The client may decide to resample if they determine that solids should not have been present.

11.4.4 Aqueous samples that are suspected to have congener levels above 40 ng/L may be screened before extraction to prevent instrument saturation or contamination.

11.4.4.1 Carefully shake the sample bottle well. Measure out 20 mL of water and place in a 40 mL vial.

11.4.4.2 Add 100 µL of the 0.2 mg/L 8082 surrogate mix to the 20 mL of water. (Refer to section 7.11.)

11.4.4.3 Add 20 mL of methylene chloride and shake the 40 mL vial for 2 minutes. Vent the vial several times during the 2 minutes to relieve any pressure that might have built up.

11.4.4.4 Allow the water layer and the methylene chloride layer to separate, swirling the vial gently to aid in separation of the layers.

11.4.4.5 Using a disposable pipet, pull the methylene chloride (bottom layer) out and filter it through a sodium sulfate funnel and into a clean 40 mL vial.

11.4.4.6 Concentrate the extract to approximately 100 µL. Bring the extract back up to 1 mL with hexane and deliver to the GC

group for screening. Refer to section 11.5.9 to 11.5.11 for details re. screening.

11.4.4.7 Refer to Knoxville SOP KNOX-QA-0002, current revision, for information on glassware cleaning procedures for extraction glassware.

11.4.4.8 For water samples, only use the glassware designated for water samples.

11.4.4.9 Place separatory funnels, one for each sample, in the separatory rotator set up in the hood.

11.4.4.10 Place a 500 mL flask directly beneath the separatory funnel.

11.4.4.11 Plug a glass funnel with glass wool and pour in some sodium sulfate (about 1 to 2 inches from the top). Rinse the sodium sulfate with methylene chloride. After the methylene chloride stops dripping, place the funnel on top of the flask that is fitted with a paper clip to aid in filtering.

11.4.4.12 Inspect the sample for solids or biphasic sample characteristics. If either condition exists, consult the project manager for further instructions.

11.4.4.13 Weigh the sample container on a balance (± 1 g); tare the sample and container. Transfer the sample to the extractor. Rinse the sample bottle with approximately 60 mL methylene chloride and transfer to the extractor. Reweigh the container. Assume a density of 1 g/mL and record the difference as the sample volume in TALS.

11.4.4.14 For brackish water or salt water, weigh 1.0 mL of the sample. If the weight is <0.98 or >1.02, adjust the sample volume based on the sample density.

11.4.5 For the method blank and the LCS (and LCSD, if required), use a 1000 mL graduated cylinder to measure 1000 mL of reagent water.

11.4.5.1 After placing the samples into separatory funnels including the method blank and the LCS the initial pH is measured with 0-14 wide range pH paper. Concentrated sulfuric acid is then carefully and slowly added in small increments, usually 1 to 2 mL at a time. Swirl the seperatory funnel to mix the sulfuric acid into the sample. The pH is then measured again and more sulfuric acid is added until the desired pH between 1 and 3 is achieved.

NOTE: Alternatively, the pH may be adjusted in the sample bottle prior to placing into the seperatory funnel.

11.4.6 Add 0.2 mL of the 10.0 ng/mL IDA spiking solution to each sample, method blank and LCS (see section 7.12.2). Additionally, add 0.2 mL of the 5.0 ng/mL LCS spiking solution to the LCS (and LCSD, MS/MSD, if required, see section 7.13.1). Record the amount of spike used and the spike standard number in TALS.

NOTE: Verify that each spiking solution has not expired.

NOTE: Each time the samples are spiked, the spiking process should be witnessed by another analyst. Refer to Appendix I for the steps that must be taken.

11.4.7 Add 60 mL of methylene chloride to the sample bottle and shake. Then add the methylene chloride to the separatory funnel. Add 60 mL of methylene chloride to the method blank, LCS (and LCSD, if required).

11.4.8 Secure the separatory funnel with the rotator retaining straps and rotate for 2 minutes.

CAUTION: Care should be used while performing this operation. Vent the separatory funnel frequently. Goggles may be worn when performing this procedure. Also, place a clean rag over the tip of the separatory funnel to catch any spray that might have vented.

11.4.9 Allow the water and the methylene chloride to separate. If it is not separated after 10 minutes, try to break up the emulsion by gently swirling the sample or tilting the separatory funnel on its side.

11.4.10 Drain the methylene chloride from the separatory funnel into the sodium sulfate funnel, allowing the extract to drip into the flask. Be careful not to allow water to escape the separatory funnel or the sodium sulfate will harden and block the flow of the extract. NOTE: If at least 10 minutes has elapsed and other ways of breaking up or reducing the size of the emulsion have failed, the following steps may be tried to reduce the impact of the emulsion on the sodium sulfate.

11.4.10.1 Place a large piece of pre-cleaned glass wool in the funnel containing the sodium sulfate.

11.4.10.2 Spread the glass wool out, covering the entire surface of the sodium sulfate to a depth of ~5 to 10 mm. If the emulsion is hard to break up and persistent, a small, additional layer of sodium sulfate may be added on top of the glass wool.

11.4.10.3 Drain the solvent and emulsion layer into the funnel, being careful to drain no more than 60 mL of volume if a clear phase layer cannot be determined. Record the formation of an emulsion in TALS.

11.4.10.4 If this procedure is used, the funnel should be rinsed immediately with an extra 30 mL of methylene chloride to ensure all analytes are rinsed into flask.

11.4.11 Repeat the extraction process two more times with fresh 60 mL portions of methylene chloride.

11.4.12 After the third methylene chloride portion has filtered through the sodium sulfate, rinse the funnel with approximately 40 mL of methylene chloride.

11.4.13 Remove the separatory funnel from the hood and pour the extracted water into the extracted waters waste container.

11.4.14 Remove the glass funnel from the top of the flask.

11.4.15 Add a few boiling stones to the 500 mL boiling flask. Place a 3-ball Snyder column on the boiling flask. For extracts that will go through GPC cleanup, first concentrate to approximately 20 mL methylene chloride. If GPC cleanup is not going to be performed immediately, solvent exchange the extracts to hexane as follows: Concentrate the extract to approximately 20mL, then add approximately 60 mL of hexane and concentrate to > 20 mL.

NOTE: Over-concentration of heated extracts is known to cause the more volatile PCB congeners to vaporize and be lost. Therefore, do not concentrate to less than 20 mL.

CAUTION: When extracting or concentrating a sample with hexane, toluene or any mixture containing these solvents, the analyst **must** add the boiling chips within 5 minutes of placing the flask on the heat source in order to prevent bumping of the solvent.

11.4.16 Transfer the extract into a solvent-rinsed 40 mL vial, rinsing the 500 mL flask three times with approximately 3 mL of the appropriate solvent (hexane or methylene chloride). Add the rinses to the 40 mL vial.

11.4.17 For extracts that will go through GPC cleanup, reduce the volume to approximately 6 mL methylene chloride on the N-EVAP concentration device. Proceed to section 11.9.6.

11.4.18 For extracts that will not go through GPC, reduce the volume to approximately 0.5 mL hexane on the N-EVAP concentration device. Do not allow the sample to go to dryness at any time. If an additional solvent exchange is needed, add 5 mL of hexane and swirl the vial. Reduce the volume of hexane to approximately 0.5 mL again to complete the solvent exchange. Adjust the final volume of the extract with hexane to 12 mL for acid cleanup or to approximately 4 to 6 mL for column cleanup. If the sample exhibits poor solubility in hexane, add approximately 1 mL of benzene to aid in dissolving the sample. Proceed to section 11.9.8.

11.5 Solid/Tissue Sample Screening and Assignment of Sample Preparation Protocols

11.5.1 Because of the sensitivity of method 1668, it is necessary to screen all solid samples and non-aqueous liquids before extraction to prevent instrument saturation and/or contamination.

11.5.2 Mix the sample well, take a 1.0 ± 0.1g aliquot of the sample material. Mix with sodium sulfate in an aluminum pan until the mixture is free flowing.

11.5.3 Transfer to a 40 mL vial and add 1.0 mL of the 0.2 mg/L 8082 surrogate mix. (Refer to section 7.11.)

11.5.4 Add 9 mL of hexane.

11.5.5 Cap tightly and secure on a platform shaker table.

11.5.6 Shake for 1 hour, minimum.

11.5.7 Transfer ~ 4 mL of extract into a fresh 12 mL vial, add ~2 mL of sulfuric acid; cap tightly, and shake the vial for 30 to 60 seconds. If necessary, centrifuge the extract.

11.5.8 Deliver the extract to the GC group for screening.

11.5.9 Perform a single point initial calibration for each sequence on the screening instrument.

11.5.10 If no pattern and no prominent peaks are present, process the sample using protocol 1.

11.5.11 If an Aroclor-like pattern is observed, calculate the estimated concentration of the largest peak expected in the determinative analysis, and assign an extraction protocol using the “PCB Protocol Selection” spreadsheet on the local area network in the MSOffice\template\org prep.

11.5.11.1 Determine the concentration of the most prominent technical mixture by comparison to Aroclor standards. Enter the Aroclor sample concentration (ppb) into the spreadsheet. Circle the amount entered on the GC hardcopy result.

11.5.11.2 For sediment samples, import the sample % moisture results.

11.5.11.3 Enter the average or selected surrogate recovery in the spreadsheet. The software will correct the PCB concentration for surrogate recovery.

11.5.11.4 The software will multiply the PCB concentration by 0.1 (the spreadsheet default) to correct for the weight percent of the largest single congener in an Aroclor mix. This value may be changed to represent special cases such as altered patterns or

unusual technical mixtures, but documentation of the rationale must accompany the spreadsheet.

11.5.11.5 The software will then multiply the PCB concentration by 4 to account for single-peak coelutions.

11.5.11.6 The resulting concentration is used as the estimate of the largest peak expected in the determinative analysis and to assign an extraction protocol for each sample.

11.5.11.7 An independent analyst should review the data entry and protocol assignments. Initial and date the spreadsheet. Attach the spreadsheet and supporting GC data to the extraction benchsheet.

11.5.11.8 During sample preparation, adjust the sample extraction amount, spiking volumes and final volume to reflect the selected protocol. Refer to Table 5.

11.6 Sample Pretreatment for Tissue Samples

11.6.1 If the sample matrix is tissue and has not been homogenized prior to sample receipt, the entire sample is homogenized prior to extraction using an industrial meat grinder, a laboratory blender, or a laboratory homogenizer. Select the equipment that is most appropriate for the size and type of tissue received.

11.7 Solid/Tissue Sample Soxhlet Extraction (samples containing more than 1% solids)

11.7.1 This section uses protocol 1 as an example. If another protocol has been assigned, the sample amount extracted, spike volumes and percent of extract used is modified based on Table 5.

11.7.2 Prepare and label the required number of Soxhlet systems. Refer to SOP KNOX-QA-0002, current revision, for information on cleaning procedures for extraction glassware. Visually inspect all glassware prior to use for scratches or cracks. Retire, repair or replace any glassware found to be damaged.

11.7.3 For soil samples, transfer a well-mixed 10 g aliquot (± 0.5 g) of the sample into a beaker or extraction thimble. Mix thoroughly with 20 g of sodium sulfate. Record the sample weight in TALS.

11.7.4 For sediment samples, adjust the amount weighed to achieve 10 to 10.5 g dry weight. Determine the amount of sediment sample to extract using the "Sediment Extraction Amounts" spreadsheet on the local area network in the MSOffice\template\Knx OrgPrep directory. Transfer a well-mixed aliquot of the sample into a beaker or extraction thimble. Mix thoroughly with 20 g of sodium sulfate. Record the sample weight in TALS

11.7.5 For tissue samples, weigh out 10 g (± 0.5 g) of homogenized tissue into a beaker or extraction thimble. Mix thoroughly with 20 g of sodium sulfate. Record the sample weight in TALS.

NOTE: If gravimetric lipids are to be determined using the tissue extracts, split the extract prior to the initiation of any cleanup steps and use 1 mL of the 10 mL extract for lipids determination. Refer to SOP KNOX-OP-0020, "Gravimetric Percent Lipids Determination", current revision.

11.7.6 Refer to the Section 9.5 for a cross-reference between the sample matrix and method blank matrix and Section 9.6 for the LCS matrix.

11.7.7 Spike each sample with 1.0 mL of the 10 ng/mL IDA spiking solution (see section 7.12.2).

11.7.8 Spike the LCS (and LCSD, MS/MSD, if required) with 1.0 mL of the 5.0 ng/mL LCS spiking solution (see section 7.13.1).

NOTE: Verify that each spiking solution has not expired. Record the amount and ID of each spiking reagent used in TALS.

NOTE: Each time the samples are spiked, the spiking process should be witnessed by another analyst. Refer to Appendix I for the steps that must be taken.

11.7.9 If needed, add a small amount of glass wool to the top of the extraction thimble.

11.7.10 Pour approximately 350 mL of the 1:1 hexane/acetone into a 500 mL flask. Place the flask in the heating mantle. Add several PTFE boiling stones.

CAUTION: When extracting or concentrating a sample with hexane, toluene or any mixture containing these solvents, the analyst **must** add the boiling chips within 5 minutes of placing the flask on the heat source in order to prevent bumping of the solvent.

11.7.11 Place the extraction thimble in the glass Soxhlet extractor.

11.7.12 Assemble the Soxhlet system and secure to the lab supports.

11.7.13 Adjust the temperature of the heating mantle to bring the solvent in the flask to a rolling boil. There should be a steady drip from the condensers so that the solvent should completely cycle at least 5 times an hour.

11.7.14 Soxhlet extract the sample in the above manner for 18-24 hours.

11.7.15 Turn off the heating mantle and allow the Soxhlet apparatus to cool.

11.7.16 Remove the condensers and allow the Soxhlet extractor chamber to empty; then remove the Soxhlet extractor from the 500 mL flask.

NOTE: If the samples appear to have a water layer or moisture, dry the extract by filtering it through a sodium sulfate filled funnel.

11.7.17 Add several PTFE boiling stones and place a solvent-rinsed 3-ball Snyder column on the 500 mL flask. Concentrate the extract to > 20 mL. Add 60 mL of hexane to the 500 mL flask and concentrate again to > 20 mL to remove any acetone that may still be present.

NOTE: Over-concentration of heated extracts is known to cause the more volatile PCB congeners to vaporize and be lost. Therefore, do not concentrate to less than 20 mL.

CAUTION: When extracting or concentrating a sample with hexane, toluene or any mixture containing these solvents, the analyst **must** add the boiling chips within 5 minutes of placing the flask on the heat source in order to prevent bumping of the solvent.

11.7.18 Transfer the extract into a solvent-rinsed 40 mL vial, rinsing the 500 mL flask three times with approximately 3 mL of hexane. Add the rinses to the 40 mL vial.

11.7.19 Place the 40 mL vial into the N-EVAP concentration device and reduce the volume to approximately 12 mL. Proceed to section 11.9.5 to perform acid cleanup.

11.8 Waste Sample Extraction

11.8.1 Organic wastes, oil, solids and non-aqueous sludge samples that will dissolve in solvent may be prepared by the waste dilution technique.

11.8.2 Add an appropriate amount of sample (based on screen results) to a solvent-rinsed 40 mL vial.

11.8.3 2 mL of hexane is used for the blank and LCS.

11.8.4 Spike each sample with 1.0 mL of the 10 ng/mL IDA spiking solution (see section 7.12.2).

11.8.5 Spike the LCS (and LCSD, MS/MSD, if required) with 1.0 mL of the 5.0 ng/mL LCS spiking solution (see section 7.1

NOTE: Verify that each spiking solution has not expired.

NOTE: Each time the samples are spiked, the spiking process should be witnessed by another analyst. Refer to Appendix I for the steps that must be taken.

11.8.6 Concentrate all extracts to approximately 0.200 mL to remove acetone from the extract.

11.8.7 Add hexane to bring the volume to 10 mL. If the sample exhibits poor solubility in hexane, add approximately 1 mL of benzene to the vial to aid in dissolving the sample. Proceed to section 11.9.5 to perform acid cleanup.

11.8.8 Record the weights and volumes of spiking reagents used in TALS.

11.9 Extract Cleanup

11.9.1 Cleanup may not be necessary for relatively clean samples (e.g., treated effluents, groundwater or drinking water). If particular circumstances require the use of a cleanup procedure, the laboratory may use any or all of the procedures below or any other appropriate procedure. Before using a cleanup procedure, the laboratory must demonstrate that the requirements of Section 13.2 can be met using the cleanup procedure.

11.9.2 Soil, sediment and tissue sample extracts are typically taken through the following cleanup procedures: Acid cleanup, GPC and silica gel cleanup. Aqueous sample extracts are typically cleaned using GPC and silica gel cleanup, but may be cleaned using silica gel only.

11.9.3 If the sample requires a split based on the protocols listed in Table 5, the split must be performed prior to the initiation of the first cleanup procedure and prior to the addition of cleanup surrogates. For protocol 1 samples, use 5 mL of the 10 mL extract. Archive the remaining 5 mL aliquot of extract.

11.9.4 Spike each sample extract with an appropriate amount (i.e., 0.5 mL or 1.0 mL for solid, tissue and waste sample extracts; 0.20 mL for aqueous sample extracts) of the 10 ng/mL cleanup surrogate spiking solution (see section 7.12.6) prior to initiating any cleanup procedures.

NOTE: Verify that each spiking solution has not expired.

11.9.5 Acid Cleanup

11.9.5.1 Slowly add 10-20 mL of concentrated sulfuric acid to the 12 mL hexane extract in the 40 mL vial and shake for 30-60 seconds. Vent the vial frequently while shaking. Let the vial stand or centrifuge to obtain well-defined separation of acid and solvent. Remove the acid layer with a glass pipet. (Optionally, the solvent layer can be removed.) Repeat the acid washing if the solvent layer is colored. Perform a maximum of four acid washings. If solvent layer is removed, step 11.9.5.2 is not performed.

11.9.5.2 Add approximately 5 mL 5% (w/v) aqueous sodium chloride to the vial and gently shake for 30 seconds. Vent the vial frequently

while shaking. Let the vial stand until a well-defined separation has occurred and remove the aqueous layer with a glass pipet. Dry the hexane extract by adding 1 to 2 grams of sodium sulfate and swirling the vial.

11.9.5.3 Place the 40 mL vial into the N-EVAP concentration device and reduce the volume to approximately 1 mL. Solvent exchange to methylene chloride and bring to approximately 6 to 8 mL. Proceed to section 11.9.6, GPC Cleanup.

11.9.6 GPC Cleanup

11.9.6.1 Gel permeation chromatography (GPC) removes high molecular weight interferences that cause GC column performance to degrade. It should be used for all soil, sediment, tissue and waste sample extracts. It may also be used for aqueous sample extracts.

11.9.6.2 Refer to SOP KNOX-OP-0022, current revision for detailed instructions for GPC cleanup.

11.9.6.3 After GPC cleanup, concentrate the extract and solvent exchange to ~6 to 8 mL hexane using low-level KD glassware. Proceed to section 11.9.7, Silica Gel Column Cleanup.

NOTE: If concentrating aqueous sample extracts, use glassware designated for water extraction and cleanup.

CAUTION: When extracting or concentrating a sample with hexane, toluene or any mixture containing these solvents, the analyst must add the boiling chips within 5 minutes of placing the flask on the heat source in order to prevent bumping of the solvent.

11.9.7 Silica Gel Column Cleanup

11.9.7.1 Prepare 20 mm columns for each extract by placing a small amount of glass wool in the bottom of each column and then solvent rinsing with hexane. Shake out the excess hexane.

11.9.7.2 Mark the level to which the column packings will be added, starting at the top of the glass wool plug and proceeding from bottom to the top.

Layer 1 – 12 mm (2 g) of 3.3% deactivated silica gel

Layer 2 – 16 mm (4 g) of acidic silica gel

Layer 3 – 12 mm (2 g) of 3.3% deactivated silica gel

Layer 4 – 10 mm of sodium sulfate

11.9.7.3 Add the column packings to the 20 mm column in the order listed above, tapping the column to settle the contents to prevent channeling.

11.9.7.4 Add 30 mL of hexane to pre-elute the column. Do not collect the pre-eluted hexane.

11.9.7.5 When the hexane reaches the top of the sodium sulfate layer, place a solvent-rinsed 250 or 500 mL flask under the column and transfer the sample extract into the column. Quickly rinse the extract vial with a small amount of hexane and add the rinse into the column. Repeat the rinse 2 more times.

11.9.7.6 As the solvent level from the last rinse reaches the top of the sodium sulfate layer, add 70 mL of hexane into the silica gel column to elute the PCB's into the flask.

11.9.7.7 When the column stops dripping, add several PTFE boiling stones to the flask and attach a solvent-rinsed Snyder column. Concentrate the extract to > 20 mL on a heating mantle.

NOTE: Over-concentration of heated extracts is known to cause the more volatile PCB congeners to vaporize and be lost. Therefore, do not concentrate to less than 20 mL.

CAUTION: When extracting or concentrating a sample with hexane, toluene or any mixture containing these solvents, the analyst must add the boiling chips within 5 minutes of placing the flask on the heat source in order to prevent bumping of the solvent.

11.9.7.8 Transfer the extract to a 40 mL vial, rinsing the flask with a small amount of hexane and add the rinse to the 40 mL vial. Repeat the rinse 2 more times.

11.9.7.9 Place the 40 mL vial into the N-EVAP concentration device and reduce the volume to approximately 10 mL. Proceed to section 11.9.8, Final Concentration.

11.9.8 Final Concentration

11.9.8.1 Place the 40 mL vial containing the extract in the N-EVAP concentration apparatus and reduce the solvent volume to approximately 0.5 to 1.0 mL.

11.9.8.2 Label a 1.1 mL tapered mini-vial with the sample ID after checking that the vial and cap fit together properly.

11.9.8.3 For aqueous samples, add 20 µL of the 100 ng/mL internal standard spiking solution (see section 7.12.4) to the mini-vial and

mark the level on the vial. Transfer the concentrated extract into the mini-vial, rinsing 2 times with small amounts of hexane. Then reduce the extract volume back down to 20 µL. Deliver the mini-vial to the HRMS lab for analysis.

NOTE: Verify that the spiking solution has not expired.

11.9.8.4 For protocol 1 solid, sediment, tissue and waste samples, add 50 µL of the 100 ng/mL internal standard spiking solution (see section 7.12.4) to the mini-vial and mark the level on the vial. Transfer the concentrated extract into the mini-vial, rinsing 2 times with small amounts of hexane. For protocol 1 samples, reduce the extract volume to 50 µL. Deliver the mini-vial to the HRMS lab for analysis.

11.9.8.5 For samples prepared according to protocols 2, 3 and 4, add 100 µL of the 100 ng/mL internal standard spiking solution and dilute the extract with nonane to achieve the final extract volume shown in Table 5. Deliver the mini-vial to the HRMS lab for analysis.

12. Data Analysis and Calculations

12.1 Not applicable.

13. Method Performance

13.1 Method Detection Limit (MDL) - An MDL must be determined for each analyte in each routine matrix prior to the analysis of any samples. The procedure for determination of the method detection limit is given in the SOP CA-Q-S006, current revision, based on 40 CFR Part 136 Appendix B. The result of the MDL determination must support the reporting limit.

13.2 Initial Demonstration of Capability – Each analyst must perform an initial demonstration of capability (IDOC) for each target analyte prior to performing the analysis independently. The IDOC is determined by analyzing four replicate spikes (e.g., LCSs) as detailed in Eurofins TestAmerica Knoxville SOP KNOX-QA-0009, current revision.

13.3 Training Qualification: The group/team leader has the responsibility to ensure that this procedure is performed by an associate who has been properly trained in its use and has the required experience. Refer to SOP KNOX-QA-0009, current revision for further requirements for performing and documenting initial and on-going demonstrations of capability.

14. Pollution Prevention

14.1 All attempts will be made to minimize the use of solvents and standard materials.

15. Waste Management

15.1 All waste will be disposed of in accordance with Federal, State and Local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment, employees will abide by this method and the policies in section 13 of the Corporate Safety Manual for "Waste Management and Pollution Prevention."

15.2 The following waste streams are produced when this method is carried out.

- Waste methylene chloride, acetone, hexane and toluene shall be placed in the flammable waste stream, contained in a steel satellite accumulation container or flammable solvent container.
- Miscellaneous disposable glassware, chemical resistant gloves, bench paper and similar materials shall be placed in the incinerable laboratory waste stream, contained in a steel or HDPE satellite accumulation container.
- Extracted solid/tissue samples, paper funnel filters, glass wool, etc., contaminated with solvents shall be placed in the incinerable laboratory waste stream, contained in a steel or HDPE satellite accumulation container.
- Contaminated sulfuric acid used during extract cleanup shall be placed in the acidic waste stream, contained in a HDPE satellite accumulation container.
- Extracted aqueous samples, contaminated with methylene chloride shall be placed in the organic water waste stream, contained in a HDPE satellite accumulation container.

16. References

16.1 Method 1668, Revision A, Chlorinated Biphenyl Congeners in Water, Soil, Sediment and Tissue by HRGC/HRMS, EPA-821-R-00-002, December 1999

16.2 Method 1668, Revision B, Chlorinated Biphenyl Congeners in Water, Soil, Sediment and Tissue by HRGC/HRMS, EPA-821-R-08-020, November 2008

16.3 Method 1668, Revision C, Chlorinated Biphenyl Congeners in Water, Soil, Sediment and Tissue by HRGC/HRMS, EPA-820-R-10-005, April 2010

16.4 Eurofins TestAmerica Knoxville SOP KNOX-ID-0013, "Analysis of Polychlorinated Biphenyl (PCB) Isomers by Isotope Dilution HRGC/HRMS", current revision

16.5 Eurofins TestAmerica Knoxville SOP KNOX-OP-0020, "Gravimetric Percent Lipids Determination" current revision

16.6 Eurofins TestAmerica Knoxville SOP KNOX-OP-0022, "GPC Cleanup", current revision

16.7 Eurofins TestAmerica Knoxville SOP KNOX-QA-0002, "Glassware Cleaning", current revision

16.8 Eurofins TestAmerica Knoxville SOP KNOX-QA-0009, "Personnel Orientation and Training", current revision

16.9 Eurofins TestAmerica Knoxville Laboratory Quality Assurance Manual (QAM), current revision

17. Miscellaneous

17.1 Modifications and enhancements from EPA Method 1668, Revisions A and B: Method 1668 is a "performance-based" method and allows for method modifications (e.g., alternate extraction, concentration and cleanup procedures, etc.) as long as the method performance criteria described in Section 13.2 are met.

17.1.1 The cleanup procedures described in this SOP have been optimized and may vary significantly from the referenced methods.

17.1.2 Additional internal standards are used in this procedure. The internal standards are listed in Table 3.

17.1.3 A solvent mixture of 1:1 hexane/acetone is used for Soxhlet extraction of solid and tissue samples that have been mixed with sodium sulfate prior to extraction.

17.1.4 Method 1668 specifies that the sample bottle is rinsed twice with 5 mL of reagent water after the sample is transferred to the separatory funnel. This procedure specifies that the sample bottle is rinsed three times with methylene chloride after the sample is transferred to the separatory funnel. This modification improves the removal of target compounds from the sample bottle.

17.1.5 The acid cleanup procedure is carried out in a 40 mL vial instead of a separatory funnel. Disposable glassware is used to decrease the risk of cross contamination. The volumes of the washes used have been adjusted for use in the 40 mL vial.

17.1.6 Extracts are stored at room temperature in the dark rather than at <10 °C. Method 1668 allows for the storage of standards at room temperature. Recovery studies performed by Cambridge Isotopes Laboratories (CIL) indicate freezing or refrigeration of standards causes problems with precipitation and irreversible adsorption to the inside surface of the vial. CIL recommends the storage of standards and extracts at room temperature as long as they are protected from exposure to UV and evaporative losses.

17.1.7 Aqueous samples with < 1% solids are extracted as received by separatory funnel. Samples with high solids content, > 1% solids, are either shaken well to thoroughly mix the sample followed by extraction by separatory funnel, or separated into two different samples for extraction and analysis, dependent upon client request.

17.1.8 Solid samples are extracted as received, with %-moisture determination

performed simultaneously. Dry weight correction to results is applied at the time of data reporting. Particle size determination and reduction as specified in method 1668 is not performed on a routine basis. These procedures are considered to be outside the scope of the laboratories routine extraction procedures and are only performed on a client specific or project specific basis. These procedures, if required, must be specified and documented in the appropriate QAPPs.

17.1.9 Tissue samples are homogenized as described in Section 11.6.1.

17.1.10 Samples are extracted immediately after spiking.

17.2 List of Tables and Appendices:

17.2.1 Table 1 – Concentration of Target Analyte PCB Congener Stock and Spiking Solutions

17.2.2 Table 2 - Concentration of $^{13}C_{12}$ Labeled Isotope Dilution Analyte (IDA) Stock and Spiking Solutions (Cambridge Mix)

17.2.3 Table 3 - Concentration of $^{13}C_{12}$ Labeled Internal Standard (IS) and Cleanup Surrogate (SU) Stock and Spiking Solutions

17.2.4 Table 4 – Acceptance Criteria for Performance Tests

17.2.5 Table 5 – Assignment of Sample Preparation Protocols for Soxhlet Extraction

17.2.6 Appendix I – Guidelines for the Spike Witnessing Process

Table 1 - Concentration of Target Analyte PCB Congener Stock and Spiking Solutions

Target analyte PCB Congener	BZ/ IUPAC	Standard Source	Catalog Number	Vendor Conc (ng/mL)	LCS Spiking Solution Conc (ng/mL)
2-MoCB	1	AccuStd	S-99994-4x	4000	5.0
4-MoCB	3	AccuStd	S-99994-4x	4000	5.0
2,2'-DiCB	4	AccuStd	S-99994-4x	4000	5.0
4,4'-DiCB	15	AccuStd	S-99994-4x	4000	5.0
2,2',6-TrCB	19	AccuStd	S-99994-4x	4000	5.0
3,4,4'-TrCB	37	AccuStd	S-99994-4x	4000	5.0
2,2',6,6'-TeCB	54	AccuStd	S-99994-4x	4000	5.0
3,3',4,4'-TeCB	77	AccuStd	S-99994-4x	4000	5.0
3,4,4',5-TeCB	81	AccuStd	S-99994-4x	4000	5.0
2,2',4,6,6'-PeCB	104	AccuStd	S-99994-4x	4000	5.0
2,3,3',4,4'-PeCB	105	AccuStd	S-99994-4x	4000	5.0
2,3,4,4',5-PeCB	114	AccuStd	S-99994-4x	4000	5.0
2,3',4,4',5-PeCB	118	AccuStd	S-99994-4x	4000	5.0
2',3,4,4',5-PeCB	123	AccuStd	S-99994-4x	4000	5.0
3,3',4,4',5-PeCB	126	AccuStd	S-99994-4x	4000	5.0
2,2',4,4',6,6'-HxCB	155	AccuStd	S-99994-4x	4000	5.0
2,3,3',4,4',5-HxCB	156	AccuStd	S-99994-4x	4000	5.0
2,3,3',4,4',5'-HxCB	157	AccuStd	S-99994-4x	4000	5.0
2,3',4,4',5,5'-HxCB	167	AccuStd	S-99994-4x	4000	5.0
3,3',4,4',5,5'-HxCB	169	AccuStd	S-99994-4x	4000	5.0
2,2',3,3',4,4',5-HpCB	170	AccuStd	S-99994-4x	4000	5.0
2,2',3,4,4',5,5'-HpCB	180	AccuStd	S-99994-4x	4000	5.0
2,2',3,4',5,6,6'-HpCB	188	AccuStd	S-99994-4x	4000	5.0
2,3,3',4,4',5,5'-HpCB	189	AccuStd	S-99994-4x	4000	5.0
2,2',3,3',5,5',6,6'-OcCB	202	AccuStd	S-99994-4x	4000	5.0
2,3,3',4,4',5,5',6-OcCB	205	AccuStd	S-99994-4x	4000	5.0
2,2',3,3',4,4',5,5',6-NoCB	206	AccuStd	S-99994-4x	4000	5.0
2,2',3,3',4',5,5',6,6'-NoCB	208	AccuStd	S-99994-4x	4000	5.0
DeCB	209	AccuStd	S-99994-4x	4000	5.0
All other CB congeners	NA	AccuStd	S-99994-4x	4000	5.0

Table 2 - Concentration of $^{13}C_{12}$ Labeled Isotope Dilution Analyte (IDA) Stock and Spiking Solutions (Cambridge Mix)

Labeled PCB Congener	BZ/ IUPAC	Standard Source	Catalog Number	Vendor Conc (ng/mL)	Stock Conc (ng/mL)	Spiking Solution Conc (ng/mL)
Isotope Dilution Analytes (IDAs)						
$^{13}C_{12}$-2-chlorobiphenyl	1L	Cambridge	EC-4977	1000	1000	10
$^{13}C_{12}$-4-chlorobiphenyl	3L	Cambridge	EC-4977	1000	1000	10
$^{13}C_{12}$-2,2'-dichlorobiphenyl	4L	Cambridge	EC-4977	1000	1000	10
$^{13}C_{12}$-4,4'-dichlorobiphenyl	15L	Cambridge	EC-4977	1000	1000	10
$^{13}C_{12}$-2,2',6-trichlorobiphenyl	19L	Cambridge	EC-4977	1000	1000	10
$^{13}C_{12}$-3,4,4'-trichlorobiphenyl	37L	Cambridge	EC-4977	1000	1000	10
$^{13}C_{12}$-2,2',6,6'-tetrachlorobiphenyl	54L	Cambridge	EC-4977	1000	1000	10
$^{13}C_{12}$-3,3',4,4'-tetrachlorobiphenyl	77L	Cambridge	EC-4977	1000	1000	10
$^{13}C_{12}$-3,4,4',5-tetrachlorobiphenyl	81L	Cambridge	EC-4977	1000	1000	10
$^{13}C_{12}$-2,2',4,6,6'-pentachlorobiphenyl	104L	Cambridge	EC-4977	1000	1000	10
$^{13}C_{12}$-2,3,3',4,4'-pentachlorobiphenyl	105L	Cambridge	EC-4977	1000	1000	10
$^{13}C_{12}$2,3,4,4',5-pentachlorobiphenyl -	114L	Cambridge	EC-4977	1000	1000	10
$^{13}C_{12}$-2,3',4,4',5-pentachlorobiphenyl	118L	Cambridge	EC-4977	1000	1000	10
$^{13}C_{12}$-2',3,4,4',5-pentachlorobiphenyl	123L	Cambridge	EC-4977	1000	1000	10
$^{13}C_{12}$-3,3',4,4',5-pentachlorobiphenyl	126L	Cambridge	EC-4977	1000	1000	10
$^{13}C_{12}$-2,2',4,4',6,6'-hexachlorobiphenyl	155L	Cambridge	EC-4977	1000	1000	10
$^{13}C_{12}$-2,3,3',4,4',5-hexachlorobiphenyl	156L	Cambridge	EC-4977	1000	1000	10
$^{13}C_{12}$-2,3,3',4,4',5'-hexachlorobiphenyl	157L	Cambridge	EC-4977	1000	1000	10
$^{13}C_{12}$-2,3',4,4',5,5'-hexachlorobiphenyl	167L	Cambridge	EC-4977	1000	1000	10
$^{13}C_{12}$-3,3',4,4',5,5'-hexachlorobiphenyl	169L	Cambridge	EC-4977	1000	1000	10
$^{13}C_{12}$-2,2',3,3',4,4',5-heptachlorobiphenyl *	**170L**	**Cambridge**	**EC-4905**	**40,000**	**1000**	**10**
$^{13}C_{12}$-2,2',3,4',5,6,6'-heptachlorobiphenyl	188L	Cambridge	EC-4977	1000	1000	10
$^{13}C_{12}$-2,3,3',4,4',5,5'-heptachlorobiphenyl	189L	Cambridge	EC-4977	1000	1000	10
$^{13}C_{12}$-2,2',3,3',5,5',6,6'-octachlorobiphenyl	202L	Cambridge	EC-4977	1000	1000	10
$^{13}C_{12}$-2,3,3',4,4',5,5',6-octachlorobiphenyl	205L	Cambridge	EC-4977	1000	1000	10
$^{13}C_{12}$-2,2',3,3',4,4',5,5',6-nonachlorobiphenyl	206L	Cambridge	EC-4977	1000	1000	10
$^{13}C_{12}$-2,2',3,3',4,5,5',6,6'-nonachlorobiphenyl	208L	Cambridge	EC-4977	1000	1000	10
$^{13}C_{12}$-2,2',3,3',4,4',5,5',6,6'-decachlorobiphenyl	209L	Cambridge	EC-4977	1000	1000	10

*PCB 170L is not included in the mix from Cambridge. It is purchased as an individual analyte and added to the mix. It is received at a concentration of 40,000 ng/mL. Bold denotes that this analyte must be added to the mix.

Table 3 - Concentration of $^{13}C_{12}$ Labeled Internal Standard (IS) and Cleanup Surrogate (SU) Stock and Spiking Solutions

Labeled PCB Congener	BZ/ IUPAC	Standard Source	Catalog Number	Vendor Conc (ng/mL)	Stock Conc (ng/mL)	Spiking Solution Conc (ng/mL)
Internal Standards						
$^{13}C_{12}$-2,5-dichlorobiphenyl	9L	Cambridge	EC-4165	40,000	1000	100
$^{13}C_{12}$-2,4',5-trichlorobiphenyl	31L	Wellington	MBP-31	50,000	1000	100
$^{13}C_{12}$-2,4',6-trichlorobiphenyl	32L	Cambridge	EC-4163	40,000	1000	100
$^{13}C_{12}$-2,2',5,5'-tetrachlorobiphenyl	52L	Cambridge	EC-1424	40,000	1000	100
$^{13}C_{12}$-2,2',4,5,5'-pentachlorobiphenyl	101L	Cambridge	EC-1405	40,000	1000	100
$^{13}C_{12}$-3,3',4,5,5'-pentachlorobiphenyl	127L	Cambridge	EC-1421	40,000	1000	100
$^{13}C_{12}$-2,2',3,4,4',5'-hexachlorobiphenyl	138L	Cambridge	EC-1436	40,000	1000	100
$^{13}C_{12}$-2,2',3,4,4',5,5'-heptachlorobiphenyl	180L	Cambridge	EC-1407	40,000	1000	100
$^{13}C_{12}$-2,2',3,3',4,4',5,5'-octachlorobiphenyl	194L	Cambridge	EC-1418	40,000	1000	100
Cleanup Surrogates						
$^{13}C_{12}$-2,4,4'-trichlorobiphenyl	28L	Cambridge	EC-1413	40,000	5000	10
$^{13}C_{12}$-2,3,3',5,5'-pentachlorobiphenyl	111L	Cambridge	EC-1415	40,000	5000	10
$^{13}C_{12}$-2,2',3,3',5,5',6-heptachlorobiphenyl	178L	Cambridge	EC-1417	40,000	5000	10

Table 4 - Acceptance Criteria for Performance Tests

Toxic & LOC Congeners	IUPAC	Test Conc (ng/mL) [1]	IDOC %RSD	IDOC %R	LCS %R
2-chlorobiphenyl	1	50	40	60-140	50-150
4-chlorobiphenyl	3	50	40	60-140	50-150
2,2'-dichlorobiphenyl	4	50	40	60-140	50-150
4,4'-dichlorobiphenyl	15	50	40	60-140	50-150
2,2',6-trichlorobiphenyl	19	50	40	60-140	50-150
3,4,4'-trichlorobiphenyl	37	50	40	60-140	50-150
2,2',6,6'-tetrachlorobiphenyl	54	50	40	60-140	50-150
3,3',4,4'-tetrachlorobiphenyl	77	50	40	60-140	50-150
3,4,4',5-tetrachlorobiphenyl	81	50	40	60-140	50-150
2,2'4,6,6'-pentachlorobiphenyl	104	50	40	60-140	50-150
2,3,3',4,4'-pentachlorobiphenyl	105	50	40	60-140	50-150
2,3,4,4',5-pentachlorobiphenyl	114	50	40	60-140	50-150
2,3',4,4',5-pentachlorobiphenyl	118	50	40	60-140	50-150
2',3',4,4',5-pentachlorobiphenyl	123	50	40	60-140	50-150
3,3',4,4',5-pentachlorobiphenyl	126	50	40	60-140	50-150
2,2',4,4',6,6'-hexachlorobiphenyl	155	50	40	60-140	50-150
2,3,3',4,4',5-hexachlorobiphenyl	156	50	40	60-140	50-150
2,3',4,4',5,5'-hexachlorobiphenyl	157	50	40	60-140	50-150
2,3,3',4,4',5'-hexachlorobiphenyl	167	50	40	60-140	50-150
3,3',4,4',5,5'-hexachlorobiphenyl	169	50	40	60-140	50-150
2,2',3,4'5,6,6'-heptachlorobiphenyl	188	50	40	60-140	50-150
2,3,3',4,4',5,5'-heptachlorobiphenyl	189	50	40	60-140	50-150
2,2',3,3',5,5',6,6'-octachlorobiphenyl	202	50	40	60-140	50-150
2,3,3',4,4',5,5',6-octachlorobiphenyl	205	50	40	60-140	50-150
2,2',3,3',4,4',5,5',6-nonachlorobiphenyl	206	50	40	60-140	50-150
2,2',3,3',4,5,5',6,6'-nonachlorobiphenyl	208	50	40	60-140	50-150
2,2',3,3',4,4',5,5',6,6'-decachlorobiphenyl	209	50	40	60-140	50-150
Isotope Dilution Analytes (IDAs)					
$^{13}C_{12}$-2-chlorobiphenyl	1L	100	50	35-135	30-140
$^{13}C_{12}$-4-chlorobiphenyl	3L	100	50	35-135	30-140
$^{13}C_{12}$-2,2'-dichlorobiphenyl	4L	100	50	35-135	30-140
$^{13}C_{12}$-4,4'-dichlorobiphenyl	15L	100	50	35-135	30-140
$^{13}C_{12}$-2,2',6-trichlorobiphenyl	19L	100	50	35-135	30-140
$^{13}C_{12}$-3,4,4'-trichlorobiphenyl	37L	100	50	35-135	30-140
$^{13}C_{12}$-2,2',6,6'-tetrachlorobiphenyl	54L	100	50	35-135	30-140
$^{13}C_{12}$-3,3',4,4'-tetrachlorobiphenyl	77L	100	50	35-135	30-140
$^{13}C_{12}$-3,4,4',5-tetrachlorobiphenyl	81L	100	50	35-135	30-140
$^{13}C_{12}$-2,2',4,6,6'-pentachlorobiphenyl	104L	100	50	35-135	30-140
$^{13}C_{12}$-2,3,3',4,4'-pentachlorobiphenyl	105L	100	50	35-135	30-140
$^{13}C_{12}$2,3,4,4',5-pentachlorobiphenyl -	114L	100	50	35-135	30-140
$^{13}C_{12}$-2,3',4,4',5-pentachlorobiphenyl	118L	100	50	35-135	30-140
$^{13}C_{12}$-2',3,4,4',5-pentachlorobiphenyl	123L	100	50	35-135	30-140
$^{13}C_{12}$-3,3',4,4',5-pentachlorobiphenyl	126L	100	50	35-135	30-140
$^{13}C_{12}$-2,2',4,4',6,6'-hexachlorobiphenyl	155L	100	50	35-135	30-140
$^{13}C_{12}$-2,3,3',4,4',5-hexachlorobiphenyl	156L	100	50	35-135	30-140
$^{13}C_{12}$-2,3,3',4,4',5'-hexachlorobiphenyl	157L	100	50	35-135	30-140
$^{13}C_{12}$-2,3',4,4',5,5'-hexachlorobiphenyl	167L	100	50	35-135	30-140
$^{13}C_{12}$-3,3',4,4',5,5'-hexachlorobiphenyl	169L	100	50	35-135	30-140
$^{13}C_{12}$-2,2',3,3',4,4',5-heptachlorobiphenyl	170L	100	50	35-135	30-140
$^{13}C_{12}$-2,2',3,4',5,6,6'-heptachlorobiphenyl	188L	100	50	35-135	30-140
$^{13}C_{12}$-2,3,3',4,4',5,5'-heptachlorobiphenyl	189L	100	50	35-135	30-140
$^{13}C_{12}$-2,2',3,3',5,5',6,6'-octachlorobiphenyl	202L	100	50	35-135	30-140

Table 4 - Acceptance Criteria for Performance Tests (continued)

Isotope Dilution Analytes (IDAs)	IUPAC	Test Conc (ng/mL) [1]	IDOC %RSD	IDOC %R	LCS %R
$^{13}C_{12}$-2,3,3',4,4',5,5',6-octachlorobiphenyl	205L	100	50	35-135	30-140
$^{13}C_{12}$-2,2',3,3',4,4',5,5',6-nonachlorobiphenyl	206L	100	50	35-135	30-140
$^{13}C_{12}$-2,2',3,3',4,5,5',6,6'-nonachlorobiphenyl	208L	100	50	35-135	30-140
$^{13}C_{12}$-2,2',3,3',4,4',5,5',6,6'-decachlorobiphenyl	209L	100	50	35-135	30-140
Cleanup Surrogates					
$^{13}C_{12}$-2,4,4'-trichlorobiphenyl	28L	50	45	45-120	40-125
$^{13}C_{12}$-2,3,3',5,5'-pentachlorobiphenyl	111L	50	45	45-120	40-125
$^{13}C_{12}$-2,2',3,3',5,5',6-heptachlorobiphenyl	178L	50	45	45-120	40-125

1 Test concentrations are based on ng/mL in the sample extract or standard solution.

Table 5 - Assignment of Sample Preparation Protocols for Soxhlet Extraction

1668 Protocol Number	Protocol Name	Sample Amount Extracted (g)	Fraction of Extract Cleaned	TALS Final Volume-Extraction Batch (mL)	TALS Initial Volume-Split Batch (mL)	Prep Split Factor	Volume IDA Added (mL)	Volume Cleanup SU Added (mL)	Volume IS Added (µL)	Extract Final Volume (µL)
1	Clean	10	1/2	20	10	2	1	0.5 (added after split)	50	50
2	Low	2	1	10	10	1	1	1	100	100
3	Medium	1.25	1/2	20	10	2	2	1 (added after split)	100	250
4	High	1	1/4	40	10	4	4	1 (added after split)	100	500

Appendix I - Guidelines for the Spike Witnessing Process

- Make sure there are no distractions, for example, phone calls, checking samples on water bath, people coming in to ask questions.
- The person spiking must tell the person who is witnessing what is being spiked and how much.
- The person witnessing should make sure they know and understand what is to be spiked and how much. Check the paperwork to verify.
- Check the syringe for air bubbles and also check the spike volume.
- Verify that the spiking solution has not expired.
- It is a good idea to also check for cracks in the glassware.
- If client service requires spiking to occur when another analyst is not available, a witness is not required. In this case, the analyst will serve as his/her own witness, and must carefully double check the spike solutions and spike amounts added to the client samples and associated quality control samples. The analyst enters his/her initials as both the analyst and witness.

Title: GAS CHROMATOGRAPHIC ANALYSIS of PCBs BASED ON METHODS 8082 and 8082A

Approvals (Signature/Date):

Technology Specialist	08/10/20 Date	Health & Safety Coordinator	08/11/20 Date
Quality Assurance Manager	08/25/20 Date	Technical Director	08/11/10 Date

This SOP was formerly known as NC-GC-045 Rev. 2, dated 04/20/18

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1. SCOPE AND APPLICATION

1.1. This SOP describes procedures to be used when SW-846 Method 8082 or 8082A are applied to the analysis of polychlorinated biphenyls (PCB) as Aroclors by GC/ECD. This SOP is applicable to extracts derived from any matrices which are prepared according to the appropriate sample extraction SOPs. PCBs are determined and quantitated as Aroclor mixes.

1.2. Table 1 lists compounds routinely determined by this method and the CAS Number. RLs are easily accessible via the Laboratory Information Management System (LIMS). Matrix interferences and/or high concentrations of PCB compounds may result in higher RLs.

1.3. Additional compounds may be amenable to this method, but they may not be reported as Ohio Voluntary Action Program (OVAP) certified data if they are not listed in 8082, and must be attested to as not-certified. If non-standard analytes are required, they must be validated.

1.4. Where requirements of this SOP are not met, including, but not limited to, Quality Control (QC) samples (Method Blank (MB) and Laboratory Control Sample (LCS)) associated to sample analytes, holding time, and preservation, the laboratory will narrate bias in the case narrative of the final report.

2. SUMMARY OF METHOD

2.1. PCBs in aqueous samples are prepared for analysis using separatory funnel extraction (SOP NC-OP-038). Solid samples are prepared using sonication or soxhlet extraction (SOP NC-OP-039 and NC-OP-040).

2.2. After the initial preparation step, the sample is introduced into the GC and the concentration of each target analyte is measured by the detector response within a defined retention time (RT) window, relative to the response of the reference standards.

3. DEFINITIONS

3.1. Refer to the Eurofins Test America Canton Quality Assurance Manual (QAM), current version, for definitions of terms and acronyms used in this document.

4. INTERFERENCES

4.1. Contamination by carryover can occur when a low concentration sample is analyzed after a high concentration sample. Co-elution of target analytes with non-targets can occur, resulting in false positives or biased high results. All glassware is cleaned per SOP NC-QA-014.

4.2. Interferences in the GC analysis can arise from many compounds which are amenable to gas chromatography and give a measurable response on the electron capture detector (ECD). Phthalate esters, which are common contaminants found in plastics, can pose a major problem in the determinations. Avoiding contact with any plastic materials minimizes interferences from phthalates.

4.3. Compounds extracted from the sample matrix to which the detector will respond, such as single-component chlorinated pesticides, including the DDT analogs (DDT, DDE, and DDD) will cause interference. A standard of the DDT analogs should be injected to determine which of the PCB or Aroclor peaks may be subject to interferences on the analytical columns used. There may be substantial DDT interference with the last major Aroclor 1254 peak in some soil and sediment samples.

4.4. All sample and associated QC extracts undergo sulfuric acid clean-up before analysis to remove non-target interfering organic compounds. Sulfur will interfere and can be removed using procedures described in SOP NC-OP-025.

4.5. Interferences co-extracted from samples will vary considerably. The presence of interferences may raise quantitation limits for individual samples. Specific cleanups may be performed on the sample extracts. These cleanup procedures are included in SOP NC-OP-025.

5. SAFETY

5.1. Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual, the Facility Addendum to the Corporate EH&S Manual, and this document. Eye protection that prevents splash, laboratory coat, and appropriate gloves must be worn while samples, standards, solvents, and reagents are being handled. Refer to the Eurofins TestAmerica Canton Corporate Environmental Health and Safety Manual for a complete description of personal protection equipment.

5.2. Cut-resistant gloves must be worn when doing any other task that presents a strong possibility of getting cut. Disposable gloves that have become contaminated must be removed and discarded; other gloves must be cleaned immediately. Nitrile gloves provide adequate protection against the solvents used in this method.

5.3. The following is a list of the materials used in this method, which have a serious or significant hazard rating. NOTE: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the Safety Data Sheet (SDS) for each of the materials listed in the table. A complete list of materials used in the method can be found in the Reagents and Standards section. Employees must review the information in the SDS for each material before using it for the first time or when there are major changes to the SDS.

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Material	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Acetone	Flammable	1000 ppm-TWA	Inhalation of vapors irritates the respiratory tract. May cause coughing, dizziness, dullness, and headache.
Hexane	Flammable Irritant	500 ppm-TWA	Inhalation of vapors irritates the respiratory tract. Overexposure may cause lightheadedness, nausea, headache, and blurred vision. Vapors may cause irritation to the skin and eyes.
Methylene Chloride	Carcinogen Irritant	25 ppm-TWA 125 ppm-STEL	Causes irritation to respiratory tract. Has a strong narcotic effect with symptoms of mental confusion, light-headedness, fatigue, nausea, vomiting and headache. Causes irritation, redness and pain to the skin and eyes. Prolonged contact can cause burns. Liquid degreases the skin. May be absorbed through skin.
Note: Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

5.4. Aroclors have been classified as a potential carcinogen under OSHA. Concentrated solutions of Aroclors must be handled with extreme care to avoid excess exposure. Contaminated gloves and clothing must be removed immediately. Contaminated skin surfaces must be washed thoroughly.

5.5. All ^{63}Ni sources (ECD) must be leak tested every six months, or in accordance with the manufacturer's general radioactive material license.

5.6. All ^{63}Ni sources must be inventoried every six months. If a detector is missing, the EH&S Director must be immediately notified and a letter sent to the NRC or local state agency.

5.7. Exposure to chemicals must be limited as much as reasonably achievable. All samples with stickers that read "Caution/Use Hood!" must be opened in the hood. Contact the EH&S Coordinator if this is not possible. Solvent and waste containers must be kept closed unless transfers are being made.

5.8. Opened containers of neat standards must be handled in a fume hood.

5.9. Sample extracts and standards, which are in a flammable solvent, must be stored in an explosion-proof refrigerator.

5.10. When using hydrogen gas as a carrier, all precautions listed in the CSM must be observed.

5.11. Standard preparation and dilution must be performed inside an operating fume hood.

5.12. The gas chromatograph contains zones that have elevated temperatures. The analyst needs to be aware of the locations of those zones, and must cool them to room temperature prior to working on them.

5.13. There are areas of high voltage in the gas chromatograph. Depending on the type of work involved, either turn the power to the instrument off, or disconnect it from its source of power.

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5.14. All work must be stopped in the event of a known or potential compromise to the health and safety of a Eurofins TestAmerica associate. The situation must be reported immediately to the EH&S Coordinator and the Laboratory Supervisor.

6. EQUIPMENT AND SUPPLIES

6.1. Gas Chromatograph (GC) equipped with Electron Capture Detectors (ECDs)

6.2. Software: ChemStation or equivalent

6.3. Refer to Table 2 for analytical columns and run conditions

6.4. Microsyringes and syringes: various sizes, for standards preparation, sample injection, and extract dilution

6.5. Solvent: Hexane

6.6. Autosampler vials, inserts, and caps

6.7. Class A volumetric flasks: various sizes

6.8. Transfer pipettes: disposable

6.9. VOA vials

6.10. Carrier gas: Hydrogen

6.11. Makeup gas: Nitrogen

7. REAGENTS AND STANDARDS

7.1. Stock Standards: Stock standards are purchased as certified solutions or prepared from pure solutions.

7.1.1. Stock standard solutions are stored as recommended by the manufacturer. All stock standards must be protected from light. Stock standard solutions must be brought to room temperature before using.

7.1.2. Stock standard solutions must be replaced after one year.

7.1.3. Expiration times for all standards are measured from the time the standard is prepared or from the time that the standard ampoule is opened, if the standard is supplied in a sealed ampoule. If vendor-supplied standard has an earlier expiration date then the vendor's expiration date is used. Refer to SOP NC-QA-017, Standards and Reagents, for additional information. The standard preparation information is detailed in the LIMS standards and reagents module.

7.2. Calibration Standards

7.2.1. PCB Calibration Standards

7.2.1.1. PCB calibration standards are prepared as dilutions of the stock standards. Surrogates and internal standards are used as specified in the method. PCB calibration solutions must be refrigerated at 4° C ± 2° C and protected from light. The standards must be replaced at least every six months or sooner if comparison with check standards indicates a problem.

7.2.1.2. See the LIMS Reagent module for details on standard concentration and preparation.

7.3. Surrogate Standards

7.3.1. Tetrachloro-m-xylene (TCMX) and decachlorobiphenyl (DCB) are the surrogate standards. Refer to the LIMS for details of surrogate standards.

7.4. Internal Standard

7.4.1. The internal standard used for PCB analysis is 1-Bromo-2-nitrobenzene (BNB)

Note: The OVAP program prohibits the use of re-verified standards. If no other standard is available, this must be narrated and the data marked as “not certified” on the affidavit.

8. SAMPLE COLLECTION, PRESERVATION AND STORAGE

8.1. Unless otherwise specified by regulatory or client programs, holding time for PCB samples (regardless of matrix) is 1 year.

8.2. Sample extracts are stored at 4 ± 2°C until analysis is complete. The holding time for PCB sample extracts is 40 days.

9. QUALITY CONTROL

9.1. Batch definition

9.1.1. The batch is a set of up to 20 samples of the same matrix processed at the same time using the same procedures and reagents. The Quality Control batch must contain a matrix spike / spike duplicate (MS/MSD), a Laboratory Control Sample (LCS), and a method blank (MB). Laboratory generated QC samples (Method Blank, LCS and MS/MSD do not count towards the maximum 20 samples in a batch. Field QC samples are included in the batch count. In some cases, at client request, the MS/MSD may be replaced with an MS and an un-spiked sample duplicate (DU).

9.2. Method Blank (MB)

9.2.1. For each batch of samples, analyze an MB. The MB consists of reagent water for aqueous PCB samples and sodium sulfate for PCB soils tests and all surrogates required for the analysis. Refer to SOPs NC-OP-038, NC-OP-039, and NC-OP-040 for details.

9.2.2. The MB must not contain any analyte of interest at, or above, the reporting limit or at, or above, 5% of the measured concentration of that analyte in the associated samples, whichever is higher.

Note: Some programs require that the MB be clean to ½ the RL. Method notes should inform the analyst if the samples are part of a special program. Analysts are responsible for checking the program requirements.

9.2.3. Corrective action

9.2.3.1. Corrective action may include re-analysis of the MB. If the re-analysis fails to meet criteria, re-extraction and re-analysis of samples associated with an unacceptable MB is required when concentrations greater than the RL are detected in the samples.

9.2.3.2. If there is no target analyte greater than the RL in the samples associated with an unacceptable MB, the data may be reported with qualifiers.

9.3. **Note:** For Ohio VAP projects, the MB results must be below the reporting limit or the samples must be re-extracted. The exceptions are as follows: (a) insufficient sample for re-extraction (b) expired holding times, or (c) the analytes detected in the blank are non-detect in the associated samples. Under the above circumstances, results may be reported. **Note:** the 10x rule does NOT apply for OVAP.Laboratory Control Samples (LCS)

9.3.1. For each batch of samples, analyze an LCS. The LCS contains a mix of Aroclors 1016 and 1260 and all surrogates required for the analysis. The LCS standard contains the same analytes as the matrix spike. If any LCS analyte is outside the laboratory established historical control limits, corrective action must occur.

9.3.2. Corrective Action

9.3.2.1. Corrective action may include re-analysis of the LCS. If the re-analysis fails to meet criteria, re-extraction and re-analysis of the batch may be needed. If the LCS is biased high, samples that have no analytes detectable above the RL may be reported with proper narration.

Note: For Ohio VAP samples, re-analyze an aliquot of the LCS to verify the outlier; if the LCS exhibits the same anomaly upon re-analysis, the sample batch must be re-extracted and re-analyzed. The exceptions are as follows: (a) insufficient sample for re-extraction (b) expired holding times, or (c) the LCS is biased high and the samples have no detections above the RL for those analytes. Under the above circumstances, results may be reported with proper narration.

9.3.3. LCS compound lists and surrogates are included in the reagent module of the LIMS.

9.4. Matrix Spikes/Spike Duplicates (MS/MSD)

9.4.1. For each QC batch, analyze an MS/MSD. Compare the percent recovery and relative percent difference (RPD) to those in the laboratory-specific historically generated limits.

9.4.2. Corrective Action

9.4.2.1. If any individual recovery or RPD falls outside the acceptable range, corrective action must occur unless samples for this compound are ND. The initial corrective action must be to check the recovery of that analyte in the LCS. Generally, if the recovery of the analyte in the LCS is within limits, adverse matrix effect is indicated, the laboratory operation is in control and analysis may proceed.

9.4.2.2. If the recovery for any component is outside QC limits for both the MS/MSD and the LCS, the laboratory process is out of control and corrective action must be taken.

9.4.3. The MS/MSD must be analyzed at the same dilution as the un-spiked sample.

9.5. Surrogates

9.5.1. Surrogates are added to all samples, and batch QC during the preparation procedure. Surrogates are added to instrument QC for the 1016/1260 Aroclor mix, which includes the calibration standards, and the CCVs. Surrogate recoveries in samples and QC samples must be assessed to ensure that recoveries are within established limits.

9.5.2. Method Blank

9.5.2.1. Surrogates are added to the MB and the MB is carried through the entire analytical procedure. The MB must have acceptable surrogate recoveries. If surrogate recoveries are not acceptable, the data must be evaluated to determine if the method blank has served the purpose of demonstrating that the analysis is free of contamination. If surrogate recoveries are low and there are reportable analytes in the associated samples, re-extraction of the MB and affected samples will normally be required. If surrogate recoveries are high, and the samples are non-detect, the data may be reported with proper narration.

9.5.3. LCS

9.5.3.1. The LCS must have acceptable surrogate recoveries. If surrogate recoveries are not acceptable, the data must be evaluated to determine if the LCS has served the purpose of demonstrating effectiveness of the extraction process. If surrogate recoveries are low, re-extraction of the LCS and affected samples will normally be required. None of the surrogate recoveries can fall below 10% in the

LCS. If surrogate recoveries are high, and the samples have no detections above the RL, the data may be reported with proper narration.

9.5.4. Instrument QC

9.5.4.1. Surrogates in the 1016/1260 calibration standards must meet the same criteria as the ICAL. See section 10 for ICAL criteria.

9.5.4.2. Surrogates in the CCVs must have a %D of <15% for Method 8082, and <20% for Method 8082A.

9.5.4.3. Corrective actions for failing surrogates in the instrument QC are discussed in section 10.

9.5.5. Samples

9.5.5.1. If either surrogate is outside limits, the following corrective actions must take place (except for dilutions greater than 5X):

9.5.5.1.1. Check all calculations for error.

9.5.5.1.2. Ensure instrument performance is acceptable.

9.5.5.1.3. Recalculate the data and/or re-analyze the extract if either of the above checks reveals a problem.

9.5.5.2. It is only necessary to re-prepare / re-analyze a sample once to demonstrate poor surrogate recovery is due to matrix effect, unless the analyst believes that the repeated out-of-control results are not due to matrix effect.

9.5.5.3. If the surrogates are out of control for the sample and MS/MSD, then matrix effect has been demonstrated for that sample and re-preparation is not necessary. If the sample is out of control and the MS/MSD is in control, then re-preparation or flagging of the data is required. Re-preparation includes the parent sample and the MS/MSD.

9.5.6. **Note:** For Ohio VAP projects all surrogates must be within acceptance criteria. For surrogate outliers, the analyst should either: (a) re-analyze, (b) re-prep/re-analyze, or (c) perform a dilution. When there is an obvious interference causing the surrogate outlier that the analyst knows a corrective action would not resolve the issue, flag the data with a qualifier indicating matrix interference. The exceptions for re-prep/re-analysis are as follows: (a) insufficient sample for re-analysis, or (b) the surrogates are biased high and the samples are non-detect. Under the above circumstances, results may be reported. All batch QC samples must pass surrogate criteria or the batch (samples and QC) must be re-analyzed if sufficient volume and holding time remains.

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9.5.7. Refer to Eurofins TestAmerica Canton QC Program document (Policy QA-003) for further details of the corrective actions.

9.6. Internal Standard (IS)

9.6.1. This procedure is an IS procedure. 1-Bromo-2-nitrobenzene (BNB) is the internal standard used.

9.6.2. Prior to analysis, the IS must be added to all standards, field sample extracts, and QC sample extracts. The concentration of the IS must be the same in all calibration standards, field sample extracts, and QC sample extracts. A concentration of 0.05 ug/mL is used.

9.6.3. The response of the IS in the ICV/CCV must be within 50-200% of the response of the IS in the CCV-level standard in the initial calibration sequence. If the response is outside of this range, the analysis of the ICV/CCV must be repeated and any samples associated with the ICV/CCV must also be re-analyzed. Repeated failure of the IS response will require recalibration.

9.6.4. The response of the IS in the samples and batch QC items must be within 50-200% of the response of the previous CCV. If the response is outside of the range, corrective action must be taken which may include re-analysis of the extract, re-spiking the extract with IS and re-analysis, or re-calibration of the analytical system. Obvious matrix interferences are qualified and noted in an NCM.

9.7. **Note:** For OVAP projects, samples with failing internal standards must be re-analyzed unless matrix interference is apparent. If matrix interference is apparent, dilute the sample with hexane. When there is obvious interference causing the IS failure that corrective action will not remedy, data must be narrated as matrix interference. If the QC has failing internal standards, the associated batch (samples and batch QC) must be re-spiked and re-analyzed.

9.8. Control Limits

9.8.1. Control limits are established by the laboratory as described in SOP NC-QA-018.

9.8.2. Laboratory control limits are internally generated and updated periodically unless method specified. Control limits are easily accessible via the LIMs.

10. CALIBRATION AND STANDARDIZATION

10.1. Internal or external calibration may be used. Prepare standards containing each analyte of interest at a minimum of five concentration levels. The low-level standard must be at, or below, the reporting limit. The other standards define the working range of the detector.

10.2. The initial PCB calibration must include at least one level with 1016 analyzed separately for pattern recognition purposes. This run does not have to be part of the calibration.

10.3. A new calibration curve must be generated after major changes to the system or when the continuing calibration criteria cannot be met. Major changes include new columns or replacing the ECD detector. A new calibration is not required after clipping the column, replacing the septum or syringe, or other minor maintenance, unless CCV criteria cannot be met.

10.4. With the exception of section 10.5 below, it is not acceptable to remove points from a calibration curve for the purpose of meeting criteria, unless the points are the highest or lowest on the curve and the reporting limit and/or linear range is adjusted accordingly. In any event, at least five points must be included in the calibration curve. Quadratic (second order) calibrations require at least six points.

Note: Quadratic calibrations are not acceptable for PCB analysis for OVAP projects.

10.5. A level may be removed from the calibration if the reason can be clearly documented (for example, a broken vial or no purge run). A minimum of five levels must remain in the calibration. The documentation must be retained with the initial calibration. Alternatively, if the analyst believes that a point on the curve is inaccurate, the point may be re-analyzed and the re-analysis used for the calibration. All initial calibration points in a single calibration curve must be analyzed without any changes to instrument conditions, and all points in a single calibration curve must be analyzed within 24 hours.

10.6. Internal Standard Calibration

10.6.1. Internal standard calibration involves the comparison of instrument responses from the target compounds in the sample extract to the response of a specific standard added to the r sample extract prior to injection. This specific standard is referred to as the internal standard (IS) because it is contained within the aliquot of the sample extract and is actually injected into the instrumentation. The ratio of the peak area (or height) of the target compound in the sample extract to peak area (or height) of the IS in the sample extract is compared to a similar ratio derived for each calibration standard. The ratio is termed the response factor (RF), or relative response factor (RRF).

10.6.2. When preparing calibration standards for use with internal standard calibration, add the same amount of internal standard solution to each calibration standard, such that the concentration of each internal standard is constant across all of the calibration standards, whereas the concentrations of the target analytes will vary. The mass of the internal standard added to each sample extract immediately prior to injection into the instrument must be the same as the mass of the internal standard in each calibration standard. The volume of the solution spiked into sample extracts should be such that minimal dilution of the extract occurs.

10.6.3. Prepare calibration standards at a minimum of five concentration levels for each parameter of interest and each surrogate. Six standards must be used for a quadratic least squares calibration. Add the appropriate amount of the IS mixture to result in a 0.05 ug/mL concentration. The low standard must be at or below the reporting limit

10.6.4. Calculate response factors (RF), average response factors, and the percent RSD of the response factors for each compound and surrogate using the equations in Section 12.

10.7. External standard calibration

10.7.1. Quantitation by the external standard method assumes a proportional relationship between the analyte or surrogate response and the concentration that is the same in all of the calibration standards and the samples. To use this approach, introduce each calibration standard into the GC using the technique that will be used for samples. The ratio of the peak height or area response to the mass or concentration injected is used to prepare a calibration curve.

10.8. Calibration Curve Fits

10.8.1. The calculations for all calibration curve fits are found in section 12.

10.8.2. Weighted linear regression, average calibration factor, non-weighted linear regression, or quadratic curves may be used to fit the data. - Average calibration factor is the preferred calibration model used for PCB.

10.9. Average calibration factor (CF) / response factor (RF)

10.9.1. The average CF (external calibration) or RF (internal standard) may be used if the average percent relative standard deviation (% RSD) of all the CFs / RFs taken together is $\leq$ 20%. The average % RSD is calculated by summing the %RSD value for each peak and dividing by the total number of peaks.

10.10. Linear Regression / Weighted Linear Regression

10.10.1. Linear / weighted linear regressions must have a minimum of 5 calibration levels with the lowest being at or below the reporting limit. The correlation coefficient (r) must be >0.990. The ICV %D must be <20%, and the CCV %D must be <15% for Method 8082. For Method 8082A, the criteria for both ICV and CCV are < 20% % D.

10.11. Quadratic Curve

10.11.1. A quadratic calibration curve must only be used if the analyst has reason to believe that a linear or average model does not fit the normal concentration-to-response behavior of the detector. A quadratic curve fit may be used only if the compounds have historically exhibited a non-linear response and cannot be used to extend the calibration range for compounds that normally exhibit a linear response, but within a narrower calibration range.

10.11.2. A quadratic calibration curve must have a minimum of 6 calibration levels with the lowest being at or below the reporting limit. The coefficient of determination (r^2) must be > 0.990.

10.12. Evaluation of Calibration Curves

10.12.1. The percent relative standard error (% RSE) from the calibration curve is used to evaluate the initial calibration. This provides a measure of how much error is associated with using the calibration curve for quantitation.

10.12.2. The least squares regression line is calculated and used to calculate the predicted concentration for each level.

Note: When average calibration factors are used, %RSE is equivalent to %RSD.

10.13. The following requirements must be met for any calibration to be used.

10.13.1. Response must increase with increasing concentration.

10.13.2. If a curve is used, the calculated intercept of the curve at zero response must be less than ± the reporting limit for the analyte.

10.13.3. The average Relative Standard Error (RSD for average response factors) of the calibration points from the curve used must be ≤ 20%.

10.13.4. Some data systems will not measure the %RSE from a linear or quadratic fit. For the linear case, the correlation coefficient may be used as an alternative to the %RSE, and must be greater than or equal to 0.990. For the quadratic case the Coefficient of Determination (COD) may be used, and must be greater or equal to 0.990.

Note: The Relative Standard Error (RSE) is superior to the Correlation Coefficient (*r*) and Coefficient of Determination (r^2) for testing the fit of a set of calibration points to a line. The lower points on a curve have little effect on *r.* As a result, a curve may have a very good correlation coefficient (>0.990) while also having > 100% error at the low point.

Note: The surrogates must be judged against these same criteria.

10.14. Weighting of Data Points

10.14.1. In linear and quadratic calibration fits, the points at the lower end of the calibration curve have less absolute variance than points at the high concentration end of the curve. This can cause severe errors in quantitation at the low end of the calibration. However, in environmental analysis, accuracy at the low end of the curve is very important. For this reason, it is preferable to increase the weighting of the lower concentration points. $1/Concentration^2$ weighting (often called $1/X^2$ weighting) will improve accuracy at the low end of the curve and must be used when an average calibration factor is not appropriate, as long as the data system has this capability.

10.15. Initial Calibration

10.15.1. A minimum five-point calibration of all Aroclors is generated. The low-level standard must be at or below the reporting limit. The other standards define the working range of the detector.

10.15.2. If any Aroclor is determined above the calibration range, the extract must be diluted and re-analyzed.

10.15.3. The surrogate calibration curve is included in the calibration from the Aroclor 1016/1260 mix.

10.16. Initial Calibration Verification (ICV)

10.16.1. The ICVs are analyzed immediately after an initial calibration. The acceptance criterion is ± 20%.

10.16.2. If the overall average percent drift of all analytes is greater than ±20%, corrective action must be taken. This may include clipping the column, changing the liner, or other minor instrument adjustments, followed by re-analyzing the standard. If the overall average percent drift still varies by more than ±20%, a new calibration curve may be required.

10.17. Continuing Calibration Verification (CCV)

10.17.1. It is not necessary to run a calibration verification standard at the beginning of the sequence if samples are analyzed immediately after the completion of the initial calibration.

10.17.2. Mid-level standards covering all Aroclors of interest are used for the calibration verifications.

10.17.3. CCVs are analyzed for all Aroclors at the beginning of each day, and at the beginning of each 12 hour shift.

10.17.4. Retention times windows are updated daily from the opening CCVs.

10.17.5. Evaluate the resolution of the triplet towards the end of the 1260 chromatogram in the opening CCV. There must be at least 25% resolution on one of the two columns used.

10.17.6. The Aroclor 1016/1260 CCV is analyzed after every 20 injections. Depending on the type of samples, it may be advisable to analyze verifications more frequently in order to minimize reruns.

Note: Various programs require a CCV every 10 injections. Analysts are responsible for checking the program requirements.

10.17.7. For Method 8082A, the CCV acceptance criterion is ± 20%. For Method 8082, the CCV acceptance criterion is ±15%.

10.17.8. A bracketing CCV is not required for IS calibrations.

10.17.9. Samples quantitated against an external standard calibration must be bracketed by a 1016/1260 CCV.

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10.17.10. Corrective Actions for Continuing Calibration Failures

10.17.10.1. If a CCV fails to meet criteria, corrective action must be taken. This may include clipping the column, changing the liner, or other minor instrument adjustments, followed by re-analyzing the standard. If the re-analysis still fails to meet criteria, a new calibration curve may be required.

10.17.10.2. Samples quantitated against an external standard calibration must be bracketed with passing CCVs. All samples analyzed immediately prior to, and immediately following the failing CCV must be re-analyzed.

10.17.10.3. Sample results that are below the reporting limit may be reported when a CCV is biased high. Such action must be addressed in the case narrative.

10.18. Retention Time Windows

10.18.1. Retention time (RT) windows must be determined for all analytes.

10.18.2. Initial determination of Retention time windows

10.18.2.1. The center of the retention time (RT) window shall be updated based on the middle level in the initial calibration or the first CCV in the daily analytical sequence, whichever is more recent.

10.18.2.2. Evaluate the deviation from expected retention time for each analyte in at least three CCV and/or LCS samples spread over at least 72 hours. Calculate the standard deviation of these retention times.

10.18.2.3. If three days of analytical data are not available, use a default RT window of 0.01 minutes. At the end of the batch evaluate all CCVs and LCS in the batch. If necessary, widen the window such that all analytes fall within the RT window. Reprocess the batch using the new RT windows.

10.18.2.4. Multiply the standard deviation by 3. This is the retention time window, unless the result is less than 0.01 min, in which case the window is set at 0.01 min.

10.18.2.5. An alternative method to determine the retention time window is to multiply the maximum deviation of all points by 1.5. The minimum retention time window is 0.01 minutes. For example, if the maximum RT deviation for a specific analyte is 0.008 min, then the RT window is set at ± 0.012 min.

Note: For multi-component analytes, like Aroclors, the maximum deviation must be evaluated for each of the 3 to 6 major peaks used for sample calculations.

10.18.2.6. If the retention time windows for analytes of interest overlap, the analyte must be confirmed on a dissimilar column.

10.18.3. Ongoing evaluation of retention time windows

10.18.3.1. Evaluate the retention time windows on an ongoing basis. The center of the RT window is updated on the first CCV of the day and verified every 12 hours. All analytes for all subsequent CCVs, LCS and matrix spikes must fall within the retention time window (except as discussed below).

10.18.3.2. Matrix spike analytes may fall outside the retention time window if there is a large non-target peak coeluting with the analyte in the matrix spike.

10.18.3.3. If any analytes fall outside the retention time window in CCVs, LCS or matrix spikes (except as discussed above for matrix spikes) then the RT windows for those analytes shall be widened to the minimum degree required for the analyte to fall within the RT window. All samples in the batch shall be reprocessed with the new RT window, and the wider RT window shall remain in place for subsequent batches.

10.18.3.4. Retention time windows should be reliably narrower than +/- 0.03 min. If RT windows wider than this are necessary, the instrument should be evaluated and maintenance performed as needed. Subsequent to maintenance, RT windows shall be narrowed to the extent that is consistent with the data obtained.

11. PROCEDURE

11.1. Procedural variations are allowed only if deemed necessary in the professional judgment of QA, operations supervisor, or designee to accommodate variation in sample matrix, chemistry, sample size, or other parameters. Any variation in procedure must be completely documented using a Nonconformance Memo. The Nonconformance Memo must be filed in the project file. The nonconformance is also addressed in the case narrative. Any unauthorized deviations from this procedure must also be documented as a nonconformance with a cause and corrective action described.

Note: Procedural deviations are not allowed for Ohio VAP Projects.

11.2. Extraction

11.2.1. Extraction procedures are referenced in the SOPs NC-OP-038, NC-OP-039, and NC-OP-040, current revisions.

11.3. Suggested gas chromatographic conditions are given in Table 2.

11.4. Allow extracts to warm to ambient temperature before injection.

11.5. Sample Analysis

11.5.1. Run between 2 and 5 instrument blanks before calibration or running the -opening CCV. The instrument blanks consist of Hexane, and are used to clean the instrument prior to analysis.

Note: Instrument blanks are not surrogated.

11.5.2. For samples analyzed by IS calibration models, transfer 100 µL of the extract to an autosampler vial and add 5 µL of the IS solution or equivalent volumes to give an IS concentration of 0.05 ug/mL. The concentration of the internal standard must be the same in all calibration samples, field samples, and QC samples. -.

11.5.3. For samples analyzed by external calibration models, transfer an adequate amount of extract into an autosampler vial and load the vials onto the instrument.

11.5.4. The sample extract must be injected using the same injection volume used for the calibration standards.

11.5.5. If highly contaminated samples are expected, it is acceptable to analyze solvent blanks or primers at any point in the run. Solvent blanks may not be routinely analyzed prior to QC samples.

11.6. Identification of Aroclors

11.6.1. The laboratory performs Update IV's recommended analysis of DDT and analogs DDD and DDE daily to assure there is no interference with major 1254 peaks.

11.6.2. Retention time windows are used for the identification of Aroclor peaks, but the "fingerprint" produced by major peaks of those analytes in the standard is used to differentiate the identity of any Aroclor that may be present. The ratios of the areas of the major peaks are also taken into consideration. Analyst judgement must weigh heavily in the identification of Aroclor patterns. Identification may be made even if the retention times of the peaks in the sample fall outside of the retention time windows of the standard, if the fingerprint (retention time and peak ratios) resembles the standard chromatogram.

11.7. Quantitation of Aroclors

11.7.1. For PCB analysis, the preferred reporting approach is the primary column approach. Dependent upon client program, the higher or lower result may be used for quantitation. Results may be report from the confirmation column when matrix interference is present and impacts either the IS or specific compounds and the RPD between columns.

11.7.2. Use 3-5 major peaks for quantitation

11.7.3. If the analyst believes that a combination of Aroclor 1254 and 1260 or a combination of 1242, 1248 and 1232 is present, then only the predominant Aroclor is quantitated and reported, but the suspicion of multiple Aroclors is discussed in the narrative. If well-separated Aroclor patterns are present, then multiple Aroclors may be quantitated and reported.

11.7.4. Every sample undergoes dual column analysis; however, reporting the second column results will only be performed when requested by the client or regulatory program. The appearance of the multiple characteristic peaks in the sample usually serves as a confirmation of the presence of an Aroclor.

11.8. Surrogate recovery results are calculated and reported for decachlorobiphenyl (DCB) and tetrachloro-m-xylene (TCMX). Corrective action is necessary if either DCB or TCMX are outside of acceptance limits. Samples may be reported when there is obvious matrix interference present; however, such action must be included in the case narrative. See section 9.5 for further information on surrogate recovery.

11.9. Dilutions

11.9.1. Samples may be screened to determine the appropriate dilution for the initial run.

11.9.2. If concentrations of any analytes exceed the working range as defined by the calibration standards, then the sample must be diluted and re-analyzed.

11.9.3. Dilutions must target the most concentrated analyte in the upper half (over 50% of the high level standard) of the calibration range when allowed by sample matrix.

11.9.4. It may be necessary to dilute samples due to matrix. Analyst judgment is required to determine the most concentrated dilution that will not result in instrument contamination.

11.9.5. If the sample is run at a dilution and only minor matrix interferences are observed, then the sample should be re-analyzed at a more concentrated dilution in an attempt to target the upper half of the calibration range.

11.9.6. The most concentrated dilution with no target compounds above the calibration range should be reported. Other dilutions may be reported at client request if the lower dilutions will not cause detector saturation, column overload, or carryover. Analyst judgment and client site history will be factors in the reporting of multiple dilutions.

11.10. Interferences

11.10.1. If peak detection is prevented by interferences, then elevation of reporting levels and/or lack of positive identification must be addressed in the case narrative.

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11.11. Reporting of Data

11.11.1. The preferred reporting approach is the primary column approach, where the analyst evaluates the opening CCVs and designates a primary column to report data from. All data is reported from the primary column in an analytical run, as long as the RPD between columns is less than 40%.

11.11.2. When the RPD between the primary and confirmation column exceeds 40%, it is an indication of likely matrix interference. Every situation is different and the analyst must rely on their experience in choosing which data to report. Two common issues are addressed below:

11.11.2.1. When there is interference with an analyte of interest, the higher result is likely biased due to matrix; therefore, the lower result is reported.

11.11.2.2. When there is interference with the IS, the results on the entire column are biased; therefore, the column without interference in the IS may be the better result. Dilution and reanalysis may be required when there is interference with the IS.

11.12. Analytical Documentation

11.12.1. Record all analytical information in LIMS, including any corrective actions or modifications to the method.

11.12.2. Record all standards and reagents in the LIMS Reagents module. All standards and reagents are assigned a unique number for identification.

Note: When making new standards, it is required that all information entered into TALS is reviewed by another analyst.

11.12.3. Record sample and associated QC information in the LIMs. Level I and Level II technical reviews are performed in LIMS.

12. DATA ANALYSIS AND CALCULATIONS

12.1. Calibration Calculations

12.1.1. Calibration Factor (CF) for external calibration and Response Factor (RF) internal standard calibration

$$CF = \frac{A_s}{C_s}$$

Where A_s = Peak area (or height) in standard

C_s = Concentration of standard injected

$$RF = \frac{A_x C_{is}}{A_{is} C_x}$$

Where: A_x = Peak Area (or height) of the analyte or surrogate

A_{is} = Peak Area (or height) of the internal standard

C_x = Concentration of the analyte or surrogate (µg/L)

C_{is} = Concentration of the internal standard (µg/L)

12.1.2. Evaluating the linearity of the initial calibration using an Average Calibration fit:

$$Mean\ CF = \overline{CF} = \frac{\sum_{i=1}^{n} CF_i}{n}$$ (external calibration)

$$Mean\ RF = \overline{RF} = \frac{\sum_{i=1}^{n} RF_i}{n}$$ (internal standard calibration)

Where n = the number of calibration standards

$$Standard\ Deviation\ (SD) = \sqrt{\frac{\sum_{i=1}^{n}(CF_i - \overline{CF})^2}{n-1}}$$ (external calibration)

$$SD = \sqrt{\frac{\sum_{i=1}^{n}(RF_i - \overline{RF})^2}{n-1}}$$ (internal standard calibration)

Where n = the number of calibration standards

$$\%RSD = \frac{SD}{\overline{CF}} \times 100$$ (external)

$$\%RSD = \frac{SD}{\overline{RF}} \times 100$$ (internal standard)

Note: If the RSD of the calibration or response factors is less than or equal to 20%, the average calibration or response factor may be used to determine sample concentrations.

12.1.3. Percent Difference (%D)

$$\%D = \frac{CF_c - \overline{CF}}{\overline{CF}} \times 100$$

Where CF_c = The calibration factor from the CCV or ICV

$\overline{CF}$ = The average calibration factors from the initial calibration

$$\%D = \frac{RF_c - \overline{RF}}{\overline{RF}} \times 100$$

Where RF_c = The response factor from the CCV or ICV

$\overline{RF}$ = The average response factors from the initial calibration

12.2. Linear regressions

y = ax + b

Where y = Instrument response (area or height)

a = Slope of the line

x = Concentration of the calibration standard

b = The intercept

12.2.1. For internal standard the equation for weighted or non-weighted linear regression is:

$$C_s = \frac{\left(\frac{A_s C_{is}}{A_{is}} - b\right)}{a}$$

Where A_s = Area (or height) of the peak for the target analyte in the sample

A_{is} = Area (or height) of the peak for the internal standard

C_s = Concentration of the target analyte in the calibration standard

C_{is} = Concentration of the internal standard

a = Slope of the line

b = The intercept

12.2.2. Correlation Coefficient

$$r = \frac{\sum d_x d_y}{\sqrt{\sum d_x^2 \sum d_y^2}}$$

Where:

d_x = deviation of x from the mean

d_y = deviation of y from the mean

12.2.3. Relative Standard Error (%RSE)

$$\%RSE = 100\% \times \sqrt{\frac{\sum_{i=1}^{n} \left[\frac{C_i - PC_i}{C_i}\right]^2}{(n-p)}}$$

Where: n = Number of points in the curve
p = Number of parameters in the curve (= 1 for average response factor, 2 for linear, 3 for quadratic)
C_i = True concentration for level i
PC_i = Predicted concentration for level i

12.2.4. Percent Drift for CCV and ICV

$$\%Drift = 100\% \times \frac{C_{actual} - C_{found}}{C_{actual}}$$

Where: C_{actaul} = Known concentration in standard

C_{found} = Measured concentration in CCV or ICV

12.2.5. Curve weighting may be beneficial for linear regressions. Curve weighting improves the linearity at the low end of the calibration curve. Weighted curve calculations are similar to the unweighted calculations above, but use 1/x or $1/x^2$ (where x = concentration) to determine the regression instead of the concentration.

12.3. Quadratic calibration fit (only to be used if applicable)

$y = ax^2 + bx + c$

Where: y = Response

x = Concentration

a = Curvature

b = Slope

c = Intercept

12.3.1. Coefficient of Determination (COD)

$$COD = r^2 = \frac{\sum_{i=1}^{n}(y_{obs} - \bar{y})^2 - \left(\frac{n-1}{n-p}\right)\sum_{i=1}^{n}(y_{obs} - y_i)^2}{\sum_{i=1}^{n}(y_{obs} - \bar{y})^2}$$

Where: y_{obs} = Observed response (area) for each concentration from each initial calibration standard

$\bar{y}$ = Mean observed response from the initial calibration

y_i = Calculated response at each concentration from the initial calibration

n = Total number of calibration points

p = Number of adjustable parameters in the polynomial equation (2 for second order polynomial)

12.4. Concentration

12.4.1. Aqueous

$$Concentration\ \left(\frac{\mu g}{L}\right)\frac{C_{ex}V_t}{V_o}$$

Where C_{ex} = On-column concentration as determined in sections 12.1-12.3. (ng/µL)

V_t = Final extract volume (µL)

V_o = Initial sample volume (mL)

12.4.2. Non-aqueous

$$Concentration\ \left(\frac{\mu g}{Kg}\right) = \frac{C_{ex}V_t}{W_s D}$$

Where C_{ex} = On-column concentration as determined in sections 12.1-12.3. (ng/µL)

V_t = Final extract volume (µL)

W_s = Initial sample mass (g)

D = (100-%moisture in sample) / 100 for dry weight basis

or

D = 1 for a wet weight basis

13. METHOD PERFORMANCE

13.1. Method Detection Limit

13.1.1. Each laboratory must generate a valid method detection limit for each analyte of interest. The MDL must be below the reporting limit for each analyte. The procedure for determination of the method detection limit is given in 40 CFR Part 136, Appendix B, and further defined in SOP CA-Q-S-006.

13.2. Training Qualification

13.2.1. The Group/Team Leader has the responsibility to ensure an analyst who has been properly trained in its use and has the required experience performs this procedure.

14. POLLUTION PREVENTION

14.1. It is Eurofins TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage, and reagent stability). Employees must abide by the policies in Section 13 of the Corporate Environmental Health and Safety Manual (CW-E-M-001) for "Waste Management and Pollution Prevention".

15. WASTE MANAGEMENT

15.1. All waste must be disposed of in accordance with Federal, State and Local laws and regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees must abide by this method and the policies in Section 13 of the Corporate Environmental Health and Safety Manual (CW-E-M-001) for "Waste Management and Pollution Prevention."

15.2. Waste Streams Produced by the Method

15.2.1. Vials containing sample extracts. These vials are placed in the vial waste located in the GC/MS laboratory.

15.2.2. **Tubes containing sample extracts, for PCBs:** these are capped and placed in the PCB/flammable waste located the GC prep laboratory.

15.2.3. Samples, standards, and all extraction materials contaminated with high levels (>50ppm) of PCB's must be segregated into their own waste stream. PCB wastes are collected in one of three waste streams, solid PCB, liquid PCB and PCB vial waste. PCB containing samples are located through a LIMS query and disposed of as PCB containing.

15.2.4. **Extracted solid samples contaminated with methylene chloride/acetone or acetone/hexane:** These materials are disposed of in the solid waste and debris in a red container located in the Extractions Lab.

15.2.5. **Discarded samples:** These samples are collected in the solid debris drum.

16. REFERENCES

16.1. Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW846, 3rd Edition, Final Update III, December 1996, and Section 8000B

16.2. Eurofins TestAmerica Canton Quality Assurance Manual (QAM), current version

16.3. Eurofins TestAmerica Corporate Environmental Health and Safety Manual, CW-E-M-001, and Eurofins TestAmerica Canton Facility Addendum and Contingency Plan, current version

16.4. Corporate Quality Management Plan (CQMP), current versions

16.5. SW846, Update III, December 1996, Method 8082

16.6. SW846, Update IV, Revision 1, February 2007, Method 8082A

16.7. Reporting Results for Methods that Require Dual Column, CA-Q-P-004

16.8. Policy for Determining RT Windows for GC/ECD Tests, CA-T-P-005

16.9. Revision History

Historical File:				
Revision 0: 03/17/14				
Revision 1: 01/14/16				
Revision 2: 04/20/18				

**4/9/19: Changed logo and copyright information. No changes made to revision number or effective date.*

16.10. Associated SOPs and Policies, current version

16.10.1. QA Policy, QA-003

16.10.2. Glassware Washing, NC-QA-014

16.10.3. Statistical Evaluation of Data and Development of Control Charts, NC-QA-018

16.10.4. Detection and Quantitation Limits, CA-Q-S-006

16.10.5. Standards and Reagents, NC-QA-017

16.10.6. Cleanup Procedures for Organic Extractable Samples, NC-OP-025

16.10.7. Manual Integration, CA-Q-S-002

16.10.8. Calibration Curves and the Selection of Calibration Points, CA-Q-P-003

16.10.9. Separatory Funnel Extraction of Organic Compounds from Waters Based on Methods SW846 3510C and 600 Series, NC-OP-038

16.10.10. Sonication Extraction of Organic Compounds from Soils Based on Method SW846 3550C NC-OP-039

16.10.11. Soxhlet (Traditional) Extraction of Organic Compounds from Soils Based on Method SW846 3540C, NC-OP-040

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17. MISCELLANEOUS

17.1. Modifications from Reference Method

17.1.1. Chapter 1 of SW-846 states the method blank must not contain any analyte of interest at, or above, the Method Detection Limit. This SOP states the Method Blank must not contain any analyte of interest at, or above, the reporting limit.

17.1.2. The extended hold time for PCBs can be applied to 8082 as per SW846 Update IV. State requirements should be consulted as applicable.

17.2. Tables

Table 1
Standard Analyte List and CAS Numbers

Compound	CAS #
Aroclor 1016	12674-11-2
Aroclor 1221	11104-28-2
Aroclor 1232	11141-16-5
Aroclor 1242	53469-21-9
Aroclor 1248	12672-29-6
Aroclor 1254	11097-69-1
Aroclor 1260	11096-82-5

TABLE 2
Suggested Instrumental Conditions for Methods 8082 and 8082A

Parameter	Recommended Conditions
Injection port temp	220°C
Detector temp	325°C
Temperature program	70°C for 0.5min, 30°C/min to 190°C, 2.5°C/min to 225, 18°C/min to 280°C, 3 min hold
Column 1	CLPesticide I, 30m, 0.53mm ID, 0.5µm
Column 2	CLPesticide II, 30m, 0.53 mm ID, 0.5µm
Injection	1-2 µL, -
Carrier gas	Hydrogen
Make up gas	Nitrogen
Y splitter	Glass, Restek or equivalent

Title: SOXHLET (TRADITIONAL) EXTRACTION OF ORGANIC COMPOUNDS FROM SOILS BASED ON METHOD SW846 3540C

[Method: SW846 3540C]

Approvals (Signature/Date):

Technology Specialist	05/27/20 Date	Health & Safety Coordinator	05/27/20 Date
Quality Assurance Manager	05/26/20 Date	Technical Director	06/01/20 Date

This SOP was previously identified as SOP No. NC-OP-040, Rev 6, dated 5/25/18

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1. SCOPE AND APPLICATION

1.1. This SOP describes procedures for preparation (extraction) of semivolatile organic analytes in soil, sediment, waste and wipe matrices for analysis by Gas Chromatography (GC) and Gas Chromatography/Mass Spectrometry (GC/MS) using Soxhlet Extraction. The procedures are based on SW846 series methodology and are applicable for measurements made to comply with the Resource Conservation and Recovery Act (RCRA) and for wastewater testing.

1.1.1. Extraction procedures for the following determinative methods are covered: 8081A, 8081B, 8082, 8082A, 8270C, 8270D, 8270E, 8015B, 8015C, and 8015D.

1.1.2. The extraction procedures herein may be appropriate for other determinative methods when appropriate spiking mixtures are used.

1.1.3. Where requirements of this SOP are not met, including, but not limited to, Quality Control (QC) samples (Method Blank (MB) and Laboratory Control Sample (LCS)) associated to sample analytes, holding time, and preservation, the laboratory will narrate potential bias in the case narrative of the final report.

2. SUMMARY OF METHOD

2.1. Soxhlet Extraction (Traditional)

2.1.1 A 30 g sample (10 g for Pesticides and PCBs) is mixed with anhydrous sodium sulfate until free flowing, or a 1 wipe sample is placed in an extraction thimble. They are extracted by refluxing with solvent.

2.2. Concentration

2.2.1 Procedures are presented for drying the extract and concentration of the extract to final volume for analysis.

3. DEFINITIONS

3.1. Definitions of terms and acronyms used in this SOP may be found in the glossary of the Eurofins TestAmerica Canton Quality Assurance Manual (QAM), current version.

4. INTERFERENCES

4.1. Method interferences may be caused by contaminants in solvents, reagents, glassware, and other processing apparatus. All of these materials must be routinely demonstrated to be free from interferences under conditions of the analysis by running laboratory method blanks as described in the Quality Control section. All

glassware is cleaned per SOP NC-QA-014, current version. Specific selection of reagents may be required to avoid introduction of contaminants.

4.2. Visual interferences or anomalies (such as foaming, emulsions, odor, etc.) must be documented.

5. SAFETY

5.1. Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual, the Facility Addendum to the Corporate EH&S Manual, and this document.

5.2. Eye protection that protects against splash, laboratory coat and appropriate gloves must be worn while samples, standards, solvents and reagents are being handled. Cut-resistant gloves must be worn doing any other task that presents a strong possibility of getting cut. Disposable gloves that have become contaminated must be removed and discarded; other gloves must be cleaned immediately.

5.3. The following analytes have been tentatively classified as known or suspected, human or mammalian carcinogens: benzo(a)anthracene, benzidine, 3,3'dichlorobenzindine, benzo(a)pyrene, alpha-BHC, beta-BHC, gamma-BHC, delta-BHC, dibenz(a,h)anthracene, N-nitrosodimethylamine, 4,4'-DDT, and polychlorinated biphenyl compounds. Primary standards of these toxic compounds must be prepared in the hood.

5.4. The following is a list of the materials used in this method, which have a serious or significant hazard rating. NOTE: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the Safety Data Sheet (SDS) for each of the materials listed in the table. A complete list of materials used in the method can be found in the Reagents and Standards section. Employees must review the information in the SDS for each material before using it for the first time or when there are major changes to the SDS.

Material	Hazards	Exposure Limit (1)	Signs and symptoms of exposure
Methylene Chloride	Carcinogen Irritant	25 ppm-TWA 125 ppm-STEL	Causes irritation to respiratory tract. Has a strong narcotic effect with symptoms of mental confusion, light-headedness, fatigue, nausea, vomiting and headache. Causes irritation, redness and pain to the skin and eyes. Prolonged contact can cause burns. Liquid degreases the skin. May be absorbed through skin.
Acetone	Flammable	1000 ppm-TWA	Inhalation of vapors irritates the respiratory tract. May cause coughing, dizziness, dullness, and headache.
Hexane	Flammable Irritant	500 ppm-TWA	Inhalation of vapors irritates the respiratory tract. Overexposure may cause lightheadedness, nausea, headache, and blurred vision. Vapors may cause irritation to the skin and eyes.
Note: Always add acid to water to prevent violent reactions.			
1 – Exposure limit refers to the OSHA regulatory exposure limit.			

5.5. Exposure to hazardous chemicals must be maintained **as low as reasonably achievable.** All samples with stickers that read "Caution/Use Hood!" **must** be opened in the hood. Contact the EH&S Coordinator if this is not possible. Solvent and waste containers must be kept closed unless transfers are being made.

5.6. The preparation of standards and reagents and glassware cleaning procedures that involve solvents such as methylene chloride must be conducted in a fume hood with the sash closed as far as the operations will permit. If more than 500 mL of methylene chloride is spilled, evacuate the area until the area has been cleaned by EH&S.

5.7. All work must be stopped in the event of a known or potential compromise to the health and safety of a Eurofins TestAmerica Canton associate. The situation must be reported **immediately** to the EH&S Coordinator and the Laboratory Supervisor.

5.8. During Kuderna-Danish (KD) concentration, do not allow the extract to boil to dryness. The solvent vapors remaining in the KD apparatus may superheat and create an explosion or fire hazard. The KD apparatus and glass separatory funnels have ground glass joints that can become stuck. Technicians must use Kevlar or other cut/puncture-resistant gloves when separating stuck joints.

6. EQUIPMENT AND SUPPLIES

6.1. Glassware must be cleaned per Glassware Washing, SOP NC-QA-014.

6.2. Equipment and supplies for extraction procedures:

EQUIPMENT AND SUPPLIES	Sox	Conc
Graduated cylinder: 1 liter. (other sizes may be used as needed)		√
Methylene chloride collection tank for waste disposal	√	
Erlenmeyer flask: 250 mL (other sizes optional)		√
Solvent dispenser pump or 100 mL graduated cylinder	√	√
Round or flat bottom: 250 mL	√	
Boiling chips: contaminant free, approximately 10/40 mesh (Teflon® PTFE, carbide or equivalent)	√	√
Cooling condensers	√	
Heating mantle: rheostat controlled or equivalent	√	
Auto-timer for heating mantle	√	
Soxgriddle or equivalent	√	
Beakers: 450mL wide-mouth glass jars		√
Balance: >100 g capacity, accurate to ±1.00 g	√	√
Soxhlet extractor	√	
Cellulose and glass thimbles	√	
Kuderna-Danish (K-D) apparatus: 500 mL		√
Concentrator tube: 10 mL, attached to K-D with clips		√
Snyder column: three-ball macro		√
Water bath: heated, with concentric ring cover, capable of temperature control (± 5°C) up to 95°C. The bath must be		√

EQUIPMENT AND SUPPLIES	Sox	Conc
used in a hood or with a solvent recovery system.		
Vials: glass, 2 mL and 40 mL capacity with Teflon®-lined screw-cap		√
Clean wipes for wipe matrix method blanks and laboratory control samples		
Nitrogen blowdown apparatus		√
Nitrogen: reagent grade.		√
Culture Tubes: 10 mL, 16 x100 mm		√
Microliter pipette and/or syringe 1 mL	√	
Glass wool	√	
Glass funnel: 75 X 75 mm	√	√
Disposable pipettes, 5 ¾ in, and 9in.	√	√
Aluminum foil	√	√

7. REAGENTS AND STANDARDS

7.1. Reagents for Extraction Procedures

7.2. All reagents must be ACS reagent grade or better, unless otherwise specified.

REAGENTS	Sox	Conc
Sodium sulfate (Na_2SO_4), Granular, Anhydrous: Purify by heating at 800°C a minimum of one hour.	√	√
Magnesium sulfate	√	
Extraction Solvents (pesticide quality or equivalent): Methylene chloride, Methylene Chloride/acetone hexane/acetone,	√	√
Hexane/Acetone:, reagent grade: Used for cleaning glassware.	√	√

7.3. Standards

7.3.1. Stock Standards

7.3.1.1. Stock standards are purchased as certified solutions. S tock standards are stored according to manufacturer's instructions. All stock standards must be protected from light. Stock standard solutions must be replaced after one year (from the time of preparation, if prepared in house, or from the time the ampoule is opened, if purchased). Standards that are cold stored must be allowed to come to room temperature before use.

7.3.2. Surrogate Spiking Standards

7.3.2.1. Prepare or purchase surrogate spiking standards at the concentrations listed in Table 2. Surrogate spiking standards are purchased or prepared as dilutions of the stock standards.

Surrogate spiking solutions must be refrigerated and protected from light, or stored according to manufacturer's instructions. The standards must be replaced every six months at a minimum, or sooner if there is reason to believe that the standard has degraded or concentrated.

7.3.3. Matrix Spiking and Laboratory Control Spiking Standards

7.3.3.1. The same spiking solution is used for the matrix spike and the Laboratory Control Sample. Prepare MS/LCS spiking standards at the concentrations listed in Table 3. Spiking standards are purchased or prepared as dilutions of the stock standards.

7.3.3.2. Spiking solutions must be refrigerated and protected from light, or stored according to manufacturer's instructions. The standards must be replaced every six months at a minimum, or sooner if there is reason to believe that the standard has degraded or concentrated.

7.3.4. See SOP NC-QA-017 for additional information on Standards and Reagents.

8. SAMPLE COLLECTION PRESERVATION AND STORAGE

8.1. Samples are not chemically preserved.

8.2. Samples are stored at 4°C ± 2°C in glass containers with Teflon®-lined caps.

8.3. Holding Times

8.3.1. The holding time for solid and waste samples is 14 days from sampling to extraction.

8.3.2. Analysis of the extracts is completed within 40 days of extraction.

8.3.3. Unless otherwise specified by regulatory or client programs, the holding time for PCB samples is 1 year from sampling to extraction.

9. QUALITY CONTROL

9.1. Quality Control Batch

9.1.1. The batch is a set of up to 20 client samples and appropriate QC that are of the same matrix and are processed together using the same procedures and reagents. The batch must contain a method blank (MB), an LCS, and a matrix spike/matrix spike duplicate (MS/MSD). (In some cases, at client request, it may be appropriate to process a matrix spike and un-spiked sample duplicate in place of the MS/MSD). If clients designate specific samples for MS/MSD,

the batch may contain multiple MS/MSD pairs. See Policy QA-003 for further definition of the batch.

9.2. Method Blank (MB)

9.2.1. An MB consisting of all reagents added to the samples must be prepared and analyzed with each batch of samples. Surrogates are spiked into the MB at the same level as the samples. See Table 2 for the appropriate amount of surrogate to use for each analytical method. The MB is used to identify any background interference or contamination of the analytical system which may lead to the reporting of elevated concentration levels or false positive data.

9.2.2. For a solid MB, use approximately 30 g of sodium sulfate spiked with the surrogates. For PCB and Pesticides, use approximately 10 g ± 0.5 g of sodium sulfate. See Table 2 for the appropriate amount of surrogate to use for each analytical method. The MB goes through the entire analytical procedure.

9.2.3. For a wipe MB, use 1 clean wipe spiked with the surrogates. See Table 2 for the appropriate amount of surrogate to use for each analytical method. The MB goes through the entire analytical procedure.

9.3. Laboratory Control Sample (LCS)

9.3.1. LCSs are well-characterized laboratory-generated samples used to monitor the laboratory's day-to-day performance of routine analytical methods. The LCS, spiked with a group of target compounds representative of the method analytes, is used to monitor the accuracy of the analytical process, independent of matrix effects. Ongoing monitoring of the LCS results provides evidence that the laboratory is performing the method within accepted QC guidelines for accuracy and precision. The LCS goes through the entire analytical procedure.

9.3.2. The LCS is made up in the same way as the MB (see Sections 9.2.1 through 9.2.3), but spiked with the LCS standard and the surrogates. See Table 3 for the appropriate amount of spike to use for each analytical method.

9.4. Surrogates

9.4.1. Surrogates are organic compounds which are similar to the target analyte(s) in chemical composition and behavior in the analytical process, but which are not normally found in environmental samples.

9.4.2. Each applicable sample, MB, LCS, and MS/MSD is spiked with surrogate standards. Surrogate spike recoveries must be evaluated by determining whether the concentration (measured as percent recovery) falls within the required recovery limits of the applicable determinative method. See Table 2 for the appropriate amount of surrogate spike to use for each analytical method.

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9.5. Matrix Spike/Matrix Spike Duplicate (MS/MSD)

9.5.1. An MS is an environmental sample to which known concentrations of target analytes have been added. An MSD is a second spiked aliquot of the same sample, which is prepared and analyzed along with the sample and MS. See Table 3 for the appropriate amount of spike to use for each analytical method.

9.6. QC requirements can be found in the various associated analytical SOPs.

9.7. Control Limits

9.7.1. Control limits are established by the laboratory as described in SOP NC-QA-018.

9.7.2. Laboratory control limits are internally generated and updated periodically, unless method specified. Control limits are easily accessible via the LIMs.

9.8. Method Detection Limits (MDLs) and MDL Verifications

9.8.1. MDLs and MDL Verifications are established and performed by the laboratory as described in SOPs CA-Q-S-006.

9.8.2. MDLs are easily accessible via the LIMs.

9.9. Nonconformance and Corrective Action

9.9.1. Any deviations from QC procedures must be documented as a nonconformance with applicable cause and corrective action.

10. CALIBRATION AND STANDARDIZATION

10.1. On a weekly basis, measure the appropriate volumes of solvents into the appropriate sized glass culture tubes gravimetrically. The “standard” glass culture tubes are sealed, and the meniscus is noted by marking a line on the tubes. The glass culture tubes containing the sample final extracts are then compared against the “standard” glass culture tubes of the appropriate volume and solvent to ensure the volumes are consistent. (See Table 1 for final volumes)The bottle top dispenser is calibrated quarterly and must be within ±5% of the target volume with an RSD ≤ 1%.

10.2. All labware, pipettes, bottle top dispensers, and balances are calibrated according to SOPs NC-QA-004 and NC-QA-015.

11. PROCEDURE

11.1. Procedural Variations

11.1.1. Procedural variations are allowed only if deemed necessary in the professional judgment of QA, operations supervisor, or designee to

accommodate variation in sample matrix, chemistry, sample size, or other parameters. Any variation in procedure must be completely documented using a Nonconformance memo and approved by a supervisor. The Nonconformance memo will be filed in the project file. Procedural variations are not allowed for Ohio VAP projects.

11.2. Soxhlet

11.2.1. Remove surrogate and matrix spiking solutions from refrigerator if cold stored, and allow to warm to room temperature.

11.2.2. If sample can be mixed easily in the sample jar, mix thoroughly by stirring with a clean plastic or wooden spoon or spatula. If the sample cannot be easily mixed (i.e., clay samples or samples of various and very different particle sizes), use the spoon or spatula to select enough separate portions from locations within the jar to produce a representative sample. Analyst judgment is important in determining how many portions and which locations are used to produce a representative aliquot. If the sample is uniform clay, at least 3 portions should be selected from different locations in the sample jar, if particle sizes or materials indicate a very non-homogenous sample, selection should be made carefully to collect an aliquot that represents the relative percentages of the various particle sizes and types in the sample jar.

11.2.3. Do not decant the water layer from sediment samples. The entire sample is used. A higher weight of sample portion must be weighed for sediment samples to account for the dry weight correction (see 11.2.3). Record and document in the LIMS if a water layer was present in the sample.

11.2.4. If the sample cannot be prepared using a Soxhlet due to matrix issues, a waste dilution may be required. Refer to SOP NC-OP-043 for the waste dilution procedure.

11.2.5. Place approximately 200mL of solvent into a 250 mL flat bottom flask containing one or two clean boiling chips. Weigh 30g ± 0.5 g of sample into a thimble or in a jar, recording the weight to the nearest 0.01g in LIMS. For PCB or Pesticides Extraction, weigh approximately 10 g of sample ± 0.5 g. Sample weights less than 30g, but over 1g, may be used if the appropriate reporting limits can be met. For sediment samples that contain excessive moisture, weigh 50 g ±0.5 g. For wipe samples, the wipe is placed in an extraction thimble. For concrete samples, weigh 5 g of sample ± 0.5g.

Note: Waste samples with difficult matrices (such as caulk) are extracted at 1g.

Note: Alternate sample volumes can be used as long as a representative sample can be obtained and the reagent levels are adjusted to maintain the sample to reagent ration. All samples and standards must be processed similarly.

11.2.6. Prepare an MB, LCS, and MS/MSD for each batch as specified in Section 9 of this SOP, using sodium sulfate or a clean wipe as the matrix for the LCS and MB. The parent sample is used for the MS/MSD. The weight of sodium sulfate used must be approximately the weight of soil used for samples.

11.2.7. Add anhydrous sodium sulfate to each solid, sediment or waste sample and mix well. The mixture must have a free-flowing texture. If not, add more sodium sulfate. Add the sample/sodium sulfate mixture to a soxhlet extractor thimble, but do not pack the mixture tightly. The soxhlet extractor must drain freely for the duration of the extraction period. A glass wool plug below the sample in the soxhlet extractor is used for the extractions. Pesticides use specific silane treated glass wool.

11.2.8. Add the appropriate amount of surrogate and matrix spiking solution as indicated in Tables 2 and 3.

11.2.9. Attach the flask to the extractor and rinse the extractor with 20-30 mL of the appropriate solvent. Attach extractor to heating griddle and extract the sample for 16-24 hours at 4-6 cycles per hour. Check the system for leaks at the ground glass joints after it has warmed up.

Note: If a reduced quantity of sample is extracted, it is usually necessary to increase the amount of sodium sulfate added or increase the solvent boiling rate to properly set the cycling rate.

Solvents:

Semivolatile GC/MS and TPH	1:1 v/v Methylene Chloride / Acetone
8270 (MS) and TPH Concrete	Methylene Chloride only
PCB and Pesticides	1:1 v/v Hexane / Acetone
8082 Concrete	1:1 v/v Methylene Chloride/Acetone

11.2.10. Allow the extract to cool after the extraction is complete then disassemble by gently twisting the soxhlet from the flask.

11.2.11. The sample is now ready for the concentration step (Section 11.3).

11.2.12. Cap the samples with flask stopper and store at 4°C ± 2°C if the extract will not be concentrated immediately. Refer to Section 11.3 for concentration.

11.3. Concentration: According to the type of sample, different solvents and final volumes will be required. Refer to Table 1 for the appropriate final volumes and concentrations.

11.3.1. Kuderna-Danish (KD) Method:

11.3.1.1. Assemble a Kuderna-Danish concentrator by attaching a 10 mL concentrator tube (CT) to the 500 mL KD flask. Label the CT and

KD. Transfer the sample to the labeled K-D flask filtering the extracts through funnels filled with sodium sulfate. Rinse the sample flasks from the soxhlet setups with approximately 10 – 20 mL of methylene chloride Transfer the rinsate through the funnel and rinse the funnel with 20-30 mL of methylene chloride to complete the quantitative transfer.

11.3.1.2. Add one or two clean boiling chips to the KD flask and attach a three-ball Snyder Column. Add approximately 1 mL of clean methylene chloride to the top of the Snyder column. **Note:** It is important to wet with MeCl to ensure that the balls in the Snyder column do not stick, and the column will work properly.

11.3.1.3. Place the KD apparatus on a water bath (90-98^{o}C) so the tip of the concentrator tube is submerged. The water level must not reach the joint between the concentrator and the KD flask. At the proper rate of distillation, the balls will actively chatter; but the chambers should not flood.

11.3.1.4. Concentrate to 15-20 mL. If the determinative method requires a solvent exchange, add the appropriate exchange solvent to the top of the Snyder Column, and then continue the water bath concentration back down to 5-8 mL. Refer to Table 1 for details of exchange solvents and final volumes. The Snyder column may be insulated if necessary to maintain the correct rate of distillation.

Note: It is very important not to concentrate to dryness as analytes will be lost.

11.3.1.5. Remove the KD apparatus from the water bath and allow to cool for a minimum of 10 minutes. If the level of the extract is above the level of the CT joint, continue to distill the solvent as necessary. Again, allow the KD flask to cool for a minimum of 10 minutes.

11.4. Nitrogen Evaporation to Final Concentration

11.4.1. Transfer the CT to the evaporation apparatus.

11.4.2. Place the tube in a warm water bath that is at least 5^{o}C below the boiling temperature of the solvent being evaporated and evaporate the solvent using a gentle stream of nitrogen. The nitrogen flow will form a slight depression on the surface of the solvent, but must not create splattering of the extract.

Boiling points of commonly used solvents are:

Methylene chloride	40^{o}C
Acetone	56^{o}C
Hexane	69^{o}C
Acetonitrile	82^{o}C

Note: It is very important not to concentrate to dryness as analytes will be lost.

11.4.3. Refer to Table 1 to determine the final volume needed for a specific test method. Evaporate to slightly less than the required final volume.

11.4.4. Rinse the CT and quantitatively transfer the extract with the rinsate to the appropriate final container, and dilute to the appropriate final volume using the "standard" glass vial noted in Section 10.1. Cap the sample and affix the appropriate label. The sample is now ready for analysis.

Note: The final concentration and volume measurement steps are critical. Use care when concentrating and make certain that the final volume measurement is accurate.

11.5. Analytical Documentation

11.5.1. Record all analytical information in LIMS, including any corrective actions or modifications to the method.

11.5.2. Record all standards and reagents in the LIMS Reagents module. All standards and reagents are assigned a unique number for identification.

11.5.3. Record sample and associated QC information into LIMs. Level I and Level II technical reviews are performed in LIMS.

12. DATA ANALYSIS AND CALCULATIONS

12.1. Not applicable

13. METHOD PERFORMANCE

13.1. Initial Demonstration

13.1.1. Each analyst must make an initial demonstration of capability (IDOC) for each individual method. This requires analysis of four QC check samples. The QC check sample is a well-characterized laboratory generated sample used to monitor method performance, which must contain all of the analytes of interest. The spiking level must be equivalent to a mid-level calibration. (For certain tests, more than one set of QC check samples may be necessary in order to demonstrate capability for the full analyte list.)

13.1.2. Four aliquots of the QC check sample are analyzed using the same procedures used to analyze samples, including sample preparation.

13.1.3. Calculations and acceptance criteria for the QC check samples are given in the determinative SOPs. See SOPs NC-GC-038, NC-MS-018, NC-MS-003, and NC-GC-007 for detailed information on the determinative methods.

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13.1.4. Method validation information (where applicable) in the form of analyst demonstrations of capabilities is maintained for this method in the analyst's training files

13.2. Training Qualification

13.2.1. The Group/Team Leader has the responsibility to ensure that this procedure is performed by an analyst who has been properly trained in its use and has the required experience.

14. POLLUTION PREVENTION

14.1. It is Eurofins TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage, and reagent stability). Employees must abide by the policies in Section 13 of the Corporate Environmental Health and Safety Manual (CW-E-M-001) for "Waste Management and Pollution Prevention".

15. WASTE MANAGEMENT

15.1. All waste will be disposed of in accordance with Federal, State, and local laws and regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this method the policies in Section 13 of the Corporate Environmental Health and Safety Manual (CW-E-M-001) for "Waste Management and Pollution Prevention."

15.2. The following waste streams are produced when this method is carried out.

15.2.1. Used sodium sulfate and glass wool or filter paper contaminated with methylene chloride/acetone or acetone/hexane from the extract drying step. These materials are disposed of in the solid waste and debris in a red container located in the Extractions Lab.

15.2.2. **Assorted flammable solvent waste from various rinses**: These wastes are put into the halogenated/non-halogenated 25 gallon solvent waste container located under the fume hood in extractions.

15.2.3. **Methylene chloride waste from various rinses:** These wastes are disposed of in the liquid-liquid separation unit.

15.2.4. **Hexane-Hexane waste:** These samples are to be disposed in the flammable waste.

15.2.5. **Waste Hexane in vials:** These vials are placed in the vial waste located in the GC prep laboratory.

15.2.6. **Waste Methylene Chloride sample vials**: These vials are placed in the vial waste located in the GC prep laboratory.

15.2.7. **Extracted solid samples contaminated with methylene chloride/acetone or acetone/hexane:** These materials are disposed of in the solid waste and debris in a red container located in the Extractions Lab.

15.2.8. Samples, standards, and all extraction materials contaminated with high levels (>50ppm) of PCBs must be segregated into their own waste stream. PCB wastes are collected in one of three waste streams, solid PCB, liquid PCB, and PCB vial waste.

15.2.9. Solvent Recovery System Waste: Methylene Chloride waste from the Solvent Recovery System is collected and disposed of in the liquid-liquid separation unit. Acetone/Methylene Chloride waste from this system is disposed of in the flammable waste containers located in the laboratory.

16. REFERENCES

16.1. References

16.1.1. Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW846, Sections 3500B, 3540C, and 3580A, current version

16.1.2. Eurofins TestAmerica Canton Quality Assurance Manual (QAM), current version

16.1.3. Eurofins TestAmerica Corporate Environmental Health and Safety Manual, CW-E-M-001, and Eurofins TestAmerica Canton Facility Addendum and Contingency Plan, current version

16.1.4. Corporate Quality Management Plan (CQMP), current version

16.1.5. Revision History

Historical File:		Revision 4.1: 10/07/03		Revision 5a: 05/25/17
(formerly CORP-OP-0001NC)		Revision 4.2: 01/30/06		Revision 6: 05/25/18
Revision 3.4: 10/16/98		Revision 0: 03/12/08 (NC-OP-032)		
Revision 3.5: 04/22/99		Revision 1: 01/07/09 (NC-OP-032)		
Revision 3.6: 05/13/99		Revision 0: 03/24/11 (NC-OP-040)		
Revision 3.7: 03/20/01		Revision 1-A: 01/24/12		
Revision 3.8: 05/23/01		Revision 2: 08/28/13		
Revision 3.9: 04/22/02		Revision 3: 08/25/14		
Revision 4.0: 02/04/03		Revision 4: 01/18/16		

**4/9/19: changed logo and copyright information. No changes made to revision number or effective date.*

16.2. Associated SOPs and Policies, current version

16.2.1. QA Policy, QA-003

16.2.2. Glassware Washing, NC-QA-014

16.2.3. Statistical Evaluation of Data and Development of Control Charts, NC-QA-018

16.2.4. Detection and Quantitation Limits, CA-Q-S-006

16.2.5. Gas Chromatographic Analysis of Pesticides Based on Methods 8081A and 8081B, NC-GC-042

16.2.6. Gas Chromatographic Analysis of Diesel Range Organics Based on Methods 8015B, 8015C, and 8015D, NC-GC-043

16.2.7. Gas Chromatographic Analysis of PCBs Based on Methods 8082 and 8082A, NC-GC-045

16.2.8. GC/MS Analysis based on Method 8270C, 8270D,and 8270E, NC-MS-018

16.2.9. Standards and Reagents, NC-QA-017

17. MISCELLANEOUS

17.1. Modifications from Reference method

17.1.1. Some surrogate spiking concentrations are modified from those recommended in SW-846, in order to make the concentrations more consistent with the calibration levels in the determinative methods.

17.1.2. Sodium sulfate is heated for 1 hour at 800°C to purify. The reference method lists a minimum of 4 hours at 400°C.

17.2. Tables

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TABLE 1
Final Volumes and Exchange Solvents

Type	Exchange Solvent for Analysis*	Final Volume for Analysis in mL
Semivolatiles	N/A	2.0 mL
PCB	Approximately 36 mL Hexane	2.0 mL
Pesticides	Approximately 18 mL Hexane	2.0 mL
TPH	N/A	5.0

Note: *PCBs and Pesticides only need the solvent exchange step when they are extracted in methylene chloride / acetone. If they are extracted in hexane / acetone, no solvent exchange is necessary.*

Note: *Different final volumes may be necessary to meet special client reporting limit requirements.*

TABLE 2
Surrogate Spiking Solutions

Analyte Group	Surrogate Spike Solution ID	Volume (mL)
BNA	20 ppm BNA	1.0
PEST	0.2 ppm DCB/TCX	1.0
TPH	o-Terphenyl	1.0
PCB	0.2 ppm DCB/TCX	1.0

TABLE 3
Matrix Spike and LCS Solutions

Analyte Group	Matrix Spike Solution ID	Volume (mL)
BNA	20 ppm BNA All-Analyte Spike	1.0
PEST	Pest NPDES Spike	1.0
PCB	10 ppm PCB Spike	1.0
TPH	Diesel Spike	1.0

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Title: Soil Processing

[Method: ASTM D6323-12, ITRC Guidance Document, Soil Fractionation Method for Michigan]

Approvals (Signature/Date):

Technology Specialist Diane Jones	12/17/20 Date	Health & Safety Coordinator Steve Jackson	12/17/20 Date
Quality Assurance Manager Mark J. Loeb	01/11/21 Date	Technology Director Raymond M. Risden	12/18/20 Date

This SOP was previously identified as NC-OP-044 Rev. 3, dated 12/05/18

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1. SCOPE AND APPLICATION

1.1. These disaggregation, mixing and particle size reduction techniques are applicable to a wide range soil, sediment, tissue, water, and waste samples. Care must be taken to match the appropriate technique with the matrix, target analytes and quality objectives.

1.2. This document accurately reflects current laboratory Standard Operating Procedures (SOP) as of the date above. All facility SOPs are maintained and updated as necessary.

Note: If foreign or quarantined solids are received, refer to SOP NC-SC-019 Canton Foreign Soils, current revision, and contact your Environmental Health and Safety Coordinator for proper handling instructions

2. SUMMARY OF METHOD

2.1. Air-Dried Soil Processing: The sample is air dried at room temperature and disaggregated to break clumps into a fine powder to facilitate obtaining a representative sub-sample and improving analyte extraction efficiency.

2.2. Soil Fractionation for Lead: Applicable to lead impacted soil and designed to distinguish the lead in the fine soil fraction from the coarse soil fraction.

2.3. Incremental Sample Wet Mixing Process: Applicable to mixing incremental samples into a single composite. These samples are NOT air dried, but rather water is added to facilitate mixing in a heavy-duty mixer.

3. DEFINITIONS

3.1. Refer to the glossary in the Eurofins TestAmerica Canton QA Manual, current version.

3.2. Mix: To thoroughly blend the sample and reduce the analyte concentration differences between different parts of the overall sample. It is most effective when the particle size and density differences within the sample are small.

3.3. Incremental Sampling Methodology: A structured composite sampling and processing protocol that reduces data variability and provides a reasonably unbiased estimate of the mean contaminant concentrations in an area/volume of soil targeted for sampling. ISM provides representative samples of specific soil areas/volumes defined as decision units (DUs) by collecting numerous increments of soil (typically 30-100 increments) that are combined, processed, and subsampled according to specific protocols.

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3.4. Grinding: A generic term for soil disaggregation or milling (ITRC Guidance Appendix E). The term grinding does not in itself represent any specific process.

3.5. Disaggregation: The act of breaking the soil clumps into individual small particles but keeping the small pebbles and hard crystalline particles intact (ITRC Guidance Appendix E).

3.6. Milling: Complete particle size reduction of all soil components including hard crystalline materials to a defined maximum particle size (e.g. <250 µm or <75 µm) (ITRC Guidance Appendix E).

3.7. Sample: For laboratory technicians, the sample is all the material delivered to the laboratory in a container collected by the field crew.

3.8. Subsample: The small representative amount removed from a field sample that selected for final analysis. Also referred to in some SOPs as “the aliquot”.

4. INTERFERENCES

4.1. Method interferences may be caused by contaminants in solvents, reagents, glassware, and other processing apparatus that lead to discrete artifacts. All of these materials must be routinely demonstrated to be free from interferences under conditions of the analysis by running laboratory method blanks as described in the Quality Control section of each analytical SOP. Specific selection of reagents may be required to avoid introduction of contaminants (i.e. samples being analyzed for VOCs should generally not come into contact with equipment that has been solvent-rinsed with acetone or methylene chloride).

4.2. Metallic components of particle size reduction equipment can contribute some metal content to the solid samples. Hence carbon steel components are usually preferable to stainless steel when necessary to minimize contamination from chromium, nickel and molybdenum. Some contamination from iron is common.

4.3. Particle size reduction equipment blanks should be generated before use. The type of blank generated may vary for each piece of equipment, and may be solid or aqueous in nature.

5. SAFETY

5.1. Employees must abide by the policies and procedures in the NDSC EH&S Manual, the Facility Addendum to the NDSC EH&S Manual, and this document.

5.2. Eye protection that protects against splash, laboratory coat, and appropriate gloves must be worn while samples, standards, solvents, and reagents are being handled. Cut-resistant gloves must be worn while doing any other task that presents a strong possibility of getting cut. Disposable gloves that have been contaminated will be removed and discarded; other gloves will be cleaned immediately.

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5.3. Exposure to chemicals must be maintained **as low as reasonably achievable**; therefore, unless they are known to be non-hazardous, all samples must be opened, transferred and prepared in a fume hood, or under other means of mechanical ventilation where possible. All samples with stickers that read "Caution/Use Hood!" **must** be opened in the hood. Contact the EH&S Coordinator if this is not possible. Solvent and waste containers will be kept closed unless transfers are being made.

5.4. The following is a list of the materials used in this method, which have a serious or significant hazard rating. NOTE: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the SDS for each of the materials listed in the table. A complete list of materials used in the method can be found in the Reagents and Standards section. Employees must review the information in the SDS for each material before using it for the first time or when there are major changes to the SDS.

Material	Hazards	Exposure Limit (1)	Signs and symptoms of exposure
Acetone	Flammable	1000 ppm-TWA	Inhalation of vapors irritates the respiratory tract. May cause coughing, dizziness, dullness, and headache.
Methanol	Flammable Poison Irritant	200 ppm-TWA	A slight irritant to the mucous membranes. Toxic effects exerted upon nervous system, particularly the optic nerve. Overexposure may include headache, drowsiness and dizziness. Methanol is a defatting agent and may cause skin to become dry and cracked. Skin absorption can occur: symptoms may be parallel to inhalation exposure. Irritant to the eyes.
Hydrochloric acid	Corrosive Poison	5ppm-Ceiling	Inhalation of vapors can cause coughing, choking, inflammation of the nose, throat, and upper respiratory tract, and in severe cases, pulmonary edema, circulatory failure, and death. Can cause redness, pain, and severe skin burns. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
Note: Always add acid to water to prevent violent reactions.			
1 – Exposure limit refers to the OSHA regulatory exposure limit.			

5.5. All work must be stopped in the event of a known or potential compromise to the health and safety of a Eurofins TestAmerica associate. The situation must be reported **immediately** to the EH&S Coordinator and the Laboratory Supervisor.

5.6. When operating the electric chopper or grinder, be sure to keep all aqueous liquids clear to prevent the risk of electrical shock from any spills.

5.7. Avoid inhalation of sample dust. Work in a ventilation hood when necessary to avoid accidental dust inhalation. Wear a dust mask or respirator if the ventilation hood does not provide sufficient dust protection. The Environmental Health and Safety Coordinator must approve any and all use of dust masks and/or respirators.

5.8. If there is any malfunction in the equipment de-energize and tag out.

5.9. All noise levels are below OSHA limits.

5.10. Training: Only trained personnel are permitted to use the equipment mentioned in this document. A list of trained personnel will be maintained with EH&S and QA.

6. EQUIPMENT AND SUPPLIES

6.1. Drying tray, plastic or aluminum

6.1.1. Half cake sheet pan, Pactiv #614255, or equivalent

6.1.2. Quarter sheet cake pan, Pactiv #604245, or equivalent

6.2. Butcher Paper

6.3. Plastic wrap

6.4. Mortar and pestle

6.5. Automated mortar grinder

6.6. Food chopper, Black and Decker Handi chopper, or DeLonghi mini food processor, or equivalent

6.7. Coffee grinder, KitchenAid BCG100 or equivalent

6.8. Automated soil disaggregator

6.9. Wooden spatula: 6 in.

6.10. Stainless steel sieves: 1 mm, #10, #20, #60, #100, ¼ inch, other sizes by request with advanced notice

6.11. Electrolux Assistant 8 qt mixer, with dough hook or equivalent

6.12. Large cookie scoop with hand actuated ejector blade

6.13. Small cookie scoop with hand actuated ejector blade

6.14. Fluoropolymer scoop, not commercially available

6.14.1. Construct by fastening the bottom half of a fluoropolymer bottle to a wooden handle with stainless steel screws.

6.15. Stainless steel pot with cover, Bain-Marie 6 qt. or equivalent

6.16. Wooden spoon, 12” long



Picture of labware for multi-increment sample wet mixing process (Section 11.3.2.5): large scoop, small scoop, wooden spoon, fluoropolymer scoop, stainless steel pot/cover

6.17. Jaw crusher, Sepor Model 150 or equivalent



6.18. Freezer for sample storage.

6.19. 8 oz. Glass jars with lids

6.20. Top loading balance capable of weighing 100 g ±0.2 g.

6.21. Aluminum foil

7. REAGENTS AND STANDARDS

7.1. Deionized water: Reagent water must be produced by a Millipore DI system or equivalent. Reagent water must be free of the analytes of interest as demonstrated through the analysis of preparation blanks.

7.2. Methanol: VOC grade

7.3. Acetone: Pesticide grade

8. SAMPLE COLLECTION, PRESERVATION, AND STORAGE

8.1. Not applicable to this procedure. Sample collection, preservation, and storage are dependent on the requested test method and analytes.

9. QUALITY CONTROL

9.1. Equipment processing blanks might be applicable for some particle size reduction or selection techniques. There is no single blank matrix that is suitable for all analytes, equipment or processes. The blank matrix should be chosen by the client/data user based on the advantages and limitations described below in Section 9. If no blank

matrix is selected by the client, reagent water should be used as the default when possible as noted in Section 9.2.

9.2. Reagent water (or organic solvents) may be used to rinse the equipment surfaces. This liquid is then analyzed for the target analytes. This process is good at monitoring residue on equipment surfaces from previously processed samples. It does not evaluate the potential contribution of the equipment surface material to a solid sample. Sand may be run through processing equipment and then analyzed to monitor for both sample carryover residue and contamination from equipment surface erosion. This process is most applicable for organic analytes. Sand always contains metals. These metals concentrations might be too high for suitable blank demonstrations. Also, the sand material is frequently more abrasive than soil and can over-estimate sample contamination due to erosion of the equipment surfaces.

9.3. Teflon boiling chips can be suitable to monitor the cleanliness of processing equipment surfaces. This option is between reagent water and sand regarding abrasion of the equipment surfaces. This material is generally non-detect at parts per billion concentrations for most organic and inorganic analytes. The Teflon material does not mimic the behavior of soil in the subsequent sample extraction or digestion procedures.

9.4. As an alternative to or in addition to equipment blanks, half of a field sample may be reprocessed with the goal of evaluating differing analyte concentrations between the single and double processed sample aliquots. The occurrence of significantly elevated analyte concentrations in the double processed sample can be a good indication of carryover or contamination due to equipment surface erosion. This is most applicable for monitoring metals contamination in soil grinders.

10. CALIBRATION AND STANDARDIZATION

10.1. Not applicable to this procedure.

11. PROCEDURE

11.1. One-time procedural variations are allowed only if deemed necessary in the professional judgment of QA, operations supervisor, or designee to accommodate variation in sample matrix, chemistry, sample size, or other parameters. Any variation in procedure shall be completely documented using a Nonconformance Memo.

11.2. Any unauthorized deviations from this procedure must also be documented as a nonconformance with a cause and corrective action described.

11.3. Sample Preparation

11.3.1. The following sections describe a variety of procedures. The client/data user must select the procedure that is most appropriate for the sample matrix, analytes, and quality objectives. The selection of the procedure(s)

can be made by the client in consultation with the appropriate laboratory representatives during project planning. There is no single default procedure that can be used in the absence of client selection.

11.3.2. Solid Sample Mixing and Particle Size Reduction Procedures

11.3.2.1. In jar mixing

11.3.2.1.1. The flowable sample is thoroughly stirred in the sample jar using a wooden blade.

11.3.2.2. Horizontal surface mixing

11.3.2.2.1. The sample is transferred to an aluminum tray or onto a sheet of paper and mixed with a wood blade.

11.3.2.3. Mortar and pestle

11.3.2.3.1. A mortar and pestle can be used for samples in the 1 to 8 mm particle size. The final reduced size is between 5 um and 8 mm. This process can be used for wet or dry, organic or inorganic substances.

11.3.2.3.2. Fill the mortar about 1/3 with sample. Grind and mix the sample with the pestle. Transfer the processed sample to a separate container, and repeat the grind-and-mix step with additional sample aliquots as needed.

11.3.2.3.3. An automated mortar grinder may be appropriate for larger samples. Set the automated mortar grinder to the desired size and allow to run until the sample has been completely ground. If the grinder is not equipped with an automated scraper/agitator, the sample may be mixed by hand. Be sure that the grinder has stopped moving completely before opening the lid.

11.3.2.4. Dry mixing (incremental sampling methodology support)

11.3.2.4.1. Air dry, disaggregate, sieve, mix

11.3.2.4.1.1. Line a standard aluminum tray with a disposable aluminum tray ($^1/_2$ or $^1/_4$ sheet depending on sample size). If aluminum is a metal of interest line the tray with butcher paper. Do not use butcher paper when TOCs are of interest.

11.3.2.4.1.2. Remove large rocks and vegetation unless client indicates otherwise. Do not decant the free water. Mix the sample and spread the entire sample in a thin layer in the tray. If necessary to perform a pre-dried total solids analysis, remove approximately 10 g. If the samples require ISM processing, an appropriate subsampling procedure should be followed, as described in the subsampling SOP.

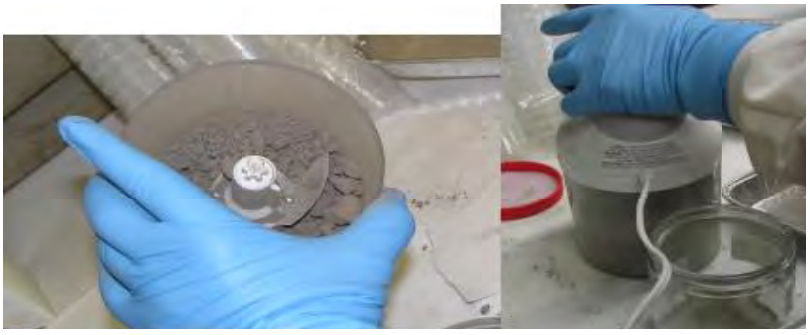
Note: it is not recommended to remove any portion of an ISM sample prior to drying and disaggregation. Any such removal from an ISM sample is considered a deviation and should be approved by the client.

11.3.2.4.1.3. Place the tray in the ventilated drying rack for up to five days at room temperature.

11.3.2.4.1.4. Periodically, stir the sample to expose moist sample to the air.

11.3.2.4.1.5. The dried sample must be crushable, and not prone to sticking together. Typically this means that sediment samples will be less than 30% moisture content and soil samples less than 15% moisture content. However, it is not necessary to do a percent moisture analysis.

11.3.2.4.1.6. Disaggregate the dried sample to break up the dried sample clumps with a bladed chopper or equivalent, but do not grind the small pebbles into powder.



11.3.2.4.1.7. If the volume capacity of the disaggregation equipment is large enough, transfer the whole sample to the disaggregator. If sample volume is too large, disaggregate the sample in sub-aliquots.

11.3.2.4.1.8. Pass the sample through a sieve to remove small pebbles and organic materials. Project specific guidelines will determine the sieve opening size. Typically, a #10 sieve is used for soils.



11.3.2.4.1.9. Combine all disaggregated sub-aliquots, and mix thoroughly by stirring, shaking or tumbling.

11.3.2.4.2. Air dry, disaggregate, mix

11.3.2.4.2.1. When the project objectives dictate that all small and medium sized materials be included in the final sample, use the procedure described in Section 11.3.2.4.1, but skip the sieving step in Section 11.3.2.4.1.8.

11.3.2.4.2.2. Collecting a representative subaliquot is more difficult when the sample has a variety of particle sizes. Refer to the subsampling SOP for recommended procedures. The one dimensional slabcake process is particularly applicable to dry, flowable samples with a wide range of particle sizes.

11.3.2.4.3. Other

11.3.2.4.3.1. Various permutations of soil disaggregation and sieving are possible depending on the needs of the project and can be accommodated but must be clearly defined in consultation with the client. These variations can include different sieve sizes and changing the order so that sieving is done prior to disaggregation. This would most likely be done to exclude a particular size of organic or rocky material.

11.3.2.5. Wet mixing (incremental sampling methodology support)

11.3.2.5.1. Weigh 100.0 +/- 0.2 g of each ¼ acre sub-sample. Stir the sample prior to and during the transfer from the original container to the weighing container. Record the weight to two decimal places.

11.3.2.5.1.1. If there are fewer than 40 sub-samples, increase the weight of sample proportionally. Calculate the new target weight as follows: target weight = 4000g/# sub-samples.

11.3.2.5.1.2. Exclude rocks, organic matter, and other debris from the weighed sub-sample by sieving through a ¼ inch sieve prior to weighing when such material is present.

11.3.2.5.1.3. The sieve may also be used to break up clay chunks. Sieving may be done either before or after weighing for this purpose. Use only when needed. Hard dry soil agglomerates should be broken up by hand crushing or chopping with a food chopper.

11.3.2.5.1.4. If a sub-sample is over ½ gravel, expand the maximum particle size from ¼ inch to 1.8 inches.

11.3.2.5.2. Transfer the weighed (and sieved as needed) sub-sample aliquot to a stainless steel pot used to collect all weighed aliquots for that composite sample. The total mass of the composite sample will be at least 4 kg.

11.3.2.5.3. Repeat Section 11.3.2.5.1 until all sub-samples have been weighed and transferred to the compositing pot.

11.3.2.5.4. Assemble the heavy-duty mixer with mixing hook. The mixing hook must be inserted high in the mounting bracket to avoid dragging on the bottom of the bowl and allow small stones to pass under the hook. Transfer the entire composite sample from the covered stainless steel pot to the stainless mixing bowl.

11.3.2.5.5. Turn on mixer. Add reagent water to ensure complete mixing of the sample. The mixed sample will have uniformly distributed water, look visually homogeneous, and have the consistency of a thick paste. Do not add so much water as to form runny slurry. Use a wooden spoon to assist the mixing process by scraping mud from the sides and directing it to the center. The picture below shows the proper consistency. Mix for three minutes after the proper consistency has been achieved.

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11.3.2.5.6. Record the volume of reagent water added and the total mixing time.

11.3.2.5.7. Scrape the mud from the wooden spoon and mixing hook.

11.3.2.5.8. Split the composite sample between 5-15 250 mL jars, depending on project requirements.

11.3.2.5.9. Use the large scoop to dispense the equal aliquots of the composite sample--one aliquot into each of 8 oz. jars. Repeat the process if there is sufficient sample for a complete set of large scoop aliquots (per Section 11.3.2.5.8).



11.3.2.5.10. Once the volume of wet composite sample in the bowl is less than the determined set of large scoops, switch to aliquoting with the small scoop. Dispense an equal amount of small scoops--one into each 8 oz. jar.

11.3.2.5.11. Use the fluoropolymer scoop to scrape inside the bowl, and dispense this part of the sample with the small scoop. If

there is insufficient sample to use full scoops, the use of replicate partial scoops is acceptable.



11.3.2.5.12. When aliquoting is complete, the composite sample has been evenly distributed among the 8 oz. jars. Note: Multiple jars are used for various analyses, quality control, and archive purposes.

11.3.2.5.13. Mix the contents of each 250 mL jar and remove about 10 g from each of the first three jars for three total solids (percent moisture) analyses. The relative percent difference (RPD) between the first two must not exceed 8%.

11.3.2.5.13.1. If the RPD exceeds 8%, repeat the total solids analysis on two fresh aliquots from the first two jars.

11.3.2.5.13.2. If the RPD still exceeds 8%, repeat the mixing process in Sections 11.3.2.5.1 to 11.3.2.5.12

11.3.2.5.14. Wipe the top of the jar to remove excess sample and install the cap. Transport all sample containers to Sample Receiving.

11.3.2.5.15. Discard the wooden spoon and spatulas. Wash the mixer bowl, hook and scoops using soap and water. Rinse with tap and reagent water.

11.3.2.6. Soil fractionation for lead analysis

11.3.2.6.1. Add sample ID label to outside of a disposable aluminum ($^1/_2$ or $^1/_4$ sheet depending on sample size). Line with butcher paper if aluminum is also an analyte of interest.

11.3.2.6.2. Transfer the soil sample to a tray. Remove large rocks and vegetation. Spreading the sample in a thin layer speeds drying and reduces the formation of hard clay chunks.



11.3.2.6.3. Place the tray in the ventilated drying rack for up to five days.

11.3.2.6.4. Periodically, stir the sample if needed to expose moist sample to the air. The butcher paper may also be changed, if it has become damp.

11.3.2.6.5. Samples with high clay content tend to form large, hard clay aggregates. To reduce the formation of these hard “bricks”, use the bottom of a clean disposable beaker to gently crush the semi-dried sample before drying is complete.

11.3.2.6.6. Allow the sample to dry until crumbly.

11.3.2.6.7. Assemble 8-inch sieve stack. The order from the bottom of the stack is collection pan, #60 sieve, and #10 sieve. Transfer sample ID label from drying pan to sieve stack.

Note: Project-specific sieve size requirements may vary from the reference method. These will be noted in LIMS, both in the project and in the method or login notes. #100 (150um) sieves are commonly used in place of #60 sieves. The smallest sieve size should be the closest to the collection pan, with the next size up above it.

11.3.2.6.8. If significant amounts of clay are present, aggregates may be broken by pressing gently with a hand-operated mortar and pestle. Note that only this gentle crushing is permitted by the reference method. Samples that are being analyzed as soil fractions for lead should not be subjected to aggressive disaggregation such as the use of a soil disaggregator or coffee grinder.

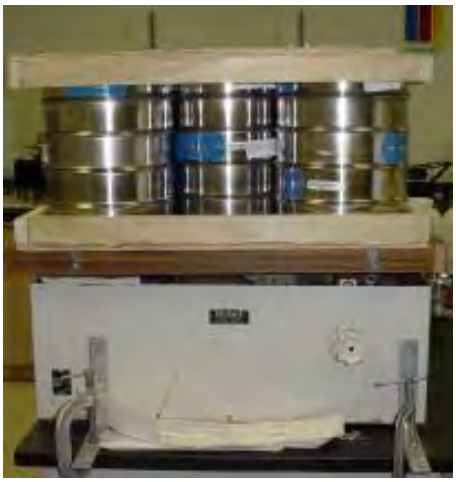
11.3.2.6.9. Transfer dried soil to the #10 sieve at the top of the sieve stack. If fine particulates are present, use sufficient ventilation to prevent the analyst from inhaling the dust. Install sieve cover.





11.3.2.6.1. Place the sieve stack on the shaker table and secure. Run the shaker for one hour. Stop the shaker and remove the lid for each sample to check on the progress of sieving. Shake the samples by hand (in a different direction than the shaker table has traveled). If any sample passes through the sieve at this point, place the sieve stack(s) back on the shaker and shake for up to one additional hour.

11.3.2.6.2. Do not extend shaking beyond two hours without conferring with the client.





11.3.2.6.3. Record the weights of the three fractions--large (did not pass through the #10 sieve), coarse (passed through #10 sieve, but not the #60 sieve), and fine (passed through both #10 and #60 sieves). If different sieve sizes have been used, record the sizes on the bench sheet.



11.3.2.6.4. To weigh each fraction, place the corresponding sieve or pan on the balance, press “Tare”, transfer the fraction, and place sieve or pan back on the balance. The weight of the fraction will read as a negative value.

11.3.2.6.5. Discard the large fraction. Mix the fine fraction. Remove approximately 10g for total solids analysis. If required, perform splitting and/or subsampling in accordance with ISM procedures. See the subsampling SOP for further information.

11.3.2.6.6. If the coarse fraction is to be analyzed separately, mix the coarse fraction and remove approximately 10g for total solids analysis. The coarse fraction should be ground in a mortar and pestle (Section 11.3.2.3) prior to removing the aliquot for metals digestion.

11.3.2.6.7. The combination of coarse and fine fractions is defined as the “total”. If the “total” is to be measured, weigh 1/10 of the coarse fraction and 1/10 of the fine fraction. Combine, mix, and remove approximately 10g for total solids analysis.

11.3.2.6.8. Bottle coarse, fine, and “total”(if needed) fractions for metals analysis.

11.3.2.6.9. For Michigan samples, the “total” result is calculated from a weighted average of the results from the fine and coarse fractions.

Total Lead = [(A x Wf) + (B x Wc)] / (Wf + Wc)

Where:

A = Concentration of Lead (mg/Kg dry) in fine fraction

B = Concentration of Lead (mg/Kg dry) in coarse fraction

Wf = Total weight of fine fraction

Wc = Total weight of coarse fraction

11.3.2.6.10. Wash the sieves with soap, tap water, and deionized water. Dislodge objects from the screen with a green scratch pad, wooden tongue blade or small screwdriver, as necessary. Dry sieves in a low-heat oven or air-dry at ambient temperature over night, depending how soon they will be needed.

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11.4. Sample Analysis

11.4.1. Not applicable to this procedure.

11.5. Analytical Documentation

11.5.1. Record all analytical information in LIMS, including any corrective actions or modifications to the method.

11.5.2. Record all standards and reagents in the LIMS Reagents module. All standards and reagents are assigned a unique number for identification.

12. DATA ANALYSIS AND CALCULATIONS

12.1. Not applicable to this procedure.

13. METHOD PERFORMANCE

13.1. The Group/Team Leader has the responsibility to ensure this procedure is performed by an associate who has been properly trained in its use and has the required experience.

14. POLLUTION PREVENTION

14.1. It is Eurofins TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage, and reagent stability). Employees must abide by the policies in Section 13 of the NDSC EH&S Manual Document No. CW-E-M-001 for "Waste management and Pollution Prevention".

15. WASTE MANAGEMENT

15.1. All waste will be disposed of in accordance with Federal, State and Local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this method and the policies in Section 13 of the NDSC EH&S Manual Document No. CW-E-M-001 for "Waste Management and Pollution Prevention".

15.2. Waste Streams Produced by the Method

15.2.1. Used wood spatulas, aluminum sheets, butcher paper; discard in solid waste.

15.2.2. Dry ice is to be melted in the sink.

15.2.3. Acetone is to be disposed of in an appropriately labeled flammable waste barrel.

15.3. Laboratory personnel assigned to perform hazardous waste disposal procedures must have a working knowledge of the established procedures and practices of Eurofins TestAmerica. They must have training on the hazardous waste disposal practices upon initial assignment to these tasks, followed by annual refresher training.

16. REFERENCES

16.1. References

16.1.1. U.S. EPA, 2000. TRW Recommendations for Sampling and Analysis of Soil at Lead (Pb) Sites. EPA-540-F-00-010. OSWER 9285.7-38. April. Available on-line at: http://www.epa.gov/superfund/programs/lead/products/sssiev.pdf

16.1.2. U.S. EPA, 2003. TRW Recommendations for Performing Human Health Risk Analysis on Small Arms Shooting Ranges. OSWER 9285.7-37. March. Available on-line at: http://www.epa.gov/superfund/programs/lead/products/firing.pdf

16.1.3. U.S. EPA, 2003. Superfund Lead-Contaminated Residential Sites handbook. OSWER 9285.7-50. August. Available on-line at: http://www.epa.gov/superfund/programs/lead/products/handbook.pdf

16.1.4. Michigan DEQ SOP #213 Revision #1, Nov. 9, 2004, Soil Fractions Preparation for Lead Analysis (Creating Total, Fine and Coarse Soil Samples).

16.1.5. ASTM D 6323-12, Laboratory Subsampling of Media Related to Waste Management Activities, 2012

16.1.6. ITRC Incremental Sampling Methodology, Available online at: http://www.itrcweb.org/ism-1/

16.1.7. Eurofins TestAmerica Canton QA Manual, current version

16.1.8. NDSC EH&S Manual Document No. CW-E-M-001, and Eurofins TestAmerica Canton Facility Addendum and Contingency Plan, current version

16.1.9. Quality Management Plan (QMP), current version

16.1.10 Revision History

Historical File:				
Revision 0: 11/27/13				
Revision 1: 01/23/15				
Revision 2: 05/31/17				
Revision 3: 12/5/18				

16.2. Associated SOPs and Policies, current version

16.2.1. QA Policy, QA-003

16.2.2. Glassware Washing, NC-QA-014

17. MISCELLANEOUS (TABLES, APPENDICES, ETC.)

17.1. Reporting limits

17.1.1. Not applicable to this procedure

17.2. Method deviations – None

Title: SUBSAMPLING

[Method: ASTM D6323-12]

Approvals (Signature/Date):

Technology Specialist Diane Jones	06/10/21 Date	Health & Safety Coordinator Steve Jackson	06/23/21 Date
Quality Assurance Manager Mark J. Loeb	06/16/21 Date	Technical Director Lucas Grossman	06/23/21 Date

This SOP was previously identified as SOP No. NC-OP-046, Rev 3, dated 06/18/19

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1. SCOPE AND APPLICATION

1.1. These mixing, subsampling, and splitting techniques are applicable to a wide range of soil, sediment, tissue, water, and waste samples. Care must be taken to match the appropriate technique with the matrix, target analytes and quality objectives.

1.2. The goal of all mixing, subsampling, and splitting techniques is to obtain representative splits or subsamples for preparation and analysis. Particle size reduction and processing can be beneficial in obtaining representative subsamples and specific processes for particle size reduction and processing can be found in the soil processing SOP (NC-OP-044) and non-soil processing SOP (NC-OP-045).

1.3. This document accurately reflects current laboratory Standard Operating Procedures (SOP) as of the date above. All facility SOPs are maintained and updated as necessary.

NOTE: If foreign or quarantined solids are received, refer to SOP NC-SC-019 Canton Foreign Soils, current revision, and contact your Environmental Health and Safety Coordinator for proper handling instructions.

2. SUMMARY OF METHOD

2.1. All samples should be mixed and subsampled appropriately for the test and analytes of interest.

2.2. Solid sample mixing procedures include in-jar mixing, horizontal surface mixing, and mortar and pestle.

2.3. Solid sample subsampling procedures include alternate scoop, one-dimensional slabcake, two-dimensional slabcake, and cone and quarter methods.

2.4. Liquid sample subsampling procedures include centrifuge, pipettes, coliwasa devices, and multiphase procedures.

3. DEFINITIONS

3.1. Refer to the glossary in the laboratory QA Manual, current version.

3.2. Mix: To thoroughly blend the sample and reduce the analyte concentration differences between different parts of the overall sample. It is most effective when the particle size and density differences within the sample are small.

3.3. Incremental Sampling Methodology: A structured composite sampling and processing protocol that reduces data variability and provides a reasonably unbiased estimate of the mean contaminant concentrations in an area/volume of soil targeted for sampling. ISM provides representative samples of specific soil areas/volumes defined as decision units

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(DUs) by collecting numerous increments of soil (typically 30-100 increments) that are combined, processed, and subsampled according to specific protocols.

3.4. Grinding: A generic term for soil disaggregation or milling.

3.5. Disaggregation: The act of breaking the soil clumps into individual small particles but keeping the small pebbles and hard crystalline particles intact.

3.6. Milling: Complete particle size reduction of all soil components including hard crystalline materials to a defined maximum particle size (e.g. <250 µm or <75 µm).

3.7. Sample: For laboratory technicians, the sample is all the material delivered to the laboratory in a container collected by the field crew.

3.8. Subsample: The small representative amount removed from a field sample selected for final analysis. This is also referred to in some SOPs as the aliquot.

3.9. Representative subsample: A subsample taken in such a way that each particle had an equal chance of being selected. The most representative subsample is one that most closely resembles the true value of the material sampled or contains the least amount of error.

4. INTERFERENCES

4.1. Method interferences may be caused by contaminants in solvents, reagents, glassware, and other processing apparatus that lead to discrete artifacts. All of these materials must be routinely demonstrated to be free from interferences under conditions of the analysis by running laboratory method blanks as described in the Quality Control section of each analytical SOP. Specific selection of reagents may be required to avoid introduction of contaminants.

4.2. Metallic components of particle size reduction equipment can contribute some metal content to the solid samples. Hence carbon steel components are usually preferable to stainless steel when necessary to minimize contamination from chromium, nickel and molybdenum. Some contamination from iron is common.

5. SAFETY

5.1. Employees must abide by the policies and procedures in the NDSC EH&S Manual, the Facility Addendum to the NDSC EH&S Manual, and this document.

5.2. Eye protection that protects against splash, laboratory coat, and appropriate gloves must be worn while samples, standards, solvents, and reagents are being handled. Cut-resistant gloves must be worn doing any other task that presents a strong possibility of getting cut. Disposable gloves that have been contaminated will be removed and discarded; other gloves will be cleaned immediately.

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5.3. Exposure to chemicals must be maintained **as low as reasonably achievable**; therefore, unless they are known to be non-hazardous, all samples must be opened, transferred and prepared in a fume hood, or under other means of mechanical ventilation where possible. All samples with stickers that read "Caution/Use Hood!" **must** be opened in the hood. Contact the EH&S Coordinator if this is not possible. Solvent and waste containers will be kept closed unless transfers are being made.

5.4. There are no materials used in this method that have a significant or serious hazard rating. A complete list of materials used in the method can be found in the reagents and standards section. Employees must review the information in the Safety Data Sheet (SDS) for each material prior to using it for the first time, or when there are major changes to the SDS.

5.5. All work must be stopped in the event of a known or potential compromise to the health and safety of a Eurofins TestAmerica associate. The situation must be reported **immediately** to the EH&S Coordinator and the Laboratory Supervisor.

5.6. When operating the electric chopper or grinder, be sure to keep all aqueous liquids clear to prevent the risk of electrical shock from any spills.

5.7. Avoid inhalation of sample dust. Work in a ventilation hood when necessary to avoid accidental dust inhalation. Wear a dust mask or respirator if the ventilation hood does not provide sufficient dust protection.

5.8. If there is any malfunction in the equipment. De-energize and tag out.

5.9. All noise levels are below OSHA limits.

5.10. Only trained personnel are permitted to use crushing equipment. A list of trained personnel will be maintained with EH&S and QA.

6. EQUIPMENT AND SUPPLIES

6.1. Butcher Paper

6.2. Plastic wrap

6.3. Aluminum foil

6.4. 8 oz. Glass jars with lids

6.5. Top-loading balance.

6.6. Appropriate containers for each subsample

7. REAGENTS AND STANDARDS

7.1. Deionized water: Reagent water must be produced by a Millipore DI system or equivalent. Reagent water must be free of the analytes of interest as demonstrated through the analysis of preparation blanks.

8. SAMPLE COLLECTION, PRESERVATION, AND STORAGE

8.1. Not applicable to this procedure. Sample collection, preservation, and storage are dependent on the requested test method and analytes.

9. QUALITY CONTROL

9.1. Equipment processing blanks might be applicable for some particle size reduction or selection techniques. There is no single blank matrix that is suitable for all analytes, equipment or processes. The blank matrix should be chosen by the client/data user based on the advantages and limitations described below in Section 9. If no blank matrix is selected by the client, reagent water should be used as the default when possible, as noted in Section 9.2.

9.2. Reagent water (or organic solvents) may be used to rinse the equipment surfaces. This liquid is then analyzed for the target analytes. This process is good at monitoring residue on equipment surfaces from previously processed samples. It does not evaluate the potential contribution of the equipment surface material to a solid sample.

9.3. Sand may be run through processing equipment and then analyzed to monitor for both sample carryover residue and contamination from equipment surface erosion. This process is most applicable for organic analytes. Sand always contains metals. These metals concentrations might be too high for suitable blank demonstrations. Also, the sand material is frequently more abrasive than soil and can over estimate sample contamination due to erosion of the equipment surfaces.

9.4. Teflon boiling chips can be suitable to monitor the cleanliness of processing equipment surfaces. This option is between reagent water and sand regarding abrasion of the equipment surfaces. This material is generally non-detect at parts per billion concentrations for most organic and inorganic analytes. The Teflon material does not mimic the behavior of soil in the subsequent sample extraction or digestion procedures.

9.5. Reprocess half of a field sample and look for differing analyte concentrations between the single and double processed sample aliquots. The occurrence of significantly elevated analyte concentrations in the double processed sample can be a good indication of carryover or contamination due to equipment surface erosion. This is most applicable for monitoring metals contamination in soil grinders.

10. CALIBRATION AND STANDARDIZATION

10.1. Not applicable to this procedure.

11. PROCEDURE

11.1. One-time procedural variations are allowed only if deemed necessary in the professional judgment of QA, operations supervisor, or designee to accommodate variation in sample matrix, chemistry, sample size, or other parameters. Any variation in procedure shall be completely documented using a Nonconformance Memo.

11.2. Any unauthorized deviations from this procedure must also be documented as a nonconformance with a cause and corrective action described.

11.3. Sample Preparation

11.3.1. The following sections describe a variety of procedures. Additional procedures for sample preparation are available in the Soil and Non-Soil Processing SOPs. The client/data user must select the procedure that is most appropriate for the sample matrix, analytes, and quality objectives. The selection of the procedure(s) can be made by the client in consultation with the appropriate laboratory representatives during project planning. There is no single default procedure that can be used in the absence of client selection.

11.3.2. Solid Sample Mixing Procedures

11.3.2.1. In jar mixing

11.3.2.1.1. The flowable sample is thoroughly stirred in the sample jar using a wooden blade.

11.3.2.2. Horizontal surface mixing

11.3.2.2.1. The sample is transferred to an aluminum tray or onto a sheet of paper and mixed with a wooden blade.

11.3.3. Solid Sample Subsampling Procedures

11.3.3.1. Multiple increments from a jar

11.3.3.1.1. Select 5-10 small sample increments from various locations within the sample container to make the entire subaliquot. The increments must come from all general areas of the container--top, sides, center, and bottom.

11.3.3.2. Two dimensional slabcake

11.3.3.2.1. Spread the sample to a consistent depth on a clean flat area covered with an appropriate disposable cover (butcher paper, aluminum tray or foil or plastic depending on the analytes of interest).

11.3.3.2.2. Select 30 or more small increments spread evenly over the sample area or as dictated by client data quality objectives.

The increments must be collected with a blunt end spatula or scoop to evenly collect from the top middle and bottom of the slabcake. A coring device of suitable material can also be used if the sample is sufficiently cohesive (e.g. moist soil).

11.3.3.2.3. If the sample has noxious odors or produces dust, the sample spreading and increment collecting should be performed in a hood or large flat bag of appropriate material. The analyst must be protected from potential inhalation hazards from the sample.

11.3.3.3. Alternate scoop

11.3.3.3.1. Wet or dry solid samples can be divided into two or more smaller aliquots. Determine the number of sub-aliquots, and prepare that many empty containers.

11.3.3.3.2. Scoops of the mixed laboratory sample are either placed in the analytical vessel or discarded.

11.3.3.3.3. Three scoops are discarded for every scoop saved. Randomly select an aliquot and place in the first container. Place the next three aliquots in the second container. Repeat as needed for additional aliquot containers.

11.3.3.3.4. Repeat the aliquoting cycle until the original sample is consumed.

11.3.3.3.5. The alternate scoop technique can also be used to subaliquot discrete samples that are to be homogenized into one composite sample.

11.3.3.3.5.1. Determine approximately the ratio of aliquot to discard scoops required to consume the sample and achieve the aliquot size required for homogenization. For example, if a 50 g aliquot is desired from a 250 g sample, a ratio of one scoop used to four scoops discarded will be used.

11.3.3.3.5.2. For each discrete sample, set up two empty containers of appropriate volume.

11.3.3.3.5.3. Stir each discrete sample in its container, or if necessary homogenize on a sheet of butcher paper and return to the jar.

11.3.3.3.5.4. Place one of the empty containers on a balance and tare it. Place one scoop of sample from the top

of the discrete sample jar into this container. This will be the portion used for a homogenized composite later.

11.3.3.3.5.5. Into the second jar, place the three or more (as determined in Section 11.3.3.3.5.1) scoops to be discarded.

11.3.3.3.5.6. Repeat Sections 11.3.3.3.5.4 and 11.3.3.3.5.5 until the sample has been consumed and the desired weight of the subaliquot is in the tared container. Record the weight of the sub-aliquot.

11.3.3.3.5.7. Repeat this process for all of the discrete samples that will be used for the composite, using approximately equal weights of each discrete sample.

11.3.3.3.5.8. After all discrete samples have been sub-aliquoted using the alternate scoop procedure, combine them on a sheet of butcher paper and homogenize. Pour the homogenized sample volume into the labeled sample jar for the composite sample.

11.3.3.4. One dimensional slabcake

11.3.3.4.1. This process is intended to produce large sub-samples from very large dry flowable solid samples, such as those collected using \incremental sampling methodology, dried and then chopped using the process described in Section 1.1.1See Reference 16.1.7.

11.3.3.4.2. Pour the dry soil sample into a long thin pile onto a clean horizontal surface. The pour height should not exceed 20 cm to minimize the formation of a dust cloud.

11.3.3.4.3. Ensure that the sample container makes at least 20 passes back and forth over the “line of sample.”

11.3.3.4.4. Using a rectangular flat-bottomed scoop, remove an increment from the “line of sample.” Ensure that a complete cross cut of the sample line includes the entire depth of that increment. Combine increments as needed to produce the needed sub-sample size.

11.3.3.5. Cone and quarter (for dry flowable samples)

11.3.3.5.1. Pour the sample into a cone on a clean flat area covered with an appropriate disposable cover (butcher paper, aluminum foil, or plastic depending on the analytes of interest).

11.3.3.5.2. Cut the cone in half in two directions to form four quadrants.

11.3.3.5.3. Return opposite quadrants to the original sample container.

11.3.3.5.4. Repeat the process in Sections 11.3.3.5.1 through 11.3.3.5.3 until the needed aliquot size has been obtained.

11.3.4. Liquid Sample Mixing Procedures

11.3.4.1. Closed container shaking

11.3.4.1.1. All liquid samples should be mixed by shaking in the original closed sample container unless a multiple layer sub-aliquoting procedure is used. This applies to emulsifiable layers such as oil and water and suspendable particulates in water. The sample must remain mixed long enough to pour out a representative aliquot. Samples that show noticeable separation in under a minute should be sub-aliquoted using an appropriate technique as described below.

11.3.4.1.2. The shaking process must be vigorous enough to mix and distribute analytes associated with different layers, particulates, or inside container walls.

11.3.5. Liquid Sample Subsampling Procedures

11.3.5.1. Pour

11.3.5.1.1. Samples that are well mixed can be sub-aliquoted by pouring an appropriate volume off the top of the sample.

11.3.5.2. Layer Subsampling

11.3.5.2.1. Some liquid samples with multiple layers separate too quickly to pour a representative sub-aliquot off the top. In some instances it is best to separate the layers and handle them as individual samples. In other instances representative aliquots of each layer must be collected and processed as a single sub-sample.

11.3.5.2.2. Unless directed otherwise, record the phase ratios (or volumes) of the layers.

11.3.5.2.3. Gravity or Centrifuge Settling

11.3.5.2.3.1. Allow the sample layers to separate based on density. Centrifugation can be used to accelerate the process if simple gravity settling is not fast enough.

11.3.5.2.4. Separatory Funnel

11.3.5.2.4.1. Gently pour the sample into a separatory funnel. Allow time for additional layer separation as needed.

11.3.5.2.4.2. Drain the layers out into separate containers one at a time.

11.3.5.2.4.3. Some oils stick to the separatory funnel sides so the draining process must be slow enough to avoid remixing the layers.

11.3.5.2.5. Pipette

11.3.5.2.5.1. Use a pipette of appropriate size to collect a sub-aliquot of a sample layer of interest and transfer to an empty container.

11.3.5.2.5.2. If the entire sample is to be separated, pipette almost all the top layer into a new container. Draw the small volume of the top layer into the pipette along with a small portion of the second layer. Allow the two layers to separate in the pipette (like a separatory funnel). Dispense the bottom layer back into original sample container with the bulk of the bottom layer. Next dispense the remaining top layer into the container that holds the top layer.

11.3.5.2.5.3. Repeat as needed for each layer of interest.

11.3.5.2.6. Coliwasa

11.3.5.2.6.1. A coliwasa is a “coring device” designed for liquid multi-layer samples. It is a long tube with a short valve at the bottom. When designed and used properly it collects representative aliquots of each layer with each sub-aliquoting immersion.

11.3.5.2.6.2. The volume collected is determined by the diameter of the coliwasa and the height of the sample. The larger the tube diameter or taller the sample height, the larger the volume of sample collected.

11.3.5.2.6.3. Place the foot of the valve and its control rod in the multi-layer sample. Slowly slide the coliwasa tube over the control rod and close the ground glass seal of the valve at the bottom.

11.3.5.2.6.4. Lift the coliwasa by the control rod to keep the valve sealed.

11.3.5.2.6.5. Place the coliwasa over the new sample aliquot container. Grasp the top of the coliwasa tube and slowly lower the control rod a few millimeters to open the foot valve and drain the sample into the container.

11.3.5.2.6.6. Repeat Sections 11.3.5.2.6.3 through 11.3.5.2.6.5 until sufficient sample aliquot has been collected.

11.3.6. Multiphase Sample Mixing Procedures

11.3.6.1. All liquid samples should be mixed by shaking unless a multiple layer subaliquoting procedure is used. This applies to emulsifiable layers such as oil and water and suspendable particulates in water. The sample must remain mixed long enough to pour out a representative aliquot. Samples that show noticeable separation in under a minute should be subaliquoted using an appropriate technique as described below.

11.3.6.2. Closed container shaking

11.3.6.2.1. The shaking process must be vigorous enough to mix and distribute analytes associated with different layers, particulates, or inside container walls.

11.3.6.3. Open container stirring

11.3.6.3.1. The sample is stirred or swirled to mix.

11.3.6.4. Wet Sediments

11.3.6.4.1. Aqueous samples for non-volatile compound analysis may contain settleable materials. If the settleable materials are to be included as part of the laboratory sample, and they will remain suspended, or can easily be re-suspended and will remain so during the subsampling operation, the sample should be handled as a liquid sample. These samples should be gently swirled for 15 seconds or slowly inverted six times to reduce heterogeneity.

11.3.6.4.2. If the liquid portion only is to be used, the settleable material must be allowed to sink to the bottom before withdrawing the subsample.

11.3.6.4.3. If the settleable material will not remain suspended and is to be included in the analysis, the sample should be treated as a multilayered sample.

11.3.7. Multi-phase Sample Subsampling Procedures

11.3.7.1. Multi-layered samples may include liquid/liquid layers, liquid/solid samples, or solid/solid samples.

11.3.7.2. If the liquid portion of a sludge sample can be re-mixed with the solid portion and will re-separate over time, the sample should be handled as a solid sample.

11.3.7.3. If the solid portion will remain suspended or can easily be re-suspended, the sample should be treated as a liquid sample. Mixing may occur by inverting the container or with slight shaking

11.3.7.4. Layer Separating

11.3.7.4.1. Gravity/Centrifuge – The solid/liquid phase separation may be achieved by gently centrifuging the unopened container or by allowing it to sit undisturbed until the solid portion is settled.

11.3.7.4.2. Pipette – For liquid/liquid layers, separate the layers using a pipette. Each layer may be either transferred into another container or directly into the analytical vessel. Each separated portion is then handled as a homogeneous liquid sample.

11.3.7.4.3. Filter/Decant – For liquid/solid layers, the liquid subsample may be obtained by filtering or decanting the liquid portion from the solid portion. The liquid portion is then handled as a homogeneous liquid sample. The solid portion is handled as a solid laboratory sample.

11.4. Sample Analysis

11.4.1. Not applicable to this procedure

11.5. Analytical Documentation

11.5.1. Record all analytical information in LIMS, including any corrective actions or modifications to the method.

11.5.2. Record all standards and reagents in the LIMS reagents module. All standards and reagents are assigned a unique number for identification.

12. DATA ANALYSIS AND CALCULATIONS

12.1. Not applicable to this procedure.

13. METHOD PERFORMANCE

13.1. The Group/Team Leader has the responsibility to ensure this procedure is performed by an associate who has been properly trained in its use and has the required experience.

14. POLLUTION PREVENTION

14.1. It is Eurofins TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage, and reagent stability). Employees must abide by the policies in Section 13 of the NDSC EH&S Manual Document No. CW-E-M-001for "Waste management and Pollution Prevention."

15. WASTE MANAGEMENT

15.1. All waste will be disposed of in accordance with Federal, State and Local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this method and the policies in Section 13 of the NDSC EH&S Manual Document No. CW-E-M-001for "Waste Management and Pollution Prevention."

15.2. Waste Streams Produced by the Method

15.2.1. Used wood spatulas, aluminum sheets, butcher paper; discard in solid waste.

15.3. Laboratory personnel assigned to perform hazardous waste disposal procedures must have a working knowledge of the established procedures and practices of Eurofins TestAmerica. They must have training on the hazardous waste disposal practices upon initial assignment to these tasks, followed by annual refresher training.

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16.1.5. ASTM D 6323-12, Laboratory Subsampling of Media Related to Waste Management Activities, 2012

16.1.6. Eurofins TestAmerica Canton QA Manual, current version

16.1.7. NDSC EH&S Manual Document No. CW-E-M-001 and Eurofins TestAmerica Canton Facility Addendum and Contingency Plan, current version

16.1.8. Quality Management Plan (QMP), current version

16.1.10 Revision History

Historical File:		Revision 0: 11/27/14		
		Revision 1: 01/23/15		
		Revision 2: 05/31/17		
		Revision 3: 06/18/19		

16.2. Associated SOPs and Policies, current version

16.2.1. QA Policy, QA-003

16.2.2. Glassware Washing, NC-QA-014

17. MISCELLANEOUS (TABLES, APPENDICES, ETC.)

17.1. Reporting limits

17.1.1. Not applicable to this procedure

17.2. Method deviations – None

Quality Assurance Manual Cover Page

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Quality Assurance Manual
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SECTION 3. INTRODUCTION, SCOPE AND APPLICABILITY

3.1 Introduction and Compliance References

Eurofins TestAmerica Canton's (QA) Manual is a document prepared to define the overall policies, organization objectives and functional responsibilities for achieving the laboratory's QA Program. Governing SOPs are in place within the organization to ensure the proper execution of this QA Manual (refer to Appendix 1). This manual and referenced documents are required reading for all personnel.

The laboratory is a team of people who work together to serve the health and environmental needs of society through science and technology. The laboratory maintains a local perspective in its scope of services and client relations and maintains a national perspective in terms of quality.

As such, this QA Manual has been prepared to assure compliance with The NELAC Institute (TNI) Standard, dated 2009 and 2016; ISO/IEC Guide 17025:2005 and 2017. The policies and procedures listed in Appendix 1 are compliant with the National Divisional Support Center (NDSC) Quality Management Plan (QMP) for Eurofins TestAmerica; Eurofins TestAmerica Canton, and the various accreditation and certification programs which are held by the laboratory to support environmental work (Appendix 2) The QMP provides a summary of Eurofins TestAmerica's quality and data integrity system. It contains requirements and general guidelines under which all Eurofins TestAmerica facilities shall conduct their operations.

Refer to Appendix 3 for a list of additional references for which this QA Manual is compliant; and Appendix 4 for the compliance crosswalk of this manual to the TNI & ISO/IEC Guide 17025 requirements.

3.2 Terms and Definitions

A QA Program is a company-wide system designed to ensure that data produced by the laboratory conforms to the standards set by state and/or federal regulations. The program functions at the local management level through company goals, from guidance at the executive management level, and at the analytical level through Standard Operating Procedures (SOPs) and quality control. Our program is designed to minimize systematic error, encourage constructive, documented problem solving, and provide a framework for continuous improvement within the organization to better serve our clients.

Refer to Appendix 5 for a list of Terms and Acronyms

3.3 .Scope / Fields of Testing

The laboratory analyzes a broad range of environmental and industrial samples. Sample matrices vary among effluent water, groundwater, hazardous waste, sludge and soils. The QA Program contains specific procedures and methods to test samples of differing matrices for chemical, physical and biological parameters. The Program also contains guidelines on maintaining documentation of analytical processes, reviewing results, servicing clients and tracking samples through the laboratory. The technical and service requirements of all analytical requests are

thoroughly evaluated before commitments are made to accept the work. Measurements are made using published reference methods or methods developed and validated by the laboratory.

The methods covered by this manual include the most frequently requested methodologies needed to provide analytical services in the United States and its territories. The specific list of test methods used by the laboratory can be found in LIMS.

Our areas of expertise include:

Standard Services	Specialty Services
• Volatiles • Semivolatiles • Metals • Pesticides/PCBs/Herbicides • Petroleum Hydrocarbons • Waste Characterization • Non-Potable Water Testing • Soil and Surface Water Testing • Sediment and Tissue Testing • Method Development • Shale Oil & Gas Analysis	• Organic Acids • Incremental sampling

NOTE: All current certificates and scopes of accreditation are available on the laboratory's website at www.testamericainc.com.

The approach of this manual is to define the minimum level of quality assurance and quality control necessary to meet these requirements. All methods performed by the laboratory shall meet these criteria as appropriate. In some instances, quality assurance project plans (QAPPs), project specific data quality objectives (DQOs) or local regulations may require criteria other than those contained in this manual. In these cases, the laboratory will abide by the requested criteria following review and acceptance of the requirements by the Laboratory Director and/or the Quality Assurance (QA) Manager. In some cases, QAPPs and DQOs may specify less stringent requirements. The Laboratory Director and the QA Manager must determine if it is in the lab's best interest to follow the less stringent requirements.

Ohio VAP requirements are listed throughout the document.

3.4 Management of the Manual

3.4.1 Review Process

The template on which this manual is based is reviewed annually by Eurofins National Divisional Support Center (NDSC) which houses the Quality Assurance leadership team for Eurofins Environment Testing America. NDSC QA will assure that the template remains in compliance with Section 3.1. This manual itself is reviewed every two years, or as required by state program, such as CA ELAP, by senior laboratory management to assure that it reflects current practices and meets the requirements of the laboratory's clients and regulators as well as the QMP. Occasionally, the manual may need changes in order to meet new or changing regulations and operations. The QA Manager will review the changes in the normal course of business and incorporate changes into revised sections of the document. All updates will be reviewed by the senior laboratory management staff. The laboratory updates and approves

such changes according to our Document Control (NC-QA-030) and Updating Procedures (NC-QA-027).

4 MANAGEMENT REQUIREMENTS

4.1 Overview

The laboratory is a local operating unit of Eurofins Environment Testing America and includes various Service Centers. Service Centers under the direct authority of Eurofins TestAmerica Canton include (but are not limited to) the Cambridge, Cincinnati, Columbus, and Dayton facilities in Ohio and the Grand Rapids and Brighton facilities in Michigan.. The laboratory operational and support staff work under the direction of the Laboratory Director. The laboratory management staff includes directors, managers and group leaders. The organizational chart of the management staff are presented in Figure 4-1. Individual departmental staff lists are maintained in the laboratory's internal intranet. Employee names are provided to demonstrate range and size of departments however the actual staff members may vary over time. The most current Organization Chart may be obtained from Quality Assurance Manager or Laboratory Director.

4.2 Roles and Responsibilities

In order for the QA Program to function properly, all members of the staff must clearly understand and meet their individual responsibilities as they relate to the quality program. The responsibility for quality resides with every employee of the laboratory. All employees have access to the QA Manual, are trained to this manual, and are responsible for upholding the standards therein. Each person carries out his/her daily tasks impartially and in a manner consistent with the goals and in accordance with the procedures in this manual and the laboratory's SOPs. The following descriptions briefly define each role in its relationship to the Quality Assurance Program.

4.2.1 Vice President of Quality and Environmental Health and Safety (VP-QA/EHS)

The Vice President (VP) of QA/EHS reports directly to Eurofins Environment Testing America Chief Operating Officer (COO). With the aid of the NDSC Quality Team Members, Business Unit Managers, and Laboratory Directors, the VP-QA/EHS has the responsibility for the establishment, general overview and Corporate maintenance of the Quality Assurance and EH&S Programs within Eurofins Environment Testing America. Additional responsibilities include:

- Review of QA/QC and EHS aspects of NDSC Official Document national projects and expansions or changes in services.
- Work with various organizations outside of the laboratory to further the development of quality standards and represent the laboratory at various trade meetings.
- Prepare monthly reports for quality and EH&S metrics across the environmental testing laboratories and a summary of any quality and EH&S related initiatives and issues.
- With the assistance of the Executive Management and the EHS Managers, maintenance and implementation of the Eurofins Environment Testing America Environmental, Health and Safety Program.

4.2.2 Quality Directors

There are three (3) Quality Directors within NDSC that report directly to the VP-QA/EHS. These Quality Directors have oversight of the general overview and maintenance of the QA Program within the Eurofins Environment Testing America laboratories. Supported tasks include:

- Monitors laboratory internal audit findings;
- Identifies common laboratory weaknesses; and monitors corrective action closures.
- Develops NDSC quality guidance documents and management tools for ensuring and improving compliance;
- Monitors and communicates DoD/DoE requirements;
- Monitors and communicates regulatory and certification requirements;
- Training and On Boarding
- Laboratory assessments, mentoring, and interventions
- Track/drive root cause investigations and corrective action plans
- Builds knowledge base for preventive actions

4.2.3 Quality Information Manager

The Quality Information works directly with the NDSC Quality Directors and EHS Managers; and reports directly to the VP-QA/EHS. The Quality Information Manager is responsible for the management of:

- NDSC Official Documents
- TALS/LIMS Certification Module Data
- Company's Intranet website
- Company's Regulatory Limits Database
- Subcontract laboratory and approved vendor information
- Internal and External client support for various company groups (e.g., Client Services, EH&S, Legal, IT, Sales) for both quality and operational functions
- Communicate regulatory information and lists

4.2.4 Ethics and Compliance Officers (ECOs)

The NDSC VP-QA/EHS and Corporate Counsel are designated Ethics and Compliance Officer (ECO) –Each ECO acts as a back-up to the other ECO and both are involved when data investigations occur. Each ECO has a direct line of communication to the entire executive management personnel and lab management staff.

The ECOs monitor and audit procedures to determine compliance with policies and to make recommendations for policy enhancements to the President COO, Laboratory Director or other appropriate individuals within the laboratory. The ECO will assist the laboratory QA Manager in the coordination of internal auditing of ethical policy related activities and processes within the laboratory, in conjunction with the laboratory's regular internal auditing function.

The ECOs will also participate in investigations of alleged violations of policies and work with the appropriate internal departments to investigate misconduct, remedy the situation, and prevent recurrence of any such activity.

4.2.5 Environmental Health and Safety (EH&S) Managers

There are 3 EH&S Managers within NDSC that report directly to the VP-QA/EHS. These EH&S Managers have oversight of the general overview and maintenance of the EH&S Program within the Eurofins Environment Testing America laboratories. Supported tasks include:

- Consolidation and tracking all safety and health-related information and reports for the company, and managing compliance activities for Eurofins Environment Testing America locations.
- Coordination/preparation of the Environmental, Health and Safety Manual Template that is used by each laboratory to prepare its own laboratory-specific Safety Manual/ CHP.
- Preparation of information and training materials for laboratory EHS Coordinators.
- Assistance in the coordination of employee exposure and medical monitoring programs to insure compliance with applicable safety and health regulations.
- Serving as Department of Transportation (D.O.T.) focal point and providing technical assistance to location management.
- Serving as Hazardous Waste Management main contact and providing technical assistance to location management.

4.2.6 Laboratory Director

The Laboratory Director is responsible for the overall quality, safety, financial, technical, human resource and service performance of the whole laboratory and reports to their respective business unit President. The Laboratory Director is also responsible for any service centers connected with their laboratory that perform analytical tests, such as short holding time analyses for pH. The Laboratory Director provides the resources necessary to implement and maintain an effective and comprehensive Quality Assurance and Data Integrity Program.

Specific responsibilities include, but are not limited to:

- Provides one or more technical directors for the appropriate fields of testing. If the Technical Director is absent for a period of time exceeding 15 consecutive calendar days, the Laboratory Director must designate another full time staff member meeting the qualifications of the Technical Director to temporarily perform this function. If the absence exceeds 35 consecutive calendar days, the primary accrediting authority must be notified in writing.
- Ensures that all analysts and supervisors have the appropriate education and training to properly carry out the duties assigned to them and ensures that this training has been documented. Works with Eurofins Environment Testing Human Resources for hiring of new personnel.
- Ensures that personnel are free from any commercial, financial and other undue pressures which might adversely affect the quality of their work.
- Ensures company human resource policies are adhered to and maintained.

- Ensures that sufficient numbers of qualified personnel are employed to supervise and perform the work of the laboratory. Assesses laboratory capacity and workload
- Ensures that appropriate corrective actions are taken to address analyses identified as requiring such actions by internal and external performance or procedural audits. Procedures that do not meet the standards set forth in the QA Manual or laboratory SOPs may be temporarily suspended by the Laboratory Director.
- Reviews and approves SOPs as directed by the Quality Assurance department prior to their implementation and ensures all approved SOPs are implemented and adhered to.
- Pursues and maintains appropriate laboratory certification and contract approvals. Supports ISO 17025 requirements.
- Ensures client specific reporting and quality control requirements are met.
- Contributes to the continuous improvement of the laboratory operations.
- Maintains an awareness of technical developments and regulatory requirements.
- Captains the management team, consisting of the QA Manager, the Technical Director, and the Operations Manager as direct reports.

4.2.7 Quality Assurance (QA) Manager or Designee

The QA Manager has responsibility and authority to ensure the continuous implementation of the quality system at the laboratory where they work. The QA Manager is also responsible for any service centers connected with their laboratory that perform analytical tests, such as short holding time analyses for pH.

The QA Manager reports directly to the Laboratory Director and their and their NDSC Quality Director. This position is able to evaluate data objectively and perform assessments without outside (e.g., managerial) influence. The NDSC QA Team may be used as a resource in dealing with regulatory requirements, certifications and other quality assurance related items. The QA Manager directs the activities of the QA office to accomplish specific responsibilities, which include, but are not limited to:

- Serves as the focal point for QA/QC in the laboratory.
- Have functions independent from laboratory operations for which he/she has quality assurance oversight.
- Have documented training and/or experience in QA/QC procedures and the laboratory's Quality System.
- Have a general knowledge of the analytical test methods for which data audit/review is performed (and/or having the means of getting this information when needed).
- Arrange for or conducting internal audits on quality systems and the technical operation
- Notifying laboratory management of deficiencies in the quality system and ensuring corrective action is taken. Procedures that do not meet the standards set forth in the QAM or laboratory SOPs shall be investigated following procedures outlined in Section 12 and if deemed necessary may be temporarily suspended during the investigation.
- Maintaining and updating the QA Manual.

- Monitoring and evaluating laboratory certifications; scheduling proficiency testing samples.
- Monitoring and communicating regulatory changes that may affect the laboratory to management.
- Training and advising the laboratory staff on quality assurance/quality control procedures that are pertinent to their daily activities.
- The laboratory QA Manager or designee will maintain records of all ethics-related training, including the type and proof of attendance.
- Maintain, improve, and evaluate the corrective action database and the corrective and preventive action systems.
- Objectively monitor standards of performance in quality control and quality assurance without outside (e.g., managerial) influence.
- Coordinating of document control of SOPs, MDLs, control limits, and miscellaneous forms and information.
- Performing technical data audits and method audits to ensure consistency and compliance with regulatory requirements.
- Review of external audit reports and data validation requests.
- Follow-up with audits to ensure client QAPP requirements are met.
- Establishment of reporting schedule and preparation of various quality reports for the Laboratory Director, clients and/or NDSC QA Team
- Development of suggestions and recommendations to improve quality systems.
- Research of current state and federal requirements and guidelines.
- Participate in the vendor and supplier approval process, including subcontractors.
- Captains the QA team to enable communication and to distribute duties and responsibilities.
- Communication to the relevant regulatory authorities when there are management or facility changes that impact the laboratory.
- Ensuring Communication & monitoring standards of performance to ensure that systems are in place to produce the level of quality as defined in this document.
- Evaluation of the thoroughness and effectiveness of training.
- Compliance with ISO 17025**.**

4.2.8 Technical Director or Designee

The Technical Director reports directly to the Laboratory Director. He/she is accountable for all analyses and analysts under their experienced supervision and for compliance with the ISO 17025:2017 Standard. The scope of responsibility ranges from the new-hire process and existing technology through the ongoing training and development programs for existing analysts and new instrumentation. Specific responsibilities include, but are not limited to:

- Exercises day-to-day supervision of laboratory operations for the appropriate field of accreditation and reporting of results. Coordinating, writing, and reviewing preparation of all test methods, i.e., SOPs, with regard to quality, integrity, regulatory and optimum and

efficient production techniques, and subsequent analyst training and interpretation of the SOPs for implementation and unusual project samples. He/she insures that the SOPs are properly managed and adhered to at the bench. He/she develops standard costing of SOPs to include supplies, labor, overhead, and capacity (design vs. demonstrated versus first-run yield) utilization.

- Reviewing and approving, with input from the QA Manager, proposals from marketing, in accordance with an established procedure for the review of requests and contracts. This procedure addresses the adequate definition of methods to be used for analysis and any limitations, the laboratory's capability and resources, the client's expectations. Differences are resolved before the contract is signed and work begins. A system documenting any significant changes is maintained, as well as pertinent discussions with the client regarding their requirements or the results of the analyses during the performance of the contract. All work subcontracted by the laboratory must be approved by the client. Any deviations from the contract must be disclosed to the client. Once the work has begun, any amendments to the contract must be discussed with the client and so documented.
- Monitoring the validity of the analyses performed and data generated in the laboratory. This activity begins with reviewing and supporting all new business contracts, insuring data quality, analyzing internal and external non-conformances to identify root cause issues and implementing the resulting corrective and preventive actions, facilitating the data review process (training, development, and accountability at the bench), and providing technical and troubleshooting expertise on routine and unusual or complex problems.
- Providing training and development programs to applicable laboratory staff as new hires and, subsequently, on a scheduled basis. Training includes instruction on calculations, instrumentation management to include troubleshooting and preventive maintenance.
- Enhancing efficiency and improving quality through technical advances and improved LIMS utilization. Capital forecasting and instrument life cycle planning for second generation methods and instruments as well as asset inventory management.
- Coordinating sample management from "cradle to grave," insuring that no time is lost in locating samples.
- Scheduling all QA/QC-related requirements for compliance, e.g., MDLs, etc.
- Captains department personnel to communicate quality, technical, personnel, and instrumental issues for a consistent team approach.
- Coordinates audit responses with the QA Manager.
- Compliance with ISO 17025.

4.2.9 Operations Manager

The Operations Manager manages and directs the analytical production sections of the laboratory. He/She reports directly to the Laboratory Director. He/She assists the Technical Director in determining the most efficient instrument utilization. More specifically, he/she:

- Evaluates the level of internal/external non-conformances for all departments.
- Continuously evaluates production capacity and improves capacity utilization.
- Continuously evaluates turnaround time and addresses any problems that may hinder meeting the required and committed turnaround time from the various departments.

- Develops and improves the training of all analysts in cooperation with the Technical Director and QA Manager and in compliance with regulatory requirements.
- Works with the Departmental Group Leaders to ensure that scheduled instrument maintenance is completed.
- Scheduling all QA/QC-related requirements for compliance, e.g., MDLs, etc.
- Captains department personnel to communicate quality, technical, personnel, and instrumental issues for a consistent team approach.
- Coordinates audit responses with the QA Manager.
- Is responsible for efficient utilization of supplies.
- Constantly monitors and modifies the processing of samples through the departments.
- Fully supports the quality system.

4.2.10 Department Group Leaders

Department Group Leaders report to the Operations Manager. Each one is responsible to:

- Ensure that analysts in their department adhere to applicable SOPs and the QA Manual. They perform frequent SOP and QA Manual review to determine if analysts are in compliance and if new, modified, and optimized measures are feasible and should be added to these documents.
- With regard to analysts, participates in the selection, training (as documented in Section 5.3), development of performance objectives and standards of performance, appraisal (measurement of objectives), scheduling, counseling, discipline, and motivation of analysts and documents these activities in accordance with systems developed by the QA and Personnel Departments. They evaluate staffing sufficiency and overtime needs. Training consists of familiarization with SOP, QC, Safety, and computer systems.
- Encourage the development of analysts to become cross-trained in various methods and/or operate multiple instruments efficiently while performing maintenance and documentation, self-supervise, and function as a department team.
- Provide guidance to analysts in resolving problems encountered daily during sample prep/analysis in conjunction with the Technical Director, Operations Manager, and/or QA Manager. Each is responsible for 100% of the data review and documentation, non-conformance and CPAR issues, the timely and accurate completion of performance evaluation samples and MDLs, for the department.
- Ensure all logbooks are maintained, current, and properly labeled or archived.
- Report all non-conformance conditions to the QA Manager, Technical Director, Operations Manager, and/or Laboratory Director.
- Ensure that preventive maintenance is performed on instrumentation as detailed in the QA Manual or SOPs. Responsible for developing and implementing a system for preventive maintenance, troubleshooting, and repairing or arranging for repair of instruments.
- Maintain adequate and valid inventory of reagents, standards, spare parts, and other relevant resources required to perform daily analysis.
- Achieve optimum turnaround time on analyses and compliance with holding times.

- Conduct efficiency and cost control evaluations on an ongoing basis to determine optimization of labor, supplies, overtime, first-run yield, capacity (designed vs. demonstrated), second- and third-generation production techniques/instruments, and long-term needs for budgetary planning.
- Develop, implement, and enhance calibration programs.
- Provide written responses to external and internal audit issues.

4.2.11 Laboratory Analysts

Laboratory analysts are responsible for conducting analysis and performing all tasks assigned to them by the group leader or supervisor. The responsibilities of the analysts are listed below:

- Perform analyses by adhering to analytical and quality control protocols prescribed by current SOPs, this QA Manual, and project-specific plans honestly, accurately, timely, safely, and in the most cost-effective manner.
- Document standard and sample preparation, instrument calibration and maintenance, data calculations, sample matrix effects, and any observed non-conformance on worklists, benchsheets, lab notebooks and/or the Non-Conformance Database.
- Report all non-conformance situations, instrument problems, matrix problems and QC failures, which might affect the reliability of the data, to their supervisor, the Technical Director, and/or the QA Manager or member of QA staff.
- Perform 100% review of the data generated prior to entering and submitting for secondary level review.
- Suggest method improvements to their supervisor, the Technical Director, and the QA Manager. These improvements, if approved, will be incorporated. Ideas for the optimum performance of their assigned area, for example, through the proper cleaning and maintenance of the assigned instruments and equipment, are encouraged.
- Work cohesively as a team in their department to achieve the goals of accurate results, optimum turnaround time, cost effectiveness, cleanliness, complete documentation, and personal knowledge of environmental analysis.

4.2.12 Project Manager (PM)

Members of the laboratory Client Services/Project Management Group are responsible for organizing and managing client projects. Clients are assigned a project manager who serves as their primary contact at the laboratory. It is the PM's responsibility to act as the client advocate by communicating client requirements to laboratory personnel and ensuring that clients provide complete information needed by the laboratory to meet those requirements – including all verbal communications.
With the overall goal of total client satisfaction, the functions of this position are outlined below:

- Scheduling sample submissions, sample container orders and sample pick-up via the laboratory courier service.
- Confirming certification status
- Coordinating and communicating turnaround time (TAT) requirements for high priority samples/projects.

- Answering common technical questions, facilitating problem resolution and coordinating technical details with the laboratory staff. Ensuring that client specifications, when known, are met by communicating project and quality assurance requirements to the laboratory.
- Notifying the supervisors of incoming projects and sample delivery schedules.
- Accountable to clients for communicating sample status reports or results prior to receipt of the final report. Monitor the status of all data package projects in-house to ensure timely and accurate delivery of reports.
- Inform clients of data package-related problems and resolve service issues.

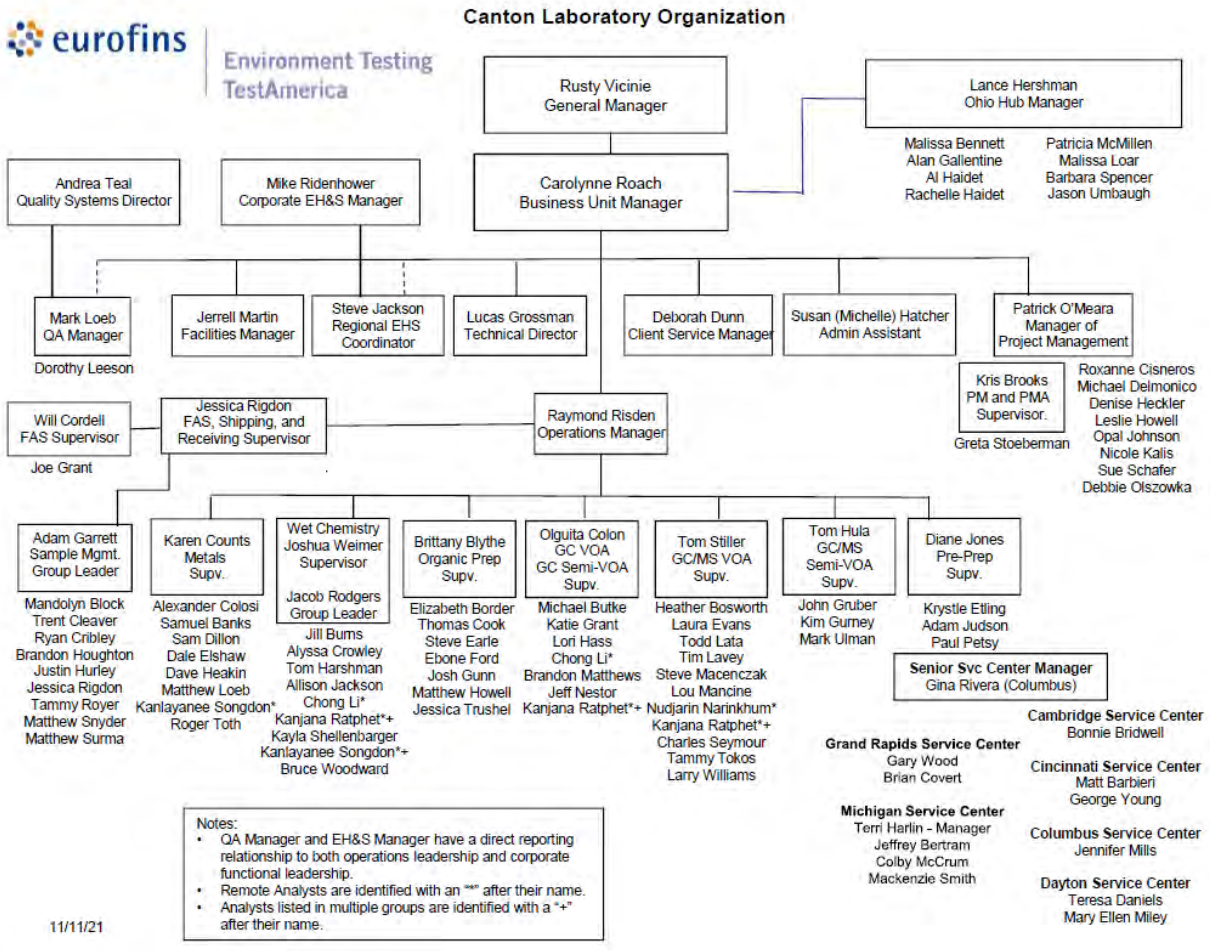
4.3 Business Continuity and Contingency Plans

Various policies and practices are in place to address continuity of business and contingency plans to ensure continued operations or minimal disruption in operations should unplanned events (natural disasters, unexpected management changes, etc.) occur. Deputies are identified for all key management personnel. Deputies would temporarily fill a role if the primary is absent for more than 15 consecutive calendar days. The deputies must meet the same qualifications as the primary person should they be required to take on the responsibilities. The QA Manager communicates to the relevant regulatory authorities when there are management or facility changes that impact the laboratory. Changes in the technical director must be communicated within a period of time and in the manner dictated by each regulatory authority.

The following table defines who assumes the responsibilities of key personnel in their absence:

Key Personnel	Deputy
Laboratory Director	Technical Director QA Manager
Operations Manager	Technical Director Laboratory Director
Quality Assurance Manager	Quality Assurance Coordinator Laboratory Director
Technical Director	Operations Manager Laboratory Group Leaders
EHS Coordinator	EHS Manager Operations Manager

Figure 4-1. Laboratory Organization Charts
Note: *Organization Charts are subject to change without notice.*



5.0 PERSONNEL

5.1 Overview

The laboratory's management believes that its highly qualified and professional staff is the single most important aspect in assuring a high level of data quality and service. The staff consists of professionals and support personnel.

All personnel must demonstrate competence in the areas where they have responsibility. Any staff that is undergoing training shall have appropriate supervision until they have demonstrated their ability to perform their job function on their own. Staff shall be qualified for their tasks based on appropriate education, training, experience and/or demonstrated skills as required.

The laboratory employs sufficient personnel with the necessary education, training, technical knowledge and experience for their assigned responsibilities.

All personnel are responsible for complying with all QA/QC requirements that pertain to the laboratory and their area of responsibility. Each staff member must have a combination of experience and education to adequately demonstrate a specific knowledge of their particular area of responsibility. Technical staff must also have a general knowledge of lab operations, test methods, QA/QC procedures and records management.

Laboratory management is responsible for formulating goals for lab staff with respect to education, training and skills and ensuring that the laboratory has a policy and procedures for identifying training needs and providing training of personnel. The training shall be relevant to the present and anticipated responsibilities of the lab staff.

The laboratory only uses personnel that are employed by or under contract to, the laboratory. Contracted personnel, when used, must meet competency standards of the laboratory and work in accordance to the laboratory's quality system.

5.2 Education and Experience Requirements for Technical Personnel

The laboratory makes every effort to hire analytical staffs that possess a college degree (AA, BA, BS) in an applied science with some chemistry in the curriculum. Exceptions can be made based upon the individual's experience and ability to learn.

Selection of qualified candidates for laboratory employment begins with documentation of minimum education, training, and experience prerequisites needed to perform the prescribed task. Minimum education and training requirements for laboratory employees are outlined in job descriptions maintained by Eurofins Environment Testing America Human Resources.

Experience and specialized training are occasionally accepted in lieu of a college degree (basic lab skills such as using a balance, colony counting, aseptic or quantitation techniques, etc., are also considered).

As a general rule for analytical staff:

Specialty	Education	Experience
Extractions, Digestions, some electrode methods (pH, DO, Redox, etc.), or Titrimetric and Gravimetric Analyses	H.S. Diploma	On the job training (OJT)
GFAA, CVAA, FLAA, Single component or short list Chromatography (e.g., Fuels, BTEX-GC, IC	A college degree in an applied science or 2 years of college and at least 1 year of college chemistry	Or 2 years prior analytical experience is required
ICP, ICPMS, Long List or complex chromatography (e.g., Pesticides, PCB, Herbicides, HPLC, etc.), GCMS	A college degree in an applied science or 2 years of college chemistry	or 5 years of prior analytical experience
Spectra Interpretation	A college degree in an applied science or 2 years of college chemistry	And 2 years relevant experience Or 5 years of prior analytical experience

Specialty	Education	Experience
Technical Managers – **General**	Bachelor's Degree in an applied science or engineering with 24 semester hours in chemistry An advanced (MS, PhD.) degree may substitute for one year of experience	And 2 years experience in environmental analysis of representative analytes for which they will oversee
Technical Managers – **Wet Chem** only (no advanced instrumentation)	Associates degree in an applied science or engineering or 2 years of college with 16 semester hours in chemistry	And 2 years relevant experience
Technical Managers - **Microbiology**	Bachelor's degree in applied science with at least 16 semester hours in general microbiology and biology An advanced (MS, PhD.) degree may substitute for one year of experience	And 2 years of relevant experience

When an analyst does not meet these requirements, they can perform a task under the direct supervision of a qualified analyst, peer reviewer or Technical Manager, and are considered an analyst in training. The person supervising an analyst in training is accountable for the quality of the analytical data and must review and approve data and associated corrective actions.

5.3 Training

The laboratory is committed to furthering the professional and technical development of employees at all levels.

Orientation to the laboratory's policies and procedures, in-house method training, and employee attendance at outside training courses and conferences all contribute toward employee proficiency. Below are examples of various areas of required employee training:

Required Training	Time Frame	Employee Type
Environmental Health & Safety	Prior to lab work	All
Ethics – New Hires	1 week of hire	All
Ethics – Comprehensive	60 days of hire	All
Data Integrity	60 days of hire	Technical and PMs
Quality Assurance	90 days of hire	All

Required Training	Time Frame	Employee Type
Ethics – Comprehensive Refresher	Annually	All
Initial Demonstration of Capability (DOC)	Prior to unsupervised method performance	Technical

The laboratory maintains records of relevant authorization/competence, education, professional qualifications, training, skills and experience of technical personnel (including contracted personnel) as well as the date that approval/authorization was given. These records are kept on file at the laboratory. Also refer to "Demonstration of Capability" in Section 19.

The training of technical staff is kept up to date by:

- Each employee must have documentation in their training file that they have read, understood and agreed to follow the most recent version of the laboratory QA Manual and SOPs in their area of responsibility. This documentation is updated as SOPs are updated.
- Documentation from any training courses or workshops on specific equipment, analytical techniques or other relevant topics.
- Documentation of proficiency (refer to Section 19).
- An Ethics Agreement signed by each staff member (renewed each year) and evidence of annual ethics training.
- A Confidentiality Agreement signed by each staff member signed at the time of employment.
- Human Resources maintains documentation and attestation forms on employment status and records; benefit programs; timekeeping/payroll; and employee conduct (e.g., ethics violations). This information is maintained in the employee's secured personnel file.

Evidence of successful training could include such items as:

- Adequate documentation of training within operational areas, including one-on-one technical training for individual technologies, and particularly for people cross-trained.
- Analysts knowledge to refer to QA Manual for quality issues.
- Analysts following SOPs, i.e., practice matches SOPs.
- Analysts regularly communicate to supervisors and QA if SOPs need revision, rather than waiting for auditors to find problems.

Further details of the laboratory's training program are described in the Laboratory Training SOP NC-QA-028.

5.4 Data Integrity and Ethics Training Program

The laboratory's Ethics and Data Integrity Program is discussed in Section 7.2. Employees are trained as to the legal and environmental repercussions that result from data misrepresentation. Key topics covered in the presentation include:

- Organizational mission and its relationship to the critical need for honesty and full disclosure in all analytical reporting.
- Ethics Policy
- How and when to report ethical/data integrity issues. Confidential reporting.

- Record keeping.
- Discussion regarding data integrity procedures.
- Specific examples of breaches of ethical behavior (e.g. peak shaving, altering data or computer clocks, improper macros, etc., accepting/offering kickbacks, illegal accounting practices, unfair competition/collusion)
- Internal monitoring. Investigations and data recalls.
- Consequences for infractions including potential for immediate termination, debarment, or criminal prosecution.
- Importance of proper written narration / data qualification by the analyst and project manager with respect to those cases where the data may still be usable but are in one sense or another partially deficient.

Additionally, a data integrity hotline (1-800-736-9407) is maintained by the NDSC

6.0 ACCOMMODATIONS AND ENVIRONMENTAL CONDITIONS

6.1 Overview

The laboratory is a 54,440 ft^2 secure laboratory facility with controlled access and designed to accommodate an efficient workflow and to provide a safe and comfortable work environment for employees. All visitors sign in and are escorted by laboratory personnel. Access is controlled by various measures.

The laboratory is equipped with structural safety features. Each employee is familiar with the location, use, and capabilities of general and specialized safety features associated with their workplace. The laboratory provides and requires the use of protective equipment including safety glasses, protective clothing, gloves, etc., OSHA and other regulatory agency guidelines regarding required amounts of bench and fume hood space, lighting, ventilation (temperature and humidity controlled), access, and safety equipment are met or exceeded.

Traffic flow through sample preparation and analysis areas is minimized to reduce the likelihood of contamination. Adequate floor space and bench top area is provided to allow unencumbered sample preparation and analysis space. Sufficient space is also provided for storage of reagents and media, glassware, and portable equipment. Ample space is also provided for refrigerated sample storage before analysis and archival storage of samples after analysis. Laboratory HVAC and deionized water systems are designed to minimize potential trace contaminants.

The laboratory is separated into specific areas for sample receiving, sample preparation, volatile organic sample analysis, non-volatile organic sample analysis, inorganic sample analysis, microbiological sample analysis, and administrative functions.

6.2 Environment

Laboratory accommodation, test areas, energy sources, and lighting are adequate to facilitate proper performance of tests. The facility is equipped with heating, ventilation, and air conditioning (HVAC) systems appropriate to the needs of environmental testing performed at this laboratory.

The environment in which these activities are undertaken does not invalidate the results or adversely affect the required accuracy of any measurements.

The laboratory provides for the effective monitoring, control and recording of environmental conditions that may affect the results of environmental tests as required by the relevant specifications, methods, and procedures. Such environmental conditions include humidity, voltage, temperature, and vibration levels in the laboratory. Spike/surge arrestors are installed to protect against power surge/sag and lightning strikes. A 30 KW natural gas-fueled backup generator is installed to provide power to the I.T. area in the event of a power failure. Additionally, this generator provides power to two walk-in sample storage coolers and several other smaller sample storage coolers. Smaller portable generators are available to provide "spot power" where needed in the event of a power failure.

When any of the method or regulatory required environmental conditions change to a point where they may adversely affect test results, analytical testing will be discontinued until the environmental conditions are returned to the required levels.

Environmental conditions of the facility housing the computer network and LIMS are regulated to protect against raw data loss.

When the laboratory performs laboratory activities at sites or facilities outside its permanent control, it shall ensure that the requirements related to facilities and environmental conditions of this document are met.

Specific requirements for facility and environmental conditions, as well as periodic monitoring of conditions, are given in the Environmental Health & Safety Manual plus each laboratory's Facility Addendum.

When the laboratory performs laboratory activities at sites or facilities outside its permanent control, it shall ensure that the requirements related to facilities and environmental conditions of this document are met.

6.3 Work Areas

There is effective separation between neighboring areas when the activities therein are incompatible with each other. Examples include:

- Microbiological culture handling and sample incubation areas
- Volatile organic chemical handling areas, including sample preparation and waste disposal, and volatile organic chemical analysis areas

Access to and use of all areas affecting the quality of analytical testing is defined and controlled by secure access to the laboratory building as described below in the Building Security section.

Adequate measures are taken to ensure good housekeeping in the laboratory and to ensure that any contamination does not adversely affect data quality. These measures include regular cleaning to control dirt and dust within the laboratory. Work areas are available to ensure an unencumbered work area. Work areas include:

- Access and entryways to the laboratory
- Sample receipt areas
- Sample storage areas
- Chemical and waste storage areas
- Data handling and storage areas
- Sample processing areas
- Sample analysis areas

Refer to the following documents and procedures for specific requirements for microbiological laboratory facility requirements

- TNI V1M5, 1.7.3.7.a

6.4 Responding to Emergencies

Employees must be aware of procedures to respond to all emergencies that might occur in the workplace. Employees must be familiar with the location and proper operation of all emergency equipment, evacuation routes and designated assembly areas for all areas where they work. Refer to the NDSC EH&S Manual Document No. CW-E-M-001. Sec. 7 and the laboratory's local EH&S addendum for complete details. These documents provide direction for situations where normal operations of the laboratory are not possible (e.g., electrical failures, heating/air conditioning failures, fire/building evacuation, computer failures, hazardous material spills, injury to employees, pandemic flu, disruption of phone service, etc.)

In the event that the building or information technology (IT) systems would be severely challenged, a designated disaster recovery team, which includes Facility Management, Maintenance, Safety, Laboratory/Executive Management, Public Relations, IT, QA and other applicable personnel depending on the scope of the disaster, would assemble at a designated area to assess the situation and formulate a plan.

6.5 Building Security

The laboratory is considered a secure facility. All outside doors (except the main lobby entrance) are locked during normal business hours to prevent unauthorized entry. Building key badges are distributed to employees as necessary.

Visitors to the laboratory sign in and out in a visitor's logbook. A visitor is defined as any person who visits the laboratory who is not an employee of the laboratory. In addition to signing into the laboratory, the Environmental Health and Safety Manual contains requirements for visitors and vendors. There are specific safety forms that must be reviewed and signed. Visitors (with the exception of company employees) are escorted by laboratory personnel at all times, or the location of the visitor is noted in the visitor's logbook.

7.0 QUALITY SYSTEM

7.1 Quality Policy Statement

The Quality Policy statement gives employees clear requirements for the production of analytical data. As an organization, all personnel are committed to high quality professional practice, testing and data, and service to our clients.

We strive to provide the highest quality data achievable by:

- Reading and understanding all of the quality documents applicable to each position and implementing the process in our work.
- Following all recordkeeping requirements; describing clearly and accurately all activities performed; recording "real time" as the task is carried out; understanding that it is never acceptable to "back date" entries and should additional information be required at a later date, the actual date and by whom the notation is made must be documented.
- Ensuring data integrity through the completeness, consistency, impartiality and accuracy of the data generated. Data is attributable, legible, contemporaneously recorded, original or a true copy, and accurate (ALCOA). This applies to manual paper documentation and electronic records.
- Providing accountability and traceability for each sample analyzed through proper sample handling, labeling, preparation, instrument calibration/qualification/validation, analysis, and reporting; establishing an audit trail (the who, what, when, and why) that identifies date, time, analyst, instrument used, instrument conditions, quality control samples (where appropriate and/or required by the method), and associated standard material.
- Emphasizing a total quality management process which provides impartiality, accuracy, and strict compliance with agency regulations and client requirements, giving the highest degree of confidence; understanding that meeting the requirements of the next employee in the work flow process is just as important as meeting the needs of the external client.
- Providing thorough documentation and explanation to qualify reported data that may not meet all requirements and specifications, but is still of use to the client; understanding this occurs only after discussion with the client on the data limitations and acceptability of this approach.
- Responding immediately to indications of questionable data, out-of-specification occurrences, equipment malfunctions, and other types of laboratory problems, with investigation and applicable corrective action; documenting these activities completely, including the reasons for the decisions made.
- Providing a work environment that ensures accessibility to all levels of management and encourages questions and expression of concerns on quality issues to management. Eurofins recognizes that the implementation of a quality assurance program requires management's commitment and support as well as the involvement of the entire staff

  Continually improve systems and manage risk to support quality improvement efforts in laboratory, administrative and managerial activities
- To comply with the ISO/IEC 17025:2005 and 2017 International Standard, the 2009 and 2016 TNI Standard and to continually improve the effectiveness of the management system.

7.2 Ethics and Data Integrity

Eurofins Environment Testing America is committed to ensuring the integrity of its data and meeting the quality needs of its clients. The laboratory operates our Ethics and Data Integrity program under the guidance of Eurofin's Key Guidance Document (KGD).The elements of our program include:

- An Ethics Policy (NDSC Document No. CW-L-P-004) and Employee Ethics Statements.

- Ethics and Compliance Officers (ECOs).
- A Training Program.
- Self-governance through disciplinary action for violations.
- A confidential mechanism for anonymously reporting alleged misconduct and a means for conducting internal investigations of all alleged misconduct. (NDSC Document No. CW-L-S-002).
- Procedures and guidance for recalling data if necessary (NDSC Document CW-Q-S-005).
- Effective external and internal monitoring system that includes procedures for internal audits (Section 15).
- Produce results, which are accurate and include QA/QC information that meets client pre-defined Data Quality Objectives (DQOs).
- Present services in a confidential, honest and forthright manner.
- Provide employees with guidelines and an understanding of the Ethical and Quality Standards of our Industry.
- Provide procedures and guidance to ensure the impartiality and confidentiality of all data and customer information.
- Operate our facilities in a manner that protects the environment and the health and safety of employees and the public.
- Obey all pertinent federal, state and local laws and regulations and encourage other members of our industry to do the same.
- Educate clients as to the extent and kinds of services available.
- Assert competency only for work for which adequate personnel and equipment are available and for which adequate preparation has been made.
- Promote the status of environmental laboratories, their employees, and the value of services rendered by them.

7.3 Quality System Documentation

The laboratory's Quality System is communicated through a variety of documents.

- Quality Assurance Manual – Each laboratory has a lab-specific QA manual.
- NDSC Official Documents – Each laboratory may use the Guidance (instructional use) documents at their discretion. Template documents are process documents that the laboratory's need to implement locally by using the document as is or as an outline to define their internal practices that meet the minimum requirements of the template. Required documents need to be implemented as is and listed in the laboratory's document control list.
- Key Guidance Documents (KGDs) - Documents compiled at the Group Service Centre (GSC) level by Functional Leaders (document owners) aimed at providing specific Eurofins groups of employees with guidelines necessary for the good conduct of their respective work.
- Work Instructions – A subset of procedural steps, tasks or forms associated with an operation of a management system (e.g., checklists, preformatted bench sheets, forms).

- Laboratory SOPs and Policies – General and Technical

7.3.1 Order of Precedence

In the event of a conflict or discrepancy between policies, the order of precedence is as follows:

- Quality Management Plan (CMP)
- NDSC Guidance Documents
- KGDs
- Laboratory Quality Assurance Manual (QA Manual)
- Laboratory SOPs and Policies
- Other (Work Instructions (WI), memos, flow charts, etc.)

NOTE: The laboratory has the responsibility and authority to operate in compliance with regulatory requirements of the jurisdiction in which the work is performed. Where the QMP conflicts with those regulatory requirements, the regulatory requirements of the jurisdiction shall hold primacy. The laboratory's QA Manual shall take precedence over the QMP in those cases.

7.4 QA/QC Objectives for the Measurement of Data

Quality Assurance (QA) is responsible for developing planned activities whose purpose is to provide assurance to all levels of management that a quality program is in place within the laboratory, and that it is functioning in an effective manner that is consistent with the requirements of NELAP and any other regulatory agencies (i.e., states) in which the laboratory maintains accreditation.

Quality Control (QC) is generally understood to be limited to the analyses of samples and to be synonymous with the term *"analytical quality control"*. QC refers to the routine application of statistically based procedures to evaluate and control the accuracy of results from analytical measurements. The QC program includes procedures for estimating and controlling precision and bias and for determining reporting limits.

Request for Proposals (RFPs) and Quality Assurance Project Plans (QAPP) provide a mechanism for the client and the laboratory to discuss the data quality objectives in order to ensure that analytical services closely correspond to client needs. In order to ensure the ability of the laboratory to meet the Data Quality Objectives (DQOs) specified in the QAPP, clients are advised to allow time for the laboratory to review the QAPP before being finalized. The client is responsible for developing the QAPP; however, the laboratory will provide support to the client for developing the sections of the QAPP that concern laboratory activities.

Historically, laboratories have described their QC objectives in terms of precision, accuracy, representativeness, comparability, completeness, selectivity and sensitivity (PARCCSS). Each laboratory SOP defines the required QC indicators.

7.4.1 Precision

The objective is to meet the performance for precision demonstrated for the methods on similar samples and to meet DQOs of the EPA and/or other regulatory programs. Precision is defined as the degree of reproducibility of measurements under a given set of analytical conditions (exclusive of field sampling variability). Precision is documented on the basis of replicate analysis, usually duplicate or matrix spike (MS) duplicate samples.

7.4.2 Accuracy

The objective is to meet the performance for accuracy demonstrated for the methods on similar samples and to meet DQOs of the EPA and/or other regulatory programs. Accuracy is defined as the degree of bias in a measurement system. Accuracy may be documented through the use of laboratory control samples (LCS) and/or MS. A statement of accuracy is expressed as an interval of acceptance recovery about the mean recovery.

7.4.3 Representativeness

The objective is to provide data which is representative of the sampled medium. Representativeness is defined as the degree to which data represent a characteristic of a population or set of samples and is a measurement of both analytical and field sampling precision. The representativeness of the analytical data is a function of the procedures used in procuring and processing the samples. The representativeness can be documented by the relative percent difference between separately procured, but otherwise identical samples or sample aliquots.

The representativeness of the data from the sampling sites depends on both the sampling procedures and the analytical procedures. Refer to laboratory SOPs for subsampling and homogenization techniques appropriate to the analytical method.

7.4.4 Comparability

The objective is to provide analytical data for which the accuracy, precision, representativeness, and reporting limit statistics are similar to these quality indicators generated by other laboratories for similar samples, and data generated by the laboratory over time.

Comparability is documented by inter-laboratory studies carried out by regulatory agencies or carried out for specific projects or contracts, by comparison of periodically generated statements of accuracy, precision, and reporting limits with those of other laboratories.

7.4.5 Completeness

The completeness objective for data is 90% (or as specified by a particular project), expressed as the ratio of the valid data to the total data over the course of the project. Data will be considered valid if they are adequate for their intended use. Data usability will be defined in a QAPP, project scope, or regulatory requirement. Data validation is the process for reviewing data to determine its usability and completeness. If the completeness objective is not met, actions will be taken internally and with the data user to improve performance. This may take the form of an audit to evaluate the methodology and procedures as possible sources for the difficulty or may result in a recommendation to use a different method.

7.4.6 Selectivity

Selectivity is defined as the capability of a test method or instrument to respond to a target substance or constituent in the presence of non-target substances. Target analytes are separated

from non-target constituents and subsequently identified/detected through one or more of the following, depending on the analytical method: extractions (separation), digestions (separation), interelement corrections (separation), use of matrix modifiers (separation), specific retention times (separation and identification), confirmations with different columns or detectors (separation and identification), specific wavelengths (identification), specific mass spectra (identification), and specific electrodes (separation and identification).

7.4.7 Sensitivity

Sensitivity refers to the amount of analyte necessary to produce a detector response that can be reliably detected (above the Method Detection Limit) or quantified (above the Reporting Limit).

7.5 Criteria for Quality Indicators

The laboratory maintains tables, housed in LIMS that summarize the precision and accuracy acceptability limits for performed analyses. This summary includes an effective date, is updated each time new limits are generated, and are managed by the laboratory's QA department. Unless otherwise noted, limits within these tables are laboratory generated. Some acceptability limits are derived from US EPA methods when they are required. Where US EPA method limits are not required, the laboratory has developed limits from evaluation of data from similar matrices. Criteria for development of control limits are contained in NC-QA-018 Statistical Evaluation of Data and Development of Control Charts and in Section 24.

7.6 Statistical Quality Control

Statistically-derived precision and accuracy limits are required by selected methods (such as SW-846) and programs [such as the Ohio Voluntary Action Plan (VAP)]. The laboratory routinely utilizes statistically-derived limits to evaluate method performance and determine when corrective action is appropriate. The analysts use the current limits entered into LIMS. The Quality Assurance department maintains an archive of all limits used within the laboratory. If a method defines the QC limits, the method limits are used.

If a method requires the generation of historical limits, the lab develops such limits from recent data in the QC database of LIMS following the guidelines described in Section 24. All calculations and limits are documented and dated when approved and effective. On occasion, a client requests contract-specified limits for a specific project.

Current QC limits are entered and maintained in the LIMS analyte database. As sample results and the related QC are entered into LIMS, the sample QC values are compared with the limits in LIMS to determine if they are within the acceptable range. The analyst then evaluates if the sample needs to be rerun or re-extracted/rerun or if a comment should be added to the report explaining the reason for the QC outlier.

7.6.1 QC Charts

The laboratory's procedures for the creation of control charts are described in laboratory SOP No. NC-QA-018, "Statistical Evaluation of Data and Development of Control Charts." Control charts are created from data stored in the LIMS. The charts are evaluated by QA and/or technical staff to determine if limits need to be updated or to assess the need for corrective actions to improve method performance.

Control charts are used to develop control limits, trouble-shoot analytical problems, and, in conjunction with the non-conformance system, to monitor for trends. Program-specific data

analysis requirements for control charts are followed as required for data generated under those programs. These additional requirements shall be documented in a QAPP.

7.7 Quality System Metrics

In addition to the QC parameters discussed above, the entire Quality System is evaluated on a monthly basis through the use of specific metrics (refer to Section 16). These metrics are used to drive continuous improvement in the laboratory's Quality System.

8.0 DOCUMENT CONTROL

8.1 Overview

The QA Department is responsible for the control of documents used in the laboratory to ensure that approved, up-to-date documents are in circulation and out-of-date (obsolete) documents are archived or destroyed. The following documents, at a minimum, must be controlled:

- Laboratory Quality Assurance (QA) Manual
- Laboratory Standard Operating Procedures (SOP)
- Laboratory Policies
- Work Instructions and Forms
- NDSC Documents[1]
- KGDs[1]

[1]Includes locally implemented documents that are document controlled within the laboratory's document control system. The NDSC and/or KGD documents are only considered controlled when they are read on the intranet site. Printed copies are considered uncontrolled unless the laboratory physically distributes them as controlled documents. A detailed description of the procedure for issuing, authorizing, controlling, distributing, and archiving NDSC Official Documents is found in Document No. CW-Q-S-001, NDSC Document Control and Archiving. The laboratory's internal document control procedure is defined in SOP NC-QA-030 Document Control and in SOP NC-QA-027 Preparation and Management of SOPs.

The laboratory QA Department also maintains access to various references and document sources integral to the operation of the laboratory. This includes reference methods and regulations. Instrument manuals (hard or electronic copies) are also maintained by the laboratory.

The laboratory maintains control of records for raw analytical data and supporting records such as audit reports and responses, logbooks, standard logs, training files, MDL studies, Proficiency Testing (PT) studies, certifications and related correspondence, and corrective action reports. Raw analytical data consists of bound logbooks, instrument printouts, any other notes, magnetic media, electronic data, and final reports.

8.2 Document Approval and Issue

The pertinent elements of the document control system include a unique document title and number, pagination, the total number of pages of the item or an 'end of document' page, the

effective date, revision number, and the laboratory's name. The QA personnel are responsible for the maintenance of this system.

Controlled documents are authorized by the QA Department. In order to develop a new document, a responsible manager submits an electronic draft to the QA Department for suggestions and approval before use. Upon approval, QA personnel add the identifying version information to the document and retains that document as the official document on file. That document is then provided to all applicable operational units. Controlled documents are identified as such and records of their distribution are kept by the QA Department. Document control may be achieved by either electronic or hardcopy distribution.

The QA Department maintains a list of the official versions of controlled documents.

Quality System Policies and Procedures will be reviewed at a minimum of every two years and revised as appropriate. State or client programs may require a different review schedule. Changes to documents occur when a procedural change warrants.

8.3 Procedures for Document Control Policy

For changes to the QA Manual, refer to SOPs NC-QA-027 and CW-Q-S-001. Uncontrolled copies must not be used within the laboratory. Previous revisions and back-up data are stored by the QA department. Electronic copies are stored on the Public server in the SOP folder for the applicable revision.

For changes to SOPs, refer to SOP No. CW-Q-S-002, Writing a Standard Operating Procedure SOP and SOP NC-QA-027 Preparation and Management of Standard Operating Procedures.. The SOPs identified above also define the process of changes to SOPs.

Forms, worksheets, work instructions and information are organized by the QA department in accordance with the procedures specified in laboratory SOP NC-QA-030.

8.4 Obsolete Documents

All invalid or obsolete documents are removed, or otherwise prevented from unintended use. The laboratory has specific procedures as described above to accomplish this. In general, obsolete documents are collected from employees according to distribution lists and are marked obsolete on the cover or destroyed. At least one copy of the obsolete document is archived according to SOP NC-QA-030.

9.0 SERVICE TO THE CLIENT

9.1 Overview

The laboratory has established procedures for the review of work requests and contracts, oral or written. The procedures include evaluation of the laboratory's capability and resources to meet the contract's requirements within the requested time period. All requirements, including the methods to be used, must be adequately defined, documented and understood. For many environmental sampling and analysis programs, testing design is site or program specific and does not necessarily fit into a standard laboratory service or product. It is the laboratory's intent to provide both standard and customized environmental laboratory services to our clients.

A thorough review of technical and QC requirements contained in contracts is performed to ensure project success. The appropriateness of requested methods, and the lab's capability to perform them must be established. Projects, proposals, and contracts are reviewed for adequately defined requirements and the laboratory's capability to meet those requirements. Alternate test methods that are capable of meeting the clients' requirements may be proposed by the lab. A review of the lab's capability to analyze non-routine analytes is also part of this review process.

All projects, proposals and contracts are reviewed for the client's requirements in terms of compound lists, test methodology requested, sensitivity (detection and reporting levels), accuracy, and precision requirements (% Recovery and RPD). The reviewer ensures that the laboratory's test methods are suitable to achieve these requirements and that the laboratory holds the appropriate certifications and approvals to perform the work. The laboratory and any potential subcontract laboratories must be certified, as required, for all proposed tests.

Electronic or hard copy deliverable requirements are evaluated against the laboratory's capacity for production of the documentation.

If the laboratory cannot provide all services but intends to subcontract such services, whether to another Eurofins facility on the same LIMS or to an outside firm, this will be documented and discussed with the client prior to contract approval. (Refer to Section 10 for Subcontracting Procedures.)

The laboratory informs the client of the results of the review if it indicates any potential conflict, deficiency, lack of accreditation, or inability of the lab to complete the work satisfactorily. Any discrepancy between the client's requirements and the laboratory's capability to meet those requirements is resolved in writing before acceptance of the contract. It is necessary that the contract be acceptable to both the laboratory and the client. Amendments initiated by the client and/or Eurofins TestAmerica Canton are documented in writing.

All contracts, QAPPs, Sampling and Analysis Plans (SAPs), contract amendments, and documented communications become part of the project record.

The same contract review process used for the initial review is repeated when there are amendments to the original contract by the client, and the participating personnel are informed of the changes

9.2 Review Sequence and Key Personnel

Appropriate personnel will review the work request at each stage of evaluation.

For routine projects and other simple tasks, a review by the Project Manager (PM) is considered adequate. The PM confirms that the laboratory has any required certifications, that it can meet the clients' data quality and reporting requirements and that the lab has the capacity to meet the clients turn around needs. It is recommended that, where there is a sales person assigned to the account, an attempt should be made to contact that sales person to inform them of the incoming samples.

For new, complex or large projects, the proposed contract is given to the Client Relationship Manager or Proposal Team, who will decide which lab will receive the work based on the scope

of work and other requirements, including certification, testing methodology, and available capacity to perform the work. This review encompasses all facets of the operation. The scope of work is distributed to the appropriate personnel, as needed based on scope of contract, to evaluate all of the requirements shown above (not necessarily in the order below)

- Contract Administrator
- Laboratory Project Manager
- Laboratory Directors and/or Technical Managers
- Account Executives
- Quality Managers Laboratory Environmental Health and Safety Managers/Directors
- The Laboratory Director reviews the formal laboratory quote and makes final acceptance for their facility.

The Sales Director, Contract Administrator, Account Executive or Proposal Coordinator then submits the final proposal to the client.

In the event that one of the above personnel is not available to review the contract, his or her back-up will fulfill the review requirements.

9.3 Balancing Laboratory Capacity and Workload

Evaluating laboratory capacity to perform specific projects is the responsibility of the Laboratory Directors and Managers, and the Client Services director and manager. Many analysts are cross-trained to perform a variety of tests, and there is redundant equipment available in case of malfunctions. This minimizes the need to evaluate small and medium size projects against capacity available to complete them. Large and complex projects are reviewed against capacity estimates before bids are submitted to ensure that the client's analysis schedule is met. Regularly scheduled meetings are held between laboratory management, PMs, Client Services and QA personnel to review progress with current projects, as well as special requirements of new work scheduled for the laboratory. Laboratory capacity and backlog is tracked on a continuous basis using information from the Laboratory Sample Information System (LIMS) including turnaround time, and work in-house.

9.4 **Documentation**

The Contracts Department maintains copies of all signed contracts. Laboratory Project Manager Assistants and/or PMs maintain electronic copies of signed contracts as needed.

Appropriate records are maintained for every contract or work request. All stages of the contract review process are documented and include records of any significant changes. Records are maintained electronically.

The contract will be distributed to and maintained by the appropriate sales/marketing personnel and the Account Executive. A copy of the contract and formal quote will be filed with the laboratory PM and the Laboratory Director.

Records are maintained of pertinent discussions with a client relating to the client's requirements or the results of the work during the period of execution of the contract. The PM keeps a phone log of conversations with the client.

9.4.1 Project-Specific Quality Planning

Communication of contract specific technical and QC criteria is an essential activity in ensuring the success of site specific testing programs. To achieve this goal, a PM is assigned to each client. It is the PM's responsibility to ensure that project-specific technical and QC requirements are effectively evaluated and communicated to the laboratory personnel before and during the project. QA department involvement may be needed to assist in the evaluation of custom QC requirements.

PM's are the primary client contact and they ensure resources are available to meet project requirements. They coordinate opportunities and work with laboratory management and supervisory staff to ensure available resources are sufficient to perform work for the client's project. Prior to work on a new project, the dissemination of project information and/or project opening meetings may occur to discuss schedules and unique aspects of the project. Items to be discussed may include the project technical profile, turnaround times, holding times, methods, analyte lists, reporting limits, deliverables, sample hazards, or other special requirements. The PM introduces new project information to maximize production and client satisfaction, while maintaining quality. Project notes may be associated with each sample batch as a reminder upon sample receipt and analytical processing.

Any change that may occur within an active project is agreed upon between the client/regulatory agency and the PM/laboratory. These changes (e.g., use of a non-standard method or modification of a method) and approvals must be documented prior to implementation. Documentation pertains to any document (e.g., letter, e-mail, variance, contract addendum), which has been signed by both parties.

Such changes are also communicated to the laboratory during operations meetings, via emails, or directly. The laboratory staff is then introduced to the modified requirements via the PM or the individual laboratory Technical Director. After the modification is implemented into the laboratory process, documentation of the modification is made in the case narrative of the data report(s).

The laboratory strongly encourages client visits to the laboratory and for formal/informal information sharing session with employees in order to effectively communicate ongoing client needs as well as project specific details for customized testing programs.

9.5 Special Services

The laboratory cooperates with clients and their representatives to monitor the laboratory's performance in relation to work performed for the client. It is the laboratory's goal to meet all client requirements in addition to statutory and regulatory requirements. The laboratory has procedures to ensure confidentiality to clients (Section 17 and 25).

The laboratory's standard procedures for reporting data are described in Section 25. Special services are also available and provided upon request. These services include:

- Reasonable access for our clients or their representatives to the relevant areas of the laboratory for the witnessing of tests performed for the client.

- Assisting client-specified third party data validators as specified in the client's contract.
- Supplemental information pertaining to the analysis of their samples. Note: An additional charge may apply for additional data/information that was not requested prior to the time of sample analysis or previously agreed upon.

When the client requests a statement of conformity to a specification or standard based on the analysis performed by the laboratory (e.g., pass/fail, in-tolerance/out-of-tolerance), the decision rule shall be clearly defined. Unless inherent in the requested specification or standard, the decision rule selected shall be communicated to the client. Associated reporting requirements are addressed in Section 25.2.18.

9.6 Client Communication

PMs are the primary communication link to the clients. They shall inform their clients of any delays in project completion as well as any non-conformances in either sample receipt or sample analysis. Project management will maintain ongoing client communication throughout the entire client project.

The Technical Director is available to discuss any technical questions or concerns that the client may have.

9.7 Reporting

The laboratory works with our clients to produce any special communication reports required by the contract.

9.8 Client Feedback and Surveys

The laboratory assesses both positive and negative client feedback. The results are used to improve overall laboratory quality and client service. Eurofins Sales and Marketing teams periodically develop lab and client specific surveys to assess client satisfaction. Client satisfaction surveys are sent routinely.

When a complaint is received, we determine, to the best of our ability, the extent of the issue and what data is in question. The person receiving the complaint documents this information and promptly forwards it to the appropriate management personnel where the work in question was performed. If a data reporting error is discovered, the final report and/or data must be regenerated with the correct value(s).

The laboratory staff is responsible for entering client concerns into iCAT. In some cases, a Corrective Action Report is initiated to address and document the situation. While an individual issue may not warrant a formal investigation, QA monitors these issues for potential trends and will issue a CAR if a trend is evident.

10.0SUBCONTRACTING OF TESTS

10.1 Overview

For the purpose of this quality manual, the phrase subcontract laboratory refers to a laboratory external to the Eurofins TestAmerica Canton laboratories. The phrase "work sharing" refers to internal transfers of samples between the Eurofins TestAmerica Canton laboratories. The term outsourcing refers to the act of subcontracting tests.

When contracting with our clients, the laboratory makes commitments regarding the services to be performed and the data quality for the results to be generated. When the need arises to outsource testing for our clients because project scope, changes in laboratory capabilities, capacity, or unforeseen circumstances, we must be assured that the subcontractors or work sharing laboratories understand the requirements and will meet the same commitments we have made to the client. Refer to the NDSC Documents on Subcontracting Procedures (CW-L-S-004). When outsourcing analytical services, the laboratory will assure, to the extent necessary, that the subcontract or work sharing laboratory maintains a program consistent with the requirements of this document, the requirements specified in TNI/ISO 17025 and/or the client's Quality Assurance Project Plan (QAPP). All QC guidelines specific to the client's analytical program are transmitted to the subcontractor and agreed upon before sending the samples to the subcontract facility. Additionally, work requiring accreditation will be placed with an appropriately accredited laboratory. The laboratory performing the subcontracted work will be identified in the final report, as will non-TNI accredited work where required.

PMs or other responsible Client Service members, for the Export Lab (i.e., the Eurofins TestAmerica laboratory that transfers samples to another laboratory) are responsible for obtaining client approval prior to subcontracting any samples. The laboratory will advise the client of a subcontract arrangement in writing and when possible approval from the client shall be obtained and retained in the project folder. Standard Eurofins TestAmerica Terms & Conditions include the flexibility to subcontract samples within the Eurofins TestAmerica laboratories. Therefore, additional advance notification to clients for intra-laboratory subcontracting is not necessary unless specifically required by a client contract.

Note: In addition to the client, some regulating agencies (e.g., USDA) or contracts require notification prior to placing such work.

10.2 Qualifying and Monitoring Subcontractors

Whenever a PM becomes aware of a client requirement or laboratory need where samples must be outsourced to another laboratory, the other laboratory(s) shall be selected based on the following:

- Subcontractors specified by the client - In these circumstances, the client assumes responsibility for the quality of the data generated from the use of a subcontractor.
- Subcontractors reviewed by Eurofins TestAmerica – Firms which have been reviewed by the company and are known to meet standards for accreditations (e.g., State, TNI and DoD/DOE); technical specifications; legal and financial information.

A listing of vendors is available on the Eurofins TestAmerica intranet site.

All Eurofins TestAmerica laboratories are pre-qualified for work sharing provided they hold the appropriate accreditations and can adhere to the project/program requirements. Client approval is not necessary unless specifically required by the contract. In these cases, the client must provide acknowledgement that the samples can be sent to that facility (an e-mail is sufficient documentation or if acknowledgement is verbal, the date, time, and name of person providing acknowledgement must be documented). The originating laboratory is responsible for communicating all technical, quality, and deliverable requirements as well as other contract needs.

10.2.1 When the potential sub-contract laboratory has not been previously approved, Account Executives or PMs may nominate a laboratory as a subcontractor based on need. The decision to nominate a laboratory must be approved by the Client Relations Manager (CRM) or Laboratory Director. The CRM or Laboratory Director requests that the QA Manager or PM begin the process of approving the subcontract laboratory. Refer to the NDSC Document No. CW-L-S-004, Subcontracting Procedures.

Once the appropriate accreditation and legal information is received by the laboratory, it is evaluated for acceptability and forwarded to the NDSC Quality Information Manager (QIM) for review. After the NDSC QIM reviews the documents for completeness, the information is forwarded to the Finance Department for formal signature and contracting with the laboratory. The approved vendor will be added to the approved subcontractor list on the intranet site, and the finance group is concurrently notified.

The client will assume responsibility for the quality of the data generated from the use of a subcontractor they have requested the lab to use. The qualified subcontractors on the intranet site are known to meet minimal standards. Eurofins TestAmerica does not certify laboratories. The subcontractors on our approved list can only be recommended to the extent that we would use them.

10.3 Oversight and Reporting

The status and performance of qualified subcontractors will be monitored by NDSC and includes an annual review process (see Subcontracting SOP CW-L-S-004). Any problems identified will be brought to the attention of Eurofins TestAmerica's Corporate Finance, Legal and Corporate Quality personnel.

- Complaints shall be investigated. Documentation of the complaint, investigation, and corrective action will be maintained in the subcontractor's file on the intranet site. Complaints are posted using the Vendor Performance Report.
- Information shall be updated on the intranet when new information is received from the subcontracted laboratories.
- Subcontractors in good standing will be retained on the intranet listing. Client Service personnel will notify all Eurofins TestAmerica laboratories, NDSC, and Corporate Contracts if any laboratory requires removal from the intranet site. This notification will be posted on the intranet site and e-mailed to all Client Service Personnel, Laboratory Directors, QA Managers, and Sales Personnel.

Prior to initially sending samples to the subcontracted laboratory, the PM confirms their certification status to determine if it's current and scope-inclusive. The information is documented within the project records.

The laboratory's certifications can be viewed on the company's website.

10.3.1 All subcontracted samples must be accompanied by a Eurofins TestAmerica Chain of Custody (COC). A copy of the original COC sent by the client must be available in LIMS for all samples workshared within Eurofins TestAmerica. Client COCs are only forwarded to external subcontractors when samples are shipped directly from the

project site to the subcontractor lab. Under routine circumstances, client COCs are not provided to external subcontractors.

Through communication with the subcontracted laboratory, the PM monitors the status of the subcontracted analyses, facilitates successful execution of the work, and ensures the timeliness and completeness of the analytical report.

Non-TNI accredited work must be identified in the subcontractor's report as appropriate. If TNI accreditation is not required, the report does not need to include this information.

Reports submitted from subcontractor laboratories are not altered and are included in their original form in the final project report. This clearly identifies the data as being produced by a subcontractor facility. If subcontract laboratory data is incorporated into the laboratory's EDD (i.e., imported), the report must explicitly indicate which lab produced the data for which methods and samples.

Note: The results submitted by a Eurofins TestAmerica work sharing laboratory may be transferred electronically and the results reported by the Eurofins TestAmerica work sharing lab are identified on the final report. The report must explicitly indicate which lab produced the data for which methods and samples. The final report must include a copy of the completed COC for all work sharing reports.

10.4 Contingency Planning

The full qualification of a subcontractor may be waived to meet emergency needs. This decision and justification must be documented in the project files, and the 'Purchase Order Terms And Conditions For Subcontracted Laboratory Services' must be sent with the samples and COC.

In the event this provision is utilized, the laboratory (e.g., PM) will be required to verify and document the applicable accreditations of the subcontractor. All other quality and accreditation requirements will still be applicable, but the subcontractor need not have signed a subcontract agreement with Eurofins TestAmerica at this time.

The use of any emergency subcontractor will require the PM to complete a New Vendor Add Form in order to process payment to the vendor and add them to LIMS. This form requires the user to define the subcontractor's category/s of testing and the reason for testing.

10.5 Use of NELAP and A2LA Logo

It is not laboratory policy to use these logos on any company letterhead, including analytical reports.

11.0 PURCHASING SERVICES AND SUPPLIES

11.1 Overview

Evaluation and selection of suppliers and vendors is performed, in part, on the basis of the quality of their products, their ability to meet the demand for their products on a continuous and short term basis, the overall quality of their services, their past history, and competitive pricing. This is achieved through evaluation of objective evidence of quality furnished by the supplier, which can include certificates of analysis, recommendations, and proof of historical compliance

with similar programs for other clients. To ensure that quality critical consumables and equipment conform to specified requirements, which may affect quality, all purchases from specific vendors are approved by a member of the supervisory or management staff.
Contracts will be signed in accordance with NDSC Document No CW-F-WI-036 Eurofins TestAmerica: Spending and Contract Authority (S&CA) Limits and Authorization Matrix for US Environment.. Request for Proposals (RFP's) will be issued where more information is required from the potential vendors than just price. RFP's allow the laboratory to determine if a vendor is capable of meeting requirements such as supplying all of the laboratory facilities, meeting required quality standards and adhering to necessary ethical and environmental standards. The RFP process also allows potential vendors to outline any additional capabilities they may offer.

11.2 Glassware

Glassware used for volumetric measurements must be Class A or verified for accuracy according to laboratory procedure. Pyrex (or equivalent) glass should be used where possible. For safety purposes, thick-wall glassware should be used where available.

11.3 Reagents, Standards & Supplies

Purchasing guidelines for equipment, consumables, and reagents must meet the requirements of the specific method and testing procedures for which they are being purchased. Solvents and acids are pre-tested in accordance with NDSC Document No. CA-Q-S-001 Solvent & Acid Lot Testing & Approval, Approval information for the solvents and acids tested under Document No. CA-Q-S-001 is stored on the SharePoint, under Solvent Approvals. A master list of all tested materials, as well as the certificates of analysis for the materials, is stored in the same location.

11.3.1 Purchasing

Chemical reagents, solvents, glassware, and general supplies are ordered as needed to maintain sufficient quantities on hand. Materials used in the analytical process must be of a known quality. The wide variety of materials and reagents available makes it advisable to specify recommendations for the name, brand, and grade of materials to be used in any determination. This information is contained in the method SOP. The analyst may check the item out of the on-site consignment system that contains items approved for laboratory use. If the item is not in consignment, the analyst must provide the master item number, item description, package size, catalogue page number, and the quantity needed. If an item being ordered is not the exact item requested, approval must be obtained from the Operations Manager or Group Leader prior to placing the order. The purchasing manager places the order.

11.3.2 Receiving

It is the responsibility of the warehouse manager to receive the shipment. It is the responsibility of the analyst who ordered the materials to document the date materials were received. Once the ordered reagents or materials are received, the analyst compares the information on the label or packaging to the original order to ensure that the purchase meets the quality level specified. This is documented through the addition of the received date and initials to the information present on the daily order log.

The purchasing manager verifies the lot numbers of received solvents and acids against the pre-approval lists. If a received material is listed as unapproved, or is not listed, it is

sequestered and returned to the vendor. Alternatively, the laboratory may test the material for the intended use, and if it is acceptable, document the approval on the approval list. Records of any testing performed locally are maintained on the shared "public" folder on the computer network.

Materials may not be released for use in the laboratory until they have been inspected, verified as suitable for use, and the inspection/verification has been documented.

Safety Data Sheets (SDSs) are available online through the Company's intranet website. Anyone may review these for relevant information on the safe handling and emergency precautions of on-site chemicals.

11.3.3 Specifications

Methods used in the laboratory specify the grade of reagent that must be used in the procedure. If the quality of the reagent is not specified, analytical reagent grade will be used. It is the responsibility of the analyst to check the procedure carefully for the suitability of grade of reagent.

Chemicals must not be used past the manufacturer's expiration date and must not be used past the expiration time noted in a method SOP. If expiration dates are not provided, the laboratory may contact the manufacturer to determine an expiration date.

The laboratory assumes a five year expiration date on inorganic dry chemicals and solvents unless noted otherwise by the manufacturer or by the reference source method. Chemicals/solvents should not be used past the manufacturer's or SOP expiration date unless verified as outlined below

- An expiration date **cannot** be extended if the dry chemical/solvent is discolored or appears otherwise physically degraded. In this case, the dry chemical/solvent must be discarded.
- Expiration dates can be extended if the dry chemical/solvent is found to be satisfactory based on acceptable performance of quality control samples (Continuing Calibration Verification (CCV), Blanks, Laboratory Control Sample (LCS), etc.).
- If the dry chemical/solvent is used for the preparation of standards, the expiration dates can be extended 6 months if the dry chemical/solvent is compared to an unexpired independent source in performing the method and the performance of the dry chemical/solvent is found to be satisfactory. The comparison must show that the dry chemical/solvent meets CCV limits. The comparison studies are maintained on-file and available for review in the reagent module of the LIMS.

  Note: Some programs (such as OVAP) do not allow the use of verified standards and/or reagents.

Wherever possible, standards must be traceable to national or international standards of measurement or to national or international reference materials. Records to that effect are available to the user.

Compressed gases in use are checked for pressure and secure positioning daily. To prevent a tank from going to dryness, or introducing potential impurities, the pressure should be closely watched as it decreases to approximately 15% of the original reading, at which point it should be replaced. For example, a standard sized laboratory gas cylinder containing 3,000 psig of gas should be replaced when it drops to approximately 500 psig. The quality of the gases must meet method or manufacturer specification or be of a grade that does not cause any analytical interference.

Water used in the preparation of samples, standards or reagents must have a specific conductivity of less than 1- µmho/cm (or specific resistivity of greater than 1.0 megohm-cm) at 25ºC. The specific conductivity is checked and recorded daily. If the water's specific conductivity is greater than the specified limit, the Facility Manager and Technical Director must be notified immediately in order to notify all departments, decide on cessation (based on intended use) of activities, and make arrangements for correction.

The laboratory may purchase reagent grade (or other similar quality) water for use in the laboratory. This water must be certified clean by the supplier for all target analytes or otherwise verified by the laboratory prior to use. This verification is documented.

Standard lots are verified before first time use if the laboratory switches manufacturers or has historically had a problem with the type of standard.

Purchased bottleware used for sampling must be certified clean and the certificates must be maintained. If uncertified sampling bottleware is purchased, all lots must be verified clean prior to use. This verification must be maintained.

Records of manufacturer's certification and traceability statements are maintained electronically in the LIMS or in binders in each laboratory section. These records include date of receipt, lot number (when applicable), and expiration date (when applicable). Incorporation of the item into the record indicates that the analyst has compared the new certificate with the previous one for the same purpose and that no difference is noted, unless approved and so documented by the Technical Director or QA Manager.

11.3.4 Storage

Reagent and chemical storage is important from the aspects of both integrity and safety. Light-sensitive reagents may be stored in brown-glass containers. Storage conditions are per the NDSC Environmental Health & Safety Manual Document, No. CW-E-M-001), the local laboratory EH&S manual addendum, and method SOPs or manufacturer instructions.

11.4 Purchase of Equipment / Instruments / Software

When a new piece of equipment is needed, either for additional capacity or for replacing inoperable equipment, the analyst or supervisor makes a supply request to the Technical Director and/or the Laboratory Director. If they agree with the request, the procedures outlined in NDSC Document No. CA-T-P-001, Qualified Products List, are followed. A decision is made as to which piece of equipment can best satisfy the requirements. The appropriate written requests are completed and purchasing places the order.

Upon receipt of a new or used piece of equipment, a unique identification name is assigned and added to the equipment list in the LIMS. IT must also be notified so that they can synchronize the instrument for back-ups. Its capability is assessed to determine if it is adequate or not for the specific application. For instruments, a calibration curve is generated, followed by MDLs, Demonstration of Capabilities (DOCs), and other relevant criteria (refer to Section 20). This can be tracked by use of the New Equipment Tracking Form (WI-NC-091). For software, its operation must be deemed reliable and evidence of instrument verification must be retained by the IT Department or QA Department. Software certificates supplied by the vendors are filed with the LIMS Administrator. The manufacturer's operation manual is retained at the bench.

11.5 Services

Service to analytical instruments (except analytical balances) is performed on an as needed basis. Routine preventative maintenance is discussed in Section 22. The need for service is determined by analysts, Group Leaders, and/or the Technical Director. The service providers that perform the services are approved by the Group Leaders or Operations Manager.

Analytical balances are serviced and calibrated annually in accordance with SOP NC-QA-015. The calibration and maintenance services are performed on-site, and the balances are returned to use immediately following successful calibration. Calibration certificates are filed for reference. If the calibration was unsuccessful, the balance is immediately removed from service and segregated pending either further maintenance or disposal.

Calibration services for support equipment such as thermometers, weight sets, autopipettors, etc., are obtained from vendors with current and valid ISO 17025 accreditation for calibration of the specific piece of equipment. Prior to utilizing the vendor's services, the vendor's accreditation status is verified. Once the equipment has been calibrated, the calibration certificates are reviewed by the QA department, and documentation of the review is filed with the calibration certificates. The equipment is then returned to service within the laboratory

11.6 Suppliers

The laboratory selects vendors through a competitive proposal / bid process, strategic business alliances or negotiated vendor partnerships (contracts). The level of control used in the selection process is dependent on the anticipated spending amount and the potential impact on the laboratory's business. Vendors that provide test and measuring equipment, solvents, standards, certified containers, instrument related service contracts or subcontract laboratory services shall be subject to more rigorous controls than vendors that provide off-the-shelf items of defined quality that meet the end use requirements. The purchasing system includes all suppliers/vendors that have been approved for use.

Evaluation of suppliers is accomplished by ensuring the supplier ships the product or material ordered and that the material is of the appropriate quality. This is documented by signing off on packing slips or other supply receipt documents. The purchasing documents contain the data that adequately describe the services and supplies ordered.

Any issues of vendor performance are to be reported immediately by the laboratory staff to the Purchasing Group by completing a Vendor Performance Report.

The Purchasing Group will work through the appropriate channels to gather the information required to clearly identify the problem and will contact the vendor to report the problem and to make any necessary arrangements for exchange, return authorization, credit, etc.

Suppliers are subject to re-evaluation, as deemed appropriate, through the use of Vendor Performance Reports used to summarize and review to determine corrective action necessary, or service improvements required by vendors

The laboratory has access to a listing of all approved suppliers of critical consumables, supplies and services. This information is provided through the purchasing system.

11.6.1 New Vendor Procedure

Laboratory employees who wish to request the addition of a new vendor must complete a Vendor Add Request Form.

New vendors are evaluated based upon criteria appropriate to the products or services provided as well as their ability to provide those products and services at a competitive cost. Vendors are also evaluated to determine if there are ethical reasons or potential conflicts of interest with laboratory employees that would make it prohibitive to do business with them as well as their financial stability. The QA Department and/or the Technical Services Director are consulted with vendor and product selection that have an impact on quality.

12.0 COMPLAINTS

12.1 Overview

The laboratory considers an effective client complaint handling processes to be of significant business and strategic value. Listening to and documenting client concerns captures client knowledge that enables our operations to continually improve processes and client satisfaction. An effective client complaint handling process also provides assurance to the data user that the laboratory will stand behind its data, service obligations and products.

A client complaint is any expression of dissatisfaction with any aspect of our business services (e.g., communications, responsiveness, data, reports, invoicing and other functions) expressed by any party, whether received verbally or in written form. Client inquiries, complaints or noted discrepancies are documented, communicated to management, and addressed promptly and thoroughly.

The laboratory has procedures for addressing both external and internal complaints with the goal of providing satisfactory resolution to complaints in a timely and professional manner.

The nature of the complaint is identified, documented and investigated, and an appropriate action is determined and taken. In cases where a client complaint indicates that an established policy or procedure was not followed, the QA Department must evaluate whether a special audit must be conducted to assist in resolving the issue. A written confirmation or letter to the client, outlining the issue and response taken is recommended as part of the overall action taken.

The process of complaint resolution and documentation utilizes the procedures outlined in Section 14 (Corrective Actions) and is documented following SOP NC-QA-029 Nonconformance

and Corrective Action System. A copy of this procedure will be made available to any interested party on request.

12.2 External Complaints

An employee that receives a complaint initiates the complaint resolution process by first documenting the complaint according to SOP NC-QA-029.

Complaints fall into two categories: correctable and non-correctable. An example of a correctable complaint would be one where a report re-issue would resolve the complaint. An example of a non-correctable complaint would be one where a client complains that their data was repeatedly late. Non-correctable complaints should be reviewed for preventive action measures to reduce the likelihood of future occurrence and mitigation of client impact.

The general steps in the complaint handling process are:

- Receiving and documenting complaints
- Acknowledging receipt of complaint, whenever possible
- Complaint investigation and service recovery
- Process improvement

The laboratory shall inform the initiator of the complaint of the results of the investigation and the corrective action taken, if any.

12.3 Internal Complaints

Internal complaints include, but are not limited to: errors and non-conformances, training issues, internal audit findings, and deviations from methods. Corrective actions may be initiated by any staff member who observes a nonconformance and shall follow the procedures outlined in Section 14. In addition, Executive Management, Sales and Marketing and IT may initiate a complaint by contacting the laboratory or through the corrective action system described in Section 14.

12.4 Management Review

The number and nature of client complaints is reported by the QA Manager to the Laboratory Director and Quality Director in the QA Monthly report. Monitoring and addressing the overall level and nature of client complaints and the effectiveness of the solutions is part of the Annual Management Systems Review (Section 18).

13 CONTROL OF NON-CONFORMING WORK

13.1 Overview

When data discrepancies are discovered or deviations and departures from laboratory SOPs, policies and/or client requests have occurred, corrective action is taken immediately. First, the laboratory evaluates the significance of the nonconforming work. Then, a corrective action plan is initiated based on the outcome of the evaluation. If it is determined that the nonconforming work is an isolated incident, the plan could be as simple as adding a qualifier to the final results and/or making a notation in the case narrative. If it is determined that the nonconforming work is a systematic or improper practices issue, the corrective action plan could include a more in depth

investigation and a possible suspension of an analytical method. In all cases, the actions taken are documented using the laboratory's corrective action system (refer to Section 14).

Due to the frequently unique nature of environmental samples, sometimes departures from documented policies and procedures are needed. When an analyst encounters such a situation, the problem is presented to the supervisor for resolution. The supervisor may elect to discuss it with the Technical Director or have a representative contact the client to decide on a logical course of action. Once an approach is agreed upon, the analyst documents it using the laboratories corrective action system described in Section 14. This information can then be supplied to the client in the form of a footnote or a case narrative with the report.

Project Management may encounter situations where a client may request that a special procedure be applied to a sample that is not standard lab practice. Based on a technical evaluation, the lab may accept or opt to reject the request based on technical or ethical merit. An example might be the need to report a compound that the lab does not normally report. The lab would not have validated the method for this compound following the procedures in Section 19. The client may request that the compound be reported based only on the calibration. Such a request would need to be approved by the Operations Manager and QA Manager, documented and included in the project folder. Deviations **must** also be noted on the final report with a statement that the compound is not reported in compliance with TNI (or the analytical method) requirements and the reason. Data being reported to a non-TNI state would need to note the change made to how the method is normally run.

13.2 Responsibilities and Authorities

Under certain circumstances, the Laboratory Director, a Technical Director, or a member of the QA team may authorize departures from documented procedures or policies. The departures may be a result of procedural changes due to the nature of the sample; a one-time procedure for a client; QC failures with insufficient sample to reanalyze, etc. In most cases, the client will be informed of the departure prior to the reporting of the data. Any departures must be well documented using the laboratory's corrective action procedures. This information may also be documented in logbooks and/or data review checklists as appropriate. Any impacted data must be referenced in a case narrative and/or flagged with an appropriate data qualifier.

Any misrepresentation or possible misrepresentation of analytical data discovered by any laboratory staff member must be reported to facility Senior Management within 24-hours. The Senior Management staff is comprised of the Laboratory Director, the QA Manager, and the Technical Director. The reporting of issues involving alleged violations of the company's Data Integrity or Manual Integration procedures must be conveyed to an ECO (e.g., the VP-QA/EHS) and the laboratory's Quality Director within 24 hours of discovery.

Whether an inaccurate result was reported due to calculation or quantitation errors, data entry errors, improper practices, or failure to follow SOPs, the data must be evaluated to determine the possible effect.

The Laboratory Director, QA Manager, ECOs, VP of Operations and the Quality Directors have the authority and responsibility to halt work, withhold final reports, or suspend an analysis for due cause as well as authorize the resumption of work.

13.3 Evaluation of Significance and Actions Taken

For each nonconforming issue reported, an evaluation of its significance and the level of management involvement needed is made. This includes reviewing its impact on the final data, whether or not it is an isolated or systematic issue, and how it relates to any special client requirements.

The NDSC Document entitled Data Recalls (CW-Q-S-005) is the procedure to be followed when it is discovered that erroneous or biased data may have been reported to clients or regulatory agencies.

The NDSC Document entitled Internal Investigations (CW-L-S-002) is the procedure to be followed for investigation and correction of situations involved alleged incidents of misconduct or violation of the company's ethics policy.

Laboratory level decisions are documented and approved using the laboratory's standard nonconformance/corrective action reporting in lieu of the data recall determination form contained in NDSC Document No. CW-Q-S-005.

13.4 Prevention of Nonconforming Work

If it is determined that the nonconforming work could recur, further corrective actions must be made following the laboratory's corrective action system. Periodically as defined by the laboratory's preventive action schedule, the QA Department evaluates non-conformances to determine if any nonconforming work has been repeated multiple times. If so, the laboratory's corrective action process may be followed.

13.5 Method Suspension / Restriction (Stop Work Procedures)

In some cases, it may be necessary to suspend/restrict the use of a method or target analyte which constitutes significant risk and/or liability to the laboratory. Suspension/restriction procedures can be initiated by any of the persons noted in Section 13.2, Paragraph 5.

Prior to suspension/restriction, confidentiality will be respected, and the problem with the required corrective and preventive action will be stated in writing and presented to the Laboratory Director.

The Laboratory Director shall arrange for the appropriate personnel to meet with the QA Manager as needed. This meeting shall be held to confirm that there is a problem, that suspension/restriction of the method is required and will be concluded with a discussion of the steps necessary to bring the method/target or test fully back on line. In some cases, that may not be necessary if all appropriate personnel have already agreed there is a problem and there is agreement on the steps needed to bring the method, target or test fully back on line. The QA Manager will also initiate a corrective action report as described in Section 14 if one has not already been started. A copy of any meeting notes and agreed upon steps should be e-mailed by the laboratory to their Business Unit President; VP-QA & EHS This e-mail acts as notification of the incident.

After suspension/restriction, the lab will hold all reports to clients pending review. No faxing, mailing or distributing through electronic means may occur. The report must not be posted for viewing on the internet. It is the responsibility of the Laboratory Director to hold all reporting and to notify all relevant laboratory personnel regarding the suspension/restriction (e.g., Project

Management, Log-in, etc.). Clients will NOT generally be notified at this time. Analysis may proceed in some instances depending on the non-conformance issue.

Within 72 hours, the QA Manager will determine if compliance is now met and reports can be released, OR determine the plan of action to bring work into compliance, and release work. A team, with all principals involved (e.g., Laboratory Director, Technical Director, QA Department, Group Leader) can devise a start-up plan to cover all steps from client notification through compliance and release of reports. Project Management and the Directors of Client Services and Sales and Marketing must be notified if clients must be notified or if the suspension/restriction affects the laboratory's ability to accept work. The QA Manager must approve start-up or elimination of any restrictions after all corrective action is complete.

14 CORRECTIVE ACTION

14.1 Overview

A major component of the laboratory's (QA) Program is the problem investigation and feedback mechanism designed to keep the laboratory staff informed on quality related issues and to provide insight to problem resolution. When nonconforming work or departures from policies and procedures in the quality system or technical operations are identified, the corrective action procedure provides a systematic approach to assess the issues, restore the laboratory's system integrity, and prevent reoccurrence. The laboratory employs two systems to manage non-conformances. Issues suspected of being systematic in nature and for which root cause analysis and a formal Corrective Action Report (CAR) are documented in the Incident Corrective Action Tracking (ICAT) database. Routine batch non-conformances, events that are understood to be isolated in nature, are documented in the LIMS non-conformance memo (NCM) system.

14.2 General

Problems within the quality system or within analytical operations may be discovered in a variety of ways, such as QC sample failures, internal or external audits, proficiency testing (PT) performance, client complaints, staff observation, etc.

The purpose of a corrective action system is to:

- Identify non-conformance events and assign responsibility for investigating.
- Resolve non-conformance events and assign responsibility for any required corrective action.
- Identify systematic problems before they become serious.
- Identify and track client complaints and provide resolution.

14.2.1 **Non-Conformance Memo (NCM)** - is used to document the following types of corrective actions:

- Deviations from an established procedure or SOP
- QC outside of limits
- Isolated reporting / calculation errors
- Client complaints
- Discrepancies in materials / goods received vs. manufacturer packing slips (Forms of documentation other than NCMs in LIMS are also acceptable)

14.2.2 **Corrective Actions Documented In the ICAT Database** –

- Internal and external audit findings
- Failed or unacceptable PT results
- Identified poor process or method performance trends
- Systematic reporting / calculation errors
- Data recall investigations
- Questionable trends that are found in the review of NCMs.
- Client complaints
- Excessive revised reports

The ICAT database is used to document background information, track the results of corrective action investigations and root cause analysis, and to provide reports of corrective action plans.

14.3 Closed Loop Corrective Action Process

Any employee in the company can initiate a corrective action. There are four main components to a closed-loop corrective action process once an issue has been identified: Cause Analysis, Selection and Implementation of Corrective Actions (both short and long term), Monitoring of the Corrective Actions, and Follow-up.

14.3.1 Cause Analysis

- Upon discovery of a non-conformance event, the event must be defined and documented. An entry into the ICAT system must be initiated, someone is assigned to investigate the issue and the event is investigated for cause. Table 12-1 provides some general guidelines on determining responsibility for assessment.
- The cause analysis step is the key to the process as a long term corrective action cannot be determined until the cause is determined.
- If the cause is not readily obvious, the Technical Director, Laboratory Director, or QA Manager (or QA designee) is consulted.

14.3.2 Selection and Implementation of Corrective Actions

- Where corrective action is needed, the laboratory shall identify potential corrective actions. The action(s) most likely to eliminate the problem and prevent recurrence are selected and implemented. Responsibility for implementation is assigned.
- Corrective actions shall be to a degree appropriate to the magnitude of the problem identified through the cause analysis.
- Whatever corrective action is determined to be appropriate, the laboratory shall document and implement the changes. The ICAT record is used for this documentation.

14.3.3 Root Cause Analysis

Root Cause Analysis is a class of problem solving (investigative) methods aimed at identifying the basic or causal factor(s) that underlie variation in performance or the occurrence of a significant failure. The root cause may be buried under seemingly innocuous events, many

steps preceding the perceived failure. At first glance, the immediate response is typically directed at a symptom and not the cause. Typically, root cause analysis would be best with three or more incidents to triangulate a weakness. NDSC Document No CA-Q-S-009, Root Cause Analysis provides guidance on this procedure.

Systematically analyze and document the root causes of the more significant problems that are reported. Identify, track, and implement the corrective actions required to reduce the likelihood of recurrence of significant incidents. Trend the root cause data from these incidents to identify root causes that, when corrected, can lead to dramatic improvements in performance by eliminating entire classes of problems.

Identify the one event associated with problem and ask why this event occurred. Brainstorm the root causes of failures; for example, by asking why events occurred or conditions existed; and then why the cause occurred consecutive times until you get to the root cause. For each of these sub events or causes, ask why it occurred. Repeat the process for the other events associated with the incident.

Root cause analysis does not mean the investigation is over. Look at technique or other systems outside the normal indicators. Often creative thinking will find root causes that ordinarily would be missed and continue to plague the laboratory or operation.

14.3.4 Monitoring of the Corrective Actions

- The Group Leader, Operations Manager, and QA Manager are responsible to ensure that the corrective action taken was effective.
- Ineffective actions are documented and re-evaluated until acceptable resolution is achieved. Group Leaders and the Operations Manager are accountable to the Laboratory Director to ensure final acceptable resolution is achieved and documented appropriately.
- The QA Manager or designee reviews monthly NCM and ICAT records for trends. Highlights are included in the QA monthly report (refer to Section 16). If a significant trend develops that adversely affects quality, an audit of the area is performed and corrective action implemented.
- Any out-of-control situations that are not addressed acceptably at the laboratory level may be reported to the NDSC Quality Director by the QA Manager, indicating the nature of the out-of-control situation and problems encountered in solving the situation.

14.3.5 Follow-up Audits

- Follow-up audits may be initiated by the QA Manager or designee and shall be performed as soon as possible when the identification of a nonconformance casts doubt on the laboratory's compliance with its own policies and procedures, or on its compliance with state or federal requirements.
- These audits often follow the implementation of the corrective actions to verify effectiveness. An additional audit would only be necessary when a critical issue or risk to business is discovered.

(Also refer to Section 17.1.4, Special Audits.)

14.4 Technical Corrective Actions

In addition to providing acceptance criteria and specific protocols for technical corrective actions in the method SOPs, the laboratory has general procedures to be followed to determine when departures from the documented policies and procedures and quality control have occurred (refer to Section 13). The documentation of these procedures is through the use of an NCM or record in the ICAT system.

Table 14-1 includes examples of general technical corrective actions. For specific criteria and corrective actions, refer to the analytical methods or specific method SOPs. The laboratory may also maintain Work Instructions on these items that are available upon request.

Table 14-1 provides some general guidelines for identifying the individual(s) responsible for assessing each QC type and initiating corrective action. The table also provides general guidance on how a data set should be treated if associated QC measurements are unacceptable. Specific procedures are included in Method SOPs, Work Instructions, QAM Sections 19 and 20. All corrective actions are reviewed monthly, at a minimum, by the QA Manager and highlights are included in the QA monthly report.

To the extent possible, samples shall be reported only if all quality control measures are acceptable. If the deficiency does not impair the usability of the results, data will be reported with an appropriate data qualifier and/or the deficiency will be noted in the case narrative. Where sample results may be impaired, the Project Manager is notified by an NCM and appropriate corrective action (e.g., reanalysis) is taken and documented**.**

14.5 Basic Corrections

When mistakes occur in records, each mistake shall be crossed-out, [not obliterated (e.g. no white-out)], and the correct value entered alongside. All such corrections shall be initialed (or signed) and dated by the person making the correction. In the case of records stored electronically, the original uncorrected file must be maintained intact and a second corrected-file is created. This same process applies to adding additional information to a record. All additions made later than the initial must also be initialed (or signed) and dated. When corrections are due to reasons other than obvious transcription errors, the reason for the corrections (or additions) shall also be documented.

Table 14-1. Example – General Corrective Action Procedures

QC Activity *(Individual Responsible for Initiation/Assessment)*	Acceptance Criteria	Recommended Corrective Action
Initial Instrument Blank *(Analyst)*	- Instrument response < RL. -See details in Method SOP	- Prepare another blank. - If same response, determine cause of contamination: reagents, environment, instrument equipment failure, etc.
Initial Calibration Standards *(Analyst, Group Leaders)*	- Correlation coefficient > 0.99 or standard concentration value. - % Recovery within acceptance range. - See details in Method SOP.	- Reanalyze standards. - If still unacceptable, remake standards and recalibrate instrument.

QC Activity *(Individual Responsible for Initiation/Assessment)*	Acceptance Criteria	Recommended Corrective Action
Independent Calibration Verification (Second Source) *(Analyst, Group Leaders*	- % Recovery within control limits.	- Remake and reanalyze standard. - If still unacceptable, then remake calibration standards or use new primary standards and recalibrate instrument.
Continuing Calibration Standards *(Analyst, Data Reviewer)*	% Recovery within control limits.	- Reanalyze standard. - If still unacceptable, then recalibrate and rerun affected samples.
Matrix Spike / Matrix Spike Duplicate (MS/MSD) *(Analyst, Data Reviewers)*	- % Recovery within limits documented in LIMS.	- If the acceptance criteria for duplicates or matrix spikes are not met because of matrix interferences, the acceptance of the analytical batch is determined by the validity of the LCS. - If the LCS is within acceptable limits the batch is acceptable. - The results of the duplicates, matrix spikes and the LCS are reported with the data set. - For matrix spike or duplicate results outside criteria the data for that sample shall be reported with qualifiers.
Laboratory Control Sample (LCS) *(Analyst, Data Reviewers)*	- % Recovery within limits specified in LIMS.	- -Check calculations and instrument performance - Re-analyze the LCS and if still outside of control limits, re-prepare and re-analyze all samples in the batch. It is acceptable to report data when the acceptance criteria for the positive control are exceeded high (i.e., high bias) and there are associated samples that are non-detects, then those non-detects may be reported. **Note:** If there is insufficient sample or the holding time cannot be met, contact the project manager for client instruction.
Surrogates *(Analyst, Data Reviewers)*	- % Recovery within limits specified in LIMS.	-Reprep and analyze the QC batch for MB surrogates below control limits -Report data with narrative if the surrogate is biased high and associated samples are < RL
Method Blank (MB)	< Reporting Limit with the exception of Common	- Reanalyze blank. - If still positive, determine source of

QC Activity *(Individual Responsible for Initiation/Assessment)*	Acceptance Criteria	Recommended Corrective Action
(Analyst, Data Reviewers)	Laboratory Contaminants listed in QA-003 QC Policy.	contamination. If necessary, reprocess (i.e. digest or extract) entire sample batch. Report blank results. - Qualify the result(s) if the concentration of a targeted analyte in the MB is at or above the reporting limit AND is > 1/10 of the amount measured in the sample. -Results are acceptable if the same contaminants were not found in the associated samples.
Proficiency Testing (PT) Samples *(QA Department,, Analysts, Group Leaders)*	- Criteria supplied by PT Supplier.	- Any failures must be investigated for cause. Failures may result in the need to repeat a PT sample to show the problem is corrected.
Internal / External Audits *(QA Department, Operations Manager, , Group Leaders, Laboratory Director)*	- Defined in Quality System documentation such as SOPs, QAM, etc.	- Non-conformances must be investigated system and necessary corrections must be made.
Reporting / Calculation Errors (Depends on issue – possible individuals include: Analysts, Data Reviewers, Project Managers, Group Leaders, QA Manager, NDSC QA,)	SOP CW-Q-S-005, Data Recall	- Corrective action is determined by type of error. Follow the procedures in SOP CW-L-S-002 or your lab's CA SOP.
Client Complaints *(Project Managers, Lab Director, QA Department, Sales and Marketing)*	-	- Corrective action is determined by the type of complaint.
QA Monthly Report (Refer to Section 16 for an example) (QA Manager, Lab Director, *Group Leaders*)		- Corrective action is determined by the type of issue.
Health and Safety Violation (EH&S Coordinator, Lab Director, Operations Manager, Group Leaders)	- Environmental Health and Safety (EHS) Manual.	- Non-conformance is investigated.

Note:
1. Except as noted below for certain compounds, the method blank should be below the detection limit. Concentrations up to five times the reporting limit will be allowed for the ubiquitous laboratory and reagent contaminants. This allowance presumes that the detection limit is significantly below any regulatory limit to which the data are to be compared and that blank subtraction will not occur.

15 PREVENTIVE ACTION / IMPROVEMENT

15.1 Overview

The laboratory's preventive action programs improve or eliminate potential causes of nonconforming product and/or nonconformance to the quality system. This preventive action process is a proactive and continuous process of improvement activities that can be initiated through feedback from clients, employees, business providers, and affiliates. The QA Department has the overall responsibility to ensure that the preventive action process is in place, and that relevant information on actions is submitted for management review.

Dedicating resources to an effective preventive action system emphasizes the laboratory's commitment to its QA Program. It is beneficial to identify and address negative trends before they develop into complaints, problems and corrective actions. Additionally, the laboratory continually strives to improve customer service and client satisfaction through continuous improvements to laboratory systems.

Opportunities for improvement may be discovered through any of the following:

- review of the monthly QA Metrics Report,
- trending NCMs,
- review of control charts and QC results,
- trending proficiency testing (PT) results,
- performance of management system reviews,
- trending client complaints,
- review of processing operations, or
- staff observations.

The monthly Management Systems Metrics Report shows performance indicators in all areas of the laboratory and quality system. These areas include revised reports, corrective actions, audit findings, internal auditing and data authenticity audits, client complaints, PT samples, holding time violations, SOPs, ethics training, etc. The metrics report is reviewed monthly by the laboratory management, NDSC QA Team, Local, and Executive Management These metrics are used in evaluating the management and quality system performance on an ongoing basis and provide a tool for identifying areas for improvement.

Items identified as continuous improvement opportunities to the management system may be issued as goals from the annual management systems review, recommendations from internal audits, white papers, Lessons Learned, Technical Services audit report, Technical Best Practices, or as Executive or management initiatives.

The laboratory's corrective action process is integral to implementation of preventive actions. A critical piece of the corrective action process is the implementation of actions to prevent further occurrence of a non-compliance event. Historical review of corrective action and non-conformances provides a valuable mechanism for identifying preventive action opportunities.

15.1.1 The following elements are part of a preventive action/process improvement system:

- Identification of an opportunity for preventive action or process improvement
- Process for the preventive action or improvement
- Define the measurements of the effectiveness of the process once undertaken
- Execution of the preventive action or improvement
- Evaluation of the plan using the defined measurements
- Verification of the effectiveness of the preventive action or improvement
- Close-Out by documenting any permanent changes to the Quality System as a result of the Preventive Action or Process Improvement. Documentation of Preventive Action/process Improvement is incorporated into the monthly QA reports, corrective action process and management review.

15.1.2 Any preventive actions/process improvement undertaken or attempted shall be taken into account during the annual Management Systems Review (Section 16). A highly detailed report is not required; however, a summary of successes and failures within the preventive action program is sufficient to provide management with a measurement for evaluation.

15.2 Management of Change

The Management of Change process is designed to manage significant events and changes that occur within the laboratory. Through these procedures, the potential risks inherent with a new event or change are identified and evaluated. The risks are minimized or eliminated through pre-planning and the development of preventive measures. The types of changes covered under this system include: Facility Changes, Major Accreditation Changes, Addition or Deletion to Division's Capabilities or Instrumentation, Key Personnel Changes, Laboratory Information Management System (LIMS) changes. The laboratory has a graded approach for managing change based on the Management Systems Review.

16. CONTROL OF RECORDS

The laboratory maintains a records management system appropriate to its needs and that complies with applicable standards or regulations as required. The system produces unequivocal, accurate records that document all laboratory activities. The laboratory retains all original observations, calculations and derived data, calibration records and a copy of the analytical report for a minimum of five years after it has been issued. Exceptions for programs with longer retention requirements are discussed in Section 14.1.2.

16.1 Overview

The laboratory has established procedures for identification, collection, indexing, access, filing, storage, maintenance and disposal of quality and technical records. A record index is listed in Table 14-1. More detailed information on retention of specific records is provided in NDSC Document No. CW-L-P-001, Records Retention Policy and CW-L-WI-001, Records Retention/Storage Schedule. Quality records are maintained by the QA department electronically. Electronic files are backed up as part of the regular laboratory backup. Records are of two types; either electronic or hard copy paper formats depending on whether the record is computer or hand generated (some records may be in both formats). Technical records are maintained by the individual analysts under the direct supervision of their group leader.

Table 16-1. Record Index[1]

	Record Types [1]:	**Retention Time:**
Technical Records	- Raw Data - Logbooks[2] - Standards - Certificates - Analytical Records - MDLs/IDLs/DOCs - Lab Reports	5 Years from analytical report issue*
Official Documents	- Quality Assurance Manual (QAM) - Work Instructions - Policies - SOPs - Policy Memorandums - Manuals - Published Methods	Indefinitely
QA Records	- Certifications - Method and Software Validation / Verification Data	Indefinitely
QA Records	- Internal & External Audits/Responses - Corrective/Preventive Actions - Management Reviews - Data Investigation	5 Years from archival* **Data Investigation:** 5 years or the life of the affected raw data storage whichever is greater (beyond 5 years if ongoing project or pending investigation)
Project Records	- Sample Receipt & COC Documents - Contracts and Amendments - Correspondence - QAPP - SAP - Telephone Logbooks - Lab Reports	5 Years from analytical report issue*
Administrative Records	Financial and Business Operations	Refer to NDSC Doc. No. CW-L-WI-001
	EH&S Manual, Permits	Indefinitely
	Disposal Records	Indefinitely
	Employee Handbook	Indefinitely
	Personnel files, Employee Signature & Initials, Administrative Training Records (e.g., Ethics)	Refer to HR Manual
	Administrative Policies	Indefinitely
	Technical Training Records	7 years
	Legal Records	Indefinitely
	HR Records	Refer to NDSC Doc. No. CW-L-WI-001
	IT Records	Refer to NDSC Doc. No.CW-L-WI-001
	NDSC Governance Records	Refer to NDSC Doc. No.CW-L-WI-001
	Sales & Marketing	5 years
	Real Estate	Indefinitely

[1] Record Types encompass hardcopy and electronic records.
[2] Examples of Logbook types: Maintenance, Instrument Run, Preparation (standard and samples), Standard and Reagent Receipt, Archiving, Balance Calibration, Temperature (hardcopy or electronic records).
** Exceptions listed in Table 16-2.*

16.1.1 All records are stored and retained in such a way that they are secure and readily retrievable at the laboratory facility or an offsite location that provides a suitable environment to prevent damage or deterioration and to prevent loss. All records shall be protected against fire, theft, loss, environmental deterioration, and vermin. In the case of electronic records, electronic or magnetic sources, storage media are protected from deterioration caused by magnetic fields and/or electronic deterioration.

Access to the data is limited to laboratory and company employees and shall be documented with an access log**.** Records archived off-site are stored in a secure location where a record is maintained of any entry into the storage facility. Whether on-site or off-site storage is used, logs are maintained in each storage box to note removal and return of records. Retention of records are maintained on-site at the laboratory for approximately one month after their generation and moved offsite for the remainder of the required storage time. Records are maintained for a minimum of five years unless otherwise specified by a client or regulatory requirement.

For raw data and project records, record retention shall be calculated from the date the project report is issued. For other records, such as NDSC and/or KGD Documents, QA, or Administrative Records, the retention time is calculated from the date the record is formally retired. Records related to the programs listed in Table 16-2 have lengthier retention requirements and are subject to the requirements in Section 16.1.3.

16.2 Programs with Longer Retention Requirements

Some regulatory programs have longer record retention requirements than the standard record retention time. These are detailed in Table 16-2 with their retention requirements. In these cases, the longer retention requirement is enacted. If special instructions exist such that client data cannot be destroyed prior to notification of the client, the container or box containing that data is marked as to who to contact for authorization prior to destroying the data.

Table 16-2. Example: Special Record Retention Requirements

Program	[1]Retention Requirement
Michigan Department of Environmental Quality – all environmental data	10 years 12 years for Lead and Copper results
Ohio VAP	10 years and State contacted prior to disposal
OSHA	30 years

[1]Note: Extended retention requirements must be noted with the archive documents or addressed in facility-specific records retention procedures.

16.2.1 The laboratory has procedures to protect and back-up records stored electronically and to prevent unauthorized access to or amendment of these records. All analytical data is maintained as hard copy or in a secure readable electronic format. For analytical reports

that are maintained as copies in PDF format, refer to Section 19.15.1 for more information.

16.2.2 The record keeping system allows for historical reconstruction of all laboratory activities that produced the analytical data, as well as rapid recovery of historical data (Records stored off site should be accessible within 2 days of a request for such records). The history of the sample from when the laboratory took possession of the samples must be readily understood through the documentation. This shall include inter-laboratory transfers of samples and/or extracts.

- The records include the identity of personnel involved in sampling, sample receipt, preparation, or testing. All analytical work contains the initials (at least) of the personnel involved. The laboratory's copy of the COC is stored with the invoice and the work order sheet generated by LIMS. The chain of custody would indicate the name of the sampler. If any sampling notes are provided with a work order, they are kept with this package.

- All information relating to the laboratory facilities' equipment, analytical test methods, and related laboratory activities, such as sample receipt, sample preparation, or data verification are documented.

- The record keeping system facilitates the retrieval of all working files and archived records for inspection and verification purposes (e.g., set format for naming electronic files, set format for what is included with a given analytical data set. SOP NC-QA-019, Records Information Management outlines this procedure. Instrument data is stored sequentially by instrument. A given day's analyses are maintained in the order of the analysis. Run logs are maintained for each instrument or method; a copy of each day's run log or instrument sequence is stored with the data to aid in re-constructing an analytical sequence. Where an analysis is performed without an instrument, electronic spreadsheets are used to record and file data, or the data is manually entered into LIMS at the time of analysis. Standard and reagent information is recorded in logbooks or entered into LIMS for each method as required.

- Changes to hardcopy records shall follow the procedures outlined in Section 12 and 19. Changes to electronic records in LIMS or instrument data are recorded in audit trails.

- The reason for a signature or initials on a document is clearly indicated in the records such as "sampled by," "prepared by," "reviewed by", or "analyzed by".

- All generated data except those that are generated by automated data collection systems, are recorded directly, promptly and legibly in permanent dark ink or entered directly into LIMS at the time the data is generated.

- Hard copy data may be scanned into PDF format for record storage as long as the scanning process can be verified in order to ensure that no data is lost and the data files and storage media must be tested to verify the laboratory's ability to retrieve the information prior to the destruction of the hard copy that was scanned.

- Also refer to Section 19.15.1 'Computer and Electronic Data Related Requirements'.

16.3 Technical and Analytical Records

16.3.1 The laboratory retains records of original observations, derived data and sufficient information to establish an audit trail, calibration records, staff records and a copy of each analytical report issued, for a minimum of five years unless otherwise specified by a client or regulatory requirement. The records for each analysis shall contain sufficient information to enable the analysis to be repeated under conditions as close as possible to the original. The records shall include the identity of laboratory personnel responsible for the sampling, performance of each analysis and reviewing results.

16.3.2 Observations, data and calculations are recorded real-time and are identifiable to the specific task.

16.3.3 Changes to hardcopy records shall follow the procedures outlined in Section 12 and 19. Changes to electronic records in LIMS or instrument data are recorded in audit trails.

The essential information to be associated with analysis, such as strip charts, tabular printouts, computer data files, analytical notebooks, and run logs, include:

- laboratory sample ID code**;**
- Date of analysis; time of analysis is also required if the holding time is seventy-two (72) hours or less, or when time critical steps are included in the analysis (e.g., drying times, incubations, etc.); instrumental analyses have the date and time of analysis recorded as part of their general operations. Where a time critical step exists in an analysis, location for such a time is included as part of the documentation in a specific logbook or on a benchsheet.
- Instrumentation identification and instrument operating conditions/parameters. Operating conditions/parameters are typically recorded in instrument maintenance logs where available.
- analysis type**;**
- all manual calculations and manual integrations**;**
- analyst's or operator's initials/signature**;**
- sample preparation including cleanup, separation protocols, incubation periods or subculture, ID codes, volumes, weights, instrument printouts, meter readings, calculations, reagents**;**
- test results**;**
- standard and reagent origin, receipt, preparation, and use**;**
- calibration criteria, frequency and acceptance criteria**;**
- data and statistical calculations, review, confirmation, interpretation, assessment and reporting conventions**;**
- quality control protocols and assessment**;**
- electronic data security, software documentation and verification, software and hardware audits, backups, and records of any changes to automated data entries
- Method performance criteria including expected quality control requirements. These are indicated both in LIMS and on specific analytical report formats**.**

16.3.4 All logbooks used during receipt, preparation, storage, analysis, and reporting of samples or monitoring of support equipment shall undergo a periodic, documented supervisory or peer review.

16.4 Laboratory Support Activities

In addition to documenting all the above-mentioned activities, the following are retained QA records and project records (previous discussions in this section relate where and how these data are stored):

- all original raw data, whether hard copy or electronic, for calibrations, samples and quality control measures, including analysts' work sheets and data output records (chromatograms, strip charts, and other instrument response readout records);
- a written description or reference to the specific test method used which includes a description of the specific computational steps used to translate parametric observations into a reportable analytical value;
- copies of final reports;
- archived SOPs;
- correspondence relating to laboratory activities for a specific project;
- all corrective action reports, audits and audit responses;
- proficiency test results and raw data; and
- results of data review, verification, and crosschecking procedures

16.4.1 Sample Handling Records

Records of all procedures to which a sample is subjected while in the possession of the laboratory are maintained. These include but are not limited to records pertaining to:

- sample preservation including appropriateness of sample container and compliance with holding time requirement
- sample identification, receipt, acceptance or rejection and login
- sample storage and tracking including shipping receipts, sample transmittal / COC forms
- procedures for the receipt and retention of samples, including all provisions necessary to protect the integrity of samples

16.5 Administrative Records

The laboratory also maintains the administrative records in either electronic or hard copy form. Refer to Table 16-1.

16.6 Records Management, Storage and Disposal

All records (including those pertaining to test equipment), certificates and reports are safely stored, held secure and in confidence to the client. Certification related records are available upon request.

All information necessary for the historical reconstruction of data is maintained by the

laboratory. Records that are stored only on electronic media must be supported by the hardware and software necessary for their retrieval.

Records that are stored or generated by computers or personal computers have hard copy, write-protected backup copies, or an electronic audit trail controlling access.

The laboratory has a record management system (a.k.a., document control) for control of laboratory notebooks, instrument logbooks, standards logbooks, and records for data reduction, validation, storage and reporting. All data are recorded sequentially within a series of sequential notebooks. Bench sheets, if used, are filed sequentially. Standards are maintained in LIMS – no logbooks are required to be used to record that data. Records are considered archived when noted as such in the records management system (a.k.a., document control.)

16.6.1 Transfer of Ownership

In the event that the laboratory transfers ownership or goes out of business, the laboratory shall ensure that the records are maintained or transferred according to client's instructions. Upon ownership transfer, record retention requirements shall be addressed in the ownership transfer agreement and the responsibility for maintaining archives is clearly established. In addition, in cases of bankruptcy, appropriate regulatory and state legal requirements concerning laboratory records must be followed. In the event of the closure of the laboratory, all records will revert to the control of NDSC. Should the entire company cease to exist, as much notice as possible will be given to clients and the accrediting bodies who have worked with the laboratory during the previous 5 years of such action.

16.6.2 Records Disposal

Records are removed from the archive and destroyed after 5 years unless otherwise specified by a client or regulatory requirement. On a project specific or program basis, clients may need to be notified prior to record destruction. Records are destroyed in a manner that ensures their confidentiality such as shredding, mutilation or incineration. (Refer to Tables 14-1 and 14-2).

Electronic copies of records must be destroyed by erasure or physically damaging off-line storage media so no records can be read.

If a third party records Management Company is hired to dispose of records, a "Certificate of Destruction" is required.

17. AUDITS

17.1 Internal Audits

Internal audits are performed to verify that laboratory operations comply with the requirements of the lab's quality system and with the external quality programs under which the laboratory operates. Audits are planned and organized by the QA staff. Personnel conducting the audits should be independent of the area being evaluated. Auditors will have sufficient authority, access to work areas, and organizational freedom necessary to observe all activities affecting quality and to report the assessments to laboratory management and, when requested, to Executive management.

Audits are conducted and documented as described in the NDSC Document No. on performing Internal Auditing, SOP No. CW-Q-S-003. The types and frequency of routine internal audits are described in Table 17-1. Special or ad hoc assessments may be conducted as needed under the direction of the QA staff.

Table 17-1. Types of Internal Audits and Frequency

Description	Performed by	Frequency
Quality Systems Audits	QA Department, QA approved designee, or NDSC QA	All areas of the laboratory annually
Method Audits QA Technical Audits	Joint responsibility: a) QA Manager or designee b) Technical Director or Designee (Refer to NDSC CW-Q-S-003)	QA Technical Audits Frequency: 50% of methods annually
SOP Method Compliance	Joint responsibility: a) QA Manager or designee b) Technical Director or Designee (Refer to NDSC CW-Q-S-003)	SOP Compliance Review Frequency: Every 2 years
Special	QA Department or Designee	Surveillance or spot checks performed as needed, e.g., to confirm corrective actions from other audits.
Performance Testing	Analysts with QA oversight	Two successful per year for each TNI-field of testing or as dictated by regulatory requirements

17.1.1 Annual Quality Systems Audit

An annual quality systems audit is required to ensure compliance to analytical methods and SOPs, Eurofins Data Integrity and Ethics Policies (see Sec. 7.2), TNI quality systems client and state requirements, and the effectiveness of the internal controls of the analytical process, including but not limited to data review, quality controls, preventive action and corrective action. The completeness of earlier corrective actions is assessed for effectiveness & sustainability. The audit is divided into sections for each operating or support area of the lab, and each section is comprehensive for a given area. The area audits may be performed on a rotating schedule throughout the year to ensure adequate coverage of all areas. This schedule may change as situations in the laboratory warrant.

17.1.2 QA Technical Audits

QA technical audits assess data authenticity and analyst integrity. These audits are based on client projects, associated sample delivery groups, and the methods performed. Reported results are compared to raw data to verify the authenticity of results. The validity of calibrations and QC results are compared to data qualifiers, footnotes, and case narratives. Documentation is assessed by examining run logs and records of manual integrations. Manual calculations are checked. Where possible, electronic audit miner programs (e.g., Chrom AuditMiner) are used to

identify unusual manipulations of the data deserving closer scrutiny. QA technical audits will include all methods within a two-year period.

17.1.3 SOP Method Compliance

Compliance of all SOPs with the source methods and compliance of the operational groups with the SOPs will be assessed by the Technical Director or qualified designee at least every two years.

17.1.4 Special Audits

Special audits are conducted on an as needed basis, generally as a follow up to specific issues such as client complaints, corrective actions, PT results, data audits, system audits, validation comments, regulatory audits or suspected ethical improprieties. Special audits are focused on a specific issue, and report format, distribution, and timeframes are designed to address the nature of the issue.

17.1.5 Performance Testing

The laboratory participates semi-annually in performance audits conducted through the analysis of PT samples provided by a third party. The laboratory generally participates in the following types of PT studies: non-potable water and soil

It is Eurofins policy that PT samples be treated as typical samples in the production process. Furthermore, where PT samples present special or unique problems, in the regular production process they may need to be treated differently, as would any special or unique request submitted by any client. The QA Manager must be consulted and in agreement with any decisions made to treat a PT sample differently due to some special circumstance.

Written investigations for unacceptable PT results are required. In some cases it may be necessary for blind QC samples to be submitted to the laboratory to show a return to control.

17.2 External Audits

External audits are performed when certifying agencies or clients conduct on-site inspections or submit performance testing samples for analysis. It is Eurofins policy to cooperate fully with regulatory authorities and clients. The laboratory makes every effort to provide the auditors with access to personnel, documentation, and assistance. Laboratory supervisors are responsible for providing corrective actions to the QA Manager who coordinates the response. Audit responses are due in the time allotted by the client or agency performing the audit.
The laboratory cooperates with clients and their representatives to monitor the laboratory's performance in relation to work performed for the client. The client may only view data and systems related directly to the client's work. All efforts are made to keep other client information confidential.

17.2.1 Confidential Business Information (CBI) Considerations

During on-site audits, auditors may come into possession of information claimed as business confidential. A business confidentiality claim is defined as "a claim or allegation that business information is entitled to confidential treatment for reasons of business confidentiality or a request for a determination that such information is entitled to such treatment." When information is claimed as business confidential, the laboratory must place on (or attach to) the information at the time it is submitted to the auditor, a cover sheet, stamped or typed legend or other suitable form of notice, employing language such as "trade secret", "proprietary" or

"company confidential". Confidential portions of documents otherwise non-confidential must be clearly identified. CBI may be purged of references to client identity by the responsible laboratory official at the time of removal from the laboratory. However, sample identifiers may not be obscured from the information. Additional information regarding CBI can be found in within the 2009 and 2016 TNI standards.

17.3 Audit Findings

Audit findings are documented using the corrective action process and database (see Section 14). The laboratory's corrective action responses may include action plans that could not be completed within a predefined timeframe. In these instances, a completion date must be set and agreed to by operations management and the QA Manager.

Developing and implementing corrective actions to findings is the responsibility of the Group Leader of the department where the finding originated. Findings that are not corrected by specified due dates are reported monthly to management in the QA monthly report.
If any audit finding casts doubt on the effectiveness of the operations or on the correctness or validity of the laboratory's test results, the laboratory shall take timely corrective action, and shall notify clients in writing if the investigations show that the laboratory results have been affected. Once corrective action is implemented, a follow-up audit is scheduled to ensure that the problem has been corrected.

Clients must be notified promptly in writing of any event such as the identification of defective measuring or test equipment that casts doubt on the validity of results given in any test report or amendment to a test report. The investigation must begin within 24 hours of discovery of the problem and all efforts are made to notify the client within two weeks after the completion of the investigation.

18. MANAGEMENT REVIEWS

18.1 Quality Assurance Report

The QA Department is responsible for preparing a comprehensive monthly metrics report to Management to keep them apprised of current quality issues. This report fosters communication, review, and refinement of the QA system to evaluate the suitability of policies and procedures to meet both regulatory and laboratory quality objectives.

The NDSC QA team compiles information from all of the Environment Testing laboratories monthly metrics reports for the Executive Management team. This report includes notable information and concerns regarding the laboratories QA program and a listing of new regulations that may potentially impact the laboratories.

18.2 Annual Management Review

The Laboratory Management team (Laboratory Director, Technical Director, QA Manager / Department, and Operations Manager) conducts a review annually of its quality systems to ensure its continuing suitability and effectiveness in meeting client and regulatory requirements and to introduce any necessary changes or improvements. It will also provide a platform for defining goals, objectives and action items that feed into the laboratory planning system. NDSC personnel may be included in this meeting at the discretion of the Laboratory Director. The LIMS

review consists of examining any audits, complaints or concerns that have been raised through the year that are related to LIMS The laboratory will summarize any critical findings that cannot be solved by the lab and report them to Corporate IT.

This management systems review (NDSC Document No. CW-Q-S-004 and Work Instruction No. CW-Q-WI-003) uses information generated during the preceding year to assess the "big picture" by ensuring that routine actions taken and reviewed on a monthly basis are not components of larger systematic concerns. The monthly review should keep the quality systems current and effective, therefore, the annual review is a formal senior management process to review specific existing documentation. Significant issues from the following documentation are compiled or summarized by the QA Manager prior to the review meeting:

- Matters arising from the previous annual review
- Prior Monthly QA Reports issues
- Laboratory QA Metrics
- Review of report reissue requests
- Review of client feedback and complaints
- Issues arising from any prior management or staff meetings
- Minutes from prior senior lab management meetings - Issues that may be raised from these meetings include:
    - Adequacy of staff, equipment and facility resources
    - Adequacy of policies and procedures
    - Future plans for resources and testing capability and capacity
- The annual internal double blind PT program sample performance (if performed),
- Compliance to the Ethics Policy and Data Integrity Plan - Including any evidence/incidents of inappropriate actions or vulnerabilities related to data Integrity
- Evaluation of overall risk, including risks to impartiality, confidentiality, reporting statements of conformity, and nonconforming work

A report is generated by the QA Manager and management. The report is distributed to the Laboratory Manager and NDSC Quality Director. The report includes, but is not limited to:

- The date of the review and the names and titles of participants
- A reference to the existing data quality related documents and topics that were reviewed
- Quality system or operational changes or improvements that will be made as a result of the review [e.g., an implementation schedule including assigned responsibilities for the changes (Action Table)

Changes to the quality systems requiring update to the laboratory QA Manual shall be included in the next revision of the QA Manual.

18.3 Potential Integrity Related Managerial Reviews

Potential integrity issues (data or business related) must be handled and reviewed in a confidential manner until such time as a follow-up evaluation, full investigation, or other appropriate actions have been completed and issues clarified. The NDSC Document No. CW-L-S-002 Internal Investigations SOP shall be followed All investigations that result in finding of inappropriate activity are documented and include any disciplinary actions involved, corrective actions taken, and all appropriate notifications of clients.

The Eurofins Environment Testing America Presidents, Business Unit Managers, Laboratory Directors, and NDSC Team, are informed of any current data integrity or data recall investigations via the monthly metrics report.

19. TEST METHODS AND METHOD VALIDATION

19.1 Overview

The laboratory uses methods that are appropriate to meet our clients' requirements and that are within the scope of the laboratory's capabilities. These include sampling, handling, transport, storage and preparation of samples, and, where appropriate, an estimation of the measurement of uncertainty as well as statistical techniques for analysis of environmental data.

Instructions are available in the laboratory for the operation of equipment as well as for the handling and preparation of samples. All instructions, Standard Operating Procedures (SOPs), reference methods and manuals relevant to the working of the laboratory are readily available to all staff. Deviations from published methods are documented (with justification) in the laboratory's approved SOPs. SOPs are submitted to clients for review at their request. Significant deviations from published methods require client approval and regulatory approval where applicable.

19.2 Standard Operating Procedures (SOPS)

The laboratory maintains SOPs that accurately reflect all phases of the laboratory such as assessing data integrity, corrective actions, handling customer complaints as well as all analytical methods and sampling procedures. The method SOPs are derived from the most recently promulgated/approved, published methods and are specifically adapted to the laboratory facility. Modifications or clarifications to published methods are clearly noted in the SOPs. All SOPs are controlled in the laboratory.

- All SOPs contain a revision number, effective date, and appropriate approval signatures. Controlled copies are available to all staff.
- Procedures for writing an SOP are incorporated by reference to NDSC Document No. CW-Q-S-002 'Writing a Standard Operating Procedure', No. or the laboratory's SOP NC-QA-027 Preparation and Management of Standard Operating Procedures.
- SOPs are reviewed at a minimum of every 2 years (annually for the state of Kentucky), and where necessary, revised to ensure continuing suitability and compliance with applicable requirements.

19.3 Laboratory Methods Manual

For each test method, the laboratory shall have available the published referenced method as well as the laboratory developed SOP.

Note: If more stringent standards or requirements are included in a mandated test method or regulation than those specified in this manual, the laboratory shall demonstrate that such requirements are met. If it is not clear which requirements are more stringent, the standard from the method or regulation is to be followed. Any exceptions or deviations from the referenced methods or regulations are noted in the specific analytical SOP.

The laboratory maintains an SOP Index for both technical and non-technical SOPs. Technical SOPs are maintained to describe a specific test method. Non-technical SOPs are maintained to describe functions and processes not related to a specific test method.

19.4 Selection of Methods

Since numerous methods and analytical techniques are available, continued communication between the client and laboratory is imperative to assure the correct methods are utilized. Once client methodology requirements are established, this and other pertinent information is summarized by the PM. These mechanisms ensure that the proper analytical methods are applied when the samples arrive for log-in. For non-routine analytical services (e.g., special matrices, non-routine compound lists), the method of choice is selected based on client needs and available technology. The methods selected should be capable of measuring the specific parameter of interest, in the concentration range of interest, and with the required precision and accuracy.

19.4.1 Sources of Methods

Routine analytical services are performed using standard EPA-approved methodology. In some cases, modification of standard approved methods may be necessary to provide accurate analyses of particularly complex matrices. When the use of specific methods for sample analysis is mandated through project or regulatory requirements, only those methods shall be used.

When clients do not specify the method to be used or methods are not required, the methods used will be clearly validated and documented in an SOP and available to clients and/or the end user of the data. Refer to Appendix 6 for a list of the currently accepted U.S. EPA analytical method references used by the laboratory.

The laboratory reviews updated versions to all the aforementioned references for adaptation based upon capabilities, instrumentation, etc., and implements them as appropriate. As such, the laboratory strives to perform only the latest versions of each approved method as regulations allow or require.

Other reference procedures for non-routine analyses may include methods established by specific states (e.g., Underground Storage Tank methods), ASTM or equipment manufacturers. Sample type, source, and the governing regulatory agency requiring the analysis will determine the method utilized.

The laboratory shall inform the client when a method proposed by the client may be inappropriate or out of date. After the client has been informed, and they wish to proceed contrary to the laboratory's recommendation, it will be documented.

19.4.1.1 Client Supplied Methods

Most of the client-supplied method requirements presented to us involve achieving specific quality control criteria, limits of quantitation (LOQ), and/or method detection limits (MDL) using standard EPA methods. These requirements are communicated to the appropriate technical groups prior to the project start up. Each technical group evaluates the scope of work and the requirements to ensure the criteria can be met using the standard EPA method. The data is monitored to ensure the criteria are met throughout the project. The PM notifies the client if there is a more appropriate method available or if the client's criteria cannot be achieved on a certain sample matrix (i.e., due to matrix or dilutions).

Occasionally, we are asked to transfer a non-standardized method from a client into our lab or to develop a new method, when one is not available. In the case of a method transfer, we set up the client's method and perform some initial evaluation. After the initial evaluation, we may make recommendations on how to improve method performance. If the method appears to be adequate, we determine linearity, specificity, precision, accuracy, MDL, and LOQ by performing calibrations, analyzing method blanks, and carrying out method detection limit and IDOC studies.

In the case of method development, we work with the client and/or data user to determine the level of validation required ensuring that the method meets its intended purpose. In addition to the elements above, we also determine standard and sample stability and robustness depending on the scope of the project. Typically, a standard operating procedure is written and submitted to the client with the results of the validation. These steps are completed prior to analysis of field samples. Data related to the setup of the method are archived.

19.4.1.2 Procedural Deviations

Analysts are required to follow a documented method for all tests performed; and any deviations from analytical methods must be documented, approved, and justified in an appropriate and consistent manner. We classify method deviations as either being a planned deviation or an unplanned deviation. In general, the following information is captured to document both types of situations:

- Description of the situation
- Reason or justification for the deviation
- Impact the deviation had on the testing
- Signature/date of analyst performing the test
- Signature/date of QA and Laboratory management approving the deviation
- Signature/date of client approval, if necessary

Deviations to written procedures are documented in raw data records, ICAT or through the ICAR (Investigation and Corrective Action Report) system. Both types of documentation require management and QA review and approval.

19.4.2 Demonstration of Capability

Before the laboratory may institute a new method and begin reporting results, the laboratory shall confirm that it can properly operate the method. In general, this demonstration does not test the performance of the method in real world samples, but in an applicable and available clean matrix sample. If the method is for the testing of analytes that are not conducive to spiking, demonstration of capability may be performed on quality control samples.

A demonstration of capability (DOC, Lab SOP NC-QA-028, Employee Orientation and Training) is performed whenever there is a change in instrument type (e.g., new instrumentation), matrix, method or personnel (e.g., analyst has not performed the test within the last 12 months).

Note: The laboratory shall have a DOC for all analytes included in the methods that the laboratory performs, and proficiency DOCs for each analyst shall include all analytes that the laboratory routinely performs. Addition of non-routine analytes does not require new DOCs for all analysts if those analysts are already qualified for routine analytes tested using identical chemistry and instrument conditions.

The initial demonstration of capability must be thoroughly documented and approved by the Group Leader and QA Manager prior to independently analyzing client samples. All associated documentation must be retained in accordance with the laboratory's archiving procedures.

The laboratory must have an approved SOP, demonstrate satisfactory performance, and conduct an MDL study (when applicable). There may be other requirements as stated within the published method or regulations (e.g., retention time window study).

Note: In some instances, a situation may arise where a client requests that an unusual analyte be reported using a method where this analyte is not normally reported. If the analyte is being reported for regulatory purposes, the method must meet all procedures outlined within this QA Manual (SOP, MDL, and Demonstration of Capability). If the client states that the information is not for regulatory purposes, the result may be reported as long as the following criteria are met:

- The instrument is calibrated for the analyte to be reported using the criteria for the method and ICV/CCV criteria are met (unless an ICV/CCV is not required by the method or criteria are per project DQOs).
- The laboratory's nominal or default reporting limit (RL) is equal to the quantitation limit (QL), must be at or above the lowest non-zero standard in the calibration curve and must be reliably determined. Project RLs are client specified reporting levels which may be higher than the QL. Results reported below the QL must be qualified as estimated values. Also see Section 19.6.1.3, Relationship of Limit of Detection (LOD) to Quantitation Limit (QL).
- The client request is documented and the lab informs the client of its procedure for working with unusual compounds. The final report must be footnoted: *Reporting Limit based on the low standard of the calibration curve.*

19.4.3 Initial Demonstration of Capability (IDOC) Procedures

At least four aliquots must be prepared (including any applicable clean-up procedures) in the same fashion, and following all of the same procedures, as client samples, and analyzed according to the test method (either concurrently or over a period of days).

Using all of the results, calculate the mean recovery in the appropriate reporting units and the standard deviations for each parameter of interest. Refer to SOP NC-QA-028, Employee Orientation and Training, for details on this procedure.

Note: Results of successive LCS analyses can be used to fulfill the DOC requirement.
Methods on line prior to the effective date of this Section shall be updated to the procedures outlined above as new analysts perform their demonstration of capability. A copy of the new record will replace that which was used for documentation in the past. At a minimum, the precision and accuracy of four mid-level laboratory control samples must have been compared to the laboratory's quality control acceptance limits.

19.5 Laboratory Developed Methods and Non-Standard Methods

Any new method developed by the laboratory must be fully defined in an SOP and validated by qualified personnel with adequate resources to perform the method. Method specifications and the relation to client requirements must be clearly conveyed to the client if the method is a non-standard method (not a published or routinely accepted method). The client must also be in agreement to the use of the non-standard method.

19.6 Validation of Methods

Validation is the confirmation by examination and the provision of objective evidence that the particular requirements for a specific intended use are fulfilled.
All non-standard methods, laboratory designed/developed methods, standard methods used outside of their scope, and major modifications to published methods must be validated to confirm they are fit for their intended use. The validation will be as extensive as necessary to meet the needs of the given application. The results are documented with the validation procedure used and contain a statement as to the fitness for use.

19.6.1 Method Validation and Verification Activities for All New Methods

While method validation can take various courses, the following activities can be required as part of method validation. Method validation records are designated QC records and are archived accordingly.

When changes are made to any validated methods, the influence of such changes shall be documented and, if appropriate, a new validation shall be performed.

19.6.1.1 Determination of Method Selectivity – Method selectivity is the demonstrated ability to discriminate the analyte(s) of interest from other compounds in the specific matrix or matrices from other analytes or interference. In some cases to achieve the required selectivity for an analyte, a confirmation analysis is required as part of the method.

19.6.1.2 Determination of Method Sensitivity – Sensitivity can be both estimated and demonstrated. Whether a study is required to estimate sensitivity depends on the level of method development required when applying a particular measurement system to a specific set of samples. Detection limit studies are conducted as described in Section 19.7 below. Where other protocols for estimations and/or demonstrations of sensitivity are required by regulation or client agreement, these shall be followed.

19.6.1.3 Relationship of Limit of Detection (LOD) to the Limit of Quantitation (LOQ) – An important characteristic of expression of sensitivity is the distinction between the LOD and the LOQ. The LOD is the minimum level at which the presence of an analyte can be reliably concluded. The LOQ is the minimum concentration of analyte that can be quantitatively determined with acceptable precision and bias, equivalent to the laboratory's routine reporting limit (RL). For most instrumental measurement systems, there is a region where semi-quantitative data is generated around the LOD (both above and below the estimated MDL or LOD) and below the LOQ. In this region, detection of an analyte may be confirmed but quantification of the analyte is unreliable within the accuracy and precision guidelines of the measurement system. When an analyte is detected below the LOQ, and the presence of the analyte is confirmed by meeting the qualitative identification criteria for the analyte, the analyte can be reliably reported, but the amount of the analyte can only be estimated. If data is to be reported in this region, it must be done so with a qualification that denotes the semi-quantitative nature of the result.

19.6.1.4 Determination of Interferences – A determination that the method is free from interferences in a blank matrix is performed.

19.6.1.5 Determination of Range – Where appropriate to the method, the quantitation range is determined by comparison of the response of an analyte in a curve to established or targeted criteria. Generally the upper quantitation limit is defined by highest acceptable calibration concentration. The lower quantitation limit or QL cannot be lower than the lowest non-zero calibration level, and can be constrained by required levels of bias and precision.

19.6.1.6 Determination of Accuracy and Precision – Accuracy and precision studies are generally performed using replicate analyses, with a resulting percent recovery and measure of reproducibility (standard deviation, relative standard deviation) calculated and measured against a set of target criteria.

19.6.1.7 Documentation of Method – The method is formally documented in an SOP. If the method is a minor modification of a standard laboratory method that is already documented in an SOP, an SOP Attachment describing the specific differences in the new method is acceptable in place of a separate SOP.

19.6.1.8 Continued Demonstration of Method Performance – Continued demonstration of method performance is addressed in the SOP. Continued demonstration of method performance is generally accomplished by batch specific QC samples such as LCS, method blanks or PT samples.

19.7 Method Detection Limits (MDL) / Limits of Detection (LOD)

The MDL is the minimum measured quantity of a substance that can be reported with 99% confidence that the concentration is distinguishable from method blank results, consistent with

40CFR Part 136 Appendix B, August, 2017. The MDL is equivalent to the TNI LOD, and is also equivalent to the DoD/DOE Quality Systems Manual (QSM) DL. The working or final MDL is the higher of the MDL value determined from spikes (MDLs) and the MDL value determined from blanks (MDLb). An initial MDL study shall be performed during the method validation process and when the method is altered in a way that can reasonably be expected to change its sensitivity. On-going data are collected during each quarter in which samples are being analyzed. At least once every 13 months the MDLs and MDLb are re-calculated and re-evaluated using data collected during the preceding period. Details of the laboratory's procedure for conducting MDL studies are given in NDSC Document No. CA-Q-S-006.

19.8 Verification of Detection Limits

If it is found during the re-evaluation of detection limit results that more than 5% of the spiked samples do not return positive numeric results that meet all method qualitative identification criteria, then then spiking level shall be increased and the initial MDL study pre-performed at the new spiking concentration.

19.9 Instrument Detection Limits (IDL)

The IDL is sometimes used to assess the reasonableness of the MDL or in some cases required by the analytical method or program requirements. IDLs are most commonly used in metals analyses but may be useful in demonstration of instrument performance in other areas.

IDLs are calculated to determine an instrument's sensitivity independent of any preparation method. IDLs are calculated either using 7 replicate spike analyses, like MDL but without sample preparation, or by the analysis of 10 instrument blanks and calculating 3 x the absolute value of the standard deviation.

19.10 Limit of Quantitation

The LOQ shall be at a concentration equivalent to the lowest calibration standard concentration, with the exception of methods using a single-point calibration, and shall be greater than the MDL. The LOQ is verified by preparing and analyzing spikes at concentrations 1-2X the selected LOQ, employing the complete analytical process.

When the laboratory establishes a quantitation limit, it must be initially verified by the analysis of a low level standard or QC sample at 1-2 times the reporting limit and annually thereafter. The annual requirement is waived for methods that have an annually verified MDL. The laboratory will comply with any regulatory requirements.

19.11 Retention Time Windows

Most organic analyses and some inorganic analyses use chromatography techniques for qualitative and quantitative determinations. For every chromatography analysis or as specified in the reference method, each analyte will have a specific time of elution from the column to the detector. This is known as the analyte's retention time. The variance in the expected time of elution is defined as the retention time window. As the key to analyte identification in chromatography, retention time windows must be established on every column for every analyte used for that method. These records are kept on-file and available for review. Complete details are available in the laboratory SOPs.

19.12 Evaluation of Selectivity

The laboratory evaluates selectivity by following the checks within the applicable analytical methods, which include mass spectral tuning, second column confirmation, ICP interelement interference checks, chromatography retention time windows, sample blanks, spectrochemical, atomic absorption or fluorescence profiles, and specific electrode response factors.

19.13 Estimation of Uncertainty of Measurement

19.13.1 Uncertainty is "a parameter associated with the result of a measurement, that characterizes the dispersion of the values that could reasonably be attributed to the measurement" (as defined by the International Vocabulary of Basic and General Terms in Metrology, ISO Geneva, 1993, ISBN 92-67-10175-1). Knowledge of the uncertainty of a measurement provides additional confidence in a result's validity. Its value accounts for all the factors which could possibly affect the result, such as adequacy of analyte definition, sampling, matrix effects and interferences, climatic conditions, variances in weights, volumes, and standards, analytical procedure, and random variation. Some national accreditation organizations require the use of an "expanded uncertainty" defined as the range within which the value of the measurand is believed to lie within at least a 95% confidence level with the coverage factor k=2.

19.13.2 Uncertainty is not error. Error is a single value (i.e., the difference between the true result and the measured result). On environmental samples, the true result is never known. The measurement is the sum of the unknown true value and the unknown error. Unknown error is a combination of systematic error, or bias, and random error. Bias varies predictably, constantly, and independently from the number of measurements. Random error is unpredictable, assumed to be Gaussian in distribution, and reducible by increasing the number of measurements.

19.13.3 The minimum uncertainty associated with results generated by the laboratory can be determined by using the Laboratory Control Sample (LCS) accuracy range for a given analyte. The LCS limits are used to assess the performance of the measurement system since they take into consideration all of the laboratory variables associated with a given test over time (except for variability associated with the sampling and the variability due to matrix effects). The percent recovery of the LCS is compared either to the method-required LCS accuracy limits or to the statistical, historical, in-house LCS accuracy limits.

19.13.4 To calculate the uncertainty for the specific result reported, multiply the result by the decimal of the lower end of the LCS range percent value for the lower end of the uncertainty range, and multiply the result by the decimal of the upper end of the LCS range percent value for the upper end of the uncertainty range. These calculated values represent uncertainties at approximately the 99% confidence level with a coverage factor of k = 3. As an example, for a reported result of 1.0 mg/L with an LCS recovery range of 50 to 150%, the estimated uncertainty in the result would be 1.0 +/- 0.5 mg/L.

19.13.5 In the case where a well-recognized test method specifies limits to the values of major sources of uncertainty of measurement (e.g., 524.2, 525, etc.) and specifies the form of presentation of calculated results, no further discussion of uncertainty is required.

19.14 Sample Reanalysis Guidelines

Because there is a certain level of uncertainty with any analytical measurement, a sample re-preparation (where appropriate) and subsequent analysis (hereafter referred to as 'reanalysis')

may result in either a higher or lower value from an initial sample analysis. There are also variables that may be present (e.g., sample homogeneity, analyte precipitation over time, etc.) that may affect the results of a reanalysis. Based on the above comments, the laboratory will reanalyze samples at a client's request with the following caveats. **Client specific Contractual Terms & Conditions for reanalysis protocols may supersede the following items.**

- Homogenous samples: If a reanalysis agrees with the original result to within the RPD limits for MS/MSD or Duplicate analyses, or within ± 1 reporting limit for samples ≤ 5x the reporting limit, the original analysis will be reported. At the client's request, both results may be reported on the same report but not on two separate reports.

- If the reanalysis does not agree (as defined above) with the original result, then the laboratory will investigate the discrepancy and reanalyze the sample a third time for confirmation if sufficient sample is available.

- Any potential charges related to reanalysis are discussed in the contract terms and conditions or discussed at the time of the request. The client will typically be charged for reanalysis unless it is determined that the lab was in error.

- Due to the potential for increased variability, reanalysis may not be applicable to Non-homogenous, Encore, and Sodium Bisulfate preserved samples. See the Area Supervisor *or* Laboratory Director if unsure.

19.15 Control of Data

The laboratory has policies and procedures in place to ensure the authenticity, integrity, and accuracy of the analytical data generated by the laboratory.

19.15.1 Computer and Electronic Data Related Requirements

The three basic objectives of our computer security procedures and policies are shown below. The laboratory is currently using the Eurofins LIMS system, which has been highly customized to meet the needs of the laboratory. It is referred to as LIMS for the remainder of this section.

19.15.1.1 Maintain the Database Integrity – Assurance that data is reliable and accurate through data verification (review) procedures, password-protecting access, anti-virus protection, data change requirements, as well as an internal LIMS permissions procedure.

- LIMS Database Integrity is achieved through data input validation, internal user controls, documentation of system failures and corrective actions taken, and data change requirements.
- Spreadsheets and other software developed in-house must be verified with documentation through hand calculations prior to use. Cells containing calculations must be lock-protected and controlled.
- Instrument hardware and software adjustments are safeguarded through maintenance logs, audit trails and controlled access.

19.15.1.2 Ensure Information Availability – Protection against loss of information or service is ensured through scheduled back-ups, stable file server network architecture, secure storage of media, line filter, Uninterruptible Power Supply (UPS), and maintaining older versions of software as revisions are implemented.

19.15.1.3 Maintain Confidentiality – Ensure data confidentiality through physical access controls such as password protection or website access approval when electronically transmitting data.

19.15.2 Data Reduction

The complexity of the data reduction depends on the analytical method and the number of discrete operations involved (e.g., extractions, dilutions, instrument readings and concentrations). The analyst calculates the final results from the raw data or uses appropriate computer programs to assist in the calculation of final reportable values.

For manual data entry, e.g., Wet Chemistry, the data is reduced by the analyst and then verified by the Group Leader or alternate analyst once the data is uploaded or entered into LIMS. The spreadsheets, or any other type of applicable documents, are initialed by both the analyst and alternate reviewer to confirm the accuracy of the manual entry(s).

Manual integration of peaks will be documented and reviewed and the raw data will be flagged using NDSG Document No. CA-Q-S-002, *Acceptable Manual Integration Practices*.

Analytical results are reduced to appropriate concentration units specified by the analytical method, taking into account factors such as dilution, sample weight or volume, etc. Blank correction will be applied only when required by the method or per manufacturer's indication; otherwise, it should not be performed. Calculations are independently verified by appropriate laboratory staff. Calculations and data reduction steps for various methods are summarized in the respective analytical SOPs or program requirements.

19.15.2.1 All raw data must be retained in the worklist folder, computer file (if appropriate), and/or run log. All criteria pertinent to the method must be recorded. The documentation is recorded at the time observations or calculations are made and must be signed or initialed/dated (month/day/year). It must be easily identifiable who performed which tasks if multiple people were involved.

19.15.2.2 In general, concentration results are reported in milligrams per liter (mg/l) or micrograms per liter (µg/l) for liquids and milligrams per kilogram (mg/kg) or micrograms per kilogram (µg/kg) for solids. For values greater than 10,000 mg/l, results can be reported in percent, i.e., 10,000 mg/l = 1**%.** Units are defined in each lab SOP.

19.15.2.3 In reporting, the analyst or the instrument output records the raw data result using values of known certainty plus one uncertain digit. If final calculations are performed external to LIMS, the results should be entered in LIMS with at least three significant figures. In general, results are reported to 2 significant figures on the final report.

19.15.2.4 For those methods that do not have an instrument printout or an instrumental output compatible with the LIMS System, the raw results and dilution factors are entered directly into LIMS by the analyst, and the software calculates the final result for the analytical report. LIMS has a formatter for significant figure criterion for each analyte.

19.15.2.5 The laboratory strives to import data directly from instruments or calculation spreadsheets to ensure that the reported data are free from transcription and calculation errors. For those analyses with an instrumental output compatible with the LIMS, the raw results and dilution factors are transferred into LIMS electronically after reviewing the quantitation report, and removing unrequested or poor spectrally-matched compounds. The analyst prints a copy of what has been entered to check for errors. This printout and the instrument's printout of calibrations, concentrations, retention times, chromatograms, and mass spectra, if applicable, are retained with the data file. The data file is stored in a monthly folder on the instrument computer; periodically, this file is transferred to the server and, eventually, to a tape file.

19.15.3 Logbook / Worksheet Use Guidelines

Logbooks and worksheets are filled out 'real time' and have enough information on them to trace the events of the applicable analysis/task. (e.g. calibrations, standards, analyst, sample ID, date, time on short holding time tests, temperatures when applicable, calculations are traceable, etc.)

- Corrections are made following the procedures outlined in Section 14.
- Logbooks are controlled by the QA department. A record is maintained of all logbooks in the lab.
- Unused portions of worksheet pages must be "Z"'d out, signed and dated. Logbooks with unused pages: The first unused page will be Z'd out, signed, and dated. A note will be added to this page stating that the logbook has been closed and no further pages are to be used.
- Worksheets are created with the approval of the QA Manager or designee at the facility. The QA Manager or designee controls all worksheets following the procedures in Section 6.

19.15.4 Review / Verification Procedures

Review procedures are outlined in several SOPs to ensure that reported data are free from calculation and transcription errors, that QC parameters have been reviewed and evaluated before data is reported. The laboratory also has an SOP discussing Manual Integrations to ensure the authenticity of the data. The general review concepts are discussed below, more specific information can be found in the SOPs.

19.15.4.1 Log-In Review - The data review process starts at the sample receipt stage. Sample control personnel review chain-of-custody forms and project instructions from the project management group. This is the basis of the sample information and analytical instructions entered into the LIMS. The log-in instructions are reviewed by the personnel entering the information, and a second level review is conducted by the project management staff.

19.15.4.2 First Level Data Review - The next level of data review occurs with the analysts. As data are generated, analysts review their work to ensure that the results meet project and SOP requirements. First level reviews include inspection of all raw data (e.g., instrument output for continuous analyzers, chromatograms, spectra, and manual integrations), evaluation of calibration/calibration verification data in the day's analytical run, evaluation of QC data, and reliability of sample results. The analyst transfers data into LIMS, data qualifiers are added as needed. All first level reviews are documented.

19.15.4.3 Second Level Data Review – All analytical data are subject to review by a second qualified analyst or supervisor. Second level reviews include inspection of all raw data (e.g., instrument output, chromatograms, and spectra) including 100% of data associated with any changes made by the primary analyst, such as manual integrations or reassignment of peaks to different analytes, or elimination of false negative analytes. The second review also includes evaluation of initial calibration/calibration verification data in the day's analytical run, evaluation of QC data, reliability of sample results, verification of units vs matrix, qualifiers and NCM narratives. Manual calculations are checked in second level review. All second level reviews are documented.

Issues that deem further review include the following:

- QC data are outside the specified control limits for accuracy and precision
- Reviewed sample data does not match with reported results
- Unusual detection limit changes are observed
- Samples having unusually high results
- Samples exceeding a known regulatory limit
- Raw data indicating some type of contamination or poor technique
- Inconsistent peak integration
- Transcription errors
- Results outside of calibration range

19.15.4.4 Unacceptable analytical results may require reanalysis of the samples. Any problems are brought to the attention of the Laboratory Director, Project Manager, Quality Director/Manager, Technical Director, or Supervisor for further investigation. Corrective action is initiated whenever necessary.

19.15.4.5 The results are then entered or directly transferred into the computer database and a hard copy (or .pdf) is printed for the client.

19.15.4.6 As a final review prior to the release of the report, the Project Manager reviews the results for appropriateness and completeness. This review and approval ensures that client requirements have been met and that the final report has been properly completed. The process includes, but is not limited to, verifying that the COC is followed, cover letters / narratives are present, flags are appropriate, reported units are appropriate for the sample matrix, and project specific requirements are met. The Project Manager may also evaluate the validity of results for different test methods given expected chemical relationships.

19.15.4.7 Any project that requires a data package is subject to a tertiary data review for transcription errors and acceptable quality control requirements. The Project Manager then electronically signs the final report which is sent out to the client.

19.15.4.8 A brief overview of sample flows and information through the laboratory, as well as data review and validation, is presented in Figure 19-1.

19.15.5 Manual Integrations

Computerized data systems provide the analyst with the ability to re-integrate raw instrument data in order to optimize the interpretation of the data. Though manual integration of data is an invaluable tool for resolving variations in instrument performance and some sample matrix problems, when used improperly, this technique would make unacceptable data appear to meet quality control acceptance limits. Improper re-integrations lead to legally indefensible data, a poor reputation, or possible laboratory decertification. Because guidelines for re-integration of data are not provided in the methods and most methods were written prior to widespread implementation of computerized data systems, the laboratory trains all analytical staff on proper manual integration techniques using NDSC Document No. CA-Q-S-002 Acceptable Manual Integration Practices.

19.15.5.1 The analyst must adjust baseline or the area of a peak in some situations, for example when two compounds are not adequately resolved or when a peak shoulder needs to be separated from the peak of interest. The analyst must use professional judgment to determine when manual integrating is required. Analysts are encouraged to ask for assistance from a senior analyst or manager when in doubt.

19.15.5.2 Analysts shall not increase or decrease peak areas for the sole purpose of achieving acceptable QC recoveries that would have otherwise been unacceptable. The intentional recording or reporting of incorrect information (or the intentional omission of correct information) is against company principles and policy and is grounds for immediate termination.

19.15.5.3 Client samples, performance evaluation samples, and quality control samples are all treated equally when determining whether or not a peak area or baseline should be manually adjusted.

19.15.5.4 All manual integrations receive a second level review. Manual integrations must be indicated on expanded scale “before” and “after” chromatograms such that the integration performed can be easily evaluated during data review. Expanded scale “before” chromatograms are also required for all manual integrations on QC parameters (calibrations, calibration verifications, laboratory control samples, internal standards, surrogates, etc.) unless the laboratory has another documented NDSC approved procedure in place that can demonstrate an active process for detection and deterrence of improper integration practices.

Figure 19-1. Example: Work Flow

Action	Personnel Involved
Bottle orders generated upon request - Bottles packed and shipped to the client under CoC	- Client Service Personnel / PM - Bottle Preparation Personnel
Samples received at laboratory - Unpacked and reconciled against client paperwork or CoC - Samples acceptance documented against completed (CoC)[1]	Sample Login Personnel
Samples are entered into LIMS - Job # assigned - Methods/Analytes linked from project - Sample labels generated - LIMS backlogs updated immediately	Sample Login Personnel
Sample Storage - Short TATs and hold-time samples are delivered directly to the laboratory for analysis - Samples stored in assigned locations (refrigerated walk-in coolers, freezers, VOA refrigerators; etc.)	Sample Login Personnel
Sample Receipt Acknowledgement sent to client (when requested)	Sample Login Personnel
Sample Acquisition/Testing - Analysts collect samples from assigned storage. - Legal CoC samples are signed in-and-out of storage - Remaining sample returned to storage.	Analysts / Login Personnel
Preparation and analysis is performed according to selected analytical method and applicable Project notes - Raw data recorded/reviewed[2] - Data imported to LIMS	Analysts
LIMS performs calculations: - Data reviewed by primary/secondary analyst - Data approved as final	Data Process (LIMS / Analyst)
Generation/review/release of reports (automated through LIMS) - Electronic copy saved in LIMS	Project Management Assistants / Reporting Group
Electronic Data Deliverables (EDDs) generated (as applicable)	EDD Group
Hard copy and electronic batch raw data is archived according to data archiving procedures.	Analysts / IT

[1]Refer to Sec. 23.3.
[2]Analyses requiring the analyst's interpretation may involve manual data reduction before entry.

SECTION 20. EQUIPMENT and CALIBRATIONS

20.1 Overview

The laboratory purchases the most technically advanced analytical instrumentation for sample analyses. Instrumentation is purchased on the basis of accuracy, dependability, efficiency and sensitivity. Each laboratory is furnished with all items of sampling, preparation, analytical testing and measurement equipment necessary to correctly perform the tests for which the laboratory has capabilities. Each piece of equipment is capable of achieving the required accuracy and complies with specifications relevant to the method being performed. Before being placed into use, the equipment (including sampling equipment) is calibrated and checked to establish that it meets its intended specification. The calibration routines for analytical instruments establish the range of quantitation. Calibration procedures are specified in laboratory SOPs. A list of available laboratory instrumentation is presented in Table 20-1.

Equipment is only operated by authorized and trained personnel. Manufacturer's instructions for equipment use are readily accessible to all appropriate laboratory personnel.

20.2 Preventive Maintenance

The laboratory follows a well-defined maintenance program to ensure proper equipment operation and to prevent the failure of laboratory equipment or instrumentation during use. This program of preventive maintenance helps to avoid delays due to instrument failure.

Routine preventive maintenance procedures and frequency, such as cleaning and replacements, should be performed according to the procedures outlined in the manufacturer's manual. Qualified personnel must also perform maintenance when there is evidence of degradation of peak resolution, a shift in the calibration curve, loss of sensitivity, or failure to continually meet one of the quality control criteria.

Scheduled routine maintenance is defined in each method SOP. It is the responsibility of each Group Leader to ensure that instrument maintenance logs are kept for all equipment in his/her department. Preventative maintenance procedures are also outlined in analytical SOPs and/or instrument manuals. (**Note:** for some equipment, the log used to monitor performance is also the maintenance log. Multiple pieces of equipment may share the same log as long as it is clear as to which instrument is associated with an entry.)

Instrument maintenance logs are controlled and are used to document instrument problems, instrument repair and maintenance activities. Maintenance logs shall be kept for all major pieces of equipment. Instrument maintenance logs may also be used to specify instrument parameters.

- Documentation must include all major maintenance activities such as contracted preventive maintenance and service and in-house activities such as the replacement of electrical components, lamps, tubing, valves, columns, detectors, cleaning and adjustments.
- Each entry in the instrument log includes the analyst's initials, the date, a detailed description of the problem (or maintenance needed/scheduled), a detailed explanation of the solution or maintenance performed, and a verification that the equipment is functioning properly (state what was used to determine a return to control. e.g. CCV run on *'date'* was acceptable, or

instrument recalibrated on 'date' with acceptable verification, etc.) must also be documented in the instrument records.

- When maintenance or repair is performed by an outside agency, service receipts detailing the service performed can be affixed into the logbooks adjacent to pages describing the maintenance performed. This stapled in page must be signed across the page entered and the logbook so that it is clear that a page is missing if only half a signature is found in the logbook.

If an instrument requires repair (subjected to overloading or mishandling), gives suspect results, or otherwise has shown to be defective or outside of specified limits it shall be taken out of operation and tagged as out-of-service or otherwise isolated until such a time as the repairs have been made and the instrument can be demonstrated as operational by calibration and/or verification or other test to demonstrate acceptable performance. The laboratory shall examine the effect of this defect on previous analyses.

In the event of equipment malfunction that cannot be resolved, service shall be obtained from the instrument vendor manufacturer, or qualified service technician, if such a service can be tendered. If on-site service is unavailable, arrangements shall be made to have the instrument shipped back to the manufacturer for repair. Back up instruments, which have been approved, for the analysis shall perform the analysis normally carried out by the malfunctioning instrument. If the back-up is not available and the analysis cannot be carried out within the needed timeframe, the samples shall be subcontracted.

At a minimum, if an instrument is sent out for service or transferred to another facility, it must be recalibrated and the laboratory MDL verified (using an MDLV) prior to return to lab operations.

20.3 Support Equipment

This section applies to all devices that may not be the actual test instrument, but are necessary to support laboratory operations. These include but are not limited to: balances, ovens, refrigerators, freezers, incubators, water baths, field sampling devices, temperature measuring devices, thermal/pressure sample preparation devices and volumetric dispensing devices if quantitative results are dependent on their accuracy, as in standard preparation and dispensing or dilution into a specified volume. All raw data records associated with the support equipment are retained to document instrument performance.

20.3.1 Weights and Balances

The accuracy of the balances used in the laboratory is checked every working day, before use. All balances are placed on stable counter tops.

Each balance is checked prior to initial serviceable use with at least two certified ASTM type 1 weights spanning its range of use (weights that have been calibrated to ASTM type 1 weights may also be used for daily verification). ASTM type 1 weights used only for calibration of other weights (and no other purpose) are inspected for corrosion, damage or nicks at least annually and if no damage is observed, they are calibrated at least every 5 years by an outside calibration laboratory. Any weights (including ASTM Type 1) used for daily balance checks or other purposes are recalibrated/recertified annually to NIST standards (this may be done internally if laboratory maintains "calibration only" ASTM type 1 weights).

All balances are serviced annually by a qualified service representative, who supplies the laboratory with a certificate that identifies traceability of the calibration to the NIST standards.

All of this information is recorded in logs, and the recalibration/recertification certificates are kept on file. Further details on balance calibrations and verifications can be found in SOP NC-QA-015 Support Equipment Calibration and Verification.

20.3.2 pH, Conductivity, and Turbidity Meters

The pH meters used in the laboratory are accurate to ± 0.1 pH units, and have a scale readability of at least 0.05 pH units. The meters automatically compensate for the temperature, and are calibrated with at least two working range buffer solutions before each use.

Conductivity meters used in the laboratory are capable of measuring conductivity with an error not exceeding 1% or one umhos/cm, whichever is greater. The meters are also calibrated before each use with a known standard.
Turbidity meters are also calibrated before each use. All of this information is documented in logs.

Consult pH, Conductivity, and Turbidity SOPs for further information.

20.3.3 Thermometers

All liquid in glass thermometers are calibrated on an annual basis with a NIST-traceable thermometer.

- If the temperature measuring device is used over a range of 10°C or less, then a single point verification within the range of use is acceptable;
- If the temperature measuring device is used over a range of greater than 10°C, then the verification must bracket the range of use.

IR thermometers, digital probes and thermocouples are calibrated quarterly.

The digital NIST thermometer is recalibrated every year (unless thermometer has been exposed to temperature extremes) by an approved outside service and the provided certificate of traceability is kept on file. The NIST thermometer(s) have increments of 0.01 degree (0.5 degree or less increments are required for drinking water microbiological laboratories), and have ranges applicable to method and certification requirements. The NIST traceable thermometer is used for no other purpose than to calibrate other thermometers.

All of this information is documented in spreadsheets, logsheets, and/or electronically. Monitoring method-specific temperatures, including incubators, heating blocks, water baths, and ovens, is documented on unit specific logsheets or in LIMS. More information on this subject can be found in SOP No. NC-QA-015 Support Equipment Calibration and Verification.

20.3.4 Refrigerators/Freezer Units, Water baths, Ovens and Incubators

The temperatures of all refrigerator units and freezers used for sample and standard storage are monitored each working day.

Ovens, water baths and incubators are monitored on days of use.

All of this equipment has a unique identification number, and is assigned a thermometer/continuous monitoring sensor for monitoring.

Sample storage refrigerator temperatures are kept between > 0ºC and ≤ 6 ºC.

Specific temperature settings/ranges for other refrigerators, ovens, water baths, and incubators can be found in method specific SOPs.

All of this information is documented on Daily Temperature Logsheets and/or electronically via LIMS or the Temperature Guard System and discussed in SOP No. NC-QA-015 Support Equipment Calibration and Verification.

20.3.5 Autopipettors, Dilutors, and Syringes

Mechanical volumetric dispensing devices including burettes (except Class A Glassware) and syringes are given unique identification numbers and the delivery volumes are verified gravimetrically, at a minimum, on a quarterly basis.

For those dispensers that are not used for analytical measurements, a label shall be applied to the device stating that it is not calibrated. Any device not regularly verified cannot be used for any quantitative measurements.

Micro-syringes are purchased from Hamilton Company. Each syringe is traceable to NIST. The laboratory keeps on file an “Accuracy and Precision Statement of Conformance” from Hamilton attesting established accuracy and considers the statement valid for the first six months. After six months, syringes that dispense volumes greater than 20 uL must be verified or replaced.

20.3.6 Field Sampling Devices (ISCO Auto Samplers)

Each Auto Sampler (ISCO) is assigned a unique identification number in order to keep track of the calibration. This number is also recorded on the sampling documentation.

The Auto Sampler is calibrated semiannually or as needed by setting the sample volume to 100ml and recording the volume received. The results are filed in a logbook/binder. The Auto Sampler is programmed to run three (3) cycles and each of the three cycles is measured into a graduated cylinder to verify 100ml are received.

If the RSD (Relative Standard Deviation) between the 3 cycles is greater than 20%, the procedure is repeated and if the result is still greater than 20%, then the Auto Sampler is taken out of service until it is repaired and calibration verification criteria can be met. The results of this check are kept in a logbook/binder.

20.4 Instrument Calibrations

Calibration of analytical instrumentation is essential to the production of quality data. Strict calibration procedures are followed for each method. These procedures are designed to

determine and document the method detection limits, the working range of the analytical instrumentation and any fluctuations that may occur from day to day.

Sufficient raw data records are retained to allow an outside party to reconstruct all facets of the initial calibration. Records contain, but are not limited to, the following: calibration date, method, instrument, analyst(s) initials or signatures, analysis date, analytes, concentration, response, and type of calibration (Avg RF, curve, or other calculations that may be used to reduce instrument responses to concentration.)

Sample results must be quantitated from the initial calibration and may not be quantitated from any continuing instrument calibration verification unless otherwise required by regulation, method or program.

If the initial calibration results are outside of the acceptance criteria, corrective action is performed and any affected samples are reanalyzed if possible. If the reanalysis is not possible, any data associated with an unacceptable initial calibration will be reported with appropriate data qualifiers (refer to Section 12).

Note: Instruments are calibrated initially and as needed after that and at least annually.

20.4.1 Calibration Standards

Calibration standards are prepared using the procedures indicated in the Reagents and Standards section of the determinative method SOP. If a reference method does not specify the number of calibration standards, a minimum number of calibration points as defined by the type of calibration curve. will be used.

Standards for instrument calibration are obtained from a variety of sources. All standards are traceable to national or international standards of measurement, or to national or international standard reference materials.

The lowest concentration calibration standard that is analyzed during an initial calibration must be at or below the stated reporting limit for the method based on the final volume of extract (or sample).

The other concentrations define the working range of the instrument/method or correspond to the expected range of concentrations found in actual samples that are also within the working range of the instrument/method. Results of samples not bracketed by initial instrument calibration standards (within calibration range to at least the same number of significant figures used to report the data) must be reported as having less certainty, e.g., defined qualifiers or flags (additional information may be included in the case narrative). The exceptions to these rules. ICP and ICPMS methods which define the working range with periodic linear dynamic range studies, rather than through the range of concentrations of daily calibration standards.

All initial calibrations are verified with a standard obtained from a second source and traceable to a national standard, when available (or vendor certified different lot if a second source is not available). For unique situations, where no other source or lot is available, a standard made by a different analyst at a different time or a different preparation would be considered a second

source. This verification occurs immediately after the calibration curve has been analyzed, and before the analysis of any samples.

20.4.1.1 Calibration Verification

The calibration relationship established during the initial calibration must be verified initially and at least daily as specified in the laboratory method SOPs in accordance with the referenced analytical methods and in the 2009 and 2016 TNI Standard. The process of calibration verification applies to both external standard and internal standard calibration techniques, as well as to linear and non-linear calibration models. Initial calibration verification (ICV) is with a standard source secondary (second source standard) to the calibration standards, but continuing calibration verifications (CCV) may use the same source standards as the calibration curve.

Note: The process of calibration verification referred to here is fundamentally different from the approach called "calibration" in some methods. As described in those methods, the calibration factors or response factors calculated during calibration are used to update the calibration factors or response factors used for sample quantitation. This approach, while employed in other EPA programs, amounts to a daily single-point calibration.

All target analytes and surrogates, including those reported as non-detects, must be included in periodic calibration verifications for purposes of retention time confirmation and to demonstrate that calibration verification criteria are being met, i.e., RPD, per 2009 and 2016 TNI Std. EL-V1M4 Sec. 1.7.2.

All samples must be bracketed by periodic analyses of standards that meet the QC acceptance criteria (e.g., calibration and retention time). The frequency is found in the determinative methods or SOPs.

Note: If an internal standard calibration is being used then bracketing calibration verification standards may not be required, only daily verifications are needed. The results from these verification standards must meet the calibration verification criteria and the retention time criteria (if applicable).

Generally, the calibrations must be verified by an ICV analyzed immediately following initial calibration and before sample analysis. The ICV may be used as the first bracketing CCV, if criteria for both are met.

A continuing instrument calibration verification (CCV) is generally analyzed at the beginning of each 12-hour analytical shift during which samples are analyzed. The 12-hour analytical shift begins with the injection of the calibration verification standard (or the MS tuning standard in MS methods). The shift ends after the completion of the analysis of the last sample, QC, or standard that can be injected within 12-hours of the beginning of the shift. For methods that have quantitation by external calibration models, a CCV is analyzed at the end of each analytical sequence. Some methods have more frequent CCV requirements. See specific SOPs. Most inorganic methods require the CCV to be analyzed after ever 10 samples or injections, including matrix or batch QC samples.

Note: If an internal standard calibration is being used (e.g., GCMS) then bracketing standards are not required, only daily verifications are needed, except as specified by program or method requirements.

If the results of a CCV are outside the established acceptance criteria and analysis of a second consecutive (and immediate) CCV fails to produce results within acceptance criteria, corrective action shall be performed. Once corrective actions have been completed and documented, the laboratory shall demonstrate acceptable instrument / method performance by analyzing two consecutive CCVs, or a new initial instrument calibration shall be performed.

Sample analyses and reporting of data may not occur or continue until the analytical system is calibrated or calibration verified. However, data associated with an unacceptable calibration verification may be fully useable reported based upon discussion and approval of the client under the following special conditions:

a). when the acceptance criteria for the CCV are exceeded high (i.e., high bias) and the associated samples within the batch are non-detects, then those non-detects may be reported with a case narrative comment explaining the high bias. Otherwise the samples affected by the unacceptable CCV shall be re-analyzed after a new calibration curve has been established, evaluated and accepted; or

b). when the acceptance criteria for the CCV are exceeded low (i.e., low bias), those sample results may be reported if they exceed a maximum regulatory limit/decision level. Otherwise the samples affected by the unacceptable CCV shall be re-analyzed after a new calibration curve has been established, evaluated and accepted.

Samples reported by the 2 conditions identified above will be appropriately flagged.

20.4.1.2 Verification of Linear and Non-Linear Calibrations

Calibration verification for calibrations involves the calculation of the percent drift or the percent difference of the instrument response between the initial calibration and each subsequent analysis of the verification standard. (These calculations are available in the laboratory method SOPs.) Verification standards are evaluated based on the % Difference from the average CF or RF of the initial calibration or based on % Drift or % Recovery if a linear or quadratic curve is used.

Regardless of whether a linear or non-linear calibration model is used, if initial verification criterion is not met, then no sample analyses may take place until the calibration has been verified or a new initial calibration is performed that meets the specifications listed in the method SOPs. If the calibration cannot be verified after the analysis of a single verification standard, then adjust the instrument operating conditions and/or perform instrument maintenance, and analyze another aliquot of the verification standard. If the calibration cannot be verified with the second standard, then a new initial calibration is performed.

- When the acceptance criteria for the calibration verification are exceeded high, i.e., high bias, and there are associated samples that are non-detects, then those non-detects may be reported. Otherwise, the samples affected by the unacceptable calibration verification shall be reanalyzed after a new calibration curve has been established, evaluated and accepted.

- When the acceptance criteria for the calibration verification are exceeded low, i.e., low bias, those sample results may be reported if they exceed a maximum regulatory limit/decision level. Otherwise, the samples affected by the unacceptable verification shall be reanalyzed after a new calibration curve has been established, evaluated and accepted. Alternatively, a reporting limit standard may be analyzed to demonstrate that the laboratory can still support non-detects at their reporting limit. For Ohio VAP samples, results may not be reported when calibration verifications fail the lower limit criterion.

20.5 Tentatively Identified Compounds (TICs) – GC/MS Analysis

For samples containing components not associated with the calibration standards, a library search may be made for the purpose of tentative identification. The necessity to perform this type of identification will be determined by the purpose of the analyses being conducted. For example, the RCRA permit or waste delisting requirements may require the reporting of non-target analytes. Only after visual comparison of sample spectra with the nearest library searches may the analyst assign a tentative identification. Data system library search routines should not use normalization routines that would misrepresent the library or unknown spectra when compared to each other. Additional details are provided in NDSC Document No. CA-Q-QM-001, Tentatively Identified Compounds (TICS) - GCMS Analysis

Note: If the TIC compound is not part of the client target analyte list but is calibrated by the laboratory and is both qualitatively and/or quantitatively identifiable, it should not be reported as a TIC. If the compound is reported on the same form as true TICs, it should be qualified and/or narrated that the reported compound is qualitatively and quantitatively (if verification in control) reported compared to a known standard that is in control (where applicable).

20.6 GC/MS Tuning

Prior to any GC/MS analytical sequence, including calibration, the instrument parameters for the tune and subsequent sample analyses within that sequence must be set.

Prior to tuning/auto-tuning the mass spectrometer, the parameters may be adjusted within the specifications set by the manufacturer or the analytical method. These generally do not need any adjustment but it may be required based on the current instrument performance. If the tune verification does not pass it may be necessary to clean the source or perform additional maintenance. Any maintenance is documented in the maintenance log.

Table 20-1. Example: Instrumentation List

Equipment Instrument	Manufacturer	Model Number	Serial Number	Year in Service	Condition When Received
MSV UX2 MSD (screen)	Hewlett Packard	5972	US00029070	1992	NEW
MSV UX2 Concentrator	OI Analytical	4560	J615460591		
MSV UX2 Sampler	Varion	Archon	12175		
MSV HP6 MSD (screen)	Hewlett Packard	5973	US00005571	1998	NEW

Equipment Instrument	Manufacturer	Model Number	Serial Number	Year in Service	Condition When Received
MSV HP6 Concentrator	OI Analytical	4560	M943460127		
MSV HP6 Sampler	OI Analytical	4552	12258		
MSV UX7 MSD (screen)	Hewlett Packard	5973	US00010937	1998	NEW
MSV UX7 Concentrator	OI Analytical	4560	N251460461		
MSV UX7 Sampler	Dynatech Precision Sampling Corp.	Archon 5100A	12019		
MSV UX8 MSD	Hewlett Packard	6890	US00027773	1999	NEW
MSV UX8 Concentrator	OI Analytical	Eclipse 4660	B444466152P		
MSV UX8 Sampler	OI Analytical	4552	14092		
MSV UX9 MSD	Hewlett Packard	5973	US00028329	2000	NEW
MSV UX9 Concentrator	OI Analytical	4560	M946460832		
MSV UX9 Sampler	Environmental Sample Technology, Inc.	Archon	13667		
MSV UX10 MSD	Hewlett Packard	5973	US00032072	2000	NEW
MSV UX10 Concentrator	OI Analytical	Eclipse 4660	BETA 6		
MSV UX10 Sampler	OI Analytical	4552	012058		
MSV UX11 MSD	Agilent	5973 Network	US00038093	2000	NEW
MSV UX11 Concentrator	OI Analytical	4560	K811460270		
MSV UX11 Sampler	OI Analytical	4552	13408		
MSV UX12 MSD	Agilent	5973 Network	US10202133	2002	NEW
MSV UX12 Concentrator	OI Analytical	4560	M041460393		
MSV UX12 Sampler	Varion	Archon	12151		
MSV UX14 MSD	Agilent	5973 Inert	CN10340027	2003	NEW
MSV UX14 Concentrator	OI Analytical	4660	D829466914P		
MSV UX14 Sampler	OI Analytical	4552	14092		
MSV UX15 MSD	Agilent	5973 Inert	CN10515062	2005	NEW
MSV UX15 Concentrator	OI Analytical	Eclipse 4660	C511466149F		
MSV UX15 Sampler	OI Analytical	4552	14368		
MSV UX16 MSD	Agilent	5973 Inert	CN10539865	2005	NEW
MSV UX16 Concentrator	OI Analytical	Eclipse 4660	D539466261P		
MSV UX16 Sampler	OI Analytical	4552	14519		
MSV UX17 MSD	Agilent	5975 Inert	US10831043	2012	NEW

Equipment Instrument	Manufacturer	Model Number	Serial Number	Year in Service	Condition When Received
MSV UX17 Concentrator	OI Analytical	Eclipse 4660	H224455292P		
MSV UX17 Sampler	OI Analytical	4552	US12160002		
MSV UX18 MSD	Hewlett Packard	5973	US00020913	2013	NEW
MSV UX18 Concentrator	OI Analytical	4560	N213460621		
MSV UX18 Sampler	Varion	Archon	12174		
MSV UX19 MS	Agilent	5977B	US1813M021	2018	NEW
MSV UX19 GC	Agilent	7890B	CN1750321		
MSV UX19 Concentrator	OI Analytical	4760	A818447017		
MSV UX19 Sampler	OI Analytical	4100	D818410016		
MSV UX20 MS	Agilent	5973 Inert	US33220199	2021	USED
MSV UX20 GC	Agilent	6890N	CN10412013		
MSV UX20 Concentrator	OI Analytical	4560	N213460321		
MSV UX20 Sampler					
MSV UX21 MS	Agilent	5973 Network	US10461280	2021	NEW
MSV UX21 GC	Agilent	6890N	US10144027		
MSV UX21 Concentrator	OI Analytical	4660	E002466503P		
MSV UX21 Sampler					
MSV UX22 MS	Agilent	5977B	US2112R016	2021	NEW
MSV UX22 GC	Agilent	7890A	CN10833028		
MSV UX22 Concentrator	EST	Evolution2	EV20143032621		
MSV UX22 Sampler	EST				
MSS HP7 MSD	Hewlett-Packard	5973-6890	US71190756	1998	NEW
MSS HP7 Sampler	Hewlett-Packard	6890 Series	US64900400		
MSS HP7 Tower	Hewlett-Packard	6890 Series	US64900400		
MSS HP7 GC	Hewlett-Packard	G1530A	US00009247		
MSS HP9 MSD	Hewlett-Packard	5973-6890	US91422379	2000	NEW
MSS HP9 Sampler	Hewlett-Packard	7683	US94606476		
MSS HP9 Tower	Hewlett-Packard	7683	US93408794		
MSS HP9 GC	ALS	Ready	US00027943		
MSS HP10 MSD	Agilent	5973	US33220074	2003	NEW
MSS HP10 Sampler	Agilent	7683 Series	US83501650		
MSS HP10 Tower	Agilent	7683 Series	CN33832656		
MSS HP10 GC	Agilent	6890N	CN10340002		

Equipment Instrument	Manufacturer	Model Number	Serial Number	Year in Service	Condition When Received
MSS AG2 MSD	Agilent	5975C Inert XL	US71235692	2007	NEW
MSS AG2 Sampler	Agilent	7683B Series	US91204712		
MSS AG2 Tower	Agilent	7683B Series	CN53827833		
MSS AG2 GC	Agilent	7890A	CN10721110		
MSS AG3 MSD	Agilent	5977A	US12203016	2014 in use:2017	NEW
MSS AG3 Sampler	Agilent	7693	CN11120088		
MSS AG3 Tower	Agilent	7693	CN11120088		
MSS AG3 GC	Agilent	7890A	CN10501006		
GC A	Agilent	6890 FID	US10402056	2004	NEW
GC O	Hewlett Packard	6890 FID	US00007206	1997	NEW
GC Y	Hewlett Packard	6890 FID	US10337062	2003	NEW
GC Z	Agilent	6890 EPC & PDD/FID	10205072	2000	NEW
GC P1	Hewlett Packard	6890 EPC & Dual ECD Y-Splitter	US00023208	1998	NEW
GC P1	Hewlett Packard	Sampler	US83401589	1998	NEW
GC P1	Hewlett Packard	Tower	CN14422923	1998	NEW
GC P2 (Screening only)	Hewlett Packard	6890 EPC & Dual ECD Y-Splitter	US83802337	1998	NEW
GC P2	Hewlett Packard	Sampler	US93806108	1998	NEW
GC P2	Hewlett Packard	Tower	US83602262	1998	NEW
GC P3	Hewlett Packard	6890 EPC & Dual ECD Y-Splitter	US00023674	1998	NEW
GC P3	Hewlett Packard	Sampler	US92205419	1998	NEW
GC P3	Hewlett Packard	Tower	CN42637504	1998	NEW
GC P4	Hewlett Packard	6890 EPC & Dual ECD Y-Splitter	US00029531	1999	NEW
GC P4	Hewlett Packard	Sampler	CN51232596	1999	NEW
GC P4	Hewlett Packard	Tower	US82401457	1999	NEW
GC P5	Hewlett Packard	6890 EPC & Dual ECD Y-Splitter	US00029508	2010	NEW
GC P5	Hewlett Packard	Sampler	CN33826455	2010	NEW
GC P5	Hewlett Packard	Tower	US92407745 US00311160	2010	NEW
GC P6	Hewlett Packard	6890 EPC & Dual ECD Y-Splitter	US00032848	2000	NEW
GC P6	Hewlett Packard	Sampler	CN43130187	2000	NEW
GC P6	Hewlett Packard	Tower	CN14422929 US82401457	2000	NEW
GC P9 (screening only)	Agilent	6890 EPC & Dual ECD Y-Splitter	US10205045	2005	NEW

Equipment Instrument	Manufacturer	Model Number	Serial Number	Year in Service	Condition When Received
GC P9	Agilent	Sampler	US01708111	2005	NEW
GC P9	Agilent	Tower	CN14523156	2005	NEW
GC P10	Agilent	6890 EPC & Dual ECD Y-Splitter	US91907177	1999	NEW
GC P10	Agilent	Sampler	CN14920067	1999	NEW
GC P10	Agilent	Tower	US83802337	1999	NEW
GC P11	Agilent	6890N EPC & Dual ECD Y-Splitter	CN10517088	2004	NEW
GC P11	Agilent	Sampler	US12411936	2004	NEW
GC P11	Agilent	Tower	CN43220375	2004	NEW
GC P12	Agilent	6890N EPC & Dual ECD Y-Splitter	CN10512025	2005	NEW
GC P12	Agilent	Sampler	US92205419	2005	NEW
GC P12	Agilent	Tower	CN51124095	2005	NEW
GC P13	Agilent	6890N EPC & Dual ECD Y-Splitter	CN10435032	2004	NEW
GC P13	Agilent	Sampler	CN42429315	2004	NEW
GC P13	Agilent	Tower	CN51825000	2004	NEW
GC P14	Agilent	7890 EPC & Dual ECD Y-Splitter	CN10281044	2012	NEW
GC P14	Agilent	Sampler	CN10220022	2012	NEW
GC P14	Agilent	Tower - front	CN10290167	2012	NEW
GC P14	Agilent	Tower - rear	CN10290169	2012	NEW
GC P15	Agilent	6890N EPC & Dual ECD Y-Splitter	CN10427010	2012	NEW
GC P15	Agilent	Sampler	US11911568	2012	NEW
GC P15	Agilent	Tower	CN43220375	2012	NEW
GC P16	Agilent	6890 EPC & Dual ECD Y-Splitter	US00025858	2014	Borrowed
GC P16	Agilent	Sampler	US83001373	2014	Borrowed
GC P16	Agilent	Tower	CN14422847	2014	Borrowed
GC P18	Agilent	6890 EPC & Dual ECD Y-Splitter	US00006438	2015	NEW
GC P18	Agilent	Sampler	US93806073	2015	NEW
GC P18	Agilent	Tower	CN53827833	2015	NEW
GC P19	Agilent	7890A Agilent	CN12021107	2019	NEW
GC P19	Agilent	G2614A Sampler	US13312527	2019	NEW
GC P19	Agilent	7683B Tower	CN71740390	2019	NEW
GC P20	Agilent	GC	US10130042	2019	USED
GC P20	Agilent	Sampler	US13312527	2019	USED
GC P20	Agilent	Tower	US12419321	2019	USED

Equipment Instrument	Manufacturer	Model Number	Serial Number	Year in Service	Condition When Received
GC HPLC L2	Agilent	HPLC 1100	US82404153	1998	NEW
GC N	Agilent	7890 Atomic Fluorescence	CN10820009	2008	NEW
Metals I-12	Thermo	Trace Analyzer 6500 Duo Ash	ICP-20101711	2013	NEW
I-12 Sampler	Elemental Scientific	SC-0500-04	FST04-TSP-090815		
I-12 Pump	Elemental Scientific	FVA	FVA-100203		
Metals I-9	Thermo	Trace Analyzer 6500 Duo Ash	ICP 20102403	2010	NEW
I-9 Sampler	Elemental Scientific	SC-4DX	X4DX-HS-TSP-16-100210		
I-9 Pump	Elemental Scientific	FVA	FVA-090915		
Metals I-14	Agilent	7800	JP18131103	2018	NEW
I-14 Sampler	Agilent	SPS4	AU17494204		
Metals I-10	Agilent	7700 ICPMS Series	JP124521145	2013	NEW
I-10 Sampler	Agilent	ASX-500	US121435A520		
Metals H2	Leeman	Hydra II AA	5VV5ZQ1	2015	NEW
H2 Sampler	Teldyne Leeman	Hydra II	4090		
Metals H3	Leeman	Hydra II AA	17010003	2015	NEW
H3 Sampler	Teldyne Leeman	Hydra II	4055		
WC Autotitrator (Severus)	Mantech	PC-1000-102/E	MT-1G8-798	2018	NEW
WC Severus Pump	Mantech	PC-1300-475	MT-117-1019		
WC Severus Burette	Mantech	PC-1000-1040	MT-1G8-219		
WC Severus Sampler	Mantech	Automatic 73	191C8020		
WC BOD (Bugsy) Sampler	Mantech	AutoMax 122	261H3N236	2013	NEW
WC Bugsy Pump	Mantech	PC-1000-443	MT-1J3-182		
WC Bugsy Pump	Mantech	PC-1000-408	MT-1K3-508		
WC Bugsy Pump	Mantech	PC-1000-475	MT-1J3-280		
WC Bugsy Interface	Mantech	PB-10030	MT-1A4-169		
WC Spec (Oscar)	Genesys	Spectronic 20	3SGK137005	2016	NEW
WC Spec (Ernie)	Genesys	Spectronic 20	3SGL226006	2008	NEW
WC IC (Veronica)	Dionex	ICS 2100	12031443	2012	NEW
WC Veronica Sampler	Thermo Sci / Dionex	AS-AP	12050895		
WC IC (Thor)	Dionex	Dionex Integrion	16081629	2016	NEW
WC Thor Sampler	Thermo Sci / Dionex	AS-AP	16110203		
WC Discrete Analyzer (Maggie)	Systea	Easy Chem Plus	07004	2013	USED

Equipment Instrument	Manufacturer	Model Number	Serial Number	Year in Service	Condition When Received
WC TOC (Clark)	OI Analytical	1030W	P428730168/ PARA	2014	NEW
WC Clark Sampler	OI Analytical	1088 AS	E427788106		
WC Block Digester (Larry)	Andrews	110-40-PA	None on unit	1999	NEW
WC Block Digester (Moe)	Andrews	110-40-PA	None on unit	1999	NEW
WC Block Digester (Curly)	Andrews	110-40-PA	None on unit	1999	NEW
WC Block Digester (Carol)	Lachat	BD46 TKN	00000993	1992	NEW
WC Spec (Snuffleupagus)	Genesys	Spectronic 20	3SGU022007	2016	NEW
WC DO Meter	Mantech	YSI 1500	13D 100737	2013	NEW
WC Conductivity (Xavier)	Orion	Star A112	J14311	2017	NEW
WC Cyanide Analyzer (CNthia)	Astoria Pacific	rAPID-T	4600-1035	2017	NEW
WC Cyanide Hotblock (Phil)	Simple Dist Micro	C8000	2017MDISW137	2017	NEW
WC Cyanide Hotblock (Lil)	Simple Dist Micro	C8000	2017MDISW136	2017	NEW
WC Flashpoint (Whitey)	Herzog	HFP 339	073390084	2007	NEW
WC pH Meter (Randolph)	Orion	320	020032	2007	NEW
WC Sulfide Distillation	Westco	Easy Dist 483-B000-01	1193	2008	NEW
WC Conductivity Screening Meter (Myron II)	Myron L Company	TechPro II	T3201412	2018	NEW
COD Block (W1)	Hach	16500-10	870512456	1998	USED
COD Block (W3)	Hach	45600	900201914	1998	USED
COD Block (W4)	Hach	16500-10	890512473	1998	USED
COD Block (W5)	Biosciences	100 003	COD-B0075	2000	NEW
COD Block (W6)	Hach	16500-10	880911479	2018	USED
MBAS Shaker	Eberbach	6010	049817	2015	USED

Note: Laboratory instrumentation, model numbers, and serial numbers are subject to change without notice.

SECTION 21. MEASUREMENT TRACEABILITY

21.1 Overview

Traceability of measurements shall be assured using a system of documentation, calibration, and analysis of reference standards. Laboratory equipment that are peripheral to analysis and whose calibration is not necessarily documented in a test method analysis or by analysis of a reference standard shall be subject to ongoing certifications of accuracy. At a minimum, these must include procedures for checking specifications of ancillary equipment: balances, thermometers, temperature, Deionized (DI) and Reverse Osmosis (RO) water systems, automatic pipettes and other volumetric measuring devices. (Refer to Section 20.3). With the exception of Class A Glassware, quarterly accuracy checks are performed for all mechanical volumetric devices. Micro syringes used to dispense volumes greater than 20 uL are verified at least semi-annually or disposed of after 6 months of use. Wherever possible, subsidiary or peripheral equipment is checked against standard equipment or standards that are traceable to national or international standards. Class A Glassware should be routinely inspected for chips, acid etching or deformity (e.g., bent needle). If the Class A glassware is suspect, the accuracy of the glassware will be assessed prior to use.

21.2 NIST-Traceable Weights and Thermometers

Reference standards of measurement shall be used for calibration only and for no other purpose, unless it can be shown that their performance as reference standards would not be invalidated.

For NIST-traceable weights and thermometers, the laboratory requires that all calibrations be conducted by a calibration laboratory accredited by A2LA, NVLAP (National Voluntary Laboratory Accreditation Program), or another accreditation organization that is a signatory to a MRA (Mutual Recognition Arrangement) of one or more of the following cooperations – ILAC (International Laboratory Accreditation Cooperation) or APLAC (Asia–Pacific Laboratory Accreditation Cooperation). A calibration certificate and scope of accreditation is kept on file at the laboratory.

21.3 Reference Standards / Materials

Reference standards/materials, where commercially available, are traceable to certified reference materials. Commercially prepared reference standards, to the extent available, are purchased from vendors that are accredited to ISO Guide 34 and ISO/IEC Guide 17025. All reference standards from commercial vendors shall be accompanied with a certificate that includes at least the following information:

- Manufacturer
- Analytes or parameters calibrated
- Identification or lot number
- Calibration method
- Concentration with associated uncertainties
- Purity

If a standard cannot be purchased from a vendor that supplies a Certificate of Analysis, the purity of the standard is documented by analysis. The receipt of all reference standards must be documented. Reference standards are labeled with a unique standard identification number and

expiration date. All documentation received with the reference standard is retained as a QC record and references the standard identification number.

All reference, primary and working standards/materials, whether commercially purchased or laboratory prepared, must be checked regularly to ensure that the variability of the standard or material from the true value does not exceed method requirements. The accuracy of calibration standards is checked by comparison with a standard from a second source. In cases where a second standard manufacturer is not available, a vendor certified different lot is acceptable for use as a second source. For unique situations, where no other source or lot is available, a standard made by a different analyst would be considered a second source. The appropriate Quality Control (QC) criteria for specific standards are defined in laboratory SOPs. In most cases, the analysis of an Initial Calibration Verification (ICV) or LCS (where there is no sample preparation) is used as the second source confirmation. These checks are generally performed as an integral part of the analysis method (e.g. calibration checks, laboratory control samples).

All standards and materials must be stored and handled according to method or manufacturer's requirements in order to prevent contamination or deterioration. Refer to the Environmental Health & Safety Manual or laboratory SOPs. For safety requirements, please refer to method SOPs and the laboratory's Environmental Health and Safety Manual.

Standards and reference materials shall not be used after their expiration dates unless their reliability is verified by the laboratory and their use is approved by the Quality Assurance Manager. The laboratory must have documented contingency procedures for re-verifying expired standards.

21.4 Documentation and Labeling of Standards, Reagents, and Reference Materials

Reagents must be at a minimum the purity required in the test method. The date of reagent receipt and the expiration date are documented. The lots for most of the common solvents and acids are tested for acceptability prior to company-wide purchase. [Refer to NDSC Document No. CA-Q-S-001 Solvent and Acid Lot Testing and Approval

All manufacturer or vendor supplied Certificate of Analysis or Purity must be retained, stored appropriately, and readily available for use and inspection. These records are maintained in LIMS or in binders in the laboratory departments. Records must be kept of the date of receipt and date of expiration of standards, reagents and reference materials. In addition, records of preparation of laboratory standards, reagents, and reference materials must be retained, stored appropriately, and be readily available for use and inspection. For detailed information on documentation and labeling, please refer to SOP NC-QA-017 Standards and Reagents.

Commercial materials purchased for preparation of calibration solutions, spike solutions, etc.., are usually accompanied with an assay certificate or the purity is noted on the label. If the assay purity is 96% or better, the weight provided by the vendor may be used without correction. If the assay purity is less than 96% a correction will be made to concentrations applied to solutions prepared from the stock commercial material. Blended gas standard cylinders use a nominal concentration if the certified value is within +/-15%, otherwise the certified values is used for the canister concentration.

21.4.1 All standards, reagents, and reference materials must be labeled in an unambiguous manner. Standards are logged into the laboratory's LIM system, and are assigned a unique identification number. The following information is typically recorded in the electronic database within the LIMS.

- Standard ID
- Description of Standard
- Department
- Preparer's name
- Final volume and number of vials prepared
- Solvent type and lot number
- Preparation Date
- Expiration Date
- Standard source type (stock or daughter)
- Standard type (spike, surrogate, other)
- Parent standard ID (if applicable)
- Parent Standard Analyte Concentration (if applicable)
- Parent Standard Amount used (if applicable)
- Component Analytes
- Final concentration of each analyte
- Comment box (text field)

Records are maintained electronically for standard and reference material preparation. These records show the traceability to purchased stocks or neat compounds. These records also include method of preparation, date of preparation, expiration date and preparer's name or initials. Preparation procedures are provided in the Method SOPs.

21.4.2 All standards, reagents, and reference materials must be clearly labeled with a minimum of the following information:

- Expiration Date (include prep date for reagents)
- Standard ID from LIMS
- Special Health/Safety warnings if applicable

Records must also be maintained of the date of receipt for commercially purchased items or date of preparation for laboratory prepared items. Special Health/Safety warnings must also be available to the analyst. This information is maintained in the analytical SOPs.

21.4.3 In addition, the following information may be helpful:

- Date opened (for multi-use containers, if applicable)
- Description of standard (if different from manufacturer's label or if standard was prepared in the laboratory)
- Recommended Storage Conditions

- Concentration (if applicable)
- Initials of analyst preparing standard or opening container

All containers of prepared reagents must include an expiration date and an ID number to trace back to preparation.

Procedures for preparation of reagents can be found in the Method SOPs.

Standard ID numbers must be traceable through associated logbooks, worksheets and preparation/analytical batch records.

All reagents and standards must be stored in accordance to the following priority: 1) with the manufacturer's recommendations; 2) with requirements in the specific analytical methods as specified in the laboratory SOP.

SECTION 22. SAMPLING

22.1 Overview

The laboratory provides sampling services. Sampling procedures are described in SOP NC-SC-006, Sample Procurement Protocol.

22.2 Sampling Containers

The laboratory offers clean sampling containers for use by clients. These containers are obtained from reputable container manufacturers and meet EPA specifications as required. Certificates of cleanliness for bottles and preservatives are provided by the supplier and are maintained at the laboratory. Alternatively, the certificates may be maintained by the supplier and available to the laboratory on-line.

22.2.1 Preservatives

Upon request, preservatives are provided to the client in pre-cleaned sampling containers. In some cases containers may be purchased pre-preserved from the container supplier. Whether prepared by the laboratory or bought pre-preserved, the grades of the preservatives are at a minimum:

- Hydrochloric Acid – Reagent ACS (Certified VOA Free) or equivalent
- Methanol – Purge and Trap grade
- Nitric Acid – Instra-Analyzed or equivalent
- Sodium Bisulfate – ACS Grade or equivalent
- Sodium Hydroxide – Instra-Analyzed or equivalent
- Sulfuric Acid – Instra-Analyzed or equivalent
- Sodium Thiosulfate – ACS Grade or equivalent

22.3 Definition of Holding Time

The date and time of sampling documented on the COC form establishes the day and time zero. As a general rule, when the maximum allowable holding time is expressed in days (e.g., 14 days, 28 days), the holding time is based on calendar day measured. Holding times expressed in hours (e.g., 6 hours, 24 hours, etc.) are measured from date and time zero. Holding times for analysis include any necessary reanalysis. However, there are some programs that determine holding time compliance based on the date and specific time of analysis compared to the time of sampling regardless of how long the holding time is.

22.4 Sampling Containers, Preservation Requirements, Holding Times

The preservation and holding time criteria specified in the laboratory SOPs are derived from the source documents for the methods. If method required holding times or preservation requirements are not met, the reports will be qualified using a flag, footnote or case narrative. As soon as possible or "ASAP" is an EPA designation for tests for which rapid analysis is advised, but for which neither EPA nor the laboratory have a basis for a holding time.

22.5 Sample Aliquots / Subsampling

Taking a representative sub-sample from a container is necessary to ensure that the analytical results are representative of the sample collected in the field. The size of the sample container, the quantity of sample fitted within the container, and the homogeneity of the sample need consideration when sub-sampling for sample preparation. It is the laboratory's responsibility to take a representative subsample or aliquot of the sample provided for analysis.

Analysts should handle each sample as if it is potentially dangerous. At a minimum, safety glasses, gloves, and lab coats must be worn when preparing aliquots for analysis.

Guidelines on taking sample aliquots and subsampling are located in each analytical SOP and in Subsampling SOP NC-OP-046.

23 HANDLING OF SAMPLES

It is the responsibility of the client to send us representative and/or homogeneous and properly preserved samples of the system from which they are drawn. The laboratory assumes that all multiple sample containers with the same designator/description and bottle type contain a homogeneous, representative sample.

The laboratory provides the appropriate sample containers, required preservative, chain-of-custody (COC) forms, shipping containers, labels, and custody seals. The laboratory also provides trip blanks and analyte-free water for field blanks. Preparation of methanol containers for field preservation of volatile soil samples is available.

Sample management procedures at the laboratory ensure that sample integrity and custody are maintained and documented from sampling/receipt through disposal.

23.1 Chain of Custody (COC)

The COC form is the written documented history of any sample and is initiated when bottles are sent to the field, or at the time of sampling. This form is completed by the sampling personnel and accompanies the samples to the laboratory where it is received and stored under the laboratory's custody. The purpose of the COC form is to provide a legal written record of the handling of samples from the time of collection until they are received at the laboratory. It also

serves as the primary written request for analyses from the client to the laboratory. The COC form acts as a purchase order for analytical services when no other contractual agreement is in effect.

23.1.1 Field Documentation

The information the sampler needs to provide at the time of sampling on the container label is:

- Sample identification
- Date and time
- Preservative

During the sampling process, the COC form is completed and must be legible. This form includes information such as:

- Client name, address, phone number and fax number (if available)
- Project name and/or number
- The sample identification
- Date, time and location of sampling
- Sample collectors name
- The matrix description
- The container description
- The total number of each type of container
- Preservatives used
- Analysis requested
- Requested turnaround time (TAT)
- Any special instructions
- Purchase Order number or billing information (e.g. quote number) if available
- The date and time that each person received or relinquished the sample(s), including their signed name.

When the sampling personnel deliver the samples directly to the laboratory personnel, the samples are stored in a cooler with ice, as applicable, and remain solely in the possession of the client's field technician until the samples are delivered to the laboratory personnel. The sample collector must assure that each container is in his/her physical possession or in his/her view at all times, or stored in such a place and manner to preclude tampering. The field technician relinquishes the samples in writing on the COC form to the sample control personnel at the laboratory or to a laboratory courier. When sampling personnel deliver the samples through a common carrier (Fed-Ex, UPS), the CoC relinquished date/time is completed by the field personnel and samples are released to the carrier. Samples are only considered to be received by the laboratory when personnel at the fixed laboratory facility have physical contact with the samples.

Note: Independent couriers are not required to sign the COC form. The COC is usually kept in the sealed sample cooler. The COC is stored with project information and the report.

23.1.2 **Legal / Evidentiary Chain-of-Custody**

If samples are identified for legal/evidentiary purposes on the COC, login will complete the custody seal retain the shipping record with the COC, and initiate an internal COC for laboratory use by analysts and a sample disposal record.

23.2 Sample Receipt

Samples are received at the laboratory by designated sample receiving personnel and a unique laboratory project identification number is assigned. Each sample container shall be assigned a unique sample identification number that is cross-referenced to the client identification number such that traceability of test samples is unambiguous and documented. Each sample container is affixed with a durable sample identification label. Sample acceptance, receipt, tracking and storage procedures are detailed in SOP NC-SC-005, Sample Receiving, and summarized in the following sections.

23.2.1 **Laboratory Receipt**

23.2.1.1 When samples arrive at the laboratory, sample receiving personnel inspect the coolers and samples. The integrity of each sample must be determined by comparing sample labels or tags with the COC and by visual checks of the container for possible damage. Any non-conformance, irregularity, or compromised sample receipt must be documented on a Cooler Receipt Form (CRF) and brought to the immediate attention of the client. The COC, shipping documents, documentation of any non-conformance, irregularity, or compromised sample receipt, record of client contact, and resulting instructions become part of the project record.

Sample Receiving personnel check and document preservation of non-volatile liquid samples in LIMS and before they are released to the laboratory for testing or placed into storage. Any checks of volatile samples and samples for oil and grease are performed and documented at the time of analysis.

23.3 Sample Acceptance Policy

The laboratory has a written sample acceptance policy outlined in SOP NC-SC-005 Sample Receiving that clearly outlines the circumstances under which samples shall be accepted or rejected. These include:

- a COC filled out completely;
- samples must be properly labeled;
- proper sample containers with adequate volume for the analysis (Sampling Guide) and necessary QC;
- samples must be preserved according to the requirements of the requested analytical method (Sampling Guide);
- sample holding times must be adhered to (Sampling Guide);
- All samples submitted for water/solid Volatile Organic analyses must have a Trip Blank submitted at the same time
- the project manager will be notified if any sample is received in damaged condition.

Data from samples which do not meet these criteria are flagged and the nature of the variation from policy is defined.

23.3.1 After inspecting the samples, the sample receiving personnel sign and date the COC form, make any necessary notes of the samples' conditions and store them in appropriate refrigerators or storage locations.

23.3.2 Any deviations from these checks that question the suitability of the sample for analysis, or incomplete documentation as to the tests required will be resolved by consultation with the client. If the sample acceptance policy criteria are not met, the laboratory shall either:

- Retain all correspondence and/or records of communications with the client regarding the disposition of rejected samples, or
- Fully document any decision to proceed with sample analysis that does not meet sample acceptance criteria.

Once sample acceptance is verified, the samples are logged into the LIMS according SOP NC-SC-005.

23.4 Sample Storage

In order to avoid deterioration, contamination or damage to a sample during storage and handling, from the time of receipt until all analyses are complete, samples are stored in refrigerators, freezers or protected locations suitable for the sample matrix. Metals samples may be unrefrigerated. In addition, samples to be analyzed for volatile organic parameters are stored in separate refrigerators designated for volatile organic parameters only. Samples are never to be stored with reagents, standards or materials that may create contamination.

To ensure the integrity of the samples during storage, refrigerator blanks are maintained in the volatile sample refrigerators and analyzed every two weeks.

Analysts and technicians retrieve the sample container allocated to their analysis from the designated refrigerator and place them on carts, analyze the sample, and return the remaining sample or empty container to the refrigerator from which it originally came. All unused portions of samples, including empty sample containers, are returned to the secure sample control area. All samples are kept in cold storage for 30 days after report generation, which meets or exceeds most sample holding times. After this time period, the samples are removed from the refrigerator shelves and prepared for disposal. Special arrangements may be made to store samples for longer periods of time.

Access to the laboratory is controlled such that sample storage need not be locked at all times unless a project specifically demands it. Samples are accessible to laboratory personnel only. Visitors to the laboratory are prohibited from entering the refrigerator and laboratory areas unless accompanied by an employee of Eurofins TestAmerica.

23.5 Hazardous Samples and Foreign Soils

To minimize exposure to personnel and to avoid potential accidents, hazardous and foreign soil samples are stored in an isolated area designated for hazardous waste only. For any sample

that is known to be hazardous at the time of receipt or, if after completion of analysis the result exceeds the acceptable regulatory levels, a Hazardous Sample Notice must be completed by the analyst. This form may be completed by Sample Control, Project Managers, or analysts and must be attached to the report. The sample itself is clearly marked with a red stamp, stamped on the sample label reading "HAZARDOUS" or "FOREIGN SOIL" and placed in a colored and/or marked bag to easily identify the sample. The date, log number, lab sample number, and the result or brief description of the hazard are all written on the Hazardous & Foreign Soil Sample Notice. A copy of the form must be included with the original COC and Work Order and the original must be given to the Sample Control Custodian. Analysts will notify Sample Control of any sample determined to be hazardous after completion of analysis by completing a Hazardous Sample Notice. All hazardous samples are either returned to the client or disposed of appropriately through a hazardous waste disposal firm that lab-packs all hazardous samples and removes them from the laboratory. Foreign soil samples are sent out for incineration by a USDA-approved waste disposal facility.

See SOP NC-SC-019 Procedure of Acceptance and Handling of USDA Regulated Domestic and Foreign Soil for further information.

23.6 Sample Shipping

In the event that the laboratory needs to ship samples, the samples are placed in a cooler with enough ice to ensure the samples remain just above freezing and at or below 6.0°C during transit. The samples are carefully surrounded by packing material to avoid breakage (yet maintain appropriate temperature). A trip blank is enclosed for those samples requiring water/solid volatile organic analyses (see Note). The chain-of-custody form is signed by the sample control technician and attached to the shipping paperwork. Samples are generally shipped overnight express or hand-delivered by a Eurofins TestAmerica courier to maintain sample integrity. All personnel involved with shipping and receiving samples must be trained to maintain the proper chain-of-custody documentation and to keep the samples intact and on ice. The Environmental, Health and Safety Manual contains additional shipping requirements.

Note: If a client does not request trip blank analysis on the COC or other paperwork, the laboratory will not analyze the trip blanks that were supplied. However, in the interest of good client service, the laboratory will advise the client at the time of sample receipt that it was noted that they did not request analysis of the trip blank; and that the laboratory is providing the notification to verify that they are not inadvertently omitting a key part of regulatory compliance testing.

23.7 Sample Disposal

Samples should be retained for a minimum of 30 days after the project report is sent, however, provisions may be made for earlier disposal of samples once the holding time is exceeded. Some samples are required to be held for longer periods based on regulatory or client requirements (e.g., 60 days after project report is sent). The laboratory must follow the longer sample retention requirements where required by regulation or client agreement. Several possibilities for sample disposal exist: the sample may be consumed completely during analysis, the sample may be returned to the customer or location of sampling for disposal, or the sample may be disposed of in accordance with the laboratory's waste disposal procedures (SOP NC-SC-005, Sample Receiving). All procedures in the laboratory's Environmental Health and Safety Manual are followed during disposal. Samples are normally maintained in the laboratory

no longer than two months from receipt unless otherwise requested. Unused portions of samples found or suspected to be hazardous according to state or federal guidelines may be returned to the client upon completion of the analytical work.

If a sample is part of a known litigation, the affected legal authority, sample data user, and/or submitter of the sample must participate in the decision about the sample's disposal. All documentation and correspondence concerning the disposal decision process must be kept on file. Pertinent information includes the date of disposal, nature of disposal (such as sample depletion, hazardous waste facility disposal, return to client), names of individuals who conducted the arrangements and physically completed the task. The laboratory will remove or deface sample labels prior to disposal unless this is accomplished through the disposal method (e.g., samples are incinerated). A Waste Disposal Record should be completed.

All incoming work will be evaluated against the criteria listed below. Where applicable, data from any samples that do not meet the criteria listed below will be noted on the laboratory report defining the nature and substance of the variation. In addition the client will be notified either by telephone, fax or e-mail ASAP after the receipt of the samples.

Per State and/or Federal Regulation, the client is responsible to ensure that samples are shipped in accordance with DOT/IATA requirements, and that radioactive materials may only be delivered to licensed facilities. Any samples containing (or suspected to contain) Source, Byproduct, or Special Nuclear Material as defined by 10 CFR should be delivered directly to facilities licensed to handle such radioactive material. Natural material or ores containing naturally occurring radionuclides may be delivered to any Eurofins TestAmerica facility or courier as long as the activity concentration of the material does not exceed 270 pCi/g alpha or 2700 pCi/g beta (49 CFR Part 173).

1) Samples must arrive with labels intact with a Chain of Custody filled out completely. The following information must be recorded.
   - *Client name, address, phone number and fax number (if available)*
   - *Project name and/or number*
   - *The sample identification*
   - *Date, time and location of sampling*
   - *The collectors name*
   - *The matrix description*
   - *The container description*
   - *The total number of each type of container*
   - *Preservatives used*
   - *Analysis requested*
   - *Requested turnaround time (TAT)*
   - *Any special instructions*
   - *Purchase Order number or billing information (e.g. quote number) if available*
   - *The date and time that each person received or relinquished the sample(s), including their signed name.*
   - *The date and time of receipt must be recorded between the last person to relinquish the samples and the person who receives the samples in the lab, and they must be exactly the same.*
   - Information must be legible

2) Samples must be properly labeled.

- Use durable labels (labels provided by Eurofins TestAmerica are preferred)
- Include a unique identification number
- Include sampling date and time & sampler ID
- Include preservative used.
- Use indelible ink
- Information must be legible

3) Proper sample containers with adequate volume for the analysis and necessary QC are required for each analysis requested. See Lab Sampling Guide.

4) Samples must be preserved according to the requirements of the requested analytical method (See Sampling Guide**.**

5) Most analytical methods require chilling samples to 4° C (other than water samples for metals analysis). For these methods, the criteria are met if the samples are chilled to below 6° C and above freezing (0°C). For methods with other temperature criteria (e.g. some bacteriological methods require ≤ 10 °C), the samples must arrive within ± 2° C of the required temperature or within the method specified range.

   - Samples that are delivered to the laboratory on the same day they are collected may not meet the requirements of Section 5. In these cases, the samples shall be considered acceptable if the samples were received on ice.
   - If sample analysis is begun within fifteen (15) minutes of collection, thermal preservation is not required.
   - Thermal preservation is not required in the field if the laboratory receives and refrigerates the sample within fifteen (15) minutes of collection.
   - Chemical preservation (pH) will be verified prior to analysis and documented, either in sample control or at the analyst's level. The project manager will be notified immediately if there is a discrepancy. If analyses will still be performed, all affected results will be flagged to indicate improper preservation.

Sample Holding Times

- Eurofins TestAmerica Canton will make every effort to analyze samples within the regulatory holding time. Samples must be received in the laboratory with enough time to perform the sample analysis. Except for short holding time samples (< 48hr HT) sample must be received with at least 48 hrs (2 working days) remaining on the holding time for us to ensure analysis.
- Analyses that are designated as "field" analyses (pH, Dissolved Oxygen, Disinfectant Residual; a.k.a. Residual Chlorine,) should be analyzed ASAP by the field sampler prior to delivering to the lab (within 15 minutes). However, if the analyses are to be performed in the laboratory, Eurofins TestAmerica Canton will make every effort to analyze the samples within 24 hours from receipt of the samples in the testing laboratory. Samples for "field" analyses received after 4:00 pm on Friday or on the weekend will be analyzed no later than the next business day after receipt (Monday unless a holiday). Samples will remain refrigerated and sealed until the time of analysis. The actual times of all "field" sample analyses are noted in the final report. Samples analyzed in the laboratory will be qualified on the final report with an 'H' to indicate holding time exceedance.

6) All samples submitted for Volatile Organic analyses should have a Trip Blank submitted at the same time. Eurofins TestAmerica Canton will supply a blank with the bottle order.

7) The project manager will be notified if any sample is received in damaged condition.

8) Recommendations for packing samples for shipment.

   - Pack samples in Ice rather than "Blue" ice packs.
   - Soil samples should be placed in plastic zip-lock bags. The containers often have dirt around the top and do not seal very well and are prone to intrusion from the water from melted ice.

- Water samples would be best if wrapped with bubble-wrap or paper (newspaper, or paper towels work) and then placed in plastic zip-lock bags.
- Fill extra cooler space with bubble wrap.

24 ASSURING THE QUALITY OF TEST RESULTS

24.1 Overview

In order to assure our clients of the validity of their data, the laboratory continuously evaluates the quality of the analytical process. The analytical process is controlled not only by instrument calibration as discussed in Section 20, but also by routine process quality control measurements (e.g. Blanks, Laboratory Control Samples (LCS), Matrix Spikes (MS), duplicates (DUP), surrogates, Internal Standards (IS)). These quality control checks are performed as required by the method or regulations to assess precision and accuracy. Quality control samples are to be treated in the exact same manner as the associated field samples being tested. In addition to the routine process quality control samples, Proficiency Testing (PT) Samples (concentrations unknown to laboratory) are analyzed to help ensure laboratory performance.

24.2 Controls

Sample preparation or pre-treatment is commonly required before analysis. Typical preparation steps include homogenization, grinding, solvent extraction, sonication, acid digestion, distillation, reflux, evaporation, drying and ashing. During these pre-treatment steps, samples are arranged into discreet manageable groups referred to as preparation (prep) batches. Prep batches provide a means to control variability in sample treatment. Control samples are added to each prep batch to monitor method performance and are processed through the entire analytical procedure with investigative/field samples.

24.3 Negative Controls

Table 24-1. Example – Negative Controls

Control Type	Details
Method Blank (MB)	are used to assess preparation and analysis for possible contamination during the preparation and processing steps.
	The specific frequency of use for method blanks during the analytical sequence is defined in the specific standard operating procedure for each analysis. Generally it is 1 for each batch of samples; not to exceed 20 environmental samples.
	The method blank is prepared from a clean matrix similar to that of the associated samples that is free from target analytes (e.g., Reagent water, Ottawa sand, glass beads, etc.) and is processed along with and under the same conditions as the associated samples. The method blank goes through all of the steps of the process (including as necessary: filtration, clean-ups, etc.).
	Reanalyze or qualify associated sample results when the concentration of a targeted analyte in the blank is at or above the reporting limit as established by the method or by regulation, AND is greater than 1/10 of the amount measured in the sample.
Calibration Blanks	are prepared and analyzed along with calibration standards where applicable. They are prepared using the same reagents that are used to prepare the standards. In some analyses the calibration blank may be included in the calibration curve.

Table 24-1. Example – Negative Controls

Control Type	Details
Instrument Blanks	are blank reagents or reagent water that may be processed during an analytical sequence in order to assess contamination in the analytical system. In general, instrument blanks are used to differentiate between contamination caused by the analytical system and that caused by the sample handling or sample prep process. Instrument blanks may also be inserted throughout the analytical sequence to minimize the effect of carryover from samples with high analyte content.
Trip Blank [1]	are required to be submitted by the client with each shipment of samples requiring aqueous and solid volatiles analyses (or as specified in the client's project plan). Additionally, trip blanks may be prepared and analyzed for volatile analysis of air samples, when required by the client. A trip blank may be purchased (certified clean) or is prepared by the laboratory by filling a clean container with pure deionized water that has been purged to remove any volatile compounds. Appropriate preservatives are also added to the container. The trip blank is sent with the bottle order and is intended to reflect the environment that the containers are subjected to throughout shipping and handling and help identify possible sources if contamination is found. The field sampler returns the trip blank in the cooler with the field samples.
Field Blanks [1]	are sometimes used for specific projects by the field samplers. A field blank prepared in the field by filling a clean container with pure reagent water and appropriate preservative, if any, for the specific sampling activity being undertaken. (EPA OSWER)
Equipment Blanks [1]	are also sometimes created in the field for specific projects. An equipment blank is a sample of analyte-free media which has been used to rinse common sampling equipment to check effectiveness of decontamination procedures. (TNI)
Holding Blanks	also referred to as refrigerator or freezer blanks, are used to monitor the sample storage units for volatile organic compounds during the storage of VOA samples in the laboratory

[1] When known, these field QC samples should not be selected for matrix QC as it does not provide information on the behavior of the target compounds in the field samples. Usually, the client sample ID will provide information to identify the field blanks with labels such as "FB", "EB", or "TB."

Evaluation criteria and corrective action for these controls are defined in the specific standard operating procedure for each analysis.

24.4 Positive Controls

Control samples (e.g., QC indicators) are analyzed with each batch of samples to evaluate data based upon (1) Method Performance (Laboratory Control Sample (LCS) or Blank Spike (BS)), which entails both the preparation and measurement steps; and (2) Matrix Effects (Matrix Spike (MS) or Sample Duplicate (MD, DUP), which evaluates field sampling accuracy, precision, representativeness, interferences, and the effect of the matrix on the method performed. Each regulatory program and each method within those programs specify the control samples that are prepared and/or analyzed with a specific batch

Note that frequency of control samples vary with specific regulatory, methodology and project specific criteria. Complete details on method control samples are as listed in each analytical SOP.

24.4.1 Method Performance Control - Laboratory Control Sample (LCS)

The LCS measures the accuracy of the method in a blank matrix and assesses method performance independent of potential field sample matrix affects in a laboratory batch.

The LCS is prepared from a clean matrix similar to that of the associated samples that is free from target analytes (for example: Reagent water, Ottawa sand, glass beads, etc.) and is

processed along with and under the same conditions as the associated samples. The LCS is spiked with verified known amounts of analytes or is made of a material containing known and verified amounts of analytes, taken through all preparation and analysis steps along with the field samples. Where there is no preparation taken for an analysis (such as in aqueous volatiles), or when all samples and standards undergo the same preparation and analysis process, a calibration verification standard is reported as the LCS. In some instances where there is no practical clean solid matrix available, aqueous LCS's may be processed for solid matrices; final results may be calculated as mg/kg or ug/kg, assuming 100% solids and a weight equivalent to the aliquot used for the corresponding field samples, to facilitate comparison with the field samples.

Certified pre-made reference material purchased from a NIST/A2LA accredited vendor may also be used for the LCS when the material represents the sample matrix or the analyte is not easily spiked (e.g. solid matrix LCS for metals, TDS, etc.).

The specific frequency of use for LCS during the analytical sequence is defined in the specific standard operating procedure for each analysis. It is generally 1 for each batch of samples; not to exceed 20 environmental samples.

If the mandated or requested test method, or project requirements, do not specify the spiking components, the laboratory shall spike all reportable components to be reported in the Laboratory Control Sample (and Matrix Spike) where applicable (e.g. no spike of pH). However, in cases where the components interfere with accurate assessment (such as simultaneously spiking chlordane, Toxaphene and PCBs in Method 608), the test method has an extremely long list of components or components are incompatible, at a minimum, a representative number of the listed components (see below) shall be used to control the test method. The selected components of each spiking mix shall represent all chemistries, elution patterns and masses, permit specified analytes and other client requested components. However, the laboratory shall ensure that all reported components are used in the spike mixture within a two-year time period.

- For methods that have 1-10 target analytes, spike all components.
- For methods that include 11-20 target analytes, spike at least 10 or 80%, whichever is greater
- For methods with more than 20 target analytes, spike at least 16 components.
- Exception: Due to analyte incompatibility in pesticides, Toxaphene and Chlordane are only spiked at client request based on specific project needs.
- Exception: Due to analyte incompatibility between the various PCB aroclors, aroclors 1016 and 1260 are used for spiking as they cover the range of all of the aroclors. Specific aroclors may be used by request on a project specific basis.

24.5 Sample Matrix Controls

Table 24-3. Sample Matrix Control

Control Type	Details	
Matrix Spikes (MS)	Use	Used to assess the effect sample matrix of the spiked sample has on the precision and accuracy of the results generated by the method used;
	Typical Frequency [1]	At a minimum, with each matrix-specific batch of samples processed, an MS is carried through the complete analytical procedure. Unless specified by the client, samples used for spiking are randomly selected and rotated between different client projects. If the mandated or requested test method does not specify the spiking components, the laboratory shall spike all reportable components to be reported in the Laboratory Control Sample and Matrix Spike. Refer to the method SOP for complete details
	Description	Essentially a sample fortified with a known amount of the test analyte(s).
Surrogate	Use	Measures method performance to sample matrix (organics only).
	Typical Frequency [1]	Are added to all samples, standards, and blanks, for all organic chromatography methods except when the matrix precludes its use or when a surrogate is not available. The recovery of the surrogates is compared to the acceptance limits for the specific method. Poor surrogate recovery may indicate a problem with sample composition and shall be reported, with data qualifiers, to the client whose sample produced poor recovery.
	Description	Are similar to matrix spikes except the analytes are compounds with properties that mimic the analyte of interest and are unlikely to be found in environment samples.
Duplicates[2]	Use	For a measure of analytical precision, with each matrix-specific batch of samples processed, a matrix duplicate (MD or DUP) sample, matrix spike duplicate (MSD), or LCS duplicate (LCSD) is carried through the complete analytical procedure.
	Typical Frequency [1]	Duplicate samples are usually analyzed with methods that do not require matrix spike analysis.
	Description	Performed by analyzing two aliquots of the same field sample independently or an additional LCS.
Internal Standards	Use	Are spiked into all environmental and quality control samples (including the initial calibration standards) to monitor the qualitative aspect of organic and some inorganic analytical measurements.
	Typical Frequency [1]	All organic and ICP methods as required by the analytical method.
	Description	Used to correct for matrix effects and to help troubleshoot variability in analytical response and are assessed after data acquisition. Possible sources of poor internal standard response are sample matrix, poor analytical technique or instrument performance.

[1] See the specific analytical SOP for type and frequency of sample matrix control samples.
[2] LCSD's are normally not performed except when regulatory agencies or client specifications require them. The recoveries for the spiked duplicate samples must meet the same laboratory established recovery limits as the accuracy QC samples. If an LCSD is analyzed both the LCS and LCSD must meet the same recovery criteria and be included in the final report. The precision measurement is reported as "Relative Percent Difference" (RPD). Poor precision between duplicates (except LCS/LCSD) may indicate non-homogeneous matrix or sampling.

24.6 Acceptance Criteria (Control Limits)

As mandated by the test method and regulation, each individual analyte in the LCS, MS, or Surrogate Spike is evaluated against the control limits published in the test method. Where there are no established acceptance criteria, the laboratory calculates in-house control limits with the use of control charts or, in some cases, utilizes client project specific control limits. When this occurs, the regulatory or project limits will supersede the laboratory's in-house limits.

Note: For methods, analytes and matrices with very limited data (e.g., unusual matrices not analyzed often), interim limits are established using available data or by analogy to similar methods or matrices.

Note: For Ohio VAP the laboratory must implement Corrective Action procedures to resolve the deviation and limit qualification of the final results. The laboratory is not permitted to deviate from its VAP approved SOP if it intends to attest under affidavit that the "results" are VAP certified. When all corrective actions listed in the SOP have been exhausted, it may be necessary to use technical judgment in which case the decision process and rationale will be presented in the final report and/or affidavit and the data will be noted as 'not VAP certified' on the affidavit.

Once control limits have been established, they are verified, reviewed, and updated if necessary on an annual basis unless the method requires more frequent updating. Control limits are established per method (as opposed to per instrument) regardless of the number of instruments utilized.

Laboratory generated % Recovery acceptance (control) limits are generally established by taking $\pm$ 3 Standard Deviations (99% confidence level) from the average recovery of a minimum of 20-30 data points (more points are preferred).

- Regardless of the calculated limit, the limit should be no tighter than the Calibration Verification (ICV/CCV) (unless the analytical method specifies a tighter limit).

- In-house limits cannot be any wider than those mandated in a regulated analytical method. Client or contract required control limits are evaluated against the laboratory's statistically derived control limits to determine if the data quality objectives (DQOs) can be achieved. If laboratory control limits are not consistent with DQOs, then alternatives must be considered, such as method improvements or use of an alternate analytical method.

- The lowest acceptable recovery limit will be 10% (the analyte must be detectable and identifiable). Exception: The lowest acceptable recovery limit for Benzidine will be 5% and the analyte must be detectable and identifiable.

- The maximum acceptable recovery limit will be 200%.

- The maximum acceptable RPD limit will be 35% for waters and 40% for soils. The minimum RPD limit is 10%.

- If either the high or low end of the control limit changes by $\leq$ 5% from previous, the control chart is visually inspected and, using professional judgment, they may be left unchanged if there is no effect on laboratory ability to meet the existing limits.

24.6.1 The lab must be able to generate a current listing of their control limits and track when the updates are performed. In addition, the laboratory must be able to recreate historical control limits. Refer to NC-QA-018, Statistical Evaluation of Data and Development of Control Charts for details.

24.6.2 A LCS that is within the acceptance criteria establishes that the analytical system is in control and is used to validate the process. Samples that are analyzed with an LCS with recoveries outside of the acceptance limits may be determined as out of control and should be reanalyzed if possible. If reanalysis is not possible, then the results for all affected analytes for samples within the same batch must be qualified when reported. The internal corrective action process (see Section 12) is also initiated if an LCS exceeds the acceptance limits. Sample results may be qualified and reported without reanalysis if:

- The analyte results are below the reporting limit and the LCS is above the upper control limit.
- If the analytical results are above the relevant regulatory limit and the LCS is below the lower control limit.

Or, for TNI work, there are an allowable number of Marginal Exceedances (ME):

<11 analytes	0 marginal exceedances are allowed.
11 – 30 Analytes	1 marginal exceedance is allowed
31-50 Analytes	2 marginal exceedances are allowed
51-70 Analytes	3 marginal exceedances are allowed
71-90 Analytes	4 marginal exceedances are allowed
> 90 Analytes	5 marginal exceedances are allowed

- Marginal exceedances are recovery exceedances between 3 SD and 4 SD from the mean recovery limit (TNI).
- Marginal exceedances must be random. If the same analyte exceeds the LCS control limit repeatedly, it is an indication of a systematic problem. The source of the error must be located and corrective action taken. The laboratory has a system to monitor marginal exceedances to ensure that they are random.

Note: Ohio VAP does not allow the use of marginal exceedance.

Though marginal exceedances may be allowed by other programs, the data must still be qualified to indicate it is outside of the normal limits.

24.6.3 If the MS/MSDs do not meet acceptance limits, the MS/MSD and the associated spiked sample is reported with a qualifier for those analytes that do not meet limits. If obvious preparation errors are suspected, or if requested by the client, unacceptable MS/MSDs are reprocessed and reanalyzed to prove matrix interference. A more detailed discussion of acceptance criteria and corrective action can be found in the lab's method SOPs and in Section 12.

24.6.4 If a surrogate standard falls outside the acceptance limits, and if there is not obvious chromatographic matrix interference, reanalyze the sample to confirm a possible matrix effect. If the recoveries confirm or there was obvious chromatographic interference, results are reported from the original analysis and a qualifier is added. If the reanalysis meets surrogate recovery criteria, the second run is reported (or both are reported if

requested by the client). Under certain circumstances, where all of the samples are from the same location and share similar chromatography, the reanalysis may be performed on a single sample rather than all of the samples and if the surrogate meets the recovery criteria in the reanalysis, all of the affected samples would require reanalysis.

24.7 Additional Procedures to Assure Quality Control

The laboratory has written and approved method SOPs to assure the accuracy of the test method including calibration (see Section 20), use of certified reference materials (see Section 21) and use of PT samples (see Section 17).

A discussion regarding MDLs, Limit of Detection (LOD) and Limit of Quantitation (LOQ) can be found in Section 19.

- Use of formulae to reduce data is discussed in the method SOPs and in Section 20.
- Selection of appropriate reagents and standards is included in Section 11 and 21.
- A discussion on selectivity of the test is included in Section 7.
- Constant and consistent test conditions are discussed in Section 6.
- The laboratories sample acceptance policy is included in Section 23.

25 REPORTING RESULTS

25.1 Overview

The results of each test are reported accurately, clearly, unambiguously, and objectively in accordance with State and Federal regulations as well as client requirements. Analytical results are issued in a format that is intended to satisfy customer and laboratory accreditation requirements as well as provide the end user with the information needed to properly evaluate the results. Where there is conflict between client requests and laboratory ethics or regulatory requirements, the laboratory's ethical and legal requirements are paramount, and the laboratory will work with the client during project set up to develop an acceptable solution. Refer to Section 9.

A variety of report formats are available to meet specific needs.

In cases where a client asks for simplified reports, there must be a written request from the client. There still must be enough information that would show any analyses that were out of conformance (QC out of limits) and there should be a reference to a full report that is made available to the client. Review of reported data is included in Section 19.

25.2 Test Reports

Analytical results are reported in a format that is satisfactory to the client and meets all requirements of applicable accrediting authorities and agencies. A variety of report formats are available to meet specific needs. The report is printed on laboratory letterhead, reviewed, and signed by the appropriate project manager. At a minimum, the standard laboratory report shall contain the following information:

25.2.1 A report title (e.g., Analytical Report)

25.2.2 The cover page shall include the laboratory name, address and telephone number.

25.2.3 A unique identification of the report (e.g., Eurofins TestAmerica Canton Job ID #) and on each page an identification in order to ensure the page is recognized as part of the report and a clear identification of the end.

Note: Page numbers of report are represented as page # of ##. Where the first number is the page number and the second is the total number of pages.

25.2.4. A copy of the chain of custody (COC)

- Any COCs involved with Subcontracting are included.

25.2.5. The name and address of client and a project name/number, if applicable.

25.2.6. Client project manager or other contact

25.2.7. Description and unambiguous identification of the tested sample(s) including the client identification code

25.2.8. Date of receipt of sample, date and time of collection, and date(s) of test preparation and performance, and time of preparation or analysis if the required holding time for either activity is less than or equal to 72 hours.

25.2.9. Date reported or date of revision, if applicable.

25.2.10. Method of analysis including method code (EPA, Standard Methods, etc.)

25.2.11. Practical quantitation limits or reporting limit.

25.2.12. Method detection limits (if requested)

25.2.13. Definition of Data qualifiers and reporting acronyms (e.g. ND)

25.2.14. Sample results

25.2.15. QC data consisting of method blank, surrogate, LCS, and MS/MSD recoveries and control limits

25.2.16. Condition of samples at receipt including temperature. This may be accomplished in a narrative or by attaching sample login sheets (Refer to Sec. 25.2.4 – Item 3 regarding additional addenda).

25.2.17. A statement expressing the validity of the results, that the source methodology was followed and all results were reviewed for error.

25.2.18. A statement to the effect that the results relate only to the items tested and the sample as received by the laboratory, except when information is provided by the

client. When data is provided by the client there shall be a clear identification of it, and a disclaimer shall be put in the report when the client supplied data can affect the validity of the test.

25.2.19. A statement that the report shall not be reproduced except in full, without prior express written approval by the laboratory coordinator.

25.2.20. A signature and title of the person(s) accepting responsibility for the content of the report and date of issue. Authorized signatories are qualified Project Managers appointed by the Manager of Project Managers.

25.2.21. When TNI accreditation is required, the lab shall certify that the test results meet all requirements of TNI or provide reasons and/or justification if they do not.

25.2.22. Where applicable, a narrative to the report that explains the issue(s) and corrective action(s) taken in the event that a specific accreditation or certification requirement was not met.

25.2.23. When soil samples are analyzed, a specific identification as to whether soils are reported on a “wet weight” or “dry weight” basis.

25.2.24. Appropriate laboratory certification number for the state of origin of the sample, if applicable.

25.2.25. If only part of the report is provided to the client (client requests some results before all of it is complete), it must be clearly indicated on the report (e.g., partial report, or how your lab identifies it). A complete report must be sent once all of the work has been completed.

25.2.26. Any non-Eurofins TestAmerica Canton subcontracted analysis results are provided as a separate report on the official letterhead of the subcontractor. All Eurofins TestAmerica Canton subcontracting is clearly identified on the report as to which laboratory performed a specific analysis.

25.2.27. A Certification Summary Report, where required, will document that, unless otherwise noted, all analytes tested and reported by the laboratory were covered by the noted certifications.

Note: Refer to the NDSC Document No. CA-I-P-002 Electronic Reporting and Signature Policy for details on internally applying electronic signatures of approval.

25.2.28. Reports for Ohio VAP work require a VAP affidavit be completed and included with the report.

25.2.29. Where the laboratory is responsible for the sampling stage, in addition to the requirements listed above, reports containing the results of sampling shall include the following, where necessary for the interpretation of test results:

- the date of sampling;

- unambiguous identification of the material sampled;
- the location of sampling plan and procedures, and deviations, addition to or exclusions from the sample procedures;
- a reference to the sampling plan and procedure, and deviations, additions to or exclusions from the sample procedures;
- details of any environmental conditions during sampling that affect the interpretation of test results;
- information required to evaluate measurement uncertainty for subsequent testing

25.3 Reporting Level or Report Type

The laboratory offers four levels of quality control reporting. Each level, in addition to its own specific requirements, contains all the information provided in the preceding level. The packages provide the following information in addition to the information described above:

- Level 1 is a report with all of the elements outlined in Section 25.2 above, excluding 25.2.15 (QC data).
- Level II is a Level I report plus summary information, including results for the method blank reported to the laboratory MDL, percent recovery for laboratory control samples and matrix spike samples, and the RPD values for all MSD and sample duplicate analyses.
- Level III contains all the information supplied in Level II, but presented on the CLP-like summary forms, and relevant calibration information. A Level II report is not included, unless specifically requested. No raw data is provided.
- Level IV is the same as Level III with the addition of all raw supporting data.

In addition to the various levels of QC packaging, the laboratory also provides reports in EDD (see below) Initial reports may be provided to clients by facsimile. Procedures used to ensure client confidentiality are outlined in Section 25.6.

25.4 Electronic Data Deliverables (EDDs)

EDDs are routinely offered as part of Eurofins TestAmerica Canton's services in addition to the test report as described in Section 25.2. When NELAP accreditation is required and both a test report and EDD are provided to the client, the official version of the test report will be the combined information of the report and the EDD. Eurofins TestAmerica Canton offers a variety of EDD formats including Environmental Restoration Information Management System (ERPIMS), Staged Electronic Data Deliverable (SEDD) Environmental Quality Information System (EQuIS), Electronic Deliverable Format (EDF), Excel and custom files.

EDD specifications are submitted to the IT department by the PM for review and undergo the contract review process. Once the facility has committed to providing data in a specific electronic format, the coding of the format may need to be performed. This coding is documented and validated. The validation of the code is retained by the IT staff coding the EDD.

EDDs shall be subject to a review to ensure their accuracy and completeness. If EDD generation is automated, review may be reduced to periodic screening if the laboratory can

demonstrate that it can routinely generate that EDD without errors. Any revisions to the EDD format must be reviewed until it is demonstrated that it can routinely be generated without errors. If the EDD can be reproduced accurately and if all subsequent EDDs can be produced error-free, each EDD does not necessarily require a review.

25.5 Supplemental Information for Test

The lab identifies any unacceptable QC analyses or any other unusual circumstances or observations such as environmental conditions and any non-standard conditions that may have affected the quality of a result. This is typically in the form of a footnote or a qualifier and/or a narrative explaining the discrepancy in the front of the report.

Numeric results with values outside of the calibration range, either high or low are qualified as estimated.

Where quality system requirements are not met, a statement of compliance/non-compliance with requirements and/or specifications is required, including identification of test results derived from any sample that did not meet TNI sample acceptance requirements such as improper container, holding time, or temperature.

Where applicable, a statement on the estimated uncertainty of measurements; information on uncertainty is needed when a client's instructions so require.

When, as requested by the client and agreed to by Eurofins TestAmerica Canton, the report includes a statement of conformity to specification or standard (see Special Services, Section 7.4), the report shall clearly identify:

- to which results the statement applies,
- which specifications, standard or parts thereof are met or not, and
- the decision rule that was applied (unless the decision rule is inherent in the requested specification or standard, taking into account the level of risk (such as false accept and false reject and statistical assumptions) associated with the decision rule.

Opinions and Interpretations - The test report contains objective information, and generally does not contain subjective information such as opinions and interpretations. If such information is required by the client, the Laboratory Director will determine if a response can be prepared. If so, the Laboratory Director will designate the appropriate member of the management team to prepare a response. The response will be fully documented, and reviewed by the Laboratory Director, before release to the client. There may be additional fees charged to the client at this time, as this is a non-routine function of the laboratory.

Note: Review of data deliverable packages for submittal to regulatory authorities requires responses to non-conforming data concerning potential impact on data quality. This necessitates a limited scope of interpretation, and this work is performed by the QA Department. This is the only form of "interpretation" of data that is routinely performed by the laboratory.

When opinions or interpretations are included in the report, the laboratory provides an explanation as to the basis upon which the opinions and interpretations have been made. Opinions and interpretations are clearly noted as such and where applicable, a comment should be added suggesting that the client verify the opinion or interpretation with their regulator.

25.6 Environmental Testing Obtained From Subcontractors

If the laboratory is not able to provide the client the requested analysis, the samples would be subcontracted following the procedures outlined in the NDSC Document No. CW-L-S-004, Subcontracting.

Data reported from analyses performed by a subcontractor laboratory are clearly identified as such on the analytical report provided to the client. Results from a subcontract laboratory outside of Eurofins TestAmerica Canton are reported to the client on the subcontract laboratory's original report stationary and the report includes any accompanying documentation.

25.7 Client Confidentiality

The laboratory will ensure the highest standards of quality and integrity of the data and services provided to our clients

The laboratory is responsible for maintaining in confidence all client information obtained or created. In situations involving the transmission of environmental test results by telephone, facsimile or other electronic means, client confidentiality must be maintained.

The laboratory will not intentionally divulge to any person (other than the client or any other person designated by the client in writing) any information regarding the services provided by the laboratory or any information disclosed to the laboratory by the client. Furthermore, information known to be potentially endangering to national security or an entity's proprietary rights will not be released.

Information about the client obtained from sources other than the client (e.g., complainant, regulators) shall be confidential between client and the laboratory. The source of this information shall be confidential to the laboratory and shall not be shared with the client, unless agreed by the source.

Note: This shall not apply to the extent that the information is required to be disclosed by the laboratory under the compulsion of legal process. The laboratory will, to the extent feasible, provide reasonable notice to the client before disclosing the information.

Note: Authorized representatives of an accrediting authority are permitted to make copies of any analyses or records relevant to the accreditation process, and copies may be removed from the laboratory for purposes of assessment.

25.7.1 Report deliverable formats are discussed with each new client. If a client requests that reports be e-mailed, the reports are to meet all requirements of this document.

25.8 Format of Reports

The format of reports is designed to accommodate each type of environmental test carried out and to minimize the possibility of misunderstanding or misuse.

25.9 Amendments to Test Reports

Corrections, additions, or deletions to reports are only made when justification arises through supplemental documentation. Justification is documented using the laboratory's corrective action system (refer to Section 12).

The revised report is retained on the Archive data server, as is the original report. The revised report is stored in the Archive data server under the sample number followed by "R" The revised report will have the word "revised" or "amended" next to the date rather than the word "reported".

When the report is re-issued, a notation of "report re-issue" is placed on the cover/signature page of the report *or at the top of the narrative page* with a brief explanation of reason for the re-issue and a reference back to the last final report generated.

25.10 Policies on Client Requests for Amendments

25.10.1 Policy on Data Omissions or Reporting Limit Increases

Fundamentally, our policy is simply to not omit previously reported results (including data qualifiers) or to not raise reporting limits and report sample results as ND. This policy has few exceptions. Exceptions are:

- Laboratory error
- Sample identification is indeterminate (confusion between COC and sample labels).
- An incorrect analysis (not analyte) was requested (e.g., COC lists 8315 but client wanted 8310). A written request for the change is required.
- Incorrect limits reported based on regulatory requirements.
- The requested change has absolutely *no possible* impact on the interpretation of the analytical results and there is *no possibility* of the change being interpreted as misrepresentation by anyone inside or outside of our company.

25.10.2 Multiple Reports

The laboratory does not issue multiple reports for the same work order where there is different information on each report (this does not refer to copies of the same report) unless required to meet regulatory needs and approved by QA.

Appendix 1. List of Governing Documents applicable to the QA Manual

(NDSC, KDG and Laboratory SOPs and Policies)

NDSC Doc. No.	Title
CA-I-P-002	Electronic Reporting and Signature Policy
CA-L-P-002	Contract Compliance Policy
CA-Q-M-002	Quality Management Plan
CA-Q-QM-001	Guidance on Tentatively Identified Compounds (TICs) – GC/MS Analysis
CA-Q-S-001	Acid and Solvent Lot Testing and Approval Program
CA-Q-S-002, NDSC-QA-SOP43862	Manual Integrations
CA-Q-S-006, NDSC-QA-SOP42091	Detection and Quantitation Limits
CA-Q-S-009	Root Cause Analysis
CA-T-P-001	Qualified Products List
CW-E-M-001, NDSC-US EHS-QP46060	Environmental Health & Safety Manual
CW-F-WI-036	Eurofins TestAmerica: Spending and Contract Authority (S&CA) Limits and Authorization Matrix for US Environment
CW-I-M-001	IT Change Control Procedure Manual
CW-L-P-001	Records Retention Policy
CW-L-P-004	Ethics Policy
CW-L-S-002,QA-QP38228	Internal Investigation
CW-L-S-004,NDSC-US-LEG-SOP44936	Subcontracting
CW-Q-S-001	Document Control and Archiving
CW-Q-S-002	Writing a Standard Operating Procedure (SOPs)
CW-Q-S-003, SOP02-QA-QP5260	Internal Auditing
CW-Q-S-004,QA-QP38702	Management Systems Review
CW-Q-S-005,QA-QP38702	Data Recall Process
CW-Q-S-001	Document Control and Archiving
Local Laboratory Document No.	**Title**
NC-QA-015	Support Equipment Calibration and Verification
NC-QA-018	Statistical Evaluation of Data and Development of Control Charts
NC-QA-019	Records Information Management

NC-QA-027	Preparation and Management of SOPs
NC-QA-028	Employee Orientation and Training
NC-QA-029	Nonconformance and Corrective Action System
NC-QA-030	Document Control
NC-SC-005	Sample Receiving and Sample Control
NC-SC-006	Sample Procurement Protocol
CA-Q-T-005	Laboratory Documentation
NC-QA-031	Internal Auditing
QA-003	Quality Control Program

Appendix 2. List of Laboratory Certifications, Accreditations, Validations

The laboratory maintains accreditations, certifications, and approvals with numerous state and national entities. Programs vary but may include on-site audits, reciprocal agreements with another entity, performance testing evaluations, review of the QA Manual, Standard Operating Procedures, Method Detection Limits, training records, etc. At the time of this QA Manual revision, the laboratory has accreditation/ certification/licensing with the following organizations:

Organization	Certificate Number	Organization	Certificate Number
California	2927	Iowa	421
Connecticut	PH-0590	New Jersey	OH001
Florida	E87225	New York	10975
Georgia	---	OVAP	CL0024
Illinois	200004	Pennsylvania	68-00340
Kansas	E-10336	USDA (Dept. of Agriculture)	P330-18-00281
Kentucky Underground Storage Tank Program	112225	Washington	C971
Minnesota	039-999-348	West Virginia	210
Texas	T104704517-20-12 Changes based on the year and month of date of issue	Virginia	460175
Oregon	4062	Kentucky Wastewater	KY98016
Minnesota Petrofund	3506		

Appendix 3. References used to prepare the QA Manual

The QAM has been prepared to be consistent with the requirements of the following documents:

- "EPA Requirements for Quality Management Programs" (QA/R-2) (EPA/240/B-01/002, May 31, 2006).
- EPA 600/4-88/039, *Methods for the Determination of Organic Compounds in Drinking Water,* EPA, Revised July 1991.

4 EPA 600/R-95/131, *Methods for the Determination of Organic Compounds in Drinking Water,* Supplement III, EPA, August 1995.

5 EPA 600/4-79-019, *Handbook for Analytical Quality Control in Water and Wastewater Laboratories,* EPA, March 1979.

6 APHA, *Standard Methods for the Examination of Water and Wastewater,* 18th Edition, 19th, 20th, 21st, and on-line Editions.

7 *Test Methods for Evaluating Solid Waste Physical/Chemical Methods (SW846), Third Edition, September 1986, Final Update I, July 1992, Final Update IIA, August 1993, Final Update II, September 1994; Final Update IIB, January 1995; Final Update III, December 1996; Final Update IV, January 2008; Final Update V, August 2015.*

8 Federal Register, 40 CFR Parts 136, 141, 172, 173, 178, 179 and 261.

9 Toxic Substances Control Act (TSCA).

Appendix 4. QA Manual Crosswalk with TNI and ISO/IEC 17025 Standards

Sec. No.	Title	2009 and 2016 TNI Standard Reference	ISO/IEC 17025:2005(E) Reference	ISO/IEC 17025:2017(E) Reference
-	QUALITY ASSURANCE MANUAL COVER PAGE	V1M2 Sec. 4.2.8.3		
1.0	Title Page			
2.0	Table of Contents	V1M2 Secs. 4.2.8.3-4.2.8.4		8.1.2, 8.2.1
3.0	Introduction, Scope and Applicability	V1M2 Sec. 4.2.8.4		
3.1	Introduction and Compliance References	V1M2 Secs. 1.1; 1.2; 2.0; 3.2; 4.1.2; 4.2.4	4.1.2; 4.2.4	5.3; 5.4; 8.2.4; 8.3.1
3.2	Terms and Definitions	V1M2 Secs. 3.0; 4.2.4	4.2.4	
3.3	Scope /Field of Testing	V1M2 Secs. 1.2; 4.2.4	4.1.2; 4.2.4	5.3; 5.4; 8.2.1; 8.2.4
3.4	Management of The Manual	V1M2 Secs. 4.2.1; 4.2.7; 4.3.3.2; 4.3.3.3	4.2.1; 4.2.7; 4.3.3.2; 4.3.3.3	5.3
4.0	Management and Responsibilities	V1M2 Sec. 4		8.2.4; 8.2.5
4.1	Overview	V1M2 Secs. 4.1.1, 4.1.3; 4.1.5	4.1.1; 4.1.3; 4.1.5; 4.2.6	5.1; 5.2; 5.5; 5.6 ; 6.2.1; 6.2.4
4.2	Roles and Responsibilities	V1M2 Secs. 4.1.4; 4.1.5; 4.1.6; 4.2.1; 4.2.6; 5.2.4	4.1.3; 4.1.5; 4.1.6; 4.2.1; 4.2.6; 5.2.4	4.1.1 to 4.1.3; 4.1.5; 5.5; 5.6; 6.2.1; 6.2.4; 6.2.6 8.2.2;
4.3	Business Continuity and Contingency Plan *(Prev. Deputies)*	V1M2 Secs. 4.1.5; 4.1.7.2; 4.2.7	4.1.5; 4.2.7	
5.0	**Error! Reference source not found.**	V1M2 Secs. 5.2; 5.2.1	5.2.1	6.1; 6.2.3
5.1	Overview	V1M2 Secs. 5.2.2; 5.2.3; 5.2.5	5.2.2; 5.2.3; 5.2.5	6.2.2
5.2	Education and Experience Requirements for Technical Staff	V1M2 Secs. 5.2.1; 5.2.3; 5.2.4	5.2.1; 5.2.3; 5.2.4	6.2.2 to 6.2.4
5.3	Training	V1M2 Sec. 5.2.5	5.2.5	4.2.1; 6.2.2; 6.2.4; 6.2.5
5.4	Data Integrity and Ethics Training Program	V1M2 Sec. 4.2.8.1; 5.2.7		4.1.1
6.0	ACCOMMODATIONS AND ENVIRONMENTAL CONDITIONS	V1M2 Sec. 5.3		6.1; 6.3.1
6.1	Overview	V1M2 Secs. 5.3.1; 5.3.3; 5.3.4; 5.3.5	5.3.1; 5.3.3; 5.3.4; 5.3.5	6.3.1
6.2	Environment	V1M2 Secs. 5.3.1; 5.3.2; 5.3.3; 5.3.4; 5.3.5	5.3.1; 5.3.2; 5.3.3; 5.3.4; 5.3.5	6.3.1 to 6.3.5
6.3	Work Area	V1M2 Secs. 5.3.3; 5.3.4; 5.3.5	5.3.3; 5.3.4; 5.3.5	6.3.1
6.4	Responding to Emergencies			
6.5	Building Security	V1M2 Sec. 5.3.4	5.3.4	6.3.4
7.0	5.0 PERSONNEL			6.1; 8.2.4

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7.3	Quality System Documentation	V1M2 Secs. 4.1.5; 4.2.2; 4.2.5	4.2.2; 4.2.5	8.2.4
7.4	QA/QC Objectives for the Measurement of Data	V1M2 Sec. 4.2.2	4.1.5; 4.2.2	6.2.4
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9.5	Special Services	V1M2 Secs. 4.7.1-4.7.2	4.7.1; 4.7.2	7.1.1.3; 7.1.1.7
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Appendix 5. Terms/Glossary and Acronyms (EL-V1M2 Sec. 3.1)

Acceptance Criteria: Specified limits placed on characteristics of an item, process, or service defined in requirement documents. (ASQC)

Accreditation: The process by which an agency or organization evaluates and recognizes a laboratory as meeting certain predetermined qualifications or standards, thereby accrediting the laboratory.

Accuracy: The degree of agreement between an observed value and an accepted reference value. Accuracy includes a combination of random error (precision) and systematic error (bias) components which are due to sampling and analytical operations; a data quality indicator. (QAMS)

Analyst: The designated individual who performs the "hands-on" analytical methods and associated techniques and who is the one responsible for applying required laboratory practices and other pertinent quality controls to meet the required level of quality.

Analytical Uncertainty: A subset of Measurement Uncertainty that includes all laboratory activities performed as part of the analysis. (TNI)

Anomaly: A condition or event, other than a deficiency, that may affect the quality of the data, whether in the laboratory's control or not.

Assessment: The evaluation process used to measure or establish the performance, effectiveness, and conformance of an organization and/or its systems to defined criteria (to the standards and requirements of laboratory accreditation). (TNI)

Audit: A systematic and independent examination of facilities, equipment, personnel, training, procedures, record-keeping, data validation, data management, and reporting aspects of a system to determine whether QA/QC and technical activities are being conducted as planned and whether these activities will effectively achieve quality objectives. (TNI)

Batch: Environmental samples that are prepared and/or analyzed together with the same process and personnel, using the same lot(s) of reagents. A **preparation batch** is composed of one (1) to twenty (20) environmental samples of the same quality systems matrix, meeting the above mentioned criteria and with a maximum time between the start of processing of the first and last sample in the batch to be twenty-four (24) hours. An **analytical batch** is composed of prepared environmental samples (extracts, digestates or concentrates) which are analyzed together as a group. An analytical batch can include prepared samples originating from various quality system matrices and can exceed twenty (20) samples. (TNI)

Bias: The systematic or persistent distortion of a measurement process, which causes errors in one direction (i.e., the expected sample measurement is different from the sample's true value). (TNI)

Blank: A sample that has not been exposed to the analyzed sample stream in order to monitor contamination during sampling, transport, storage or analysis. The blank is subjected to the usual analytical and measurement process to establish a zero baseline or background value and is sometimes used to adjust or correct routine analytical results. (ASQC)

Calibration: A set of operations that establish, under specified conditions, the relationship between values of quantities indicated by a measuring instrument or measuring system, or values represented by a material measure or a reference material, and the corresponding values realized by standards. (TNI)

1) In calibration of support equipment the values realized by standards are established through the use of reference standards that are traceable to the International System of Units (SI).

2) In calibration according to methods, the values realized by standards are typically established through the use of Reference Materials that are either purchased by the laboratory with a certificate of analysis or purity, or prepared by the laboratory using support equipment that has been calibrated or verified to meet specifications.

Calibration Curve: The mathematical relationship between the known values, such as concentrations, of a series of calibration standards and their instrument response. (TNI)

Calibration Standard: A substance or reference material used to calibrate an instrument (QAMS)

Certified Reference Material (CRM): A reference material accompanied by a certificate, having a value, measurement uncertainty, and stated metrological traceability chain to a national metrology institute. (TNI)

Chain of Custody (COC) Form: Record that documents the possession of the samples from the time of collection to receipt in the laboratory. This record generally includes: the number and types of containers; the mode of collection; the collector; time of collection; preservation; and requested analyses. (TNI)

Compromised Samples: Those samples which are improperly sampled, insufficiently documented (chain of custody and other sample records and/or labels), improperly preserved, collected in improper containers, or exceeding holding times when delivered to a laboratory. Under normal conditions, compromised samples are not analyzed. If emergency situation require analysis, the results must be appropriately qualified.

Confidential Business Information (CBI): Information that an organization designates as having the potential of providing a competitor with inappropriate insight into its management, operation or products. TNI and its representatives agree to safeguard identified CBI and to maintain all information identified as such in full confidentiality.

Confirmation: Verification of the identity of a component through the use of an approach with a different scientific principle from the original method. These may include, but are not limited to Second Column Confirmation; Alternate wavelength; Derivatization; Mass spectral interpretation; Alternative detectors or Additional Cleanup procedures. (TNI)

Conformance: An affirmative indication or judgment that a product or service has met the requirements of the relevant specifications, contract, or regulation; also the state of meeting the requirements. (ANSI/ASQC E4-1994)

Correction: Actions necessary to correct or repair analysis specific non-conformances. The acceptance criteria for method specific QC and protocols as well as the associated corrective actions. The analyst will most frequently be the one to identify the need for this action as a result of calibration checks and QC sample analysis**.** No significant action is taken to change behavior, process or procedure.

Corrective Action: The action taken to eliminate the causes of an existing nonconformity, defect or other undesirable situation in order to prevent recurrence. (ISO 8402)

Data Integrity: The condition that exists when data are sound, correct, and complete, and accurately reflect activities and requirements. (TNI)

Data Audit: A qualitative and quantitative evaluation of the documentation and procedures associated with environmental measurements to verify that the resulting data re of acceptable quality (i.e., that they meet specified acceptance criteria).

Data Reduction: The process of transforming the number of data items by arithmetic or statistical calculations, standard curves, and concentration factors, and collation into a more useable form. (TNI)

Deficiency: An unauthorized deviation from acceptable procedures or practices, or a defect in an item (ASQC), whether in the laboratory's control or not.

Demonstration of Capability: A procedure to establish the ability of the analyst to generate analytical results of acceptable accuracy and precision. (TNI)

Document Control: The act of ensuring that documents (and revisions thereto) are proposed, reviewed for accuracy, approved for release by authorized personnel, distributed properly, and controlled to ensure use of the correct version at the location where the prescribed activity if performed. (ASQC)

Duplicate Analyses: The analyses or measurements of the variable of interest performed identically on two subsamples of the same sample. The results from duplicate analyses are used to evaluate analytical or measurement precision but not the precision of sampling, preservation or storage internal to the laboratory. (EPA-QAD)

Equipment Blank: Sample of analyte-free media which has been used to rinse common sampling equipment to check effectiveness of decontamination procedures.

External Standard Calibration: Calibrations for methods that do not utilize internal standards to compensate for changes in instrument conditions.

Field Blank: Blank prepared in the field by filing a clean container with pure de-ionized water and appropriate preservative, if any, for the specific sampling activity being undertaken (EPA OSWER)

Field of Accreditation: Those matrix, technology/method, and analyte combinations for which the accreditation body offers accreditation.

Holding Times: The maximum time that samples may be held prior to analyses and still be considered valid or not compromised. (40 CFR Part 136)

Internal Standard: A known amount of standard added to a test portion of a sample as a reference for evaluating and controlling the precision and bias of the applied analytical test method. (TNI)

Internal Standard Calibration: Calibrations for methods that utilize internal standards to compensate for changes in instrument conditions.

Instrument Blank: A clean sample (e.g., distilled water) processed through the instrumental steps of the measurement process; used to determine instrument contamination. (EPA-QAD)

Instrument Detection Limit (IDL): The minimum amount of a substance that can be measured with a specified degree of confidence that the amount is greater than zero using a specific instrument. The IDL is associated with the instrumental portion of a specific method only, and sample preparation steps are not considered in its derivation. The IDL is a statistical estimation at a specified confidence interval of the concentration at which the relative uncertainty is ± 100%. The IDL represents a range where qualitative detection occurs on a specific instrument. Quantitative results are not produced in this range.

Laboratory Control Sample (however named, such as laboratory fortified blank, spiked blank, or QC check sample): A sample matrix, free from the analytes of interest, spiked with verified known amounts of analytes or a material containing known and verified amounts of analytes, taken through all preparation and analysis steps of the procedure unless otherwise noted in a reference method. It is

generally used to establish intra-laboratory or analyst specific precision and bias or to assess the performance of all or a portion of the measurement system.

An LCS shall be prepared at a minimum of 1 per batch of 20 or less samples per matrix type per sample extraction or preparation method except for analytes for which spiking solutions are not available such as total suspended solids, total dissolved solids, total volatile solids, total solids, pH, color, odor, temperature, dissolved oxygen or turbidity. The results of these samples shall be used to determine batch acceptance.

Least Squares Regression (1st Order Curve): The least squares regression is a mathematical calculation of a straight line over two axes. The y axis represents the instrument response (or Response ratio) of a standard or sample and the x axis represents the concentration. The regression calculation will generate a correlation coefficient (r) that is a measure of the "goodness of fit" of the regression line to the data. A value of 1.00 indicates a perfect fit. In order to be used for quantitative purposes, r must be greater than or equal to 0.99 for organics and 0.995 for inorganics.

Limit(s) of Detection (LOD) [a.k.a., Method Detection Limit (MDL)]: The MDL is the minimum measured quantity of a substance that can be reported with 99% confidence that the concentration is distinguishable from method blank results, consistent with 40CFR Part 136 Appendix B, August, 2017.

Limit(s) of Quantitation (LOQ) [a.k.a., Reporting Limit]: The minimum levels, concentrations, or quantities of a target variable (e.g., target analyte) that can be reported with a specified degree of confidence.

(QS) Matrix: The component or substrate that contains the analyte of interest. For purposes of batch and QC requirement determinations, the following matrix distinctions shall be used:

Aqueous: Any aqueous sample excluded from the definition of Drinking Water or Saline/Estuarine. Includes surface water, groundwater effluents, and TCLP or other extracts.

Drinking Water: Any aqueous sample that has been designated as a potable or potential potable water source.

Saline/Estuarine: Any aqueous sample from an ocean or estuary, or other salt water source such as the Great Salt Lake.

Non-Aqueous Liquid: Any organic liquid with <15% settleable solids.

Biological Tissue: Any sample of a biological origin such as fish tissue, shellfish, or plant material. Such samples shall be grouped according to origin.

Solids: Includes soils, sediments, sludges, and other matrices with >15% settleable solids.

Chemical Waste: A product or by-product of an industrial process that results in a matrix not previously defined.

Air & Emissions: Whole gas or vapor samples including those contained in flexible or rigid wall containers and the extracted concentrated analytes of interest from a gas or vapor that are collected with a sorbant tube, impinger solution, filter, or other device. (TNI)

Matrix Spike (spiked sample or fortified sample): A sample prepared, taken through all sample preparation and analytical steps of the procedure unless otherwise noted in a referenced method, by adding a known amount of target analyte to a specified amount of sample for which an independent test

result of target analyte concentration is available. Matrix spikes are used, for example, to determine the effect of the matrix on a method's recovery efficiency.

Matrix Spike Duplicate (spiked sample or fortified sample duplicate): A replicate matrix spike prepared and analyzed to obtain a measure of the precision of the recovery for each analyte.

Method Blank: A sample of a matrix similar to the batch of associated samples (when available) that is free from the analytes of interest and is processed simultaneously with and under the same conditions as samples through all steps of the analytical procedures, and in which no target analytes or interferences are present at concentrations that impact the analytical results for sample analyses.

Method Detection Limit: See Limit of Detection (LOD)

Negative Control: Measures taken to ensure that a test, its components, or the environment do not cause undesired effects, or produce incorrect test results.

Non-conformance: An indication, judgment, or state of not having met the requirements of the relevant specifications, contract, or regulation.

Observation: A record of phenomena that (1) may assist in evaluation of the sample data; (2) may be of importance to the project manager and/or the client, and yet not at the time of the observation have any known effect on quality.

Performance Audit: The routine comparison of independently obtained qualitative and quantitative measurement system data with routinely obtained data in order to evaluate the proficiency of an analyst or laboratory.

Positive Control: Measures taken to ensure that a test and/or its components are working properly and producing correct or expected results from positive test subjects.

Precision: The degree to which a set of observations or measurements of the same property, obtained under similar conditions, conform to themselves; a data quality indicator. Precision is usually expressed as standard deviation, variance or range, in either absolute or relative terms. (TNI)

Preservation: Any conditions under which a sample must be kept in order to maintain chemical and/or biological integrity prior to analysis. (TNI)

Proficiency Testing: A means of evaluating a laboratory's performance under controlled conditions relative to a given set of criteria through analysis of unknown samples provided by an external source. (TNI)

Proficiency Testing Program: The aggregate of providing rigorously controlled and standardized environmental samples to a laboratory for analysis, reporting of results, statistical evaluation of the results and the collective demographics and results summary of all participating laboratories. (TNI)

Proficiency Test Sample (PT): A sample, the composition of which is unknown to the laboratory and is provided to test whether the laboratory can produce analytical results within specified acceptance criteria. (TNI)

Quality Assurance: An integrated system of management activities involving planning, implementation, assessment, reporting and quality improvement to ensure that a process, item or service is of the type of quality needed and expected by the client. (TNI)

Quality Assurance [Project] Plan (QAPP): A formal document describing the detailed quality control procedures by which the quality requirements defined for the data and decisions pertaining to a specific project are to be achieved. (EAP-QAD)

Quality Control: The overall system of technical activities that measures the attributes and performance of a process, item, or service against defined standards to verify that they meet the stated requirements established by the customer; operational techniques and activities that are used to fulfill requirements for quality; also the system of activities and checks used to ensure that measurement systems are maintained within prescribed limits, providing protection against "out of control" conditions and ensuring that the results are of acceptable quality. (TNI)

Quality Control Sample: A sample used to assess the performance of all or a portion of the measurement system. One of any number of samples, such as Certified Reference Materials, a quality system matrix fortified by spiking, or actual samples fortified by spiking, intended to demonstrate that a measurement system or activity is in control. (TNI)

Quality Manual: A document stating the management policies, objectives, principles, organizational structure and authority, responsibilities, accountability, and implementation of an agency, organization, or laboratory, to ensure the quality of its product and the utility of its product to its users. (TNI)

Quality System: A structured and documented management system describing the policies, objectives, principles, organizational authority, responsibilities, accountability, and implementation plan of an organization for ensuring quality in its work processes, products (items), and services. The quality system provides the framework for planning, implementing, and assessing work performed by the organization and for carrying out required QA and QC activities. (TNI)

Raw Data: The documentation generated during sampling and analysis. This documentation includes, but is not limited to, field notes, electronic data, magnetic tapes, untabulated sample results, QC sample results, print outs of chromatograms, instrument outputs, and handwritten records. (TNI)

Record Retention: The systematic collection, indexing and storing of documented information under secure conditions.

Reference Material: Material or substance one or more properties of which are sufficiently homogeneous and well established to be used for the calibration of an apparatus, the assessment of a measurement method, or for assigning values to materials. (TNI)

Reference Standard: Standard used for the calibration of working measurement standards in a given organization or a given location. (TNI)

Sampling: Activity related to obtaining a representative sample of the object of conformity assessment, according to a procedure.

Second Order Polynomial Curve (Quadratic): The 2nd order curves are a mathematical calculation of a slightly curved line over two axis. The y axis represents the instrument response (or Response ratio) of a standard or sample and the x axis represents the concentration. The 2nd order regression will generate a coefficient of determination (COD or r^2) that is a measure of the "goodness of fit" of the quadratic curvature the data. A value of 1.00 indicates a perfect fit. In order to be used for quantitative purposes, r^2 must be greater than or equal to 0.99.

Selectivity: The ability to analyze, distinguish, and determine a specific analyte or parameter from another component that may be a potential interferent or that may behave similarly to the target analyte or parameter within the measurement system. (TNI)

Sensitivity: The capability of a method or instrument to discriminate between measurement responses representing different levels (e.g., concentrations) of a variable of interest. (TNI)

Spike: A known mass of target analyte added to a blank, sample or sub-sample; used to determine recovery efficiency or for other quality control purposes.

Standard: The document describing the elements of laboratory accreditation that has been developed and established within the consensus principles of standard setting and meets the approval requirements of standard adoption organizations procedures and policies. (TNI)

Standard Operating Procedures (SOPs): A written document which details the method for an operation, analysis, or action, with thoroughly prescribed techniques and steps. SOPs are officially approved as the methods for performing certain routine or repetitive tasks. (TNI)

Storage Blank: A blank matrix stored with field samples of a similar matrix (volatiles only) that measures storage contribution to any source of contamination.

Surrogate: A substance with properties that mimic the analyte of interest. It is unlikely to be found in environment samples and is added to them for quality control purposes.

Surrogate compounds must be added to all samples, standards, and blanks, for all organic chromatography methods except when the matrix precludes its use or when a surrogate is not available. Poor surrogate recovery may indicate a problem with sample composition and shall be reported to the client whose sample produced poor recovery. (QAMS)

Systems Audit (also Technical Systems Audit): A thorough, systematic, qualitative on-site assessment of the facilities, equipment, personnel, training, procedures, record keeping, data validation, data management, and reporting aspects of a total measurement system. (EPA-QAD)

Technical Director: A member of the staff of an environmental laboratory who exercises actual day-to-day supervision of laboratory operations for the appropriate fields of accreditation and reporting of results

Technology: A specific arrangement of analytical instruments, detection systems, and/or preparation techniques.

Traceability: The ability to trace the history, application, or location of an entity by means of recorded identifications. In a calibration sense, traceability relates measuring equipment to national or international standards, primary standards, basic physical constants or properties, or reference materials. In a data collection sense, it relates calculations and data generated throughout the project back to the requirements for the quality of the project. (TNI)

Trip Blank: A blank matrix placed in a sealed container at the laboratory that is shipped, held unopened in the field, and returned to the laboratory in the shipping container with the field samples.

Uncertainty: A parameter associated with the result of a measurement that characterizes the dispersion of the value that could reasonably be attributed to the measured value.

Acronyms:

CAR – Corrective Action Report
CCV – Continuing Calibration Verification
CF – Calibration Factor
CFR – Code of Federal Regulations
COC – Chain of Custody
DOC – Demonstration of Capability
DQO – Data Quality Objectives
DUP - Duplicate
EHS – Environment, Health and Safety
EPA – Environmental Protection Agency
GC - Gas Chromatography
GC/MS - Gas Chromatography/Mass Spectrometry
HPLC - High Performance Liquid Chromatography
ICP - Inductively Coupled Plasma Atomic Emission Spectroscopy
ICP/MS – ICP/Mass Spectrometry
ICV – Initial Calibration Verification
IDL – Instrument Detection Limit
IH – Industrial Hygiene
IS – Internal Standard
LCS – Laboratory Control Sample
LCSD – Laboratory Control Sample Duplicate
LIMS – Laboratory Information Management System
LOD – Limit of Detection
LOQ – Limit of Quantitation
MDL – Method Detection Limit
MDLCK – MDL Check Standard
MDLV – MDL Verification Check Standard
MRL – Method Reporting Limit Check Standard
MS – Matrix Spike
MSD – Matrix Spike Duplicate
SDS - Safety Data Sheet
NELAP - National Environmental Laboratory Accreditation Program
PT – Performance Testing
TNI – The NELAC Institute
QAM – Quality Assurance Manual
QA/QC – Quality Assurance / Quality Control
QAPP – Quality Assurance Project Plan
RF – Response Factor
RPD – Relative Percent Difference
RSD – Relative Standard Deviation
SD – Standard Deviation
SOP – Standard Operating Procedure
TAT – Turn-Around-Time
VOA – Volatiles
VOC – Volatile Organic Compound

Appendix 6. Analytical Method References

Reference methods include:

- *Guidelines Establishing Test Procedures for the Analysis of Pollutants Under the Clean Water Act; Analysis and Sampling Procedures; 40CFR Part 136 as amended by Method Update Rule; August 28, 2017.*
- *Methods for Chemical Analysis of Water and Wastes, EPA 600 (4-79-020), 1983.*
- *Methods for the Determination of Inorganic Substances in Environmental Samples, EPA-600/R-93/100, August 1993.*
- *Methods for the Determination of Metals in Environmental Samples, EPA/600/4-91/010, June 1991. Supplement I: EPA-600/R-94/111, May 1994.*
- *Standard Methods for the Examination of Water and Wastewater, 18th/19th /20th/ on-line edition; Eaton, A.D. Clesceri, L.S. Greenberg, A.E. Eds; American Water Works Association, Water Pollution Control Federation, American Public Health Association: Washington, D.C.*
- *Test Methods for Evaluating Solid Waste Physical/Chemical Methods (SW846), Third Edition, September 1986, Final Update I, July 1992, Final Update IIA, August 1993, Final Update II, September 1994; Final Update IIB, January 1995; Final Update III, December 1996; Final Update IV, January 2008; Final Update V, August 2015.*
- *Annual Book of ASTM Standards, American Society for Testing & Materials (ASTM), Philadelphia, PA.*
- *Code of Federal Regulations (CFR) 40, Parts 136, 141, 172, 173, 178, 179 and 261*
- *Methods for Chemical Analysis of Water and Wastes, EPA 600 (4-79-020), 1983.*
- *Methods for the Determination of Metals in Environmental Samples, EPA/600/4-91/010, June 1991. Supplement I: EPA-600/R-94/111, May 1994.*
- *Standard Methods for the Examination of Water and Wastewater, 18th/19th /20th/ on-line edition; Eaton, A.D. Clesceri, L.S. Greenberg, A.E. Eds; American Water Works Association, Water Pollution Control Federation, American Public Health Association: Washington, D.C.*
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- *Annual Book of ASTM Standards, American Society for Testing & Materials (ASTM), Philadelphia, PA.*
- *Code of Federal Regulations (CFR) 40, Parts 136, 141, 172, 173, 178, 179 and 261*
- *Method 1664, Revision A: N-Hexane Extractable Material (HEM; Oil and Grease) and Silica Gel Treated N-Hexane Extractable Material (SGT-HEM); Non-polar Material) by Extraction and Gravimetry, EPA-821-R-98-002, February 1999*
- *Method 1630, Methyl Mercury in Water by Distillation, Aqueous Ethylation, Purge and Trap and CVAFS, August, 1998.*
- *Method 1631, Revision E: Mercury in Water by Oxidation, Purge and Trap, and Cold Vapor Atomic Fluorescence Spectrometry, EPA-821-R-02-19, August 2002.*

Title: Polychlorinated Biphenyls (PCBs) and PCBs as Congeners by GC/ECD

Method: SW-846 8082 and 8082A

Approvals (Signature/Date):

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Sharon Bacha Date	Steve Jackson Date
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5/8/2020	5/18/2020
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1.0 SCOPE AND APPLICATION

1.1 This SOP describes the procedure for the determination of concentrations of polychlorinated biphenyls (PCB) as Aroclors and as individual Congeners (see Appendix A) using the methodology prescribed in EPA SW-846 Methods 8082 and 8082A.

1.2 This procedure is applicable to the analysis of aqueous, solid, sediment, biological, and waste/oil samples. When utilized for the analysis of waste/oils, additional cleanup procedures may be required.

1.3 This SOP does not include the procedures for extracting environmental samples. Refer to TestAmerica SOPs PT-OP-001 for aqueous and PT-OP-026 for solid and waste sample preparation procedures.

1.4 Table 1 at the end of this document lists the specific Aroclors that are determined using this procedure and their associated reporting limits (RLs).

1.5 On occasion clients may request slight modifications to this SOP. Any modification to this procedure must be approved by the QA department of the Laboratory or Technical Director, and documented fully in an NCM.

2.0 SUMMARY OF METHOD

2.1 Preparation

The extraction procedure is detailed in SOP PT-OP-001 for aqueous samples and PT-OP-026 for solids matrices and wipes.

Extract clean-up procedures are addressed in SOP PT-OP-028.

2.2 Analysis

Samples are analyzed using a gas chromatograph with dual electron capture detectors (ECDs). Specific Aroclor mixtures are identified by the pattern of peaks compared to chromatograms of reference standards. The concentrations of Aroclors in the sample extract are determined using an internal standard calibration.

3.0 DEFINITIONS

3.1 Polychlorinated biphenyls (PCBs): PCBs are a class of organic compounds with 1 to 10 chlorine atoms attached to biphenyl, with a general chemical formula of $C_{12}H_{10-x}Cl_x$. There are 209 possible congeners.

3.2 Aroclor: PCBs were produced as technical mixtures by the chlorination of biphenyl. Production processes were designed to produce mixtures with characteristic chlorine contents. In the United States, most of the PCBs in the environment are in the form of Aroclors, which were produced by Monsanto from the 1930s through 1977. Each Aroclor mixture is identified by a four-digit number, the first two digits of which indicate the number of carbons in the biphenyl ring, i.e., 12, and the second two of which indicate the weight percent of chlorine. For example, Aroclor 1254 has 12 carbons and 54% by weight chlorine. The exception is Aroclor 1016, which has 12 carbons and 41% by weight chlorine.

Each specific Aroclor produces a characteristic gas chromatographic pattern that represents the relative amounts of PCB congeners in the formulation. The

formulation of the mixtures from batch to batch was fairly consistent, but never exactly the same. In almost all cases, the gas chromatogram can be used as a fingerprint to identify the specific Aroclor. Exceptions occurred for Aroclors 1254 and 1221. In each case, at least one batch was produced under different conditions, which resulted in an Aroclor mixture with the same approximate chlorine content, but with a significantly different distribution of congeners. These odd batches of 1254 and 1221 produce chromatographic patterns that are very different from the typical formulations. Standards for these odd batch Aroclors can be used to aid in the qualitative identification of Aroclors in environmental samples.

3.3 TALS: TestAmerica Laboratory Information Management System

3.4 NCM: Non-Conformance Memo – a system within TALS for documenting and communicating QC failures or anomalies encountered during sample receipt, preparation or analysis.

3.5 Please see the glossary of the TestAmerica Pittsburgh Quality Assurance Manual (PT-QA-M-001) for additional definitions.

4.0 INTERFERENCES

4.1 Hydrocarbons can co-elute and thereby mask the Aroclor pattern. The laboratory uses acid cleanup with concentrated sulfuric acid to remove hydrocarbons from solid and oil sample extracts, and for water samples when extracts have noticeable color or whenever there is clear evidence of interferences in the initial sample chromatograms. Acid cleanup removes low-to-medium molecular weight polar organic interferences from sample extracts. Detailed instructions for performing acid cleanup are provided in SOP PT-OP-028.

4.2 Sulfur will interfere and can be removed using procedures described in SOP PT-OP-028.

4.3 Carboprep 90 Cleanup – This method may be performed to remove potential interference prior to analysis if the sample extract is colored.

NOTE: When the Carboprep Cartridge Clean-up Method is used, the recovery of the TCMX surrogate is poor.

4.3.1 Cartridge Method

4.3.1.1 Place approximately 2 ml of sample extract into a test tube and mark the sample volume on the tube.

4.3.1.2 Condition the cartridge by adding 2 ml of methylene chloride and allowing it to drip through the cartridge. Do not allow the cartridge packing to go dry in this or any subsequent step, until the final rinse has been completed.

4.3.1.3 Add 2 ml of hexane/methylene chloride (80%/20%) mixture and allow it to drip through the cartridge until almost empty.

4.3.1.4 Add the sample extract to the cartridge and place the test tube under the cartridge to collect the liquid as it drips through.

4.3.1.5 Rinse 3 times with 2 ml aliquots of hexane/methylene chloride (80%/20%) mixture, while not allowing the cartridge to go dry. After the final rinse, use a pipette bulb to force out all of the remaining liquid in the cartridge.

4.3.1.6 Concentrate the sample extract back down to the original volume according to the mark on the test tube. The extract is now ready for analysis.

4.3.2 Quick Method

4.3.2.1 Add a half scoop of Carboprep 90 to approximately 2 ml of sample extract. Swirl for about one minute and allow the extract to settle.

4.3.2.2 Add a half scoop of Copper Granules to 1 mL of sample or a whole scoop to 2 mL of sample, cap the vial and shake vigorously for 2 minutes.

4.3.2.3 Pipette out an aliquot of extract and filter through a 0.45 µm syringe filter. The extract is now ready for analysis.

4.4 Contamination by carryover can occur when a low concentration sample is analyzed after a high concentration sample. Carryover may also occur with a positive or negative interferrant as well as a target analyte. Any samples analyzed after a samples with high concentrations of target analytes, or high recovery of IS or surrogate compounds, which show the possible effects of carryover must re-analyzed.

4.5 Interferences in the GC analysis arise from many compounds amenable to gas chromatography that give a measurable response on the electron capture detector. Phthalate esters, which are common plasticizers, can pose a major problem in the determinations. Interferences from phthalates are minimized by avoiding contact with any plastic materials.

5.0 SAFETY

5.1 Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual (CW-E-M-001), the Pittsburgh Facility Addendum EH&S Manual (PT-HS-001) and this document. This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use.

5.2 It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

5.3 Specific Safety Concerns and Requirements

5.3.1 Eye protection that protects against splash, laboratory coat, and nitrile gloves must be worn while handling samples, standards, solvents, and reagents. Disposable gloves that have been contaminated must be removed and discarded; non-disposable gloves must be cleaned immediately.

5.3.2 The gas chromatograph contains zones that have elevated temperatures. The analyst needs to be aware of the locations of those zones, and must cool them to room temperature prior to working on them.

5.3.3 There are areas of high voltage in the gas chromatograph. Depending on the type of work involved, either turn the power to the instrument off, or disconnect it from its source of power.

5.3.4 All ^{63}Ni sources shall be leak tested every six months, or in accordance with the manufacturer's general radioactive material license. All ^{63}Ni sources shall be inventoried every six months. If a detector is missing, the TestAmerica Denver Radiation Safety Officer and the TestAmerica Corporate EH&S Director shall be immediately notified and a letter sent to the Colorado Department of Public Health and Environment.

5.4 Primary Materials Used

The following is a list of the materials used in this method, which have a serious or significant hazard rating. This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the SDS for each of the materials listed in the table. A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the SDS for each material before using it for the first time or when there are major changes to the SDS.

Material (1)	**Hazards**	**Exposure Limit** (2)	**Signs and Symptoms of Exposure**
Acetone	Flammable	1000 ppm (TWA)	Inhalation of vapors irritates the respiratory tract. May cause coughing, dizziness, dullness, and headache.
Hexane	Flammable Irritant	500 ppm (TWA)	Inhalation of vapors irritates the respiratory tract. Overexposure may cause lightheadedness, nausea, headache, and blurred vision. Vapors may cause irritation to the skin and eyes.
Methylene Chloride	Carcinogen Irritant	25 ppm (TWA) 125 ppm (STEL)	Causes irritation to respiratory tract. Has a strong narcotic effect with symptoms of mental confusion, light-headedness, fatigue, nausea, vomiting, and headache. Causes irritation, redness and pain to the skin and eyes. Prolonged contact can cause burns. Liquid degreases the skin. May be absorbed through skin.
Hydrogen Gas	Explosive	None	The main hazard is flammability. Exposure to moderate concentrations may cause dizziness, headache, nausea, and unconsciousness. Exposures to atmospheres less than 8 to 10% oxygen will bring about sudden unconsciousness, leaving individuals unable to protect themselves. Lack of sufficient oxygen may cause serious injury or death.

(1) Always add acid to water to prevent violent reactions.
(2) Exposure limit refers to the OSHA regulatory exposure limit.

6.0 EQUIPMENT AND SUPPLIES

The following items are recommended for performing this procedure. Equivalent items should only be used when they result in an improvement in quality, efficiency, productivity, or cost. An item can be considered equivalent if with its use, the analytical and QA/QC requirements in this SOP can be met.

6.1 Instrumentation

6.1.1 A gas chromatographic system with dual columns and dual ECD (^{63}Ni) detectors, and a data system capable of measuring peak area and/or height.

6.2 Supplies

6.2.1 Columns

- Primary Column: RTX-CLP1, 30 m x 0.53 mm id, 0.5 µm coating.
- Secondary Column: RTX-CLP2, 30 m x 0.53 mm id, 0.42 µm coating.
- Additional columns that can be used for confirmation include 30m x 0.53mm id MR1, MR2, RTX5, RTX50 or RTX-1701.

6.3 Autosampler vials, crimp caps with PTFE-faced septa

6.4 Microsyringes, various sizes, for standards preparation, sample injection, and extract dilution

7.0 REAGENTS AND STANDARDS

7.1 The following items are recommended for performing this procedure. Equivalent items should only be used when they result in an improvement in quality, efficiency, productivity, or cost. An item can be considered equivalent if with its use, the analytical and QA/QC requirements in this SOP can be met.

7.2 Reagents

7.2.1 Acetone, 99.4% for organic residue analysis

7.2.2 Hexane, pesticide grade.

7.2.3 Carrier Gas: ≥ 99.99999% pure hydrogen

7.2.4 Make-up Gas: ≥ 99.99980% pure nitrogen

7.3 Standards

7.3.1 Stock Standards

7.3.1.1 All standards must be refrigerated at >0.0 ºC but ≤6.0 ºC. All stock standards must be protected from light. Stock standard solutions should be brought to room temperature before using.

7.3.1.2 Stock standards are monitored for signs of degradation or evaporation. The standards must be replaced annually from the date of receipt or earlier if the vendor indicates an earlier date.

7.3.1.3 Dilutions from stock standards cannot have a later expiration date than the date assigned to the parent stock solutions. The standards must

be replaced at least every six months, or sooner, if comparison with check standards indicates a problem.

7.3.2 PCB, Surrogate and Internal Standard Stock Calibration Standards

7.3.2.1 Stock A

For each of the Aroclors listed in Table 1, a commercially prepared stock standard solution is obtained. Each stock standard contains the specific Aroclor in isooctane at a concentration of 1,000 µg/mL. (Note: At times, specific Aroclor stock standards may also be purchased at various concentration levels, for example 2nd source stock standards are purchased at 200 ug/mL concentration.)

7.3.2.2 Surrogate Stock B

A commercially prepared stock standard solution is obtained that contains the surrogate compounds tetrachloro-m-xylene (TCMX) and decachlorobiphenyl (DCB) in acetone, each at a concentration of 200 µg/mL.

7.3.2.3 Internal Standard Stock Standard

A commercially prepared stock standard solution is obtained that contains the internal standard compound, 1-bromo-2-nitrobenzene, at a concentration of 1000 ug/mL.

7.3.3 Intermediate and Working Level Calibration Standard Solutions

7.3.3.1 Stock C (Level 7 Calibration) Standard Solutions

A Stock C standard solution is prepared for the various Aroclors or combination of Aroclors as summarized in the following table. In each case, the Stock C standard solution is also the highest concentration (i.e., Level 7) calibration standard.

Stock C	**Recipe**	**Conc. (µg/mL)**	**Final Volume (mL)**	**Final Concentrations (µg/mL)**	
AR1660	0.1 mL of Aroclor 1016 Stock A 0.1 mL of Aroclor 1260 Stock A	100	10	Aroclor 1016 Aroclor 1260	4.0 4.0
		200	10	TCMX	0.2
	0.2 mL of surrogate Stock B	200	10	DCB	0.2
AR2154	0.125 mL of Aroclor 1221 Stock A	1000	250	Aroclor 1221	0.5
	0.125 mL of Aroclor 1254 Stock A	1000	250	Aroclor 1254	0.5
AR3262	0.125 mL of Aroclor 1232 Stock A	1000	250	Aroclor 1232	0.5
	0.125 mL of Aroclor 1262 Stock A	1000	250	Aroclor 1262	0.5
AR4268	0.125 mL of Aroclor 1242 Stock A	1000	250	Aroclor 1242	0.5
	0.125 mL of Aroclor 1268 Stock A	1000	250	Aroclor 1268	0.5
AR1248	0.125 mL of Aroclor 1248 Stock A	1000	250	Aroclor 1248	0.5

7.3.4 AR1660 Calibration Levels

7.3.4.1 A total of 7 calibration standards are prepared for AR1660 as summarized in the following table. As needed, the following table can be used to prepare calibration standards for any of the Aroclors, but only the

AR1660 calibration standards include the surrogates. In all cases, measured volumes of the Stock C standard are diluted using pesticide-grade hexane to the final volume indicated in the following table.

Level	Volume of Stock C Used (mL)	Final Volume (mL)	Final PCB Conc (µg/mL)	Final Surrogate Conc (µg/mL)*
1	0.01	100	0.01	0.0005
2	0.10	100	0.10	0.005
3	0.25	100	0.25	0.0125
4 (CCV)	1.0	200	0.50	0.025
5	2.0	200	1.0	0.050
6	2.0	100	2.0	0.100
7	4.0	100	4.0	0.200
*Surrogates are in the AR1660 calibration solutions only. None of the other Aroclor calibration solutions contain the surrogate compounds.				

7.3.5 Working Single-Point PCB Calibration Standards

The Level 4 standard in the table above is used for single-point calibrations of the individual Aroclors. These standards are also used as pattern recognition standards.

7.4 Second-Source Standards for Initial Calibration Verification (ICV)

These standards are purchased from a vendor different from the one that supplied the stock calibration standards.

7.4.1 Second-Source Stock A Aroclor Standard Solutions

Commercially prepared solutions in pesticide-grade hexane (or isooctane) are routinely obtained for Aroclors 1016 and 1260. The Aroclor concentration in each solution is 200 µg/mL. Additionally, a second source standard is obtained for all other Aroclors.

7.4.2 Second-Source Working Level Standards

The working level second-source ICV standard is prepared by combining 0.5 mL of Aroclor 1016 Stock A' and 0.5 mL of Aroclor 1260 Stock A' diluting to a final volume of 200 mL with pesticide-grade hexane. This results in a concentration of 0.5 µg/mL for each of the Aroclors. Note: Surrogates are not added to any 2nd source working level standards.

7.5 Continuing Calibration Verification Standard (CCV), 0.5 µg/mL

The working CCV solution is the same as the Level 4 initial calibration standard, as shown in the table in Section 7.3.4.1.

7.6 Laboratory Control Standard (LCS) Spiking Solution (AR1660)

7.6.1 The LCS/MS spiking solution is prepared and used as part of the scope of the organic preparation SOP PT-OP-001. The following information is provided for reference only.

7.6.2 The working LCS spike solution is prepared in a volumetric flask by combining 1 mL of the Aroclor 1016 Stock standard (10,000 ug/ml) and 1 mL of the Aroclor 1260 Stock standard (10,000 ug/ml), and diluting to a 250 mL final volume with acetone. The final concentration of each Aroclor is 40.0 µg/mL.

7.7 Matrix Spike (MS) Spiking Solution:

The working matrix spike solution is the same as the LCS spike solution. The MS duplicate (MSD) is prepared in the same way using a third aliquot of the selected sample.

Matrix Spike and LCS Solutions		
Analyte Group	Matrix spike Solution ID	Volume added in mL
AR1660	40 ug/mL PCB Spike	1.0 - waters
		0.5 - soils
		0.025 - low-level waters
		0.025 – low-level soils
		1.0 – tissues (40 mL F.V.)
		0.5 – tissues (20 mL F.V.)

7.8 Surrogate Spike Solution

7.8.1 Stock Surrogate Spike Solution: A commercially prepared solution containing 200 µg/mL each of decachlorobiphenyl (DCB) and tetrachloro-m-xylene (TCMX) in acetone is purchased.

7.8.2 Working Surrogate Spike Solution: Samples are spiked with the surrogate compounds during sample preparation, which is described in the organic preparation SOP. The following information is provided for reference only.

7.8.3 The working surrogate spike solution is prepared in a volumetric flask by adding 1.6 mL of the DCB (1000 ug/mL) stock surrogate spike solution, 0.80 mL of the TCMX (2000 ug/mL) stock surrogate spike solution and diluting to a final volume of 2000 mL with acetone. The surrogate compounds are added to all field and QC samples as follows:

Surrogate Spiking Solutions		
Analyte Group	**Surrogate Spike Solution ID**	**Volume added in mL**
PCB Aroclors	0.8 ug/mL DCB/TCMX	1.0 - Waters
		1.0 - Tissues (40 mL F.V.)
		0.5 - Tissues (20 mL F.V.)
		0.5 - Solids
	0.2 ug/mL DCB/TCMX	0.1 - low-level waters
		0.1 - low-level soils

7.9 Internal standard

7.9.1 Stock - A commercially prepared stock standard solution is obtained that contains the internal standard compounds, 1-bromo-2-nitrobenzene and BZ-205, at concentrations of 1000 ug/ml and 100 ug/ml respectively.

7.9.2 A working standard for the internal standard, 1-bromo-2-nitrobenzene and BZ-205, is prepared at a concentration of 10 ug/ml. This standard is added to all samples and standards to result in a final concentration of 0.1 ug/ml (20 ul of 1 ug/ml to 200 ul; 10 ul to 100 ul; etc.) after any cleanups or dilutions that may be necessary but before injection.

7.10 Aroclor mixes

The following Aroclors can be analyzed together for calibration and calibration

verification: 1016/1260, 1221/1254, 1232/1262, 1242/1268. If standards are mixed

together, concentrations need to be taken into account (initial concentrations and

final volumes). A calibration curve will be analyzed for 1660 and midpoint

standards will be analyzed for all other Aroclors.

7.11 RT standard

It is an option to make a multi Aroclor standard for the purposes of the daily RT update.

8.0 SAMPLE COLLECTION, PRESERVATION, SHIPMENT AND STORAGE

8.1 Water samples are collected in pre-cleaned amber glass bottles fitted with a Teflon-lined caps. To achieve routine reporting limits, a full one liter of sample is required for regular level extraction and 250mL for LVI extraction*. Because the entire sample volume is used for extraction, additional bottles are needed to satisfy the requirements for matrix spike and matrix spike duplicate analyses.

8.2 Soil samples are collected in 8-ounce, wide-mouth jars with a Teflon-lined lid.

Matrix	Sample Container	Preservation	Extraction Holding Time	Analysis Holding Time	Reference
Waters	Amber glass	Cool >0.0 but ≤6.0°C	1 year	1 year from extraction	40 CFR Part 136.3
Soils	Glass	Cool >0.0 but ≤6.0°C	14[1] Days	40[1] Days from extraction	N/A
Tissues	Borosilicate glass, PTFE, quartz, aluminum foil	Freeze at < - 10°C can be stored this way for 1 year prior to extraction	14 Days from date of thawing	40 Days from extraction	PSEP [2]

[1] Some states will accept a 1 year holding time from sampling to extraction and from extraction to analysis for Solid samples. Consult the Project Manager or the QA Department for specific project requirements before exceeding 14/40 days.

[2]This maximum holding time is also recommended by the PSEP (1990). The California Department of Fish and Game (1990), and the USGS National Water Quality Assessment Program (Crawford and

Luoma, 1993) recommend a more conservative maximum holding time of 1 year at < -10℃ for dioxins/furans. NOAA recommends a maximum holding time of 3 months.

*NOTE: LVI cannot be used for samples from South Carolina. The 1 Liter bottles must be used for sampling.

9.0 QUALITY CONTROL

9.1 Refer to the TestAmerica Pittsburgh QC program document (PT-QA-021) for further details on criteria and corrective actions. Refer to "project checklist" for project specific requirements. More stringent project-specific requirements will supersede the requirements presented in this section when there is a written agreement between the laboratory and the client, and the source of those requirements should be described in the project documents. Project-specific requirements are communicated to the analyst via special instructions in the LIMS.

9.2 Any QC result that fails to meet control criteria must be documented in a Nonconformance Memo (NCM). The NCM is approved by the supervisor and then automatically sent to the laboratory Project Manager by e-mail so that the client can be notified as appropriate. The QA group also receives NCMs by e-mail for tracking and trending purposes. The NCM process is described in more detail in SOP PT-QA-016. This is in addition to the corrective actions described in the following sections.

9.3 If dual column reporting is required, the choice of which column to report the QC from is made in the same manner as for the associated samples.

9.4 The following Quality Controls are required:

Quality Controls	Frequency	Acceptance Limits
Initial Calibration: Minimum 5 points for 1016/1260, One point for all other Aroclors Minimum 6 points for Congeners	Every 6 months, or more often as needed	Average RF: ≤ 20%RSD Linear Regression: Linear Correlation Coefficient ≥0.990 and intercept <1/2 RL
ICV (second source)	After each ICAL	≤ 20%D (80-120% recovery)
CCV[1]	Beginning of run and every 20 samples	8082 ≤ 15%D (85-115% recovery) 8082A ≤ 20%D (80-120% recovery)
RT Windows	With each ICAL (mid-point updated daily with CCVIS)	± 3X SD / ± 0.01 min
Internal Standard (IS)	All standards, samples, and QC	50 – 150% of the response in mid-level calibration standard
Method Blank (MB)	1 per preparation batch	<RL
Laboratory Control Sample (LCS)	1 per preparation batch	Statistical control limits are maintained in TALS.
Matrix Spike (MS)	1 per preparation batch	

Quality Controls	Frequency	Acceptance Limits
		Statistical control limits are maintained in TALS. MS is not used for batch control.
Matrix Spike Duplicate (MSD)	1 per preparation batch	Statistical control limits are maintained in TALS. MSD is not used for batch control.
Surrogates	All samples and QC	Statistical control limits are maintained in TALS.
Dual column agreement	All positive results	40% RPD (see section 11.4 for reporting instructions)

[1]If the analysis includes the use of internal standards, a closing CCV is not required at the end of the analytical run.

9.5 Method blank

9.5.1 A method blank (MB) is prepared and analyzed with each batch of 20 or fewer samples. The method blank consists of reagent water (for aqueous sample batches) or 50:50 sodium sulfate : magnesium sulfate mixture (for solid sample batches) to which the surrogate compounds are added (see SOP PT-OP-001 or PT-OP-026). The method blank is subject to the entire extraction, cleanup and analysis process. The method blank must be analyzed daily prior to sample analysis to demonstrate that the chromatographic system is contaminant free.

9.5.2 The method blank must not contain any analyte of interest at or above the reporting limit (RL). For analytes detected above the MDL, all associated results will be reported with B qualifiers. If the MB is found to be contaminated above the RL, the sample batch must be re-extracted / reanalyzed unless one of the following criteria is met:

- Evaluate the associated sample results. If sample results are non-detect for the Aroclor found in the MB, results may be reported using the “Method Blank – Report, ND” NCM.
- If the associated sample concentrations are >10x the MB concentration, results for these samples may be reported using the “Method Blank – Report, 10X” NCM.
- If neither of the above situations exists, and re-extraction is not possible due to limited sample volume or holding time, the lab must notify the Project Manager. Instructions must be sought from the client on how to proceed with reporting these results. Results that are reported must be properly qualified and an NCM created to document the situation for the case narrative.

9.6 Laboratory Control Sample (LCS)

9.6.1 One LCS is prepared and analyzed with each batch of 20 or fewer samples. The LCS is prepared as described in Section 7.6, using the same matrix as for the MB. The LCS is subject to the entire extraction, cleanup and analysis process.

9.6.2 The LCS recovery must be within the established control limits. The laboratory's standard control limits are set at ± 3 standard deviations around the historical mean, unless project requirements dictate otherwise. Current control limits are maintained in the LIMS. Unless otherwise requested by the client, the PCB LCS contains only Aroclors 1016 and 1260.

9.6.3 For batches that include samples from South Carolina, the LCS control limits are static at 70-130%.

9.6.4 Laboratory generated Control Limits are maintained in TALS and can be provided upon request.

9.6.5 If any analyte is outside the control limits in the LCS, the batch must be re-extracted / reanalyzed unless the following criterion is met:

- If the LCS recovery is above the upper control limit and sample results are non-detect, those associated sample results may be reported with the appropriate qualifier and using the “LCS/LCSD - %R High” NCM.
- If the above situation does not exist, and re-extraction is not possible due to limited sample volume or holding time, the lab must notify the Project Manager. Instructions must be sought from the client on how to proceed with reporting these results. Results that are reported must be properly qualified and an NCM created to document the situation for the case narrative.

9.7 Matrix Spike (MS) and Matrix Spike Duplicate Samples (MSD)

9.7.1 One MS/MSD pair is required with each analytical batch of 20 or fewer samples. Note that some programs (e.g., North Carolina and South Carolina) require preparation and analysis of an MS/MSD pair at a 10% frequency. This shall be noted in the project when required. Preparation of the MS is described in Section 7.7. The MSD is another aliquot of the sample selected for the MS that is spiked in the same manner as the MS.

9.7.2 The MS and MSD recoveries must fall within the established LCS control limits. The relative percent difference (RPD) between the MS and MSD must be less than the established limit, which is based on statistical analysis of past results, unless otherwise dictated by project requirements. Current control limits are maintained in the LIMS.

Laboratory generated Control Limits are maintained in TALS and can be provided upon request.

9.7.3 If analyte recovery or RPD falls outside the acceptance range, but the associated LCS recovery is in control, and all other QC criteria (e.g., continuing calibration verification) are met, then there is no evidence of analytical problems. This may indicate that the sample matrix itself has an adverse effect on recovering the analyte.

- If a matrix effect is suspected, the parent sample is qualified appropriately, and results are reported using the "MS/MSD - %R, LCS Pass" NCM.
- If the recovery for any component is outside control limits for both the MS and the LCS, the laboratory is out of control and corrective action must be taken. Refer to section 9.6.4.
- If an MS/MSD is not possible due to limited sample, then an LCS duplicate (LCSD) may be analyzed to monitor batch precision.

9.7.4 The MS/MSD must be analyzed at the same dilution level as the parent sample, unless the matrix spike components would then be above the calibration range. In this case, would require a dilution greater than the parent sample to bring the spiked compounds within the calibration range.

9.7.4.1 For PCB analysis, the matrix spike added is diluted to concentrations below the RL at a dilution of 40X. MS/MSD that are diluted above 40X must be reported with a "D" qualifier on the spiked compounds to indicate that recovery is not able to be accurately calculated.

9.7.4.2 If the parent sample concentration is >4X, it may not be possible to accurately calculate %recoveries. Results will be reported with the "MS/MSD – High Targets" NCM.

9.8 Surrogates

9.8.1 Each field sample, QC sample, and calibration standard, is spiked with surrogate compounds decachlorobiphenyl (DCB) and tetrachloro-m-xylene (TCMX). The surrogate spike solution is prepared as described in Section 7.8.

9.8.2 For field samples and batch QC, the surrogate recoveries must be within the established control limits, which are set at ± 3 standard deviations around the historical mean, unless project requirements dictate otherwise. Surrogate recovery limits are maintained in TALS.

9.8.2.1 Surrogate recoveries for both DCB and TCMX in the MB and LCS both must pass on at least one column. If the recovery of the surrogates in MB or LCS's is outside of the control limits, check for calculation or instrument problems.

- Surrogate recoveries (one or both) outside of control limits on both columns for the MB or LCS require re-preparation and reanalysis of the entire preparation batch.

9.8.2.2 For field samples, surrogate recovery is calculated and reported for both DCB and TCMX. At least one surrogate must pass on the one column from which Aroclors are identified and reported, and that column must have a passing CCV.

9.8.2.3 For CCV standards, Aroclors 1016 and1260 and surrogates must pass on the reporting column, for the data to be considered reportable.

9.8.2.4 If both surrogates fail on both columns for a sample, and matrix interference is not obvious from the initial analysis, it is only necessary to re-prepare and reanalyze a sample once to demonstrate that poor surrogate recovery is due to matrix effects, as long as the extraction/instrument system is proven to be working properly according to the batch and instrument QC.

9.8.2.5 For PCB analysis, the surrogate spike added is diluted to concentrations below the RL at a dilution of 40X. Samples that are diluted above 40X must be reported with a "D" qualifier on the surrogates to indicate that recovery is not able to be accurately calculated.

9.9 Initial Calibration

9.9.1 See section 10.3 below for initial calibration options and evaluation requirements.

9.9.2 A full calibration (minimum 5 points) is required on both columns for Aroclor 1016 and 1260 and the two surrogates plus internal standards. A single point is used for all other Aroclors.

9.9.3 Each peak in the Aroclor (a minimum of 5 peaks is required for all Aroclors, except 1221, which requires a minimum of 3 peaks) is intergrated using peak height.

9.9.4 The calibration must be verified by the analysis of a second source standard (ICV) near the mid-point of the calibration range. The %Drift/Difference for this standard must be $\leq$ 20% from the ICAL. (The response for each individual peak for the Aroclors in the ICV standard must be within ± 20.0% of the expected value.

If the ICV response does not meet this limit, repeat the ICV analysis once with a freshly made standard. If failure repeats, the initial calibration and ICV standards must be prepared fresh and calibration repeated.

9.10 Continuing Calibration Verification (CCVIS)

NOTE: It is not necessary to run a CCV standard at the beginning of the sequence if samples are analyzed immediately after the completion of the initial calibration and ICV.

9.10.1 A mid-level calibration standard of 1016/1260 is analyzed as the continuing calibration verification (CCVIS) standard at the beginning of the analytical sequence, and after every 20 samples (all injections except solvent blanks count as samples), or every 12 hours, whichever is more frequent. Depending on the type of samples, it may be advisable to analyze verifications more frequently in order to minimize reruns. If 12 hours elapsed since the last CCV was analyzed, a new sequence is started with a fresh CCV.

9.10.2 A mid-range CCV standard of each Aroclor must be analyzed at least every 24 hours, for retention time updating.

9.10.3 If Aroclors other than 1016 or 1260 are detected in any sample, a CCV of the associated Aroclor(s) must be analyzed within 12 hours of the sample and pass criteria in order for the sample data to be reported. If a CCV is not run within 12 hours of the sample, or if the CCV does not meet the method CCV criteria, the sample must be re-analyzed along with a passing CCV for that compound. CCV's of other Aroclors may be run every 20 samples/12 hours along with the 1016/1260 CCVs in a routine analysis sequence to meet this requirement. If the analysis includes the use of internal standards, a closing CCV is not required at the end of the analytical run.

9.10.4 Acceptance criteria for CCVs is < 15%D for method 8082 and <20%D for 8082A. All Aroclors (each individual peak) and surrogates must meet these recovery limits on both columns, unless only reporting from one column. If a CCV does not meet the acceptance criteria explained above, associated samples must be re-analyzed unless one of the following conditions is met:

- If the CCV (or individual peak) %D fails high, >15.0% for 8082 or >20.0% for 8082A, and the sample results are ND for that compound, the data may be reported with the appropriate qualifier and the "CCV - %D, High, Samples ND" NCM.
- If the CCV % D fails low, and samples results are ND, then a low level standard can be analyzed to prove that the instrument is sensitive enough to call the sample "Not Detected" and the sample can be reported. This low level standard must be analyzed after all batch samples to prove that sample matrices have not affected the ability to recover compounds of interest at the reporting limit. Results may be reported with proper qualification and using the "CCV – Low Biased, ND, RL Check Passes" NCM.
- If the CCV fails on the non-reporting, or confirmation, column, and that failure does not exceed 40%D, results may be reported without qualification.
- If a continuing batch CCV fails, and there is evidence that the sample matrix has affected the column performance, samples must be rerun once to confirm this affect. Results may be reported from the first or second analysis, with proper qualification and using the "CCV - %D, Capping, RR Confirms" NCM.
- If more than 5 (or 3 for 1221) peaks are used for

identification/quantitation, and a single peak fails to meet CCV criteria, that peak may be removed from the quantitation for the affected CCV and all associated samples and QC, as long as the minimum number of peaks remain.

9.11 Retention Time (RT) Windows

9.11.1 Absolute retention times are used for the identification of PCBs as Aroclors. However, in addition to retention times, peak patterns play a large role in the identification of Aroclors.

9.11.2 The center of the retention time (RT) window shall be updated based on the middle level in the initial calibration or the first CCV (CCVIS) in the daily analytical sequence, whichever is more recent,.

9.11.3 Before establishing windows, make sure the GC system is within optimum operating conditions. Evaluate the deviation from expected RT for each analyte in at least three CCV and/or LCS samples spread over at least 72 hours.

- If three days of analytical data are not available, use a default RT window of 0.010 minutes. At the end of the batch, evaluate all CCVs and LCS in the batch. If necessary, widen the window such that all analytes fall within the RT window. Reprocess the batch using the new RT windows.
- Multiply the maximum deviation from the set by 1.5. This is the retention time window, unless the result is less than 0.010 minute, in which case the window is set at 0.010 minute.
- Determine the retention time (RT) windows for the 3-5 major peaks selected for each Aroclor. The AR1016 windows will be used to establish retention time windows for PCBs AR1221, AR1016, AR1232, AR1242, and AR1248. The AR1260 windows will be used to establish retention time windows for PCBs AR1254, AR1260, AR1262, and AR1268.
- Retention time windows for analytes of interest must not overlap.

9.11.4 Evaluate the retention time windows on an ongoing basis. The center of the RT window is updated on the first CCV (CCVIS) of the day. All analytes for all subsequent CCVs, LCS and matrix spikes must fall within the RT window, except as discussed below.

Matrix spike analytes may fall outside the RT window if there is a large, non-target peak co-eluting, or overlapping, with the analyte in the matrix.

9.11.5 If any analyte falls outside the RT window in CCV, LCS or matrix spike (except as discussed above for matrix spikes), then the RT window for that analyte shall be widened to the minimum degree required for the analyte to fall within the RT window. All samples in the batch shall be reprocessed with the new RT window, and the wider RT window shall remain in place for subsequent batches.

9.11.6 Retention time windows should be reliably narrower than +/- 0.03

minute. If RT windows wider than this are necessary, the instrument should be evaluated and maintenance performed as needed. Subsequent to maintenance, RT windows shall be narrowed to the extent that is consistent with the data obtained and the foregoing requirements.

9.11.7 The laboratory must calculate retention time windows for each analyte on each GC column and whenever a new type of GC column is installed. The data must be retained by the laboratory and available for review.

9.12 Control Limits: In-house historical control limits must be determined for surrogates, matrix spikes, and laboratory control samples (LCS). These limits must be reviewed at least annually. The recovery limits are mean recovery +/- 3 standard deviations, unless that limit is tighter than the calibration criteria, in which case limits may be widened. Refer to policy PT-QA-021 for more details.

9.12.1 Surrogate and matrix spike recoveries will be reported unless the dilution is more than 40X.

9.13 Percent Moisture

9.13.1 Analytical results may be reported as dry or wet weight, as required by the client. Percent moisture must be determined on a separate portion of the sample if results will be reported based on dry weight.

9.14 Internal Standard Criteria

9.14.1 If the internal standard response in the continuing calibration is more than 150% or less than 50% of the response in the mid-level calibration standard, the chromatographic system must be inspected for malfunctions and corrected. Reanalysis of samples analyzed while the system was malfunctioning is required.

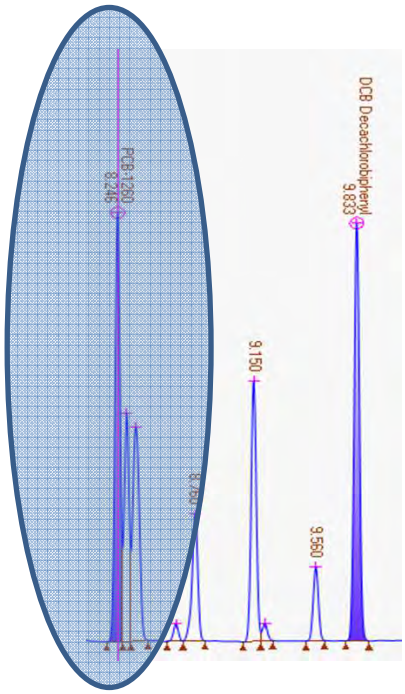
9.14.2 Any samples or standards that do not meet the internal standard criteria of > 50% and <150% of the response of the mid-level calibration standard must be evaluated for validity. Samples that are reported with internal standard exceedances must have documentation supporting matrix effect. Where the matrix effect is well established it may be reported with narration, otherwise the samples must be reanalyzed to confirm matrix effect is required. If the internal standard exceedance is deemed to be due to an instrumental problem, instrument maintenance will be done and all affected samples must be reanalyzed after the problem is corrected.

9.15 Resolution Check requirement

9.15.1 The circled triplet of peaks in the figure below is observed towards the end of the 1260 pattern on columns such as CLP 1. Minimum resolution (degree of overlap) requirement between peak 1 / 2 and peak 2 / 3 is <75%. This chromatogram shows overlap of about 50% between peak 2 and 3, and 30% between peak 1 and 2.

9.15.2 Resolution (degree of overlap) is calculated as follows:

[Height of the valley / (Sum of the two peak heights / 2)] x 100%



9.16 Procedural Variations

9.16.1 Any variation in procedure shall be completely documented using a Nonconformance Memo and approved by a supervisor and QA Manager. If contractually required, the client shall be notified. The Nonconformance Memo shall be filed in the project file. The nonconformance is also addressed in the case narrative. Any unauthorized deviations from this procedure must also be documented as a nonconformance, with a cause and corrective action described.

10.0 PROCEDURE

10.1 Sample Preparation

10.1.1 Samples are extracted and prepared for analysis as described in SOPs PT-OP-001 and PT-OP-026. Allow extracts to warm to room temperature before injecting.

10.1.2 If needed, extracts are cleaned up using the procedure in section 4.3 of this SOP or one of the clean-up procedures in SOP PT-OP-028. Any clean-up performed is documented either within the method chain or using an NCM.

10.1.3 Internal standard is added to each extract prior to loading the extract on the instrument for analysis. Add 1µL of Internal Standard Spike (7.9) per 100µL of extract or standard.

10.2 Calibration

Instrument conditions for this method are presented in Table 2.

10.2.1 Use the ChemStation interface to establish instrument operating conditions for the GC.

10.2.2 Raw data obtained by the ChemStation software is transferred to the CHROM DB(database) for further processing. The data analysis method, including peak processing and integration parameters, calibration, RT windows, and compound identification parameters, is set up in the CHROM DB software.

10.2.3 Transfer calibration standard solutions into autosampler vials and load into the GC autosampler. Use the ChemStation software to set up the analytical sequence.

10.2.4 Unprocessed calibration data are transferred to the CHROM DB database for processing. After processing the calibration data, print the calibration report and review it using the calibration review checklist (GC and HPLC Data Review Checklist - ICAL). Submit the calibration report to a qualified peer or the group leader for final review. The completed calibration reports are scanned and stored as Adobe Acrobat files on the Public Drive.

10.2.5 A new calibration curve must be generated initially, after major changes to the system, or when continuing calibration criteria cannot be met. Major changes include installation of new type of column and replacing the ECD detector. Initial Calibration will be performed at least every 6 months.

10.3 Initial Calibration (ICAL)

10.3.1 An initial standard calibration using seven concentration levels of the AR1660 mixture is routinely performed. (At least five calibration levels are required.) Table 3 provides concentration levels for Aroclor 1016, Aroclor 1260, and the surrogate compounds DCB and TCMX.

10.3.2 All initial calibration points must be analyzed without any changes to instrument conditions, and all points must be analyzed within 24 hours.

10.3.3 The calibration curves for Aroclors 1016 and 1260 and the surrogate compounds, relative to the internal standard response, are modeled either as average response factors (RF) or as calibration curves using a systematic approach to selecting the optimum calibration function. PCB's are well established linear responding compounds and an average curve fit should be the norm. Use of linear regression is acceptable, however the individual points should be evaluated to determine if there has been a change in the instrument sensitivity or if there is a potential issue with the standards.

10.3.4 The calibration for each of the other Aroclors (see Tables 1 & 3) is initially determined using a single, mid-level calibration standard. As needed, the laboratory may generate a multi-point calibration for other commonly detected Aroclors, such as 1221, 1254, and 1248. When additional multi-point calibrations are developed for the other Aroclors, a second-source ICV standard is also analyzed, see section 9.9.

NOTE: Samples from sites known to be contaminated with specific Aroclors should be analyzed using a multi-point calibration curve for the identified Aroclors. This information is provided to the analyst through special instructions in the LIMS.

NOTE: Generally, it is NOT acceptable to remove points from a calibration for the purposes of meeting calibration criteria, unless the points are the highest or lowest on the curve AND the reporting limit and/or the linear range is adjusted accordingly. A minimum of five levels must remain in the calibration for a linear regression. The documentation must be retained with the initial calibration. Alternatively, if the analyst believes that a point on the curve is inaccurate, the point may be reanalyzed and the reanalysis used for the calibration.

10.3.5 The high and low standard for the initial calibration of the AR1660 mixture defines the acceptable quantitation range for all of the Aroclors. If a sample extract contains any Aroclor at a concentration that exceeds the upper range of the calibration, then the extract must be diluted and reanalyzed.

10.3.6 Select 5 major peaks (at least 25% peak height of the largest peak in the aroclor) in the analyte pattern (only 3 peaks are usable for Aroclor 1221). The response factors or curves will be generated for each of these major peaks for each Aroclor. The results are based off of an average of the calculated concentrations of these major peaks.

NOTE: A minimum of five accurate peaks must be used to quantify an Aroclor (three peaks for Aroclor 1221).

NOTE: See Appendix A for more details on peak choice and identification of individual Aroclors.

10.4 Internal Standard Calibration

10.4.1 Internal standard calibration involves the comparison of instrument responses from the samples to the responses from the target compounds in the calibration standards. The area (or height) of a peak and the area (or height) of the internal standard in a sample chromatogram is compared to the area (or height) of the peak and the area (or height) of the internal standard in the standard chromatograms that appears at the same retention time. The ratio of the detector response to the concentration of the target analyte in the calibration standard is defined as the response factor (RF) and is calculated as follows:

$$RF = \frac{Ax \times Cis}{}$$

$$\frac{A_{is} \times C_x}{A_x}$$

Where:

A_x = Area (or height) of the peak to be measured

A_{is} = Area (or height) of the peak for the internal standard

C_{is} = Concentration of the internal standard

C_x = Concentration of the peak being measured

NOTE: It is also possible to use the concentration of the standard rather than the mass injected.

10.5 Establishing the Calibration Function

10.5.1 Calibrations are modeled either as average response factors (RF) or as linear regression curves, using a systematic approach to select the optimum calibration function. Start with the simplest model, i.e., average RF, and progress through the other options until calibration acceptance criteria are met. PCB's are well established linear responding compounds and an average curve fit should be the norm. If linear regression is utilized, it is acceptable, however the individual points should be evaluated to determine if there has been a change in the instrument sensitivity or if there is a potential issue with the standards. Different calibration models can be applied to each of the major peaks chosen to represent the Aroclor.

NOTE: Each individual peak used for each Aroclor must meet the calibration acceptance criteria.

10.5.2 Linear Calibration Using Average Response Factor (RF)

The response factor is a measure of the slope of the calibration line, assuming that the line passes through the origin. Under ideal conditions, the factors calculated for each calibration level will not vary with the concentration of the standard. In practice, some variation can be expected. When the variation, measured as the relative standard deviation, is relatively small (e.g., ≤ 20%), the use of the straight line through the origin model is generally appropriate.

The average response factor is calculated as follows:

$$\text{Average RF} = \overline{RF} = \frac{\sum_{i=1}^{n} RF_i}{n}$$

Where:

RF_i = The response factor for the i^{th} calibration level.

n = The number of calibration levels.

The relative standard deviation (RSD) is calculated as follows:

$$RSD = \frac{SD}{\overline{RF}} \times 100\%$$

Where SD is the standard deviation of the average RF, which is calculated as follows:

$$SD = \sqrt{\frac{\sum_{i=1}^{n} \left(RF_i - \overline{RF}\right)^2}{n-1}}$$

10.5.3 Evaluation of the Average Response Factor:

Plot the calibration curve using the average RF as the slope of a line that passes through the origin. Examine the residuals, i.e., the difference between the actual calibration points and the plotted line. Particular attention should be paid to the residuals for the highest points, and if the residual values are relatively large, a linear regression should be considered.

- The RSD for each peak chosen to represent the aroclor must be ≤ 20.0% for both 8082 and 8082A.
- If the %RSD is <20.0% (8082/8082A), alternative curve fits may still be considered.
- If the RSD exceeds the limit, linearity through the origin cannot be assumed, and alternative curve fits must be considered.

10.5.4 Linear Calibration Using Least-Squares Regression (weighted) - Evaluation of the Linear Least-Squares Regression Calibration Function

With an unweighted linear regression, points at the lower end of the calibration curve have less weight in determining the curve than points at the high concentration end of the curve. For this reason, inverse weighting of the linear function is recommended to optimize the accuracy at low concentrations. Note that the August 7, 1998 EPA memorandum "Clarification Regarding Use of SW-846 Methods", Attachment 2, Page 9, includes the statement "The Agency further recommends the use of this for weighted regression over the use of an unweighted regression."

- To avoid bias in low level results, the absolute value of the y-intercept must be significantly less than reporting limit (RL), and preferably less than the MDL.
- Also examine the residuals, but with particular attention to the residuals at the bottom of the curve. If the intercept or the residuals are large, the calibration should be repeated since a higher order regression is not allowed for this method.
- If weighted linear regression is used 1/(Amt) or 1/(Amt)2 a minimum of 5 points are required. The coefficient of determination (r2) must be >

0.990. If the correlation of determination falls below the acceptance limit, linear regression cannot be used and a second-order regression should be attempted.

- If the correlation coefficient falls below the acceptance limit, the linear regression is unacceptable and the calibration should be repeated since a higher order regression is not allowed for this method.
  - For non-detects, reanalysis is not required, and flagging is not required.

10.5.5 Other curve types

Due to the stability of the compounds, non-linear calibration curves (quadratic) and unweighted linear regression curves are not applicable for this method.

10.5.6 Calibration point read-back criteria

Whichever calibration mode above is selected, it should be subjected to an additional check to establish the representativeness of the data that were used to produce it. This check is the refitting of each calibration point response back to the calibration model, or the comparison of the calculated amount of the standard against the expected amount.

- CHROM software provides an Initial Calibration %Drift report which shows the % Error for each calibration point. This report must be reviewed in addition to the %RSD / Linear Response Factor.
- The absolute value of the % Error for each calibration point should be $\leq$ 30%. For the lowest calibration point, the % Error may be $\leq$ 50%.

10.5.7 Removal of calibration points and/or levels

10.5.7.1 Certain analytes that have particularly high or low sensitivity may need to have the high or low calibration points removed in order to optimize the calibration curve. Removal of high or low calibration points is allowed, but the working range of the calibration must be adjusted accordingly. The reporting limit must be supported by the lowest point remaining in the calibration and any samples with concentrations above the highest point in the calibration must be diluted and reanalyzed.

10.5.7.2 Points for individual compounds from the interior levels of the calibration may not be removed. A complete level may be removed if the laboratory can document a clear reason for incorrect response in the standard (for example, analysis of the incorrect level, failure to add internal standards or instrument malfunction).

10.6 Second-Source Initial Calibration Verification (ICV)

10.6.1 Second-source ICV standards are analyzed for all Aroclors. The stock

standards are obtained from a source different than that of the standards used for the calibration. The ICV standards are analyzed immediately following the initial calibration. Refer to section 9.9.4 for acceptance criteria.

10.7 Sample Analysis

10.7.1 An autosampler is used to introduce samples into the chromatographic system by direct injection of 1 or 2 µL of the sample extract.

10.7.2 Samples, standards, and QC samples must be introduced using the same procedure.

10.7.3 All extracts and standards are allowed to warm to room temperature before injection.

10.7.4 Use the ChemStation interface to set up and run the analytical sequence. Sample injection and analysis are automated and may proceed unattended.

10.8 Analytical Sequence

10.8.1 An analytical sequence starts with a minimum five-level initial calibration (ICAL) or a daily calibration verification. The daily run sequence is generated electronically using the ChemStation software.

The following is the typical analytical sequence for routine sample analysis:

Suggested Analytical Sequence		
Initial Calibration	(not required daily)	
Injection No.		
1	Solvent Blank (optional)	
2	Aroclor 2154	Level 4
3	Aroclor 3262	Level 4
4	Aroclor 4268	Level 4
5	Aroclor 1248	Level 4
6	Aroclor 1016/1260	Level 1
7	Aroclor 1016/1260	Level 2
8	Aroclor 1016/1260	Level 3
9	Aroclor 1016/1260	Level 4
10	Aroclor 1016/1260	Level 5
11	Aroclor 1016/1260	Level 6
12	Aroclor 1016/1260	Level 7
13-17	ICV's (2^{nd} source standard of all Aroclors)	
18	Method Blank	
19-38	Sample 1-20 (or 12 hours)	
39	Aroclor 1016/1260	Level 4
At least every 24 hours, counting from the start of the initial calibration or from the start of the last daily calibration, the retention time windows must be updated. CCVs for 1016/1260 must be run every 20 samples or 12 hours, whichever is more frequent. Mid-level standards of any other Aroclors expected to be present in the samples are also injected. For sequences without an initial calibration, start with CCVIS 1660 and CCVs for 1248 and 2154, method blank plus 19 samples, then CCVIS 1660 and CCVs for 3262 and 4268, method blank plus 19 samples. This way all standards are run within 24 hours for RT's and samples with detections other than 1660 have CCVs within 12 hours.		

10.9 When a sample result exceeds the upper calibration range, then that sample extract is diluted to obtain a result in the upper half of the calibration range and reanalyzed. Any samples that were analyzed immediately following the high sample are evaluated for carryover. If the samples had target analyte detections at or above the RL, the samples must be reanalyzed to rule out carryover.

10.10 Upon completion of the analytical sequence, transfer the raw chromatography data to the CHROM DB database for further processing. Review chromatograms online and determine whether manual data manipulations are necessary. All manual integrations must be justified and documented. See CA-Q-S-002 for requirements for manual integration.

10.11 Manual integrations may be processed using an automated macro, which prints the before and after chromatograms and the reason for the change, and attaches the analyst's electronic signature. Alternatively, the manual integration may be processed manually. In the latter case, print both the both the before and after chromatograms and record the reason for the change and initial and date the after chromatogram. Before and after chromatograms must be of sufficient scale to allow an independent reviewer to evaluate the manual integration.

10.12 Compile the raw data for all the samples and QC samples in a batch. The analytical batch is defined as containing no more than 20 samples, which include field samples and the MS and MSD. Perform a level 1 data review and document the review on the data review checklist (GC Data Review Checklist). Submit the data package and review checklist to a peer analyst for the level 2 review. The data review process is explained in SOP PT-QA-018.

11.0 CALCULATIONS / DATA REDUCTION

11.1 Qualitative Identification of Aroclors

Retention time windows are used for identification of Aroclors, but the "fingerprint" produced by major peaks of those analytes in the standard is used in tandem with the retention times for identification. The ratios of the areas of the major peaks are also taken into consideration. Identification may be made even if the retention times of the peaks in the sample fall outside of the retention time windows of the standard, if in the analyst's judgment the fingerprint (retention time and peak ratios) resembles the standard chromatogram. If there are any anomalies in the pattern due to the presence of multiple PCBs or weathering of peaks the analyst should discuss this with another analyst or technical director to come to a consensus about which PCB(s) should be identified. An NCM should be completed indicating the rationale behind the decision made. Sometimes Aroclors 1262 and 1268 are not requested as target analytes. If these patterns are observed, an NCM should be completed informing the project manager of possible PCB detection.

NOTE: See Appendix A for more details on peak choice and identification of individual Aroclors.

11.2 Quantitation of Aroclors

Quantitation of Aroclors is accomplished using 5 major peaks. The peaks must be within the established retention time windows. If there is an interference that affects the accuracy of results, the analyst must use 5 major peaks (3 peaks for Aroclor 1221). The same peaks that are used for sample quantitation must be used for standards and QC quantitation. The concentrations of each of the major peak are averaged for a final concentration result. When using this option, it is appropriate to remove peaks that appear to be co-eluting with contaminant peaks from the quantitation (i.e. peaks that are significantly larger than would be expected from the rest of the pattern or peaks that have been significantly weathered and are smaller than would be expected). This should be done if there are peaks that differ from the others by a factor of 2 to 5 depending on where the results fall within the calibration curve.

11.3 Second column confirmation of Aroclors is always performed and is reported when requested by the client.

11.4 Dual Column Quantitation

11.4.1 Dual column quantitation is routinely performed for PCB analysis and reported as such when requested by the client.

11.4.2 When reporting the analytical results for field samples of dual column analysis, the higher of the two results is normally reported. The result from the second column confirmation is reported if any of the following is true:

- There is obvious chromatographic interference on the initial quantitation column.
- The difference between the result on the initial quantitation column and the result on the second column is > 40% and chromatographic interference is evident.
- A continuing or bracketing standard fails on the initial quantitation column, but is acceptable on the second column. However, if the difference between the initial quantitation column and second column results is > 40% and the initial quantitation column calibration verification fails, then the sample must be evaluated for reanalysis.

11.4.3 Dual Column Results with > 40% RPD:

- If the relative percent difference (RPD) between the responses on the two columns is greater than 40%, the lower of the two results is reported unless there is obvious interference documented on the chromatogram. If the result is < the RL it will be reported as estimated with a “J” flag.
- If there is visible positive interference, e.g., co-eluting peaks, elevated baseline, etc., for one column and not the other, then report the results from the column without the interference with the appropriate data qualifier flag, footnote, and/or narrative comment in the final report.

- If there is visible positive interference for both columns, then report the lower of the two results with the appropriate flag, footnote, and/or narrative comment in the final report.
- If the relative percent difference (RPD) between the results on the two columns is greater than 40%, or if the opinion of an experienced analyst is that the complexity of the matrix is resulting in false positives, the confirmation is suspect and the results are qualified. The RPD is calculated as follows:

$$\%RPD = \frac{|R_1 - R_2|}{1/2(R_1 + R_2)} \times 100\%$$

Where:

R_1 = initial quantitation column result

R_2 = second column result

NOTE: Wisconsin requires when results are greater than the LOQ (RL) and the %RPD between column results is greater than 40%, that the HIGHER of the two results must be reported unless the analyst defensible documents that the higher result is biased due to an interference.

11.5 Calibration Range and Sample Dilutions

11.5.1 If the concentration of any analyte exceeds the working range as defined by the calibration standards, then the sample must be diluted and reanalyzed. Dilutions should target the most concentrated analyte in the upper half (over 50% of the high level standard) of the calibration range. Samples that were analyzed immediately following the high sample must be evaluated for carryover. If the samples have results at or above the RL for any analyte, they must be reanalyzed to rule out carryover. It may also be necessary to dilute samples because of matrix interferences.

11.5.2 If the initial diluted run has no hits or hits below 20% of the calibration range, and the matrix allows for analysis at a lesser dilution, then the sample should be reanalyzed at a dilution targeted to bring the largest hit above 50% of the calibration range.

11.5.3 Guidance for Dilutions Due to Matrix Interference

If the sample is initially run at a dilution and only minor matrix peaks are present, then the sample should be reanalyzed at a more concentrated dilution. Analyst judgment is required to determine the most concentrated dilution that will not result in instrument contamination. Ideally, the dilution chosen will make the response of the matrix interferences equal to approximately half the response of the mid-level calibration standard.

11.5.4 Reporting Dilutions
Some programs (e.g., South Carolina) and some projects require reporting of multiple dilutions (check special requirements in LIMS). In other cases, the most concentrated dilution with no target compounds above the calibration range will be reported. If results are reported from a diluted run, the cause for the dilution should be narrated in an NCM. For dilutions greater than 40X report the surrogates diluted out ("D").

11.6 Interferences in Observed in Samples

11.6.1 Dual column analysis does not entirely eliminate interfering compounds. Complex samples with high background levels of interfering organic compounds can produce false positive and/or false negative results. The analyst must use appropriate judgment to take action as the situation warrants.

11.6.2 Suspected Negative Interferences: If peak detection is prevented by interferences, further cleanup should be attempted. Elevation of reporting levels and/or lack of positive identification must be addressed in the case narrative.

11.7 Concentration of Analyte in Sample Extract

11.7.1 Depending on the calibration function used, the concentration of the analyte in the sample extract is calculated as follows (see Section 10 for details on establishing the calibration function):

Average Response Factor: $C_{ex} = \frac{R_x C_{is}}{R_{is} RF}$

Linear Regression: $C_{ex} = A + B\frac{(R_x C_{is})}{R_{is}}$

Where:

Cex = Concentration in the extract, µg/mL

Rx = Response for the analyte

Ris = Response for the internal standard

Cis = Concentration of the internal standard

A = Intercept of linear calibration line

RF = Average response factor

B = Slope of linear calibration line Concentration of Analyte in Original Sample

11.7.2 The concentration of the analyte in the original sample is calculated as follows:

$$C_{sample} = \frac{C_e}{1000\frac{ng}{\mu g}} \times \frac{V_e}{V_s} \times DF$$

Where:

C_{sample} = Concentration of analyte in original sample (µg/L or µg/kg).

C_e = Concentration of analyte in sample extract injected in GC (ng/mL).

$1000\frac{ng}{\mu g}$ = Factor to convert ng/mL to µg/mL.

V_e = Volume of sample extract (mL).

V_s = Volume (or weight) of original sample (L or kg).

DF = Dilution Factor (post extraction dilutions)

11.7.3 Spike Recovery Calculation

$$\%\text{Recovery} = \frac{\text{Measured Concentration}}{\text{Spiked Concentration}} \times 100\%$$

11.7.4 MS/MSD RPD Calculation

$$RPD = \frac{\left|MS - MSD\right|}{1/2\left(MS + MSD\right)} \times 100\%$$

12.0 METHOD PERFORMANCE

12.1 The supervisor has responsibility to ensure that an analyst who performs this procedure is properly trained in its use and has the required experience. Performance is monitored through internal QC and outside performance evaluation samples. Please refer to the QA Manual for additional information concerning Precision and Accuracy.

12.2 Demonstration of Capabilities – Prior to the analysis of samples, a Demonstration of Capabilities (DOC) as described in the QA Manual, must be performed initially, annually and any time a significant change is made to the analytical system.

12.3 A Method Detection Limit (MDL) Study, as described in the Detection Limits SOP, PT-QA-007, must be performed initially and whenever a significant change is made to the analytical system. The MDL must be re-evaluated from quarterly MDL points at least every 12 months.

12.4 Lower Limit of Quantitation Verification – The lowest calibration standard analyzed establishes the LLOQ or Reporting Limit. The capability to reliably detect this concentration through the preparation, clean-up and analytical procedure is verified through the annual analysis of a standard at the LLOQ/RL. The LLOQ verification shall also be performed whenever significant changes are made to the preparation and/or analytical procedure.

12.4.1 The LLOQ verification standard shall be prepared at a concentration 0.5-2 times the LLOQ/RL, and be taken through all preparation and clean-up methods which samples would be.

12.4.2 The LLOQ verification standard for aqueous matrix shall be prepared using laboratory deionized water and for the solid matrix using clean Ottawa sand. Other clean matrices may be used in addition, for project specific requirements.

12.4.3 The LLOQ shall be verified annually on each instrument used for client sample analysis.

12.4.4 Recovery of each analyte must meet the laboratory established LCS recovery limits ± 20%. (For example, if the LCS recovery limits are 70-130%, the LLOQ verification must meet recovery limits of 50-150%.) Once sufficient points have been generated, LLOQ based statistical limits may be used in place of limits based on LCS recovery.

NOTE: The lower recovery limit for the LLOQ can be no lower than 10%.

12.4.5 If the LLOQ cannot be verified, it will be necessary to raise the RL to a concentration level that can be carried through the preparation and clean-up steps to meet recovery limits.

13.0 POLLUTION CONTROL

13.1 It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage and reagent stability). Employees must abide by the policies in Section 13 of the Corporate Environmental Health and Safety Manual (CW-E-M-001) for "Waste Management and Pollution Prevention" and the Pittsburgh Facility Addendum EH&S Manual (PT-HS-001).

14.0 WASTE MANAGEMENT

14.1 Waste management practices are conducted consistent with all applicable rules and regulations. Excess reagents, samples and method process wastes are disposed of in an accepted manner. Waste description rules and land disposal restrictions are followed. Waste disposal procedures are incorporated by reference to PT-HS-001. The following waste streams are produced when this method is carried out.

14.2 Methylene Chloride in vials. This waste is placed in waste container identified as "Vials & Extracts", Waste #7.

14.3 Flammable solvents in vials. This waste is placed in waste container identified as "Vials & Extracts", Waste #7.

14.4 Waste flammable solvents. This waste is collected in a waste container identified as "Mixed Flammable Solvent Waste", Waste #3.

14.5 Expired primary and working PCB standards. This waste is placed in a waste container identified as "PCB Standard Waste", Waste #8.

14.6 Samples containing polychlorinated biphenyls (PCB's) at concentrations ≥50 ppm are regulated under the Toxic Substance Control Act (TSCA) and must be segregated from all other waste streams. Analysts are responsible for contacting the Group Leader, Sample Control, and the Health and Safety Coordinator immediately if a sample falls into the TSCA category.

15.0 REFERENCES

15.1 Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846 Method 8082, Polychlorinated Biphenyls (PCBs) by Gas Chromatograph, Revision 0, December, 1996

15.2 Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846 Method 8082A, Polychlorinated Biphenyls (PCBs) by Gas Chromatograph, Revision 1, February, 2007

15.3 SW 846, Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, Update V, Method 8000D, Determinative Chromatographic Separations, Revision 4, October 2012.

15.4 SOP PT-OP-001, Extraction of Organic Compounds from Waters, SW-846 3500 Series and EPA 600 Series Methods

15.5 SOP PT-OP-026, Extraction of Organic Compounds from Solids, Sediments, Tissues, and Wipes, SW-846 3500 Series Methods

15.6 SOP PT-OP-028, Clean-up of Organic Extracts by SW-846 3600 Series Methods

15.7 SOP PT-QA-001, Employee Orientation and Training

15.8 SOP PT-QA-016, Nonconformance & Corrective Action System

15.9 SOP PT-QA-018, Technical Data Review Requirements

15.10 SOP PT-QA-021, Quality Control Program

15.11 SOP PT-QA-031, Internal Chain of Custody

15.12 PT-QA-M-001, Pittsburgh Laboratory Quality Assurance Manual

15.13 Corporate SOP CA-Q-S-002, Manual Integration

15.14 Corporate Policy No, CA-T-P-005, Revision 0, Policy for Determining RT Windows for GC/ECD Tests, May 26, 2016

15.15 Corporate Policy No. CA-T-P-003, Revision 2, Reporting Results for Methods that Require 2nd Column Confirmation, January 2, 2013

15.16 SOP PT-QA-007, Detection Limits

15.17 SOP PT-HS-001, Pittsburgh Facility Addendum EH&S Manual, to the Corporate Environmental Health and Safety Manual (CW-E-M-001) for "Waste Management and Pollution Prevention".

16.0 METHOD MODIFICATIONS

16.1 None

17.0 ATTACHMENTS

17.1 Table 1 - Analyte List and Standard Reporting Limits

17.2 Table 2 - Typical Instrument Conditions
17.3 Table 3 - Calibration Levels (µg/mL)
17.4 Table 4 - LCS/Matrix Spike and Surrogate Spike Levels (µg/L)
17.5 Table 5 - Preparation of Calibration Standards
17.6 Appendix A – Aroclor Identification
17.7 Appendix B - Analysis of PCB Congeners

18.0 REVISION HISTORY

18.1 Revision 1, 7/27/2009
18.2 Revision 2, 8/5/2011
18.3 Revision 3, 7/24/2011
18.4 Revision 4, 10/16/2012
18.5 Revision 5, 4/25/2014
18.6 Revision 6, 9/28/2015
18.7 Revision 7, 7/19/2016
18.8 Revision 8, 12/12/2016
18.9 Revision 9, 3/10/2017
18.10 Revision 10, 11/28/2017
18.11 Revision 11, 5/13/2019
18.12 Changes to current revision

SOP section	Change from	Change to	Reason
7.3.4.1 Table		Correct Levels 2 & 3 PCB and all surrogate concentrations	Correction
9.10.4	Prior to	After	Incorporating SOP amendment
12.3 & 15.16	CA-Q-S-006	PT-QA-007	SOP update
Table 3		Correct Levels & 3 PCB and surrogate concentrations	Correction

Table 1				
Standard Analyte List and Reporting Limits				
Compound	**CAS #**	**Reporting Limit, µg/L, µg/wipe or µg/kg**		
		Water/Wipe Regular/ Low Level	**Soil/ Tissue Regular/Low Level**	**Waste**
Aroclor 1016	12674-11-2	0.4/0.01	16.67/0.833	500
Aroclor 1221	11104-28-2	0.4/0.01	16.67/0.833	500
Aroclor 1232	11141-16-5	0.4/0.01	16.67/0.833	500
Aroclor 1242	53469-21-9	0.4/0.01	16.67/0.833	500
Aroclor 1248	12672-29-6	0.4/0.01	16.67/0.833	500
Aroclor 1254	11097-69-1	0.4/0.01	16.67/0.833	500
Aroclor 1260	11096-82-5	0.4/0.01	16.67/0.833	500
Optional Compounds:				
Aroclor 1262	37324-23-5	0.4/0.01	16.67/0.833	500
Aroclor 1268	11100-14-4	0.4/0.01	16.67/0.833	500

The following concentration factors are assumed in calculating the Reporting Limits:

	Extraction Vol.	**Final Vol.**	**Low Level Vol.**
Groundwater	1000 mL	40 mL	1 mL
Wipe	1 wipe	40 mL	NA
Low-Level Soil	30 g / 15 g	40 mL / 20 mL	1 mL
High-Level Soil/Waste	1 g	40 mL	NA
Tissue	6 g	2 mL (1 mL with GPC clean-up)	NA

Table 2	
Typical Instrument Conditions	
Parameter	Recommended Conditions
Injection Port Temperature:	220 °C
Detector Temperature:	325 °C
Temperature Program:	120 °C for 0.75 minute 18 °C/min to 260 °C 1 minute hold 20 °C/min to 300 °C, 1.5 minute hold
Column 1:	RTX-CLP1, 30 m x 0.53 mm id, 0.5 µm
Column 2:	RTX-CLP2, 30 m x 0.53 mm id, 0.42 µm
Injection:	1 or 2 µL
Carrier Gas:	Hydrogen or Helium
Make-up Gas:	Nitrogen

Table 3							
Calibration Levels ng/mL							
	Level 1	Level 2	Level 3	Level 4	Level 5	Level 6	Level 7[1]
AR1660	10	100	250	500	1000	2000	4000
AR1242[2]				500			
AR2154[2]				500			
AR1232[2]				500			
AR1248[2]				500			
Surrogates are included with all the calibration mixes at the following levels:							
TCMX	0.5	5	12.5	25	50	100	200
DCB	0.5	5	12.5	25	50	100	200

[1] Level 7 is optional and should only be used if linearity can be maintained on the instrument to this level.

[2] Aroclors may be quantitated within the range 100 to 2000 ng/mL (4000 ng/mL if the level 7 1016/1260 standard is included). If the Aroclor is more concentrated, it must be reanalyzed at a dilution.

Table 4			
LCS/Matrix Spike and Surrogate Spike Levels for Aroclor Analysis µg/L, µg/wipe or µg/kg			
	Aqueous/Wipe	Soil/Tissue	Waste
Aroclor 1016/1260	10	333	10,000
TCMX (surrogate)	0.20	6.67	200
DCB (surrogate)	0.20	6.67	200

Table 5					
Preparation of Calibration Standards					
Calibration Level	1016/1260 Intermediate (µL)	1221 + 1254 Stock (µL)	1232 Stock (µL)	1242 Stock (µL)	1248 Stock (µL)
Level 1	4				
Level 2	20				
Level 3	80				
Level 4	1000	250	250	250	250
Level 5	400				
Level 6	800				
Level 7	1600				

The surrogate stock is purchased (Decachlorobiphenyl and Tetrachloro-m-xylene) at 200 ug/mL.

The Aroclor 1016 and 1260 stock standards are purchased as certified standards in isooctane at 1000 ug/mL. The other five Aroclor stock standards are purchased at 200 ug/mL.

For Aroclors 1016 and 1260, an intermediate standard is prepared by diluting 1.0 mL of each of the stock standards and 0.25 mL of the surrogate stock standard to 10.0 mL in hexane. The intermediate Aroclor 1016/1260 standard concentrations are 100 ug/mL for each Aroclor and 5 ug/mL for each surrogate.

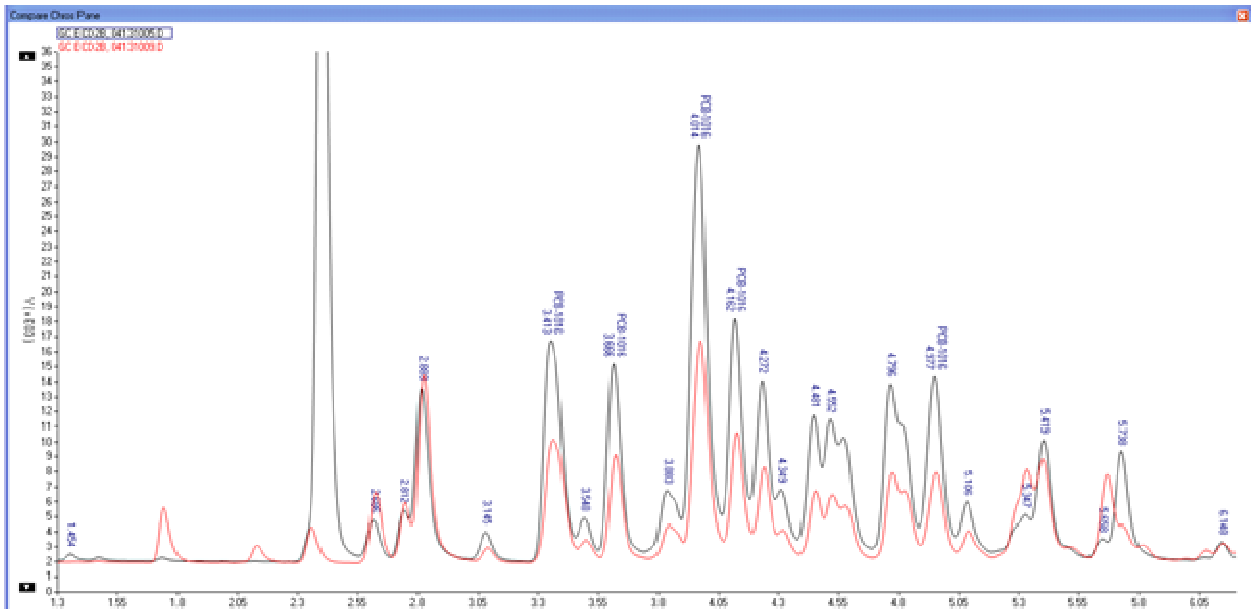
The Aroclor 1016/1260 calibration standards are prepared by diluting the volumes noted in Table C-6 to a 40.0 mL final volume in hexane except for the Level 3 standard, which is taken to a 200mL final volume in hexane.

The mid-level (Level 3) calibration standards for each of the other five Aroclors (1221, 1232, 1242, 1248, 1254) are prepared by diluting 0.25 mL of the appropriate stock standard to a final volume of 100 mL in hexane. Aroclors 1221 and 1254 are combined into one standard and Aroclors 1232, 1242, and 1248 are prepared individually.

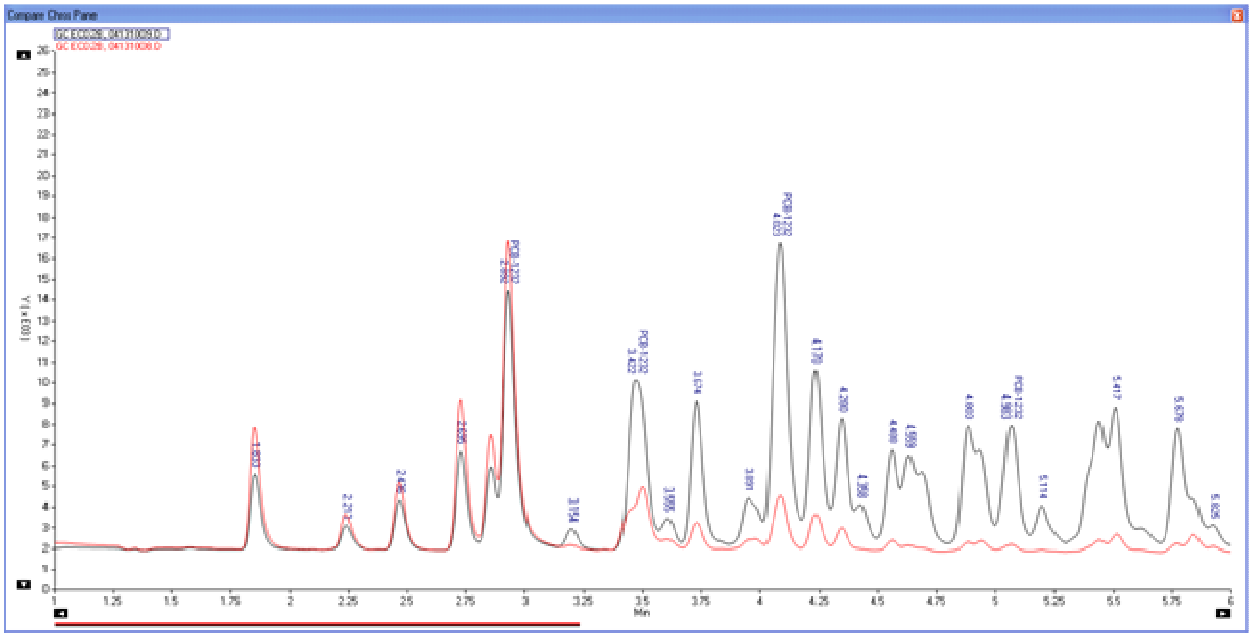
Appendix A

Aroclor Identification

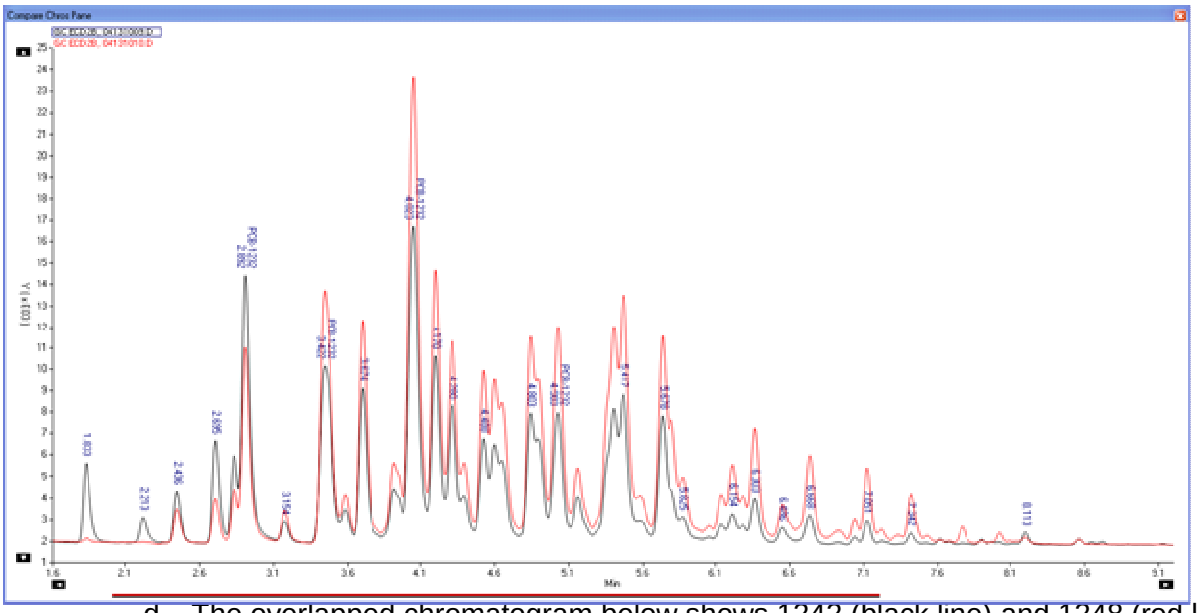
1. Aroclors are mixtures of individual PCBs, which is why multiple peaks are present for each Aroclor standard. The different Aroclors can contain the same peaks, therefore a specific set of peaks, as well as the pattern of the peaks are used for identification. The percentage of each PCB in the Aroclor mix will also be different between Aroclors, therefore the ratio of the peak heights/areas will be different.
2. Within the 12xx designated Aroclors, the higher the number, the more chlorines are part of the compounds that comprise the Aroclor, therefore the molecular weights are higher, and the later the peaks of the mix elute.
3. When choosing the five (or three for 1221) peaks to be used for identifying and quantitating each Aroclor, choose prominent peaks from across the pattern. The previous standard patterns should be followed, and an experienced analyst should be consulted before changing the peaks used.
4. Standards of the separate individual Aroclors are injected periodically to verify the peak retention times and patterns.
5. Below are some examples of peaks/patterns for Aroclors:
    a. The overlapped chromatogram below shows a mixture of 1016 and 1260 (black line) with 1232 (red line). The large peak around 2.35 minutes is TCMX surrogate. 1016 and 1260 patterns are completely separate. 1016 and 1232 have similar patterns, however the very early peaks that appear in 1232 are not present in 1016, and the front end of the pattern is stronger (higher peaks) where 1016 is stronger on the back end of the pattern.



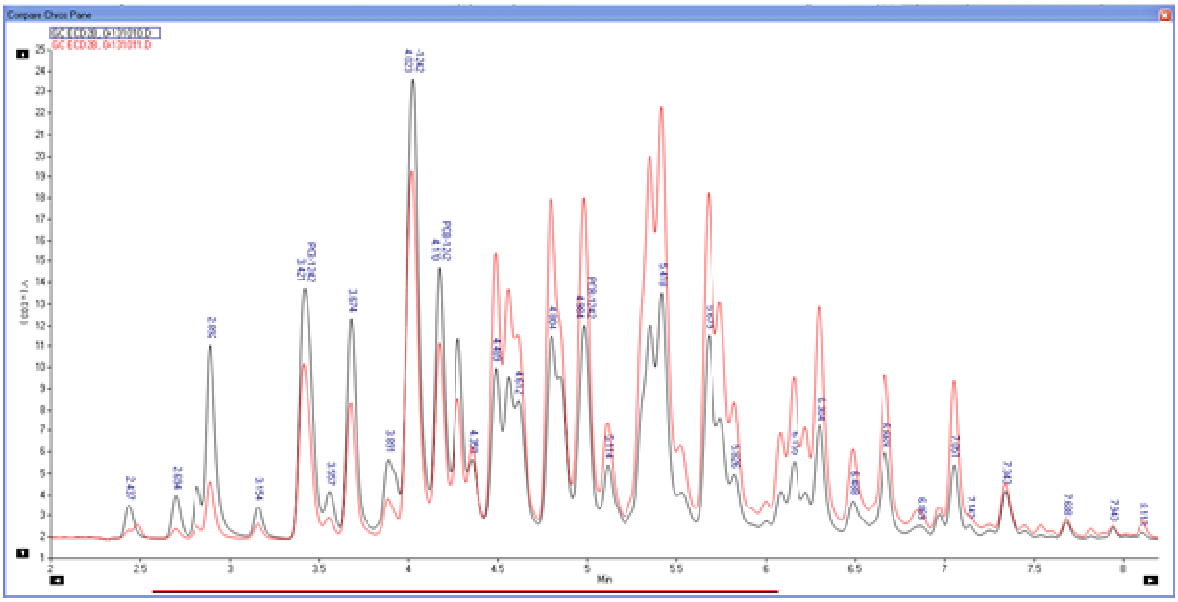
b. The overlapped chromatogram below shows a mixture of 1232 (black line) and 1221 (red line). The front end of the patterns are identical, but 1232 has later peaks that are not present in 1221.



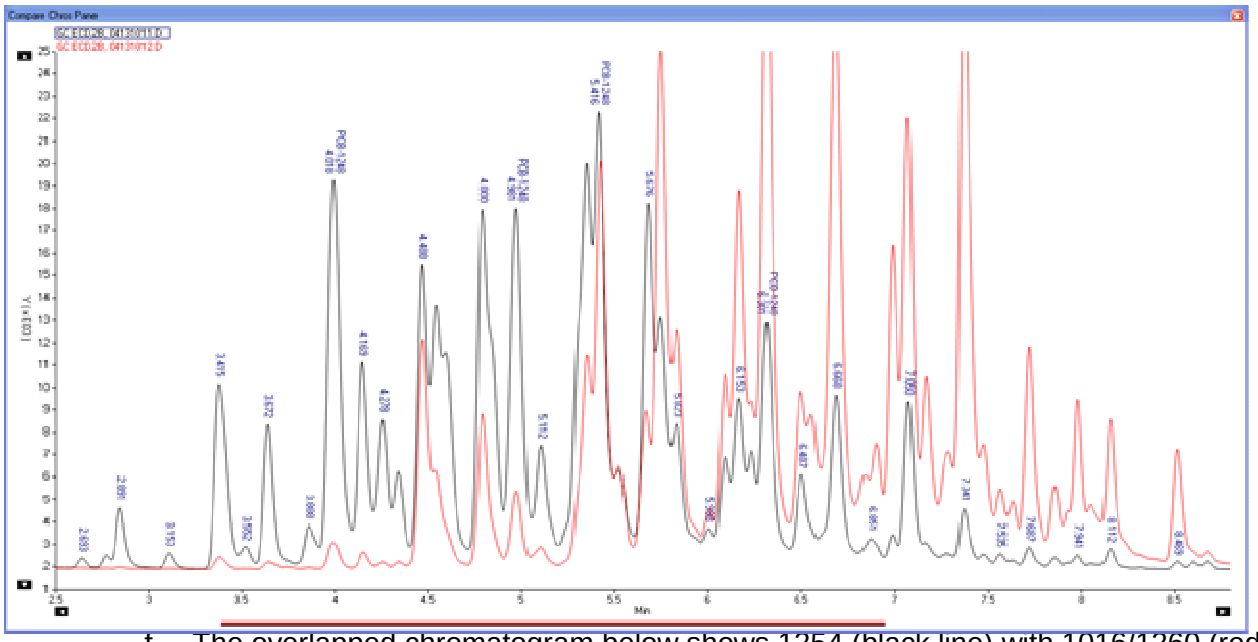
c. The overlapped chromatogram below shows 1232 (black line) and 1242 (red line). The very early peaks that appear in 1232 are not present in 1242, also the later eluting peaks of 1242 are stronger.



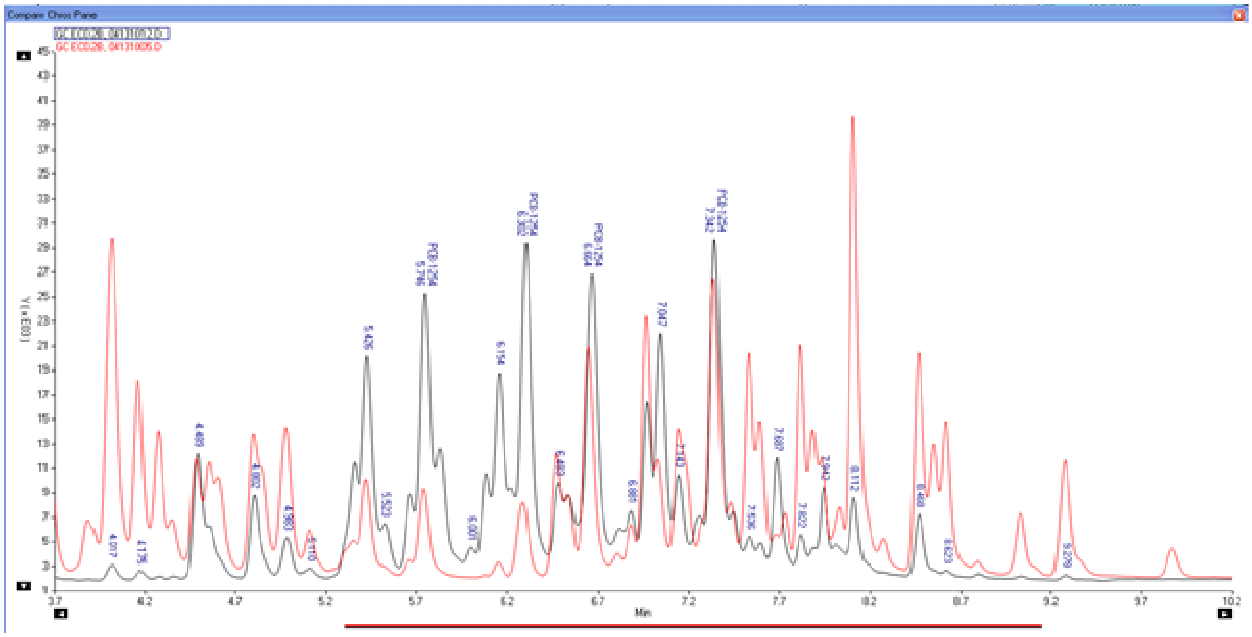
d. The overlapped chromatogram below shows 1242 (black line) and 1248 (red line). These Aroclors share all of the same peaks, so the relative strengths of the front and back of the pattern is the only way to distinguish the two. If this pattern is present, consult historical project data and an experienced analyst for the identification. Aroclors 1242 and 1248 are never reported together in a sample for this reason.



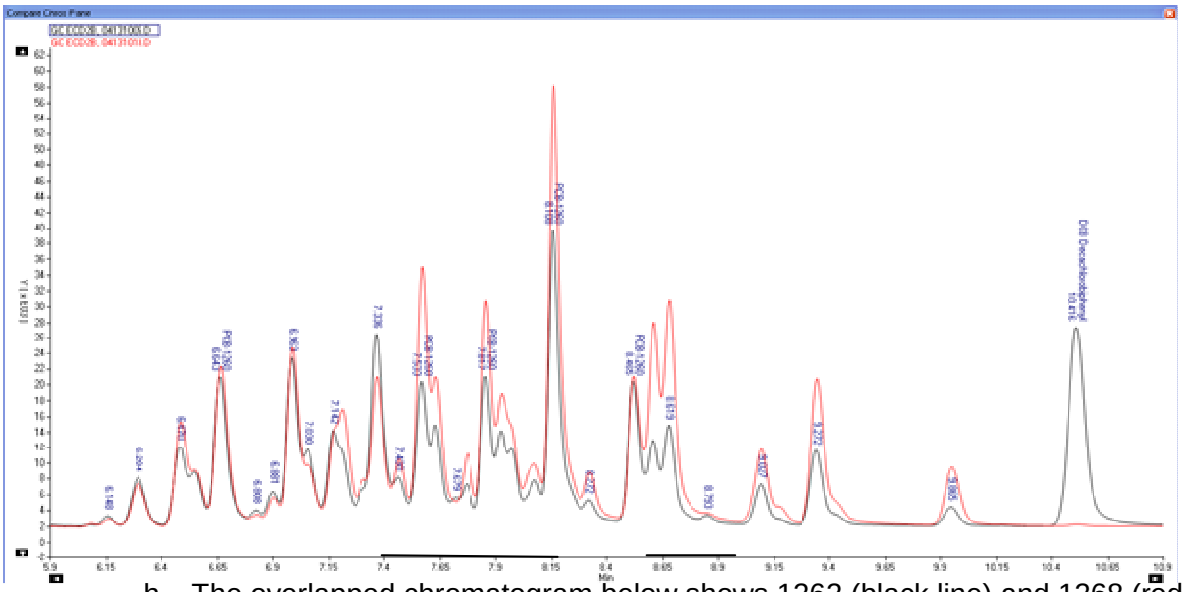
e. The overlapped chromatogram below shows 1248 (black line) and 1254 (red line), which are relatively easy to differentiate, as the front end of the pattern is much stronger in 1248.



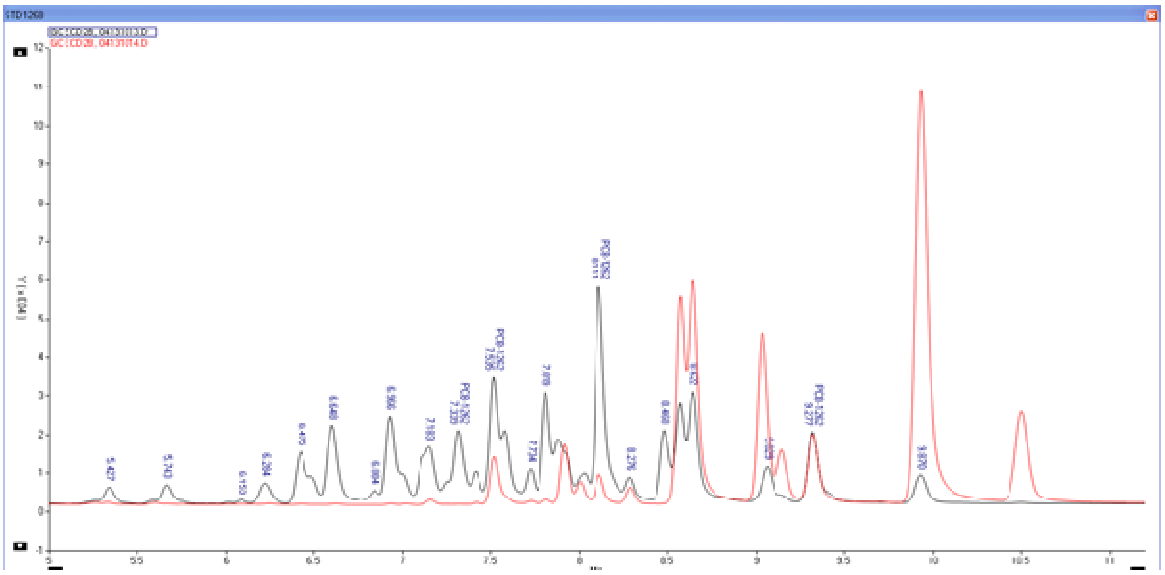
f. The overlapped chromatogram below shows 1254 (black line) with 1016/1260 (red line). While 1254 and 1260 share peaks, the relative strength of 1254 at the front end, and 1260 at the back end serve as the indicator between the two.



g. The overlapped chromatogram below shows 1260 (black line) and 1262 (red line). The ratio of the three peaks between 7-8 minutes and 8.4-8.7 minutes are the differences that indicate the identification for these Aroclors. For 1260, the first 3 show a slight decreasing pattern, and the second three a V shaped pattern, while for 1262 the first three sets are more of an arched pattern and the second three show a pattern of increasing height.

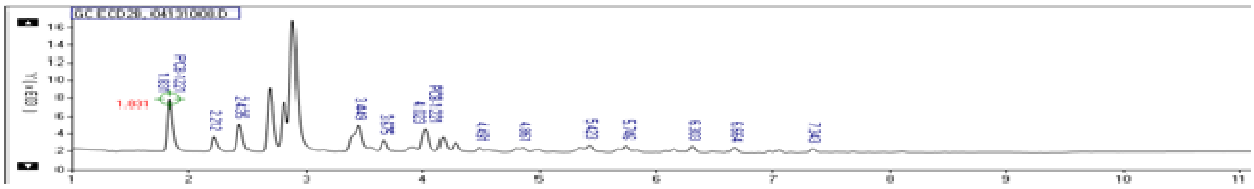


h. The overlapped chromatogram below shows 1262 (black line) and 1268 (red line). Clearly there are peaks present early in 1262 that are missing from 1268, and the back end of 1268 is much stronger.

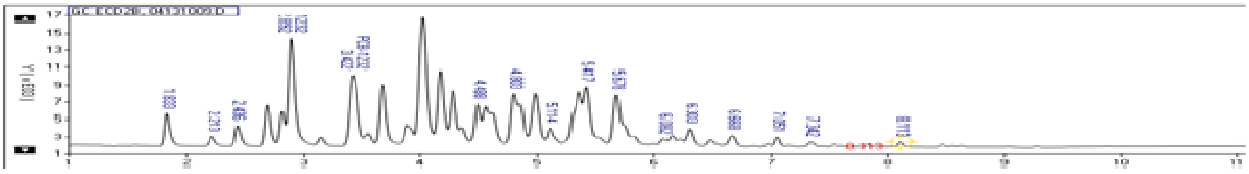


6. Example Individual Aroclor patterns:

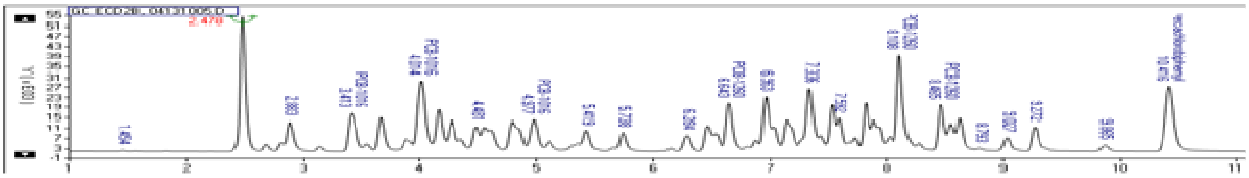
   a. Aroclor 1221



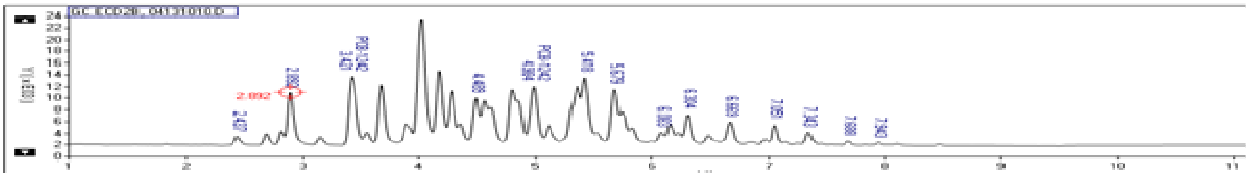
b. Aroclor 1232



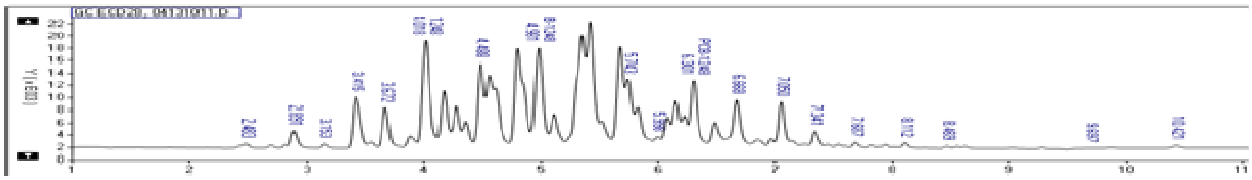
c. Aroclor 1016



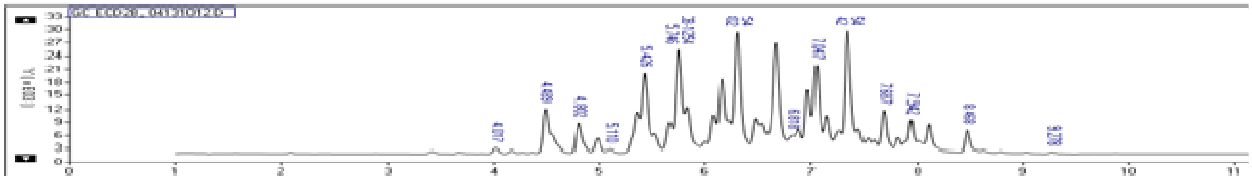
d. Aroclor 1242



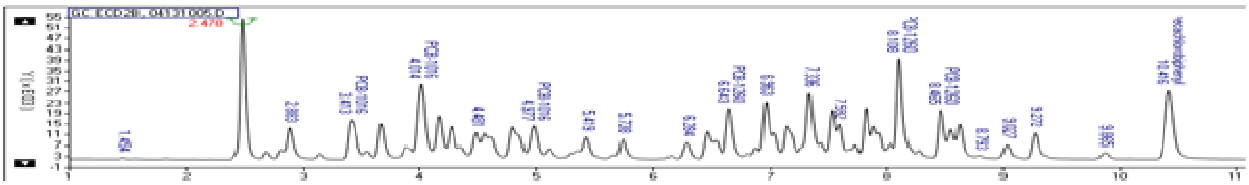
e. Aroclor 1248



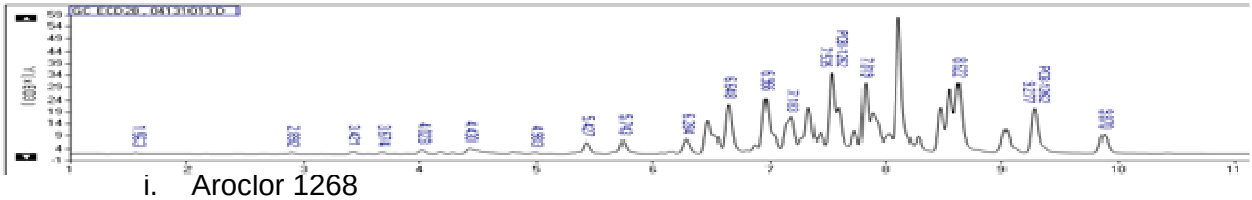
f. Aroclor 1254



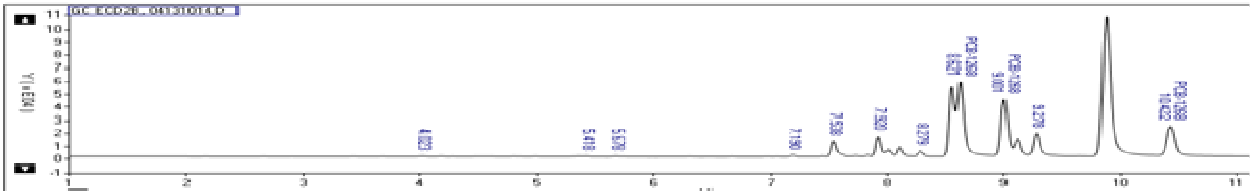
g. Aroclor 1260



h. Aroclor 1262



i. Aroclor 1268



j.

7. When a mixture of Aroclors is present in a sample, unless the pattern of each is very clearly evident, consult historical project data and an experienced analyst for identification for reporting. Note that Aroclors 1242 and 1248 are never reported together in a sample.

    a. When a mixture of overlapping Aroclors is present, and the predominant Aroclor only is reported, it must be accompanied by the following narrative statement: "The following sample(s) contained more than one Aroclor with insufficient separation to quantify individually. The PCBs present are quantitated as the predominant Aroclor for reporting."

8. Most PCBs that are present in the environment have been there for years are "weathered". The patterns in environmental samples may not match the patterns present in standards. The relative intensity of the peaks may be different, or 1 or 2 peaks of the chosen pattern may be missing. Identification of weathered Aroclors take experience with reviewing these chromatograms, so consult a more experienced analyst or the laboratory Technical Director for instructions.

    a. To aid in the identification, make sure that the extracts have had cleanup performed.

    b. Compare to recent individual Aroclor standards.

    c. When reporting weathered Aroclors, the following statement (or similar) must be included in the case narrative: "The following sample(s) appear(s) to containe PCBs, however, due to weathering or other environmental processes, the PCBs in the sample(s) do not closely match any of the laboratory's Aroclor standards used for instrument calibration. The sample(s) has(have) been quantified and reported as Aroclor <XXXX> (OR as a mixture of Aroclors XXXX and YYYY). Due to the poor match with the Aroclor standard(s), there is increased qualitative and quantitative uncertainty associated with the result(s)."

Appendix B

Analysis of PCB Congeners Based on Method 8082A

1.0 SCOPE OF METHOD

1.1 This SOP Appendix describes procedures to be used when SW-846 Method 8000B is applied to the analysis of polychlorinated biphenyls (PCB) congeners by GC/ECD. This Appendix is to be applied when SW-846 Method 8082A is requested, and is applicable to extracts derived from any matrix, which are prepared according to the appropriate TestAmerica sample extraction SOP (PT-OP-001 or PT-OP-026). The PCBs are determined and quantitated as individual PCB congeners.

1.2 Table A-1 lists the congeners, which are routinely determined by this method and gives the Reporting Limits (RL) for each matrix. RLs given are based on the low level standard and the sample preparation concentration factors. Matrix interferences may result in higher RLs than those listed.

2.0 SUMMARY OF METHOD

2.1 This method presents conditions for the analysis of prepared extracts for PCB congeners. The PCBs are injected onto the GC column(s) and separated and detected by electron capture detection. Quantitation is by the external standard method.

3.0 DEFINITIONS

3.1 Refer to Section 3 of the main body of this SOP for definitions.

4.0 INTERFERENCES

4.1 Refer to Section 4 of the main body of this SOP for information regarding chromatographic interferences.

5.0 SAFETY

5.1 Refer to Section 5 of the main body of this SOP for general safety requirements.

5.2 PCB congeners have been classified as a potential carcinogen under OSHA. Concentrated solutions of PCB congeners must be handled with extreme care to avoid excess exposure. Contaminated gloves and clothing must be removed immediately. Contaminated skin surfaces must be washed thoroughly.

6.0 EQUIPMENT AND SUPPLIES

6.1 Refer to Section 6 of the main body of this SOP for equipment and supplies.

6.2 Refer to Table B-2 for analytical column requirements

7.0 REAGENTS AND STANDARDS

7.1 Reagents

7.1.1 Acetone, 99.4% for organic residue analysis.

7.1.2 Hexane, pesticide grade.

7.1.3 Carrier Gas: ≥ 99.99999% pure hydrogen.

7.1.4 Make-up Gas: ≥ 99.99980% pure nitrogen.

7.2 Standards

7.2.1 Stock Standards

7.2.1.1 All standards must be refrigerated at >0.0˚C but ≤6.0℃. All stock standards must be protected from light. Stock standard solutions should be brought to room temperature before use.

7.2.1.2 Stock standards are monitored for signs of degradation or evaporation. The standards must be replaced annually or earlier if the vendor indicates an earlier date.

7.2.1.3 Dilutions from stock standards cannot have a later expiration date than the date assigned to the parent stock solutions. The standards must be replaced at least every six months, or sooner, if comparison with check standards indicates a problem.

7.2.2 PCB Congener and Surrogate Stock Standards

7.2.2.1 PCB Congener Stock Mix 1: For the PCB Congeners listed in Table B-1, except BZ-205 (surrogate), a commercially prepared stock standard solution is obtained. The concentration of the PCB Congeners contained in Mix 1 is 4.0 ug/mL and the concentration of the TCMX (surrogate) is 6.6 ug/mL PCB Congener Stock Add-on Mix: This stock standard is prepared by adding 400 uL of 100 ug/mL of the following individual congener standards brought to a final volume of 10 mL with Hexane and resulting in a final concentration of 4 ug/mL: BZ-205 (surrogate).

7.2.2.2 TCMX (2000 ug/mL) and BZ-205 (100 ug/mL) Surrogate Stock Standards are commercially purchased standards.

7.2.2.3 PCB Congener Matrix Spike Stock Standard is a commercially purchased standard containing BZ-8, BZ-18, BZ-28, BZ-44, BZ-52, BZ-66, BZ-77, BZ-101, BZ-105, BZ-118, BZ-126, BZ-128, BZ-138, BZ-153, BZ-170, BZ-180, BZ-187, BZ-195 and BZ-206 at a concentration of 100 ug/mL. (NOTE: BZ-49, BZ-81, BZ-87, BZ-114, BZ-123, BZ-156, BZ-157, BZ-167, BZ-169, BZ-187 and BZ-189 are also used as needed, however these are separate commercially purchased stock solutions also at a concentration of 100 ug/mL).

7.2.3 Calibration Curve Standard Solutions

The calibration standards are prepared as follows:

Calibration Level	Recipe	Stock Concentration (ug/mL)	Final Volume (ml of Hexane)
1	5 uL of PCB Congener Stock Mix 1 and 5 uL of PCB Congener Stock Add-on Mix	4 ug/mL (TCMX 6.6 ug/mL)	40
2	10 uL of PCB Congener Stock Mix 1 and 10 uL of PCB Congener Stock Add-on Mix	4 ug/mL (TCMX 6.6 ug/mL)	40
3	25 uL of PCB Congener Stock Mix 1 and 25 uL of PCB Congener Stock Add-on Mix	4 ug/mL (TCMX 6.6 ug/mL)	40
4	50 uL of PCB Congener Stock Mix 1 and 50 uL of PCB Congener Stock Add-on Mix	4 ug/mL (TCMX 6.6 ug/mL)	40
5	100 uL of PCB Congener Stock Mix 1 and 100 uL of PCB Congener Stock Add-on Mix	4 ug/mL (TCMX 6.6 ug/mL)	40
6	200 uL of PCB Congener Stock Mix 1 and 200 uL of PCB Congener Stock Add-on Mix	4 ug/mL (TCMX 6.6 ug/mL)	40

7.2.3.1 Refer to Table B-3 for details of calibration standard concentrations.

7.2.4 Surrogate Standards

7.2.4.1 The Working Surrogate Solution is made as follows: 2.5 uL of the 2000 ug/mL commercially purchased TCMX stock and 50 uL of the 100 ug/mL commercially purchased BZ-205 stock brought to a final volume of 20 mL with Acetone (see section 7.2.2.3). The final concentration of the Working Surrogate Solution is 0.025 ug/mL. Refer to Table A-3 for details of surrogate standard concentrations in the calibration standards.

NOTE: The Working Surrogate Solution is prepared and used as part of the scope of the organic preparation SOP PT-OP-001 and PT-OP-026. The preceding information is provided for reference only.

7.2.5 Laboratory Control Standard (LCS) and Matrix Spiking (MS) Solution

The working LCS/MS spiking solution is prepared by combining 1mL of the PCB Congener Matrix Spike Stock Standard and 1 mL each, as needed, of the BZ-49, BZ-81, BZ-87, BZ-114, BZ-123, BZ-156, BZ-157, BZ-167, BZ-169, BZ-187 and BZ-189 stock standards brought to a 100 mL final volume with Acetone. The final concentration of the working LCS/MS spiking solution is 1.0 ug/mL.

NOTE: The LCS/MS spiking solution is prepared and used as part of the scope of the organic preparation SOP SOP PT-OP-001 and PT-OP-026.

8.0 SAMPLE COLLECTION, PRESERVATION, SHIPMENT AND STORAGE

8.1 Refer to Section 8 of the main body of this SOP.

9.0 QUALITY CONTROL

9.1 Refer to Section 9 of the main body of this SOP.

10.0 PROCEDURE

10.1 Calibration and Standardization

10.1.1 PCB Congeners uses an External Standard Calibration as follows:

External standard calibration involves the comparison of instrument responses from the samples to the responses from the target compounds in the calibration standards. The area (or height) of a peak in a sample chromatogram is compared to the area (or height) of the peak in the standard chromatograms that appears at the same retention time. The ratio of the detector response to the concentration of the target analyte in the calibration standard is defined as the calibration factor (CF) and is calculated as follows:

$$CF = \frac{Area\ or\ Height\ of\ Peak}{Mass\ Injected}$$

Note: It is also possible to use the concentration of the standard rather than the mass injected.

10.1.2 Refer to Section 10 of the main body of this SOP for the other general calibration requirements.

10.2 Initial Calibration

10.2.1 Refer to Table B-5 for the initial calibration analytical sequence.

10.2.2 The response for each PCB congener will be calculated by the procedures described in the general method for 8082A analysis, with the following modifications.

- A minimum five-point calibration of each of the individual congeners mixes is generated. At least 2 separate mixes are prepared to ensure that there is complete resolution of all congeners in the mixes. Calibration levels are listed in Table B-3.

10.3 Initial Calibration Verification

10.3.1 The ICV will consist of second source standards of all congeners of interest. The ICV is prepared from a second source stock standard of a concentration of 4ppm for all congeners of interest. 25 uL of the ICV stock standard is added to Hexane and brought to a final volume of 40 mL for a concentration of 0.0025 ppm.

10.3.2 Refer to section 9.9.4 in the main body of this SOP for acceptance criteria and corrective actions for failures.

10.3.3 12 hour Calibration

The 12-hour calibration verification must be analyzed within 12 hours of the start of the initial calibration and at least once every 12 hours thereafter if

samples are being analyzed. If there is a break in the analytical sequence of greater than 12 hours, then a new continuing calibration run must be analyzed before proceeding with the sequence. If more than 12 hours have elapsed since the injection of the last sample in the analytical sequence, a new analytical sequence must be started with a 12-hour calibration.

The retention time windows for any analytes included in the daily calibration are updated.

For this method, samples must be <u>bracketed</u> with successful calibration verification runs.

10.3.4 Calibration Verification

10.3.4.1 A mid-level calibration mix is analyzed as the calibration verification standard. This is analyzed after every 20 samples or 12 hours, including matrix spikes, LCS, and method blanks. (Depending on the type of samples, it may be advisable to analyze verifications more frequently in order to minimize reruns.).

10.3.4.2 The daily CCV analysis at a concentration other than the mid-level (to meet NELAC requirements) will consist of all of the congeners of interest. All other CCVs will be mid-level calibration standards.

10.3.5 Procedure - Refer to Section 10 of the main body of this SOP for general procedural requirements.

10.3.6 Suggested gas chromatographic conditions are given in Table B-2.

10.3.7 The suggested analytical sequence is given in Table B-5.

11.0 CALCULATIONS / DATA REDUCTION

11.1 Identification of Congeners

Retention time windows are used for identification of PCB congeners. Second column confirmation must be performed.

11.2 Refer to section 11 of the main body of this SOP for calculation details.

12.0 METHOD PERFORMANCE

12.1 Refer to section 12.3 for initial demonstration of capability requirements under Section 12 of the main body of this SOP. The LCS spiking level is used for IDOC.

13.0 POLLUTION CONTROL

13.1 Refer to Section 13 of the main body of this SOP.

14.0 WASTE MANAGEMENT

14.1 Refer to Section 14 of the main body of this SOP.

15.0 REFERENCES

15.1 Refer to Section 15 of the main body of this SOP.

16.0 METHOD MODIFICATIONS

16.1 Refer to Section 16 of the main body of this SOP.

17.0 ATTACHMENTS

17.1 Table B-1: Standard Analyte List and Reporting Limits
17.2 Table B-2: Recommended GC Operating Conditions
17.3 Table B-3: Calibration Levels
17.4 Table B-4: LCS/Matrix Spike and Surrogate Spike Levels
17.5 Table B-5: Suggested Analytical Sequence

18.0 REVISION HISTORY

18.1 See section 18 of the Main Body of this SOP.

Table B-1
Standard Analyte List and Reporting Limits

Compound *	CAS #	Reporting Limit			
		Water ng/L	Soil/Sediment/Tissue ug/kg	Wipe ng/wipe	Waste ug/kg
BZ-8	34883-43-7	1.0	1.0	1.0	10
BZ-18	37680-65-2	1.0	1.0	1.0	10
BZ-28	7012-37-5	1.0	1.0	1.0	10
BZ-44	41464-39-5	1.0	1.0	1.0	10
BZ-49	41464-40-8	1.0	1.0	1.0	10
BZ-52	35693-99-3	1.0	1.0	1.0	10
BZ-66	32598-10-0	1.0	1.0	1.0	10
BZ-77	32598-13-3	1.0	1.0	1.0	10
BZ-81	70362-50-4	1.0	1.0	1.0	10
BZ-87	38380-02-8	1.0	1.0	1.0	10
BZ-101	37680-73-2	1.0	1.0	1.0	10
BZ-105	32598-14-4	1.0	1.0	1.0	10
BZ-114	74472-37-0	1.0	1.0	1.0	10
BZ-118	31508-00-6	1.0	1.0	1.0	10
BZ-123	65510-44-3	1.0	1.0	1.0	10
BZ-126	57465-28-8	1.0	1.0	1.0	10
BZ-128	38380-07-3	1.0	1.0	1.0	10
BZ-138	35065-28-2	1.0	1.0	1.0	10
BZ-153	35065-27-1	1.0	1.0	1.0	10
BZ-156	38380-08-4	1.0	1.0	1.0	10
BZ-157	69782-90-7	1.0	1.0	1.0	10
BZ-167	52663-72-6	1.0	1.0	1.0	10
BZ-169	32774-16-6	1.0	1.0	1.0	10
BZ-170	35065-30-6	1.0	1.0	1.0	10
BZ-180	35065-29-3	1.0	1.0	1.0	10
BZ-183	52663-69-1	1.0	1.0	1.0	10
BZ-184	74472-48-3	1.0	1.0	1.0	10
BZ-187	52663-68-0	1.0	1.0	1.0	10
BZ-189	39635-31-9	1.0	1.0	1.0	10
BZ-195	52663-78-2	1.0	1.0	1.0	10
BZ-206	40186-72-9	1.0	1.0	1.0	10
BZ-209	2051-24-3	1.0	1.0	1.0	10

* The congener identifications are consistent with the short-hand identifications recommended by Ballschmiter and Zell (1980).

The following concentration factors are assumed in calculating the Reporting Limits:

	Extraction Vol.	**Final Volume**	**Dilution Factor**
Groundwater	1000 mL	2 mL	1
Soil/Tissue/Sediment	10 g	20 mL	1
Waste	1 g	20 mL	1
Wipe	1 wipe	2 mL	1

Table B-2	
Parameter	**Recommended Conditions**
Injection port temp	225^{o}C
Detector temp	325^{o}C
Temperature program	80^{o}C ramping 30^{o}C/min to 190^{o}C, then 20^{o}C/min to 230^{o}C, then 4^{o}C/min to 270^{o}C and finally 20^{o}C/min to 300^{o}C holding for 5 minutes (22 minute run-time)
Column 1	ZB 50, 30 m, 0.25 mm id, 0.25 µm FT
Column 2	ZB 1701, 30 m, 0.25 mm id, 0.25 µm FT
Injection	1-2µL
Carrier gas	Hydrogen
Make up gas	Nitrogen

Table B-3						
Calibration Levels ug/mL						
	Level 1	Level 2	Level 3	Level 4	Level 5	Level 6
BZ-8	0.0005	0.001	0.0025	0.0050	0.010	0.020
BZ-18	0.0005	0.001	0.0025	0.0050	0.010	0.020
BZ-28	0.0005	0.001	0.0025	0.0050	0.010	0.020
BZ-44	0.0005	0.001	0.0025	0.0050	0.010	0.020
BZ-49	0.0005	0.001	0.0025	0.0050	0.010	0.020
BZ-52	0.0005	0.001	0.0025	0.0050	0.010	0.020
BZ-66	0.0005	0.001	0.0025	0.0050	0.010	0.020
BZ-77	0.0005	0.001	0.0025	0.0050	0.010	0.020
BZ-81	0.0005	0.001	0.0025	0.0050	0.010	0.020
BZ-87	0.0005	0.001	0.0025	0.0050	0.010	0.020
BZ-101	0.0005	0.001	0.0025	0.0050	0.010	0.020
BZ-105	0.0005	0.001	0.0025	0.0050	0.010	0.020
BZ-114	0.0005	0.001	0.0025	0.0050	0.010	0.020
BZ-118	0.0005	0.001	0.0025	0.0050	0.010	0.020
BZ-123	0.0005	0.001	0.0025	0.0050	0.010	0.020
BZ-126	0.0005	0.001	0.0025	0.0050	0.010	0.020
BZ-128	0.0005	0.001	0.0025	0.0050	0.010	0.020
BZ-138	0.0005	0.001	0.0025	0.0050	0.010	0.020
BZ-153	0.0005	0.001	0.0025	0.0050	0.010	0.020
BZ-156	0.0005	0.001	0.0025	0.0050	0.010	0.020
BZ-157	0.0005	0.001	0.0025	0.0050	0.010	0.020
BZ-167	0.0005	0.001	0.0025	0.0050	0.010	0.020
BZ-169	0.0005	0.001	0.0025	0.0050	0.010	0.020
BZ-170	0.0005	0.001	0.0025	0.0050	0.010	0.020
BZ-180	0.0005	0.001	0.0025	0.0050	0.010	0.020
BZ-183	0.0005	0.001	0.0025	0.0050	0.010	0.020
BZ-184	0.0005	0.001	0.0025	0.0050	0.010	0.020
BZ-187	0.0005	0.001	0.0025	0.0050	0.010	0.020
BZ-189	0.0005	0.001	0.0025	0.0050	0.010	0.020
BZ-195	0.0005	0.001	0.0025	0.0050	0.010	0.020
BZ-206	0.0005	0.001	0.0025	0.0050	0.010	0.020
BZ-209	0.0005	0.001	0.0025	0.0050	0.010	0.020
SURROGATES						
TCMX	0.000825	0.00165	0.004125	0.00825	0.0165	0.033
BZ-205	0.0005	0.001	0.0025	0.0050	0.010	0.020

Table B-4				
LCS/Matrix Spike and Surrogate Spike Levels for Congener Analysis[1]				
ng/L, ng/wipe or ug/kg				
Compound	Aqueous	Soil/Sediment/Tissue	Wipe	Waste
BZ-8	20	20	20	200
BZ-18	20	20	20	200
BZ-28	20	20	20	200
BZ-44	20	20	20	200
BZ-49	20	20	20	200
BZ-52	20	20	20	200
BZ-66	20	20	20	200
BZ-77	20	20	20	200
BZ-81	20	20	20	200
BZ-87	20	20	20	200
BZ-101	20	20	20	200
BZ-105	20	20	20	200
BZ-114	20	20	20	200
BZ-118	20	20	20	200
BZ-126	20	20	20	200
BZ-123	20	20	20	200
BZ-128	20	20	20	200
BZ-138	20	20	20	200
BZ-153	20	20	20	200
BZ-156	20	20	20	200
BZ-157	20	20	20	200
BZ-167	20	20	20	200
BZ-169	20	20	20	200
BZ-170	20	20	20	200
BZ-180	20	20	20	200
BZ-183	20	20	20	200
BZ-184	20	20	20	200
BZ-187	20	20	20	200
BZ-189	20	20	20	200
BZ-195	20	20	20	200
BZ-206	20	20	20	200
BZ-209	20	20	20	200
Surrogates				
TCMX	5	5	5	50
BZ-205	5	5	5	50

[1]Individual Congeners are spiked as needed.

Table B-5		
Suggested Analytical Sequence		
Initial Calibration		
Injection #		
Solvent blank (optional)		
PCB Congener Mix 1	5-point	6-point
ICV (second source standard(s) of all congeners of interest)		
Samples 1-20 (or 12 hours, whichever is more frequent)		
Solvent blank (optional)		
PCB Congener Mix 1		Level 3
After 12 hours:		
PCB Congener Mix 1		Level 3
Samples 1-20 (or 12 hours)		
Solvent blank (optional)		
PCB Congener Mix 1		Level 3
12 hour Calibration		

At least every 24 hours, counting from the start of the initial calibration, or from the start of the last daily calibration, the retention time windows must be updated.

Title: Determination of Inorganic Anions by Ion Chromatography

Method(s): EPA Method 300.0 and SW846 Method 9056A

Approvals (Signature/Date):

11/20/2020	11/23/2020
Sharon Bacha Date	Steve Jackson Date
Organics Department Manager	Regional Safety Coordinator
11/20/2020	11/23/2020
Virginia Zusman Date	Deborah L. Lowe Date
Quality Assurance Manager	Laboratory Director

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1. SCOPE AND APPLICATION

1.1. This method covers the determination of fluoride, chloride, nitrite-nitrogen, bromide, nitrate-nitrogen, ortho-phosphate and sulfate in drinking water, surface water, mixed domestic and industrial wastewaters, groundwater, reagent waters, solids (after extraction 10.1.2) and leachates (when no acetic acid is used).

1.2. On occasion clients may request slight modifications to this SOP. Any modification to this procedure must be approved by the QA department of the Laboratory or Technical Director, and documented fully in an NCM.

1.3. The reporting limits for each anion are listed below. Limits for soil are based on an extraction of 1 unit of soil / 10 of water.

Analyte	Water RL (mg/L)	Soil RL (mg/Kg)
Bromide	0.10	2.0
Chloride	1.0	10.0
Fluoride	0.10	1.0
Nitrate-N	0.10	0.50
Nitrite-N	0.05	0.50
Ortho-Phosphorous	0.50	5.0
Sulfate	1.0	10.0

2. SUMMARY OF METHOD

2.1. A 10uL (INTEGRION, 2100A and 2100B) or 50uL (IC2000) volume of sample is introduced into the ion chromatograph. The sample is pumped through three different ion exchange columns and into a conductivity detector. The first two columns, a pre-column or guard column and a separator column, are packed with low-capacity, strongly basic anion exchange resin. Ions are separated into discrete bands based on their affinity for the exchange sites of the resin. The last column is a suppresser column that reduces the background conductivity of the eluent to a low or negligible level and converts the anions in the sample to their corresponding acids. The separated anions in their acid form are measured using an electrical conductivity cell. Anions are identified based on their retention times compared to known standards. Quantitation is accomplished by measuring the peak height or area and comparing it to a linear calibration curve generated from known standards.

3. DEFINITIONS

3.1. TALS – Eurofins TestAmerica Laboratory Information Management System

3.2. NCM - Non-Conformance Memo – a system within TALS for the lab to communicate to project management and others when there is an anomaly seen with the samples or batch, or a QC failure.

3.3. Refer to the glossary in the Pittsburgh Laboratory Quality Manual (PT-QA-M-001) for additional definitions.

4. INTERFERENCES

4.1. Interferences can be caused by substances with retention times that are similar to and overlap those of the anion of interest. Large amounts of an anion can interfere with the peak resolution of an adjacent anion. Sample dilution and/or fortification can be used to solve most interference problems associated with retention times.

4.2. The water dip or negative peak that elutes near, and can interfere with, the fluoride peak can usually be eliminated by the addition of concentrated eluent to each standard and sample. Due to the chromatographic conditions used, the addition of eluent to standards and samples is typically not necessary because the water dip is sufficiently resolved from the earliest eluting target anion (fluoride).

4.3. Method interferences may be caused by contaminants in the reagent water, reagents, glassware and other sample processing apparatus that lead to discrete artifacts or an elevated baseline in the ion chromatograms.

4.4. Any anion that is not retained by the column or only slightly retained will elute in the area of fluoride and interfere. Known co-elution is caused by carbonate and other small organic anions. At concentrations of fluoride above 1.5 mg/L, this interference may not be significant; however, it is the responsibility of the user to generate precision and accuracy information in each sample matrix.

4.5. The acetate anion elutes early during the chromatographic run. The retention times of the anions also seem to differ when large amounts of acetate are present. Therefore, this method is not recommended for leachates of solid samples when acetic acid is used for pH adjustment.

5. SAFETY

5.1. Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual (CW-E-M-001), the Pittsburgh Facility Addendum EH&S Manual (PT-HS-001) and this document. This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

5.2. Specific Safety Concerns or Requirements

5.2.1. Exercise caution when using syringes with attached filter assemblies. Application of excessive force has, upon occasion, caused a filter disc to burst during the process.

5.3. Primary Materials Used

5.3.1. A complete list of materials used in the method can be found in the reagents and materials section. No chemicals used with this method pose any safety concerns.

5.4. Employees must review the information in the SDS for each material before using it for the first time or when there are major changes to the SDS. Eye protection that protects against splash, laboratory coat, and appropriate gloves must be worn while samples, standards, solvents, and reagents are being handled. Cut resistant gloves must be worn doing any other task that presents a strong possibility of getting cut. Disposable gloves that have been contaminated will be removed and discarded; other gloves will be cleaned immediately.

5.5. Eye protection against splashing, laboratory coat, and appropriate gloves must be worn while samples, standards, solvents, and reagents are being handled. Cut resistant gloves must be worn when performing any task that presents a strong possibility of getting cut. Disposable gloves that have been contaminated must be removed and properly discarded. Other types of contaminated gloves will be cleaned immediately.

5.6. Exposure to chemicals must be maintained as low as reasonably achievable, therefore; unless they are known to be non-hazardous, all samples must be opened, transferred and prepared in a fume hood, or under other means of mechanical ventilation when possible. Solvent and waste containers will be kept closed transfers are being made.

5.7. All work must be stopped in the event of a known or potential compromise to the health and safety of a Eurofins TestAmerica associate. The situation must be reported immediately to a laboratory supervisor or the EH&S coordinator.

6. EQUIPMENT AND SUPPLIES

The following items are recommended for performing this procedure. Equivalent items should only be used when they result in an improvement in quality, efficiency, productivity, or cost. An item can be considered equivalent if with its use, the analytical and QA/QC requirements in this SOP can be met.

6.1. Instrumentation

6.1.1. Ion Chromatograph -- Analytical system complete with injector/autosampler, analytical columns, detectors and all necessary accessories.

Anion guard column: A protector of the separator column, usually packed with same substrate as the separator column. If the guard column is omitted from the system the retention times will be shorter.

- For INTEGRION, IC2100A & B - 2 x 50 mm, Dionex IonPac AG18, P/N 060555
- For IC2000 – 4 x 50 mm, Dionex IonPac AG22, P/N 064139

Anion separator column:

- For INTEGRION, IC2100A & B - 2 x 250mm, Dionex IonPac AS18, P/N 060553
- For IC2000 – 4 x 250mm, Dionex IonPac AS22, P/N 064141

Anion suppresser device: Dionex AERS 500 Carbonate Electrolytically Regenerated Suppressor, 2mm for IC2100A & B (P/N 085028), 4mm for IC2000 (P/N 085029). This suppressor eliminates the need for a regeneration solution.

Detector -- Conductivity cell: approximately 1.25 uL internal volume

Dionex –Chromeleon Data Chromatography Software

CHROM data processing software is used for data reporting

6.1.2. Eurofins TestAmerica Pittsburgh currently has three Dionex Ion Chromatographs

Models ICS2000 (IC2000), ICS2100 (2100A and 2100B) and Dionex Integrion HPIC (INTEGRION) all have an eluent generator, which allows facilitation of isocratic or gradient elution via the instrumentation's software program. This eluent gradiation may be performed to improve the chromatography of the instrument and to reduce the analysis runtime.

Note: At this time, IC2000 is using manually prepared eluent with an isocratic program.

6.1.3. Refer to PT-QA-022, Equipment Maintenance SOP, for routine instrument maintenance procedures.

6.2. Supplies

6.2.1. Analytical Balance capable of accurately weighing to the nearest 0.0001 g

6.2.2. Volumetric flasks, Class A, various sizes

6.2.3. Fixed or variable volume pipettors, calibration verified quarterly

7. REAGENTS AND STANDARDS

The following items are recommended for performing this procedure. Equivalent items should only be used when they result in an improvement in quality, efficiency, productivity, or cost. An item can be considered equivalent if with its use, the analytical and QA/QC requirements of this SOP can be met.

Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

7.1. Reagent water: Distilled or deionized water, free of the anions of interest. Water should contain particles no larger than 0.20 microns.

7.2. For the IC2000, prepare a 100x concentrated eluent solution by dissolving 8.40g of sodium bicarbonate ($NaHCO_3$) and 37.10g sodium carbonate (Na_2CO_3) in reagent water and dilute to 1000mL. Dilute 10.0mL of this concentrated solution to 1000 mL with reagent water to make the 1.0mM sodium bicarbonate/3.5mM sodium carbonate working eluent. The 100x concentrated eluent may also be purchased.

7.3. Dionex EGC III KOH Eluent Generator Cartridge, purchased from Thermo Scientific or equivalent vendor, is used by INTEGRION, IC2100A and IC2100B.

7.4. All stocks are purchased from commercial sources. Primary and secondary sources are required for each target analyte.

7.4.1. Primary Commercial IC solution A (CCV/LCS): F^- - 125 mg/L, Cl^- - 2500 mg/L, Br^- - 500 mg/L, NO_3^- - N- 125 mg/L, PO_4^{-3} – P - 125 mg/L, SO_4^{-2} - 2500 mg/L

7.4.2. Primary Commercial IC solution B (CCV/LCS): NO_2^- - N- 125 mg/L

7.4.3. Secondary Commercial stock solution A (ICV): F^- - 25 mg/L, Cl^- - 500 mg/L, Br^- - 100 mg/L, NO_3^- - N- 25 mg/L, PO_4^{-3} – P - 25 mg/L, SO_4^{-2} - 500 mg/L

7.4.4. Secondary Commercial stock solution B (ICV): NO_2^- - N- 25 mg/L

7.5. Working standards: Prepare calibration standards fresh daily in reagent grade water. Final concentrations of working standards are shown below:

NOTE: The following standard preparation tables are for example purposes and equivalent preparation volumes may be used.

Calibration Standard #1: take 0.05 mL of calibration STD #6 and dilute to 5.0 mL final volume.

Analyte	Final Conc.
Fluoride	0.025 mg/L
Chloride	0.50 mg/L
Nitrite	0.025 mg/L
Bromide	0.100 mg/L
Nitrate	0.025 mg/L
Ortho-Phosphate	0.025 mg/L
Sulfate	0.50 mg/L

Calibration Standard #2: take 0.1 mL of calibration STD #6 and dilute to 5.0 mL final volume.

Analyte	Final Conc.
Fluoride	0.050 mg/L
Chloride	1.00 mg/L
Nitrite	0.050 mg/L
Bromide	0.200 mg/L
Nitrate	0.050 mg/L
Ortho-Phosphate	0.050 mg/L
Sulfate	1.00 mg/L

Calibration Standard #3: take 0.5 mL of calibration STD #6 and dilute to 5.0 mL final volume.

Analyte	Final Conc.
Fluoride	0.250 mg/L
Chloride	5.00 mg/L
Nitrite	0.250 mg/L
Bromide	1.00 mg/L
Nitrate	0.250 mg/L
Ortho-Phosphate	0.250 mg/L
Sulfate	5.00 mg/L

Calibration Standard #4: take 0.5 mL of calibration STD #8 and dilute to 5.0 mL final volume.

Analyte	Final Conc.
Fluoride	0.500 mg/L
Chloride	10.00 mg/L
Nitrite	0.500 mg/L
Bromide	2.00 mg/L
Nitrate	0.500 mg/L
Ortho-Phosphate	0.500 mg/L
Sulfate	10.00 mg/L

Calibration Standard #5: take 1.0 mL of calibration STD #8 and dilute to 5.0 mL final volume.

Analyte	Final Conc.
Fluoride	1.00 mg/L
Chloride	20.00 mg/L
Nitrite	1.00 mg/L
Bromide	4.00 mg/L
Nitrate	1.00 mg/L
Ortho-Phosphate	1.00 mg/L
Sulfate	20.00 mg/L

Calibration Standard #6: take 0.1 mL of Primary solution A (7.4.1) and 0.1 mL of Primary solution B (7.4.2) and dilute to 5.0 mL final volume.

Analyte	Final Conc.
Fluoride	2.50 mg/L
Chloride	50.00 mg/L
Nitrite	2.50 mg/L
Bromide	10.00 mg/L
Nitrate	2.50 mg/L
Ortho-phosphate	2.50 mg/L
Sulfate	50.00 mg/L

Calibration Standard #7 for IC2000: take 0.15 mL of Primary solution A (7.4.1) and 0.15 mL of Primary solution B (7.4.2) and dilute to 5.0 mL final volume.

Analyte	Final Conc.
Fluoride	3.75 mg/L
Chloride	75.00 mg/L
Nitrite	3.75 mg/L
Bromide	15.00 mg/L
Nitrate	3.75 mg/L
Ortho-phosphate	3.75 mg/L
Sulfate	75.00 mg/L

Calibration Standard #8 for INTEGRION, IC2100A&B (Standard #8 for IC2000): take 0.2 mL of Primary solution A (7.4.1) and 0.2 mL of Primary solution B (7.4.2) and dilute to 5.0 mL final volume.

Analyte	Final Conc.
Fluoride	5.00 mg/L
Chloride	100.00 mg/L
Nitrite	5.00 mg/L
Bromide	20.00 mg/L
Nitrate	5.00 mg/L
Ortho-phosphate	5.00 mg/L
Sulfate	100.00 mg/L

Calibration Standard #9 for INTEGRION, IC2100A&B: take 0.6 mL of Primary solution A (7.4.1) and 0.6 mL of Primary solution B (7.4.2) and dilute to 10.0 mL final volume.

Analyte	Final Conc.
Fluoride	7.50 mg/L
Chloride	150.0 mg/L
Nitrite	7.50 mg/L
Bromide	30.0 mg/L
Nitrate	7.50 mg/L
Ortho-phosphate	7.50 mg/L
Sulfate	150.0 mg/L

Calibration Standard #10 for INTEGRION, IC2100A&B only: take 0.8 mL of Primary solution A (7.4.1) and 0.8 mL of Primary solution B (7.4.2) and dilute to 10.0 mL final volume.

Analyte	Final Conc.
Fluoride	10.0 mg/L
Chloride	200.0 mg/L
Bromide	40.0 mg/L
Nitrate	10.0 mg/L
Ortho-phosphate	10.0 mg/L
Sulfate	200.0 mg/L

NOTE: Standard 10 may not be used for Fluoride or Nitrite

7.5.1. **Continuing Calibration Verification**

Dilute 0.30mL of primary stock standards 7.4.1 and 7.4.2 to 15mL to prepare the CCV solution with the concentrations listed below. Prepare fresh daily.

CCV Solution Concentrations

Analyte	Final Conc.
Fluoride	2.5 mg/L
Chloride	50.0 mg/L
Nitrite	2.5 mg/L
Bromide	10.0 mg/L
Nitrate	2.5 mg/L
Ortho Phosphate	2.5 mg/L
Sulfate	50.0 mg/L

7.5.2. **Laboratory Control Sample**

Dilute 0.50mL of secondary stock standards 7.4.3 and 7.4.4 to 10.0mL to prepare the LCS solution with the concentrations listed below for IC2100A and IC2100B. For IC2000, LCS concentrations are twice as high, prepared by

diluting 1.0mL of the secondary stock standards to 10.0mL.. For soil sample batches, the LCS is taken through the leaching process along with the samples. Prepare LCS solution fresh daily.

LCS Solution Concentrations

Analyte	Final Conc. INTEGRION IC2100A & 2100B	Final Conc. IC2000
Fluoride	1.25 mg/L	2.50 mg/L
Chloride	25.0 mg/L	50.0 mg/L
Nitrite	1.25 mg/L	2.50 mg/L
Bromide	5.0 mg/L	10.0 mg/L
Nitrate	1.25 mg/L	2.50 mg/L
Ortho Phosphate	1.25 mg/L	2.50 mg/L
Sulfate	25.0 mg/L	50.0 mg/L

7.5.3. Matrix Spike Solution: Add 0.150 mL of primary stocks (7.4.1, and 7.4.2) dilute to 15 mL with sample for IC2100A and IC2100B. For IC2000, add 0.30mL of primary stocks to 15mL of sample. Final spike concentrations are the same as for the LCS listed in the table above.

7.5.4. Using the secondary stock standard(s) described in Section 7.4.3 and 7.4.4. Dilute these stock standards to as indicated in the table below to prepare the mixture to be used for the ICV solution. Prepare fresh daily.

ICV Solution:

Analyte	ml of Stock	Stock source	Final Vol.	Final Conc. mg/L
Fluoride	0.6 mL	7.4.3	5 mL	3.0
Chloride	0.6 mL	7.4.3	5 mL	60
Nitrite	0.6 mL	7.4.4	5 mL	3.0
Bromide	0.6 mL	7.4.3	5 mL	12
Nitrate	0.6 mL	7.4.3	5 mL	3.0
Ortho Phosphate	0.6 mL	7.4.3	5 mL	3.0
Sulfate	0.6 mL	7.4.3	5 mL	60

7.6.5 The ability to accurately detect concentrations at the reporting limit must be verified at a minimum with each calibration curve. The Reporting Limit Standard, (LLOQ, ICVL, or CCVL) is prepared by diluting 0.10mL of primary stock standards 7.4.1 and 7.4.2 to 250mL. The concentrations of this solution are listed below. Prepare fresh daily.

LLOQ/CCVL Solution Concentrations

Analyte	Final Conc.
Fluoride	0.05 mg/L
Chloride	1.00 mg/L
Nitrite	0.05 mg/L
Bromide	0.20 mg/L
Nitrate	0.05 mg/L
Ortho Phosphate	0.05 mg/L
Sulfate	1.00 mg/L

NOTE: All standards should be stored in the dark at > 0.0°C but ≤6.0 °C. Replace these standards when instrument response indicates target analyte degradation may have occurred or after the standard has expired, whichever occurs first. Stock standards are purchased from a vendor and have vendor assigned expiration dates. The dilutions made from the stocks are prepared weekly. Nitrite and ortho-phosphate are particularly light and oxygen sensitive and should be prepared daily if needed.

8. SAMPLE COLLECTION, PRESERVATION, SHIPMENT AND STORAGE

8.1. Samples should be collected in plastic bottles. All bottles must be thoroughly cleaned and rinsed with reagent water. The volume collected should be sufficient to ensure a representative sample, allow for replicate analysis, if required, and minimize waste disposal.

8.2. Sample preservation and holding times for the anions that can be determined by this method for water samples are as follows:

Method	Analyte	Preservation	Holding Time	Reference
300.0/9056A	Fluoride	>0.0°C but ≤6.0 °C	28 days	40CFR Part 136 Table II
300.0/9056A	Chloride	>0.0°C but ≤6.0 °C	28 days	40CFR Part 136 Table II
300.0/9056A	Nitrite	>0.0°C but ≤6.0 °C	48 hours	40CFR Part 136 Table II
300.0/9056A	Bromide	>0.0°C but ≤6.0 °C	28 days	40CFR Part 136 Table II
300.0/9056A	Nitrate	>0.0°C but ≤6.0 °C	48 hours	40CFR Part 136 Table II
300.0/9056A	Ortho-Phosphate	>0.0°C but ≤6.0 °C	48 hours; filter sample within 15 minutes of collection	40CFR Part 136 Table II
300.0/9056A	Sulfate	>0.0°C but ≤6.0 °C	28 days	40CFR Part 136 Table II

Note: Soil leachates (i.e DI Leach, ASTM Leach, SPLP, etc.) will follow the same holding times as the water samples; starting from the time of extraction. See Section 10.1.2 for the soil extraction procedure. If project specific requirements exist for sample handling, follow the project specific protocols.

9. QUALITY CONTROL

9.1. The following quality control samples are prepared and analyzed with each batch of 20 or fewer samples of the same matrix.

QC Samples	Frequency	Acceptance Criteria	Corrective Action
Method Blank (MB)	1 per batch of 20 samples per matrix	< MDL – Method 300.0 < RL – Method 9056A	Rerun once, reanalyze all samples since the last acceptable MB
Laboratory Control Sample (LCS)	1 per batch of 20 samples	90-110% - Method 300.0 80-120% - Method 9056A	Reanalyze all samples associated with unacceptable LCS
Matrix Spike Sample (MS/MSD)[1]	For Method 300.0: 1 MS/MSD pair per 10 samples For Method 9056A: 1 MS/MSD pair per batch or 20 samples	Meets laboratory limits Method 300.0: 90-110% ±20% RPD Method 9056A: 80-120% ±15% RPD	Supervisor's technical judgment

[1]The parent sample for MS/MSD is randomly selected, unless specifically requested by a client.

9.1.1. Method Blank (MB): One method blank must be processed with each batch of 20 or fewer samples of similar matrix. The method blank for aqueous samples consists of reagent grade water that is filtered along with the samples. The method blank is used to identify any system and process interferences or contamination of the analytical system that may lead to the reporting of elevated analyte concentrations or false positive data. Because there is no preparation step for aqueous samples, the method blank and CCBs are the same for this analysis. The method blank should not contain any analyte of interest above the MDL for method 300.0, or above the RL for method 9056A. All samples are associated with the immediately preceding MB/CCB.

NOTE: A solid matrix (simulated using glass or plastic beads or Teflon chips because purified sand still contains chloride contamination) will be prepared and tumbled with soils extracted using the 1-Hour DI Leaching procedure found in SOP PT-WC-033.

- If the MB contains any of the target anions above the MDL/RL, the source of the contamination should be investigated and corrected, and all associated samples reanalyzed, unless one of the following situations exists:
  - If the MB is contaminated, but associated samples results are >10x the MB concentration, results may be reported using the "Method Blank – Report, 10X" NCM.

- If the contamination anion is not detected above the MDL/RL in the associated samples, results may be reported with qualifier and using the "Method Blank – Report, ND" NCM.
- If neither of the above criteria are met, but a reanalysis will be outside the holding time, contact the Project Manager for approval from the client to perform the reanalysis.

9.1.2. Laboratory Control Sample (LCS): One LCS must be processed with each batch of 20 or fewer samples of similar matrix. The LCS is filtered along with samples. The LCS is used to monitor the accuracy of the analytical process. On-going monitoring of the LCS results provides evidence that the laboratory is performing the method within acceptable accuracy and precision guidelines. The LCS consists of reagent grade water* containing a known amount of target analytes. The LCS is prepared from the secondary stock standard used to prepare ICV. The LCS recovery must be within 90-110% of the true value for method 300.0 and 80-120% for method 9056A. Because the LCS is prepared from the primary source standard, has no additional prep procedures and must meet the same acceptance criteria, the LCS can serves as a CCV for method 300.0. For QC batch purposes all samples are associated with the immediately preceding LCS/CCV.

***NOTE**: A solid matrix (simulated using plastic beads or Teflon chips) LCS will be prepared with soils extracted using the 1-Hour DI Leaching procedure found in SOP PT-WC-033.

- If the LCS recovery falls outside the method acceptance limits, all associated samples must be reanalyzed with an acceptable LCS unless one of the following situations exists:
  - If the LCS fails with recovery above the upper control limit, and the associated sample result is non-detect, results may be reported with qualifier and the "LCS/LCSD - %R High" NCM.
  - If the reanalysis will be outside the holding time, contact the Project Manager for approval from the client to perform reanalysis.

9.1.3. Matrix Spike/Matrix Spike Duplicate (MS/MSD): For method 9056A, one MS/MSD pair must be processed for each batch of 20 or fewer samples of similar matrix. For method 300.0 an MS/MSD pair must be analyzed for every 10 samples analyzed. A matrix spike (MS) is a field sample to which a known concentration of target analyte has been added. A matrix spike duplicate (MSD) is a second aliquot of the same sample (spiked identically as the MS) prepared and analyzed along with the sample and matrix spike. Some client specific DQO's may require the use of sample duplicates in place of or in addition to MS/MSD's. The MS/MSD results are used to determine the effect of a matrix on the precision and accuracy of the analytical process. Due to the potential variability of the matrix of each sample, these results may have immediate bearing only on the specific sample spiked. Spiking levels should be similar to the concentrations expected in the samples, if known, otherwise, the MS/MSD shall be spiked the same as the LCS values.

- If the recovery of the MS or MSD is outside the limits, corrective action must be taken. Corrective action may include re-preparation and reanalysis of the parent sample and MS/MSD.
    - If the LCS recovery is within control limits, MS failure may be due to a matrix interference. Results may be reported with qualifier and the "MS/MSD- %R, LCS Pass" NCM.
    - If the MS/MSD recovery or RPD falls outside the acceptance range, the recovery of the analyte must be in control for the LCS. If the native analyte concentration in the MS/MSD exceeds 4x the spike level for that analyte, the recovery data are reported with qualifier and the failure explained in the case narrative.
    - If a MS/MSD is not possible due to limited sample volume then a LCS duplicate may be analyzed. The RPD of the LCS and LCSD must meet the MD/MSD %RPD limit. If the MS/MSD pair cannot be run, the "MS/MSD/DUP – Insufficient Volume" NCM must be applied to the batch.

9.1.4 **If samples in the batch are filtered prior to analysis, the MB and LCS must also be filtered using the same method. If the parent sample is filtered, the MS and MSD must also be filtered using the same method.**

9.2. Instrument QC

9.2.1. Initial Calibration Verification (ICV): An ICV must be analyzed at the beginning of the sequence each day the instrument is used and immediately following the initial calibration of the instrument. The ICV is used to monitor the initial calibration curve. If the result is outside established control limits, the system is out of control and corrective action must occur. The ICV consists of reagent grade water containing a known amount of target analytes that has been injected into the ion chromatography system. The ICV is prepared from a separate stock standard from a different manufacturer than the stock used to prepare the calibration standards. ICV recovery must be within 90-110% of the true value.

9.2.2. Continuing Calibration Verification (CCV): Continuing calibration stability is verified by analyzing a near mid-range calibration verification standard after every ten (10) samples and at the end of the analytical sequence. CCV recovery must be within 90-110%.
If a CCV falls outside control limits for a target anion to be reported, all samples after the last acceptable CCV must be re-analyzed, unless the recovery is high and the associated sample results are non-detect. If results are associated with a failed CCV, they must be reported with qualifier in all cases.

9.2.3. A Continuing Calibration Blank (CCB) is analyzed immediately following each CCV to monitor low level accuracy and system cleanliness. CCB results must

be < MDL. Corrective actions for CCB failures are the same as for Method Blanks (see section 9.1.1).

9.3. Lower Limit of Quantitation (LLOQ): the lowest concentration level that can be measured within stated accuracy limits, varies for each individual analyte anion and as a function of sample size. The LLOQ standard is analyzed after an initial calibration curve. The LLOQ recoveries must be within ±50% of the true value to verify the reporting limit.

10. PROCEDURE

10.1. Sample Preparation

10.1.1. No sample preparation is required for aqueous samples. Aqueous samples are pre-screened for conductivity to determine if any dilutions are required for sample detections to fall within the calibrated range and to avoid overloading the analytical column (see Table 2).

10.1.2. See SOP PT-WC-033 for solid sample preparation using the 1-Hour DI Leaching procedure. Filter the resulting slurry before injecting using a 0.45 um membrane type filter. A Leach Blank and a solid matrix LCS will also be prepared and analyzed along with solid samples.

10.1.3 All samples and QC (MB, LCS and MS/MSD) are filtered using a 0.45 um filter before loading onto the instrument.

10.2. Calibration and Standardization

10.2.1. Establish ion chromatographic operating parameters equivalent to those indicated in Table 1. When using the fast chromatographic run for chloride, bromide, nitrate and sulfate only the guard column is used. Refer to Table 3 for analyte elution order on each column type.

10.2.2. For each analyte of interest, prepare a calibration blank and 6 calibration standards (IC2000) or 8 calibration standards (INTEGRION, IC2100A and IC2100B) by adding accurately measured volumes of one or more stock standards to a volumetric flask and dilution to volume with reagent water. If a sample analyte concentration exceeds the calibration range the sample may be diluted to fall within the range. If this is not possible then three new calibration concentrations must be chosen, two of which must bracket the concentration of the sample analyte of interest. Each attenuation range of the instrument used to analyze a sample must be calibrated individually.

10.2.3. Using an injection volume of 50 uL (IC2000) or 10 uL (INTEGRION, IC2100A and IC2100B) of each calibration standard, tabulate peak height or area responses against the concentration and prepare a calibration curve for each analyte. During this procedure, retention times must be recorded.

10.2.4. Calibration curves are plotted by the instrument software as either peak height or peak area vs concentration. Peak height is preferred for anions that produce sharp, symmetrical peaks though-out the calibrated range. Otherwise, peak area is used.

10.2.5. Curves plots are calculated using either weighted or un-weighted linear regression. The calibration model is chosen based on the best fit to the data to meet the criteria listed below.

- Calculate the %Error for each standard read back to the plotted curve. CHROM software will perform this calculation. The %E for each point above the lowest must be $\leq$ 20%, for the lowest calibration standard the %E must be $\leq$50%.
- The linear correlation coefficient (r^2), must be $\geq$0.995. The absolute value of the intercept must not be above the reporting limit and should be < ½ RL.
- The progression of curve models by preference is:
  Weighted Linear ($1/x^2$)
  Un-weighted Linear
  Linear Forced through Zero

  NOTE: Curves associated with the analysis of any samples from Wisconsin may not be forced through zero. It may be necessary to shorten the calibrated range to achieve linearity with good precision at lower concentrations without forcing the curve plot through the origin.

10.2.6 It is expected that the same curve model should be used for an anion/column combination for each calibration. A need to change the calibration model may indicate that instrument or column maintenance is required.

10.2.7 Retention Time Monitoring

10.2.7.1 The mean retention times for the analytes in the calibration standards will be determined and the retention times of the ICV and CCV will be compared to them.

10.2.7.2 If the retention times of any analyte(s) in the ICV or CCV vary by more than ± 10% from the mean retention time determined from the initial calibration, a new calibration curve will need to be analyzed and all samples not bracketed by a compliant ICV/CCV or CCV/CCV pair will need to be reanalyzed.

10.2.7.3 Daily Retention Time Windows

Establish the center of the retention time windows from the calibration verification at the beginning of the analytical shift. For samples run during the same shift as an initial calibration, use the retention time of the mid-point standard of the initial calibration. Retention time windows can be updated every 12 hours. However, they are usually only updated at the onset of a continuing calibration sequence or after maintenance has been performed.

10.3. Sample Analysis

10.3.1. Any unauthorized deviations from this procedure must also be documented as a nonconformance, with a cause and corrective action described.

10.3.2. Table 1 summarizes the recommended operating conditions for the ion chromatograph. Included in this table are estimated retention times that can be achieved by this method. Other columns, chromatographic conditions, or detectors may be used if the requirements of the QC Table under section 9 are met.

10.3.3. Check system calibration daily and, if required, recalibrate as described in Section 10.2.

10.3.4. Load and inject a fixed amount (10 uL or 50 uL) of settled & filtered sample. Refer to PT-QA-024 for subsampling procedures. If the sample is cloudy then it should be filtered prior to loading into the autosampler polyvial. Filtration will be performed using a 0.45 um filter. If samples are filtered, the method blank and LCS are also filtered. Use the same size loop for standards and samples. Record the resulting peak size in area or peak height units. An automated constant volume injection system may also be used.

10.3.5. The width of the retention time window used to make identifications should be based upon measurements of actual retention time variations of various concentrations. Three times the standard deviation of a retention time can be

used to calculate a suggested window size for each analyte. However, the experience of the analyst should weigh heavily in the interpretation of chromatograms since retention time is concentration dependent for most analytes.

10.3.6. If the response for the peak exceeds the working range of the system, dilute the sample with an appropriate amount of reagent water and reanalyze.

10.3.7. If the resulting chromatogram fails to produce adequate resolution, or if identification of specific anions is questionable, fortify the sample with an appropriate amount of standard and reanalyze.

NOTE: Retention time is affected by concentration. Nitrate and sulfate exhibit the greatest amount of change, although all anions are affected to some degree. In some cases this peak migration may produce poor resolution or identification.

10.3.8. Should more complete resolution be needed between peaks, the first step would be diluted analysis of the sample. In extreme cases, the eluent (7.3 or 7.4) can be diluted. This will spread out the run but will also cause the later eluting anions to be retained longer. The analyst must determine to what extent the eluent is diluted. A calibration curve must be run with the diluted eluent prior to sample analysis. This dilution should not be considered a deviation from the method.

11. CALCULATIONS / DATA REDUCTION

11.1. Prepare a calibration curve for each analyte by plotting instrument response against standard concentration. Compute sample concentration by comparing sample response with the standard curve. Multiply answer by appropriate dilution factor.

11.2. Report only those values that fall between the lowest and the highest calibration standards. Samples exceeding the highest standard should be diluted and reanalyzed.

11.3. Report results in mg/L for aqueous samples and mg/kg for 1-hour leachates and mg/L for 18-hour leachates of solid samples.

11.4. Report

NO2- as N
NO_3^- as N
PO_4^{-3} as P

11.5. When performing manual integration, the reason for it must be clearly documented. For manual integration requirements and documentation refer to SOP CA-Q-S-002.

11.6. If samples for ortho-phosphate analysis have not been filtered in the field within 15 minutes of sampling, results must be qualified as not meeting preservation/holding time requirments.

12. METHOD PERFORMANCE

12.1. The supervisor has responsibility to ensure that an analyst who performs this procedure is properly trained in its use and has the required experience. Performance

is monitored through internal QC and outside performance evaluation samples. Please refer to the QA Manual for additional information concerning Precision and Accuracy.

12.2. Demonstration of Capabilities – Prior to the analysis of samples, a Demonstration of Capabilities (DOC) as described in the QA Manual, must be performed initially, annually and any time a significant change is made to the analytical system.

12.3. A Method Detection Limit (MDL) study, as described in the Detection Limit SOP, PT-QA-007, must be performed initially and whenever a significant change is made to the analytical system. The MDL must be re-evaluated from quarterly MDL points at least every 12 months.

12.4. For Method 300.0, the Linear Calibration Range must be verified every 6 months to ensure that the instrument response is linear beyond the regular calibration range. The criteria used to evaluate the Linear Calibration Range standard is ± 10% of the true value of the standard.

13. POLLUTION CONTROL

13.1. It is Eurofins TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage and reagent stability). Employees must abide by the policies in Section 13 of the Corporate Environmental Health and Safety Manual (CW-E-M-001) for "Waste Management and Pollution Prevention." and the Pittsburgh Facility Addendum EH&S Manual (PT-HS-001).

13.2. This method does not contain any specific modifications that serve to minimize or prevent pollution.

14. WASTE MANAGEMENT

14.1. Waste management practices are conducted consistent with all applicable rules and regulations. Excess reagents, samples and method process wastes are disposed of in an accepted manner. Waste description rules and land disposal restrictions are followed. Waste disposal procedures are incorporated by reference to SOP PT-HS-001. The following waste streams are produced when this method is carried out. The following waste streams are produced when this method is carried out.

14.1.1. Alkaline and/or acidic waste generated by the analysis. This waste is collected in a waste container identified as "Waste Water". This waste is checked with pH paper and neutralized to a final pH between 6 and 9, if needed, and discharged down into a lab sink.

14.2. Contaminated plastic materials such as IC syringes, filters, caps and vials utilized for sample preparation. This waste is placed in trash containers and disposed as regular trash.

15. REFERENCES

15.1. Method 300.0, “Determination of Inorganic Anions by Ion Chromatography”, Environmental Monitoring Systems Laboratory, Office of Research and Development, U.S. Environmental Protection Agency, Cincinnati, Ohio, Revision 2.1, August 1993

15.2. Method 9056A, “Determination of Inorganic Anions by Ion Chromatography”, SW846, Test Methods for Evaluating Solid Waste, Revision 1, February 2007

15.3. SOP PT-QA-006, Procurement of Standards and Materials; Labeling and Traceability

15.4. SOP PT-QA-015, Maintaining Time Integrity

15.5. SOP PT-QA-016, Nonconformance & Corrective Action System

15.6. SOP PT-QA-022, Equipment Maintenance

15.7. SOP PT-QA-031, Internal Chain of Custody

15.8. SOP PT-QA-024, Subsampling

15.9. SOP PT-QA-021, Quality Control Program

15.10. Corporate SOP CA-Q-S-002, Manual Integration

15.11. SOP PT-QA-007, Detection Limits

15.12. SOP PT-WC-033, DI Leachate Procedure for Solids (1 Hour Routine DI Leach and 18 Hour ASTM DI Leachate Procedure)

15.13. PT-QA-M-001, Pittsburgh Laboratory Quality Manual

15.14. PT-HS-001, Pittsburgh Facility Addendum EH&S Manual

16. METHOD MODIFICATIONS

16.1. None

17. ATTACHMENTS

17.1. Attachment 1 - Method Flow Chart

17.2. Table 1 - Standard Instrument Operating Parameters

17.3. Table 2 – TestAmerica Pittsburgh IC Dilution Table Based on Conductivity

17.4. Table 3 – Elution Order for Column Types

18. REVISION HISTORY

18.1. Revision 8, 09/14/07

18.2. Revision 9, 07/14/08

18.3. Revision 10, 7/27/2009

18.4. Revision 11, 7/28/2010

18.5. Revision 12, 7/3/2012

18.6. Revision 13, 11/12/2012

18.7. Revision 14, 5/29/2014

18.8. Revision 15, 3/3/2015

18.9. Revision 16, 4/19/2017

18.10. Revision 17, 4/6/2017

18.11. Revision 18, 5/31/2019

18.12. Revision 19, 4/28/2020

18.13. Revision 20, 5/25/2020

18.14. Changes to current revision

SOP section	Change from	Change to	Reason
1.3	Br W RL 0.5 mg/L Br S RL 5.0 mg/kg	Br W RL 0.1 mg/L Br S RL 1.0 mg/kg	Update to current procedure
Entire SOP		Added reference for new IC INTEGRION	Correction
Entire SOP	Removed reference to LL Bromide		Correction
7.5	Cal Std. #4 - 0.5 mL of Std. #7 Cal Std. #5 – 1.0 mL of Std. #7	Cal Std. #4 - 0.5 mL of Std. #8 Cal Std. #4 – 1.0 mL of Std. #8	Correction
9.1.2	LCS prepped from primary stock standard	LCS prepped from secondary stock standard used to prep the ICV	Correction

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TABLE 1

Standard Instrument Operating Parameters

Standard Conditions for IC2000:

Eluent Pump Rate:	1.20 - 1.50 mL/min
Sample Loop:	50 uL (partial loop injection)
Eluent:	1.0mM sodium bicarbonate, 3.5mM sodium carbonate
Detector Output:	Baseline conductivity 15 - 30 uS prior to sample analysis.
Columns:	AS22 (4 x 250mm)

Standard Conditions for INTEGRION, IC2100A and IC2100B:

Eluent Pump Rate:	0.30 mL/min
Sample Loop:	10 uL (partial loop injection)
Eluent:	range of 28 to 36 mM KOH using the Eluent Generator Cartridge
Detector Output:	Total conductivity < 1.0 uS prior to sample analysis.
Columns:	AS18 (2 x 250mm)

Table 2

IC Dilution Table Based on Conductivity

Conductivity(µS)	Dilution Required	Dilution Formula[1]
IC2000		**AS22 Column (4 x 250 mm)**
1 - 300	Undiluted	1 mL of undiluted sample
--	2.5	400 uL sample : 600 uL DI water
300 - 750	5	200 uL sample : 800 uL DI water
750 - 1250	10	100 uL sample : 900 uL DI water
1250 - 2500	25	40 uL sample : 960 uL DI water
2500 - 5000	50	20 uL sample : 980 uL DI water
5000 - 10000	100	10 uL sample : 990 uL DI water
10000 - 20000	250	100 uL(40 uL sample : 960 uL DI water) : 900 uL DI Water
20000 - 40000	500	100 uL(20 uL sample : 980 uL DI water) : 900 uL DI water
IC2100A/IC2100B		**AS18 Column (2 x 250 mm)**
1 - 600	Undiluted	1 mL of undiluted sample
--	2.5	400 uL sample : 600 uL DI water
600 - 1500	5	200 uL sample : 800 uL DI water
1500 - 2500	10	100 uL sample : 900 uL DI water
2500 - 5000	25	40 uL sample : 960 uL DI water
5000 - 10000	50	20 uL sample : 980 uL DI water
10000 - 20000	100	10 uL sample : 990 uL DI water
20000 - 40000	250	100 uL(40 uL sample : 960 uL DI water) : 900 uL DI Water
40000 - 80000	500	100 uL(20 uL sample : 980 uL DI water) : 900 uL DI water
80000 - 160000	1000	100 uL(10 uL sample : 990 uL DI water) : 900 uL DI water
QC Sample	**Dilution Required**	**QC Reagent Formula**
ICV	NA	600 uL of Secondary A STD + 600 uL of Secondary B STD + 3.8 mL DI Water
CCV	NA	300 uL Primary A STD + 300 uL Primary B STD + 14.4 mL DI Water
LCS	NA	100 uL Primary A STD + 100 uL Primary B STD + 9.8 mL DI Water
MS/MSD	Undiluted	300 uL Matrix Spiking Solution[2] + 14.7 mL of sample
MS/MSD	2.5	300 uL Matrix Spiking Solution[2] + 8.7 mL DI water + 6 mL of sample
MS/MSD	5	300 uL Matrix Spiking Solution[2] + 11.7 mL DI water + 3 mL of sample
MS/MSD	10	300 uL Matrix Spiking Solution[2] + 13.2 mL DI water + 1.5 mL of sample
MS/MSD	25	300 uL Matrix Spiking Solution[2] + 14.1 mL DI water + 600 uL of sample
MS/MSD	50	300 uL Matrix Spiking Solution[2] + 14.4 mL DI water + 300 uL of sample
MS/MSD	100	300 uL Matrix Spiking Solution[2] + 14.55 mL DI water + 150 uL of sample
MS/MSD	250	300 uL Matrix Spiking Solution[2] + 14.64 mL DI water + 60 uL of sample
MS/MSD	500	300 uL Matrix Spiking Solution[2] + 14.67 mL DI water + 30 uL of sample

[1]Primary dilutions are contained within the innermost parentheses. Secondary dilutions bracket the primary dilutions. If the analyst does not see the dilution listed on the work instruction; the dilution performed must be indicated on the raw data, including the dilution factor, in the “sample Info”: field of the quantitation report.

[2] The Matrix Spiking Solution consists of 150 uL of Primary A Standard and 150 uL of Primary B Standard.

TABLE 3

ELUTION ORDER FOR COLUMN TYPE

AS22 COLUMN ELUTION ORDER	AS18 COLUMN ELUTION ORDER
Fluoride	Fluoride
Chloride	Chloride
Nitrite	Nitrite
Bromide	Sulfate
Nitrate	Bromide
ortho-Phosphate	Nitrate
Sulfate	ortho-Phosphate

SOP # with Revision: PT-MT-005 Revision 20
SOP Title: Preparation and Analysis of Mercury by CVAA
Amendment Number: 1

Effective Date of Amendment: 10/13/2020
Append this form to the front of the SOP copy.

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The following SOP change is in effect as of the stated date. *This form will remain attached to the referenced SOP until such a time that the SOP is updated, approved, and redistributed, at which time it will become part of the historical SOP record.*

1. Briefly explain the reason for the update: The autodigestor was moved and the change of location affected the temperature of the enclosure and block.

2. Category for SOP Change:

☐ Typographical Corrections (Non-Technical) – Retraining Not Required.

☐ Typographical Corrections (Technical- define below) – Retraining is required.

X Procedural Changes (Define Below) – Retraining Required.

3. Summary of Procedure Change (circle to indicate if there are attachments to this form: (No) Yes: # pages attached = ___)

Section 10.3.9 bullet 5 and NOTE under bullet 6 – change temperature setting from 108°C to 96°C.

Does this change result in a Method Modification? Yes (No)
If yes, please explain below.

Any method modification will be incorporated into section 16 of the SOP at the next revision.

10.14.20
Approval Signature/Date: Technical

10-13-20
Approval Signature/Date: QA Manager or Designee

TITLE: PREPARATION AND ANALYSIS OF MERCURY BY COLD VAPOR ATOMIC ABSORPTION

METHODS: EPA 245.1, SW846 7470A AND 7471A & B

Approvals (Signature/Date):

3/30/2020	3/23/2020
Roseann Ruyechan Date	Steve Jackson Date
Inorganics Department Manager	Regional Safety Coordinator
3/18/2020	4/1/2020
Virginia Zusman Date	Deborah L. Lowe Date
Quality Assurance Manager	Laboratory Director

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1. SCOPE AND APPLICATION

1.1. This procedure describes the preparation and analysis of mercury (Hg, CAS # 7439-97-6) by Cold Vapor Atomic Absorption Spectroscopy (CVAA) using SW-846 Methods 7470A 7471A and 7471B and MCAWW Method 245.1. The TestAmerica Pittsburgh facility utilizes hot block digestion for all preparations.

1.2. CVAA analysis provides for the determination of total mercury (organic and inorganic). The combination of the oxidants, potassium permanganate and potassium persulfate, has been found to give 100% recovery with both types of compounds. Detection limits, sensitivity and optimum concentration ranges for mercury analysis will vary with the matrices, instrumentation and volume of sample used.

1.3. Method 7470A is applicable to the preparation and analysis of mercury in ground water, aqueous samples, wastes, wipes, TCLP, SPLP and other leachates/extracts. Certain solid and sludge type wastes may also be analyzed using this method. Methods 7471A and 7471B are applicable to the preparation and analysis of mercury in soils, sediments, bottom deposits, sludge-type materials wipes, and tissue matrices. All matrices require sample preparation prior to analysis.

1.4. Method 245.1 is applicable to the determination of mercury in drinking, surface and saline waters, domestic and industrial wastes. All matrices require sample preparation prior to analysis.

1.5. The TestAmerica reporting limit for mercury in aqueous matrices is 0.0002 mg/L. The reporting limit in solid matrices is 0.033mg/kg based on a 0.6g sample aliquot (wet weight). The reporting limit for wipes is 0.02 ug/wipe.

1.6. On occasion clients may request modifications to this SOP. Any modification to this procedure must be approved by the QA department of the Laboratory or Technical Director, and documented fully in an NCM.

2. SUMMARY OF METHOD

2.1. This SOP describes a technique for the determination of mercury in solution. The procedure is a physical method based on the absorption of radiation at 253.7 nm by mercury vapor. A representative portion of the sample is digested in acid. Organic mercury compounds are oxidized with potassium permanganate and potassium persulfate and the mercury reduced to its elemental state with stannous chloride and aerated from solution in a closed system. The mercury vapor passes through a cell positioned in the light path of an atomic absorption spectrophotometer. Absorbance is measured as a function of mercury concentration. Concentration of the analyte in the sample is determined by comparison of the sample absorbance to the calibration curve (absorbance vs. concentration).

3. DEFINITIONS

3.1. Dissolved Metals - Those elements which pass through a 0.45 um membrane. (Sample is acidified after filtration).

3.2. Suspended Metals - Those elements which are retained by a 0.45 um membrane.

3.3. Total Metals - The concentration determined on an unfiltered sample following digestion.

3.4. TALS – TestAmerica Laboratory Information Management System

3.5. NCM – Non-Conformance Memo – a system within TALS for the lab to communicate to project management and others when there is an anomaly seen with the samples or batch, or a QC failure.

3.6. Refer to the glossary in the Laboratory Quality Manual (PT-QA-M-001) for additional definitions.

4. INTERFERENCES

4.1. Sulfide may pose an interference with this analysis. Potassium permanganate, which is used to breakdown organic mercury compounds, also eliminates possible interferences from sulfide. Concentrations as high as 20 mg/L of sulfide, as sodium sulfide, do not interfere with the recovery of inorganic mercury from reagent water.

4.2. Copper has also been reported to interfere; however, copper concentrations as high as 10 mg/L had no effect on the recovery of mercury from spiked samples.

4.3. Chlorides can cause a positive interference. Seawaters, brines and industrial effluents high in chlorides require additional permanganate (as much as 25 mL) because, during the oxidation step, chlorides are converted to free chlorine, which also absorbs radiation at 253.7 nm. Care must be taken to ensure that free chlorine is absent before the mercury is reduced and swept into the cell. This is accomplished by adding excess hydroxylamine reagent (25 mL) and purging the sample head space before stannous chloride is added. Both inorganic and organic mercury spikes have been quantitatively recovered from seawater using this technique. Alternatively, samples may be allowed to stand for at least one hour under a hood (without active purging) to remove the chlorine.

Note: Sufficient addition of permanganate is apparent when the purple color persists at least 15 minutes. Some samples may require dilution prior to digestion due to extremely high concentrations of chloride.

4.4. Interference from certain volatile organic materials that absorb at this wavelength may also occur. If suspected, a preliminary run without stannous chloride can determine if this type of

interference is present. While the possibility of absorption from certain organic substances present in the sample does exist, this problem is not routinely encountered. This is mentioned only to caution the analyst of the possibility. If this condition is found to exist, the mercury concentration in the sample can be determined by subtracting the result of the sample run without the reducing reagent (stannous chloride) from that obtained with the reducing reagent.

4.5. Samples containing high concentrations of oxidizable organic materials, as evidenced by high COD levels, may not be completely oxidized by this procedure. When this occurs the recovery of mercury will be low. The problem can be eliminated by reducing the volume of original sample used.

4.6. The most common interference is laboratory contamination, which may arise from impure reagents, dirty glassware, improper sample transfers, dirty work areas, etc. Be aware of potential sources of contamination and take appropriate measures to minimize or avoid them.

5. SAFETY

5.1. Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual (CW-E-M-001), the Pittsburgh Facility Addendum EH&S Manual (PT-HS-001) and this document. This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

5.2. Samples that contain high concentrations of carbonates or organic material, or samples that are at elevated pH, can react violently when acids are added.

5.3. Do not look directly into the beam of the Hg lamp. The UV light that these lamps radiate is harmful to the eyes.

5.4. The following is a list of the materials used in this method, which have a serious or significant hazard rating. This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the SDS for each of the materials listed in the table. A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the SDS for each material before using it for the first time or when there are major changes to the SDS.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Mercury (1,000 PPM in Reagent)	Oxidizer Corrosive Poison	0.1 mg/m^3 Ceiling (Mercury Compounds)	Extremely toxic. Causes irritation to the respiratory tract. Causes irritation. Symptoms include redness and pain. May cause burns. May cause sensitization. Can be absorbed through the skin with symptoms to parallel ingestion. May affect the central nervous system. Causes irritation and burns to eyes. Symptoms include redness, pain, and blurred vision; may cause serious and permanent eye damage.
Sulfuric Acid	Corrosive Oxidizer Dehydrator Poison	1 mg/m^3-TWA	Inhalation produces damaging effects on the mucous membranes and upper respiratory tract. Symptoms may include irritation of the nose and throat, and labored breathing. Symptoms of redness, pain, and severe burn can occur. Contact can cause blurred vision, redness, pain and severe tissue burns. Can cause blindness.
Nitric Acid	Corrosive Oxidizer Poison	2 ppm-TWA 4 ppm-STEL	Nitric acid is extremely hazardous; it is corrosive, reactive, an oxidizer, and a poison. Inhalation of vapors can cause breathing difficulties and lead to pneumonia and pulmonary edema, which may be fatal. Other symptoms may include coughing, choking, and irritation of the nose, throat, and respiratory tract. Can cause redness, pain, and severe skin burns. Concentrated solutions cause deep ulcers and stain skin a yellow or yellow-brown color. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.

Hydrochloric Acid	Corrosive Poison	5 PPM-Ceiling	Inhalation of vapors can cause coughing, choking, inflammation of the nose, throat, and upper respiratory tract, and in severe cases, pulmonary edema, circulatory failure, and death. Can cause redness, pain, and severe skin burns. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
Potassium Permanganate	Oxidizer	5 mg/m^3 for Mn Compounds	Causes irritation to the respiratory tract. Symptoms may include coughing, shortness of breath. Dry crystals and concentrated solutions are caustic causing redness, pain, severe burns, brown stains in the contact area and possible hardening of outer skin layer. Diluted solutions are only mildly irritating to the skin. Eye contact with crystals (dusts) and concentrated solutions causes severe irritation, redness, and blurred vision and can cause severe damage, possibly permanent.
Stannous Chloride	Irritant	2 mg/m^3 TWA as Tin	Causes irritation to the respiratory tract. Can irritate skin and eyes. Symptoms include coughing and shortness of breath. Contact with skin and/or eyes may cause redness, itching and pain.
Potassium Persulfate	Oxidizer	None	Causes irritation to the respiratory tract. Symptoms may include coughing, shortness of breath. Causes irritation to skin and eyes. Symptoms include redness, itching, and pain. May cause dermatitis, burns, and moderate skin necrosis.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

5.5. Eye protection that protects against splash, laboratory coat, and appropriate gloves must be worn while samples, standards, solvents, and reagents are being handled. Cut resistant gloves must be worn doing any other task that presents a strong possibility of getting cut. Disposable gloves that have been contaminated will be removed and discarded; other gloves will be cleaned immediately.

5.6. Mercury is a highly toxic element that must be handled with care. The analyst must be aware of the handling and clean-up techniques before working with mercury. Since

mercury vapor is toxic, precaution must be taken to avoid its inhalation, ingestion or absorption through skin. All lines should be checked for leakage and the mercury vapor must be vented into a hood or passed through a mercury absorbing media.

5.6.1. Instrument carrier gas passes through a solid potassium permanganate trap before being released into the lab atmosphere.

5.7. Exposure to chemicals must be maintained as low as reasonably achievable. Therefore, unless they are known to be non-hazardous, all samples should be opened, transferred and prepared in a fume hood, or under other means of mechanical ventilation. Solvent and waste containers will be kept closed unless transfers are being made.

5.8. The preparation of standards and reagents will be conducted in a fume hood with the sash closed as far as the operation will permit.

5.9. All work must be stopped in the event of a known or potential compromise to the health and safety of a TestAmerica associate. The situation must be reported immediately to a laboratory supervisor or EH&S coordinator.

5.10. Cylinders of compressed gas must be handled with caution, in accordance with local regulations. It is recommended that, wherever possible, cylinders be located outside the laboratory and the gas led to the instrument through approved lines.

5.11. The CVAA apparatus must be properly vented to remove potentially harmful fumes generated during sample analysis. See 5.6.1 above.

6. EQUIPMENT AND SUPPLIES

The following items are recommended for performing this procedure. Equivalent items should be used only when they result in an improvement in quality, efficiency, productivity, or cost. An item can be considered equivalent if, with its use, the analytical and QA/QC requirements in this SOP can be met.

6.1. Temperature controlled hot blocks, capable of maintaining a temperature of 95 ± 3℃ for 2 hours

6.2. Environmental Express Autoblock automated sample digestion system with Autoblock Plus software

6.3. Automated Mercury Analysis System, Leeman Quick Trace M-7600 or equivalent

6.4. Disposable, sealable sample containers (Environmental Express)

6.5. Argon gas supply (ultrahigh purity-grade)

6.6. Calibrated automatic pipettes or Class A glass volumetric pipettes

6.7. Class A volumetric flasks

6.8. Thermometer (capable of accurate readings at 95°C)

6.9. Disposable cups or tubes, and disposable watch glasses

6.10. Filter paper, Whatman grade 41 or Fisher Scientific grade Q8

6.11. Class A graduated cylinders

6.12. Balance capable of weighing to the nearest 0.01 g.

7. REAGENTS AND STANDARDS

The following items are recommended for performing this procedure. Equivalent items should be used only when they result in an improvement in quality, efficiency, productivity, or cost. An item can be considered equivalent if with its use, the analytical and QA/QC requirements in this SOP can be met. Please refer to the SDS prior to the use of any reagent or standard.

Reagent grade, or higher, chemicals must be used unless otherwise indicated.

7.1. Reagent water must be produced by a Millipore DI system or equivalent. Reagent water must be free of the analytes of interest as demonstrated through the analysis of method blanks.

7.2. Stock (1000 ppm) mercury standards (in 2% HNO_3) are purchased as custom TestAmerica solutions. A standard must be purchased from two separate sources, one for calibration and spiking and another for calibration verification. All standards must be stored in FEP fluorocarbon or previously unused polyethylene or polypropylene bottles. Stock standard solutions must be replaced prior to the expiration date provided by the manufacturer. If no expiration date is provided, the stock solutions may be used for up to one year and must be replaced sooner if verification from an independent source indicates a problem.

7.3. Intermediate mercury standard (10 ppm): Take 1 mL of the stock mercury standard (7.2) and dilute to 100 mL with reagent water. The intermediate standard must be prepared daily and must be prepared in a matrix of 2% HNO_3. This acid (2 mL of concentrated HNO_3) must be added to the flask/bottle before the addition of the stock standard aliquot.

7.3.1. A Second Source Intermediate standard at 10 ppm is prepared in the same way, using the second source stock standard, for use in preparing the Initial Calibration Verification (ICV) standard.

7.4. Working mercury standard (0.1 ppm): Take 1 mL of the intermediate mercury standard (7.3) and dilute to 100 mL with reagent water. The working mercury standard must be made daily and must be prepared in a matrix of 0.15% HNO_3. This acid (150 uL of concentrated HNO_3) must be added to the flask/bottle before the addition of the stock standard aliquot. A second source working standard is prepared at 0.1 ppm for preparation of the ICV. Working standard must be prepared fresh daily.

7.5. The calibration standards listed in Table I must be prepared fresh daily from the working standard (7.4) by transferring 0, 0.2, 0.5, 1.0, 5.0 and 10.0 mL aliquots of the working mercury standard into 100 mL flasks and diluting to volume with reagent water. See Table 1 for the preparation of the ICV, CCV and RLV/CRA standards. Calibration and QC standards must be prepared fresh daily.

Note: Alternate approaches to standard preparation may be taken and alternate volumes of standard may be prepared as long as the accuracy and final standard concentrations as detailed in Table I are maintained. For example, automated mercury systems do not require 100 mL of standard and therefore smaller volumes may be generated to reduce waste generation.

7.6. The initial calibration verification standard (ICV) must be made from a different stock solution than that of the calibration standards and prepared daily.

7.7. Nitric acid (HNO_3), concentrated, trace metal grade or better.

Note: If a high reagent blank is obtained, it may be necessary to distill the nitric acid.

7.8. Sulfuric acid (H_2SO_4), concentrated, trace metal grade or better.

7.8.1. Sulfuric acid, 0.5 N: Dilute 14.0 mL of concentrated H_2SO_4 to 1 liter with reagent water.

7.9. Hydrochloric acid (HCl), concentrated, trace metal grade or better.

7.10. Aqua regia: Prepare by carefully adding three parts concentrated HCl to one part concentrated HNO_3. Must be prepared fresh every 48 hours or sooner in aliquots of 500 to 1000 mls.

7.11. Stannous chloride solution: Add 200 g of stannous chloride to 2 L of 10% hydrochloric acid. Expiration date for the stannous chloride solution is 3 months from the date of preparation.

7.12. Sodium chloride, NaCl

7.13. Hydroxylamine hydrochloride, $HONH_2 \bullet HCl$,

7.13.1. Sodium chloride-hydroxylamine hydrochloride - Clearing Solution: Add 12 g of sodium chloride and 12 g of hydroxylamine hydrochloride to every 100 mL of reagent water. Expiration date for the sodium chloride-hydroxylamine solution is 3 months from the date of preparation.

7.14. Potassium permanganate, $KMnO_4$

7.14.1. Potassium permanganate, 5% solution (w/v): Dissolve 5 g of potassium permanganate for every 100 mL of reagent water. Expiration date for the potassium permanganate 5% solution is 3 months from the date of preparation.

7.15. Potassium persulfate, $K_2S_2O_8$

7.15.1. Potassium persulfate, 5% solution (w/v): Dissolve 5 g of potassium persulfate for every 100 mL of reagent water. Solution should be gently heated to dissolve crystals. Expiration date for the potassium persulfate 5% solution is 3 months from the date of preparation.

8. SAMPLE COLLECTION, PRESERVATION, SHIPMENT AND STORAGE

8.1. Listed below are the holding times and preservation requirements:

Matrix	Sample Container	Min. Sample Size	Preservation	Holding Time
Water	Plastic or glass	250 mL	pH <2 with HNO3	28 days from sampling to analysis
TCLP Leachates	Plastic	250 mL	pH <2 with HNO3	28 days from start of TCLP Leach
Soils/ Sediments	4oz Jar	5 grams	Cool to >0 but ≤ 6.0ºC	28 Days from collection to analysis
Wipes	2oz Jar	1 wipe	Cool to >0 but ≤ 6.0ºC	28 Days from collection to analysis
Tissues	4oz Jar	5 grams	Frozen	Freeze up to 1 year, then 28 Days from thaw date to analysis

8.2. For dissolved mercury analysis, the samples should be filtered through a 0.45 µm filter prior to preservation. Filtration should be done in the field within 15 minutes of collection. If the laboratory filters the samples, this process will occur as soon as possible after receipt. A filtered blank must be processed with laboratory filtered samples. The "Filtration – Field Filtered" NCM shall be completed if the laboratory filters the samples.

NOTE: If a sample being analyzed for dissolved mercury is found to contain sediment the analyst should contact their supervisor or group leader. The client should be notified of the problem to decide how to treat the sample.

8.3. Dissolved mercury samples that are filtered directly into a nitric acid preserved bottle may be digested immediately.

9. QUALITY CONTROL

9.1. The following quality control samples are prepared with each batch of samples.

QC PARAMETER	FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTION
ICV	Beginning of every analytical run	%Recovery: 7470A/7471A/B: 90 - 110% 245.1: 95 - 105%	Re-inject once, if failure repeats, terminate analysis; correct the problem; recalibrate, re-prepare batch
ICB	Beginning of every analytical run, immediately following the ICV	The result must be within +/- RL from zero	Re-inject once, if failure repeats, terminate analysis; correct the problem; recalibrate, re-prepare batch
RLV/CRA	Beginning of every analytical run, immediately following the ICB	70 – 130% recovery for 7470A 50-150% for 245.1 & 7471A/B	Re-inject once, if failure repeats, terminate analysis; correct the problem; recalibrate, re-prepare batch Note exception under section 9.7

QC PARAMETER	FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTION
CCV	Every 10 samples and at the end of the run	90 - 110 % recovery	Re-inject once, if failure repeats, terminate analysis; Correct the problem; Recalibrate or rerun all samples not bracketed by acceptable CCV or re-prepare batch Note exceptions under section 9.6.
CCB	Immediately following each CCV	The result must be within +/- RL from zero	Re-inject once, if failure repeats, terminate analysis; Correct the problem; Recalibrate or rerun all samples not bracketed by acceptable CCB or re-prepare batch Note exceptions under section 9.6.
Method Blank	One per sample preparation batch of up to 20 samples	The result must be $\leq$ the RL for method 7470A & 7471A/B or $\leq$ 2.2 x MDL for 245.1. Sample results greater than 10x the blank concentration are acceptable.	Re-inject once, if failure repeats, re-prepare and reanalyze affected samples. Note exceptions under section 9.2.
Laboratory Control Sample (LCS)	One per sample preparation batch of up to 20 samples.	%Recovery Limits: 7470A/7471A/B: 80 - 120 % 245.1: 85 – 115%	Re-inject once, if failure repeats, re-prepare and reanalyze all samples associated with the LCS. Note exceptions under section 9.3.

QC PARAMETER	FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTION
Matrix Spike	One per sample preparation batch of up to 20 samples for 7470A and 7471A/B and one per 10 samples for 245.1	%Recovery Limits: 7470A 75 – 125% 7471A/B 80 – 120% 245.1 70 – 130%	In the absence of client specific requirements, flag the data. Note exceptions under section 9.4.
Matrix Spike Duplicate	See Matrix Spike	% Recovery as for MS above; RPD ≤ 20%.	See Corrective Action for Matrix Spike.

9.2. Method Blank (MB) - The method blank is carried through the entire analytical procedure, including preparation and analysis. The method blank is used to identify any system and process interferences or contamination of the analytical system that may lead to the reporting of elevated analyte concentrations or false positive data. The method blank is prepared using a known clean matrix, laboratory reagent water along with aqueous samples or Teflon boiling chips along with solid samples. The method blank should not contain any mercury at or above the reporting limit for methods 7470A and 7471A/B, or above 2.2 x the MDL for method 245.1. If the MB result is above the required method limit, re-inject it once. If failure repeats, follow corrective actions below.

- If the method blank is contaminated and associated sample concentrations are > 10x the MB concentration, results may be reported and using the “Method Blank – Report, 10X” NCM.
- If mercury is not detected above the RL in the samples associated with an unacceptable method blank, the data may be reported with qualifiers and using the “Method Blank – Report, ND” NCM.
- If neither of the above situations applies, and reanalysis is not possible due to sample volume or holding time, the client must be notified. Results must be properly qualified in the report and the nonconformance described in the case narrative.

NOTE: See Appendix C for information on contamination control.

9.3. Laboratory Control Sample (LCS) - The LCS (referred to as a Laboratory Fortified Blank in 245.1) is a blank that is spiked with a known concentration of mercury and carried through the same preparation and analysis procedures as the samples. The LCS is used to monitor the accuracy of the analytical process and is prepared in the same clean matrix as the Method Blank. On-going monitoring of the LCS results provides evidence that the laboratory is performing the method within acceptable accuracy and precision guidelines. CCV results can be reported as LCS results since all CCVs (as well as all other standards) are processed through the sample preparation step with the field samples. No more than 20 samples can be associated with one CCV used for the purpose of reporting LCS data.

- If the LCS is outside established control limits given in the table above, re-inject the LCS once. If failure repeats, follow corrective actions below.
- In the instance where the LCS recovery is > 120% (7470A/7471A/B) or > 115% (245.1), but below 140%, and sample results are < RL, reanalysis is not required and the data may be reported with qualifiers and using the “LCS/CLSD - %R High” NCM.
- Corrective action will be re-preparation and reanalysis of the batch unless the client agrees that other corrective action is acceptable. If reanalysis is not possible due to sample volume or holding time, the client must be notified. Results must be properly qualified in the report and the nonconformance described in the case narrative.

9.4. Matrix Spike/Matrix Spike Duplicate (MS/MSD) - A matrix spike (MS) is a field sample to which known concentrations of target analytes have been added (referred to as a Laboratory Fortified Matrix in 245.1). A matrix spike duplicate (MSD) is a second aliquot of the same sample (spiked identically as the MS) prepared and analyzed along with the sample and matrix spike. Some client specific data quality objectives (DQO’s) may require the use of sample duplicates in place of or in addition to MS/MSD’s. The MS/MSD results are used to determine the effect of a matrix on the precision and accuracy of the analytical process. Due to the potential variability of the matrix of each sample, these results may have immediate bearing only on the specific sample spiked. Samples identified as field blanks cannot be used for MS/MSD analysis. Spiking levels are provided in Table I.

- If analyte recovery or RPD falls outside the acceptance ranges given in the table above, the recovery of that analyte must be in control for the LCS. If the LCS recovery is within limits, then the laboratory operation is in control and the results may be accepted. If the recovery of the LCS is outside limits, corrective action must be taken. Corrective action will include re-preparation and reanalysis of the batch. MS/MSD results which fall outside the control limits must be addressed in the narrative and qualified.

- If the parent sample concentration is >4x the spiked concentration, the spike cannot be accurately recovered. The results may be reported using the "MS/MSD – High Targets" NCM.

- If there is insufficient sample volume to perform and MS and MSD with the batch, an LCSD should be performed and the situation documented with the "MS/MSD/DUP – Insufficient Volume" NCM.

9.5. Initial Calibration Verification (ICV/ICB) - Calibration accuracy is verified by analyzing a second source standard (ICV). An ICB is analyzed immediately following the ICV to monitor low level accuracy and system cleanliness. (The ICV is equivalent to the Quality Control Sample (QCS) and the first Initial Performance Check (IPC) specified in method 245.1.) See the Table above for control limits.

- If either the ICV or ICB fail to meet criteria, it should be re-injected once. If failure repeats, the analysis should be terminated, the problem corrected and the instrument recalibrated. If the cause of the ICV or ICB failure was not directly instrument related the corrective action will include re-preparation of the associated samples.

9.6. Continuing Calibration Verification (CCV/CCB) - Calibration accuracy is monitored throughout the analytical run through the analysis of a known standard and blank after every 10 samples and at the end of the analytical sequence. The CCV must be a mid-range standard at a concentration different than that of the ICV. Each CCV and CCB analyzed must reflect the conditions of analysis of all associated samples. Sample results may only be reported when bracketed by valid ICV/CCV and ICB/CCB pairs. If a mid-run CCV or CCB fails, it should be re-injected once. If failure repeats, the analysis must be terminated, the problem corrected, the instrument recalibrated, the calibration verified and the affected samples reanalyzed. If the cause of the CCV or CCB failure was not directly instrument related the corrective action will include re-preparation of the associated samples.

- If a bracketing CCB is contaminated, but the associated samples are non-detect, results may be reported with qualification and NCM similar to section 9.2 for MB.

- If a bracketing CCV has recovery above 110%, but the associated samples are non-detect, results may be reported with qualification and NCM similar to section 9.3 for LCS.

9.7. A Reporting Limit Verification Standard (RLV) or Contract Required Quantitation Standard (CRA) is used to evaluate the calibration accuracy at the laboratory reporting limit. A standard at the reporting limit concentration is injected immediately after the analysis of the ICB. This standard may also be analyzed at the end of the analytical sequence. Recovery must be within 70-130% for method 7470A, or 50-150% for methods 245.1 and 7471A/B. If the RLV/CRA recovery is outside control limits, re-inject the standard once. If failure

repeats, follow corrective actions below. Recalibrate the instrument, and re-digest the batch if recalibration does not correct the failure.

- If recovery is low, all samples must be reanalyzed.
- If recovery is high, samples with non-detect results may be reported with the "Low-Level Std – Outside Criteria" NCM. All samples that have results above the RL must be re-analyzed.

9.8. Method of Standard Addition (MSA) -This technique involves adding known amounts of standard to one or more aliquots of the sample prior to preparation. This technique compensates for a sample interferent that may enhance or depress the analyte signal, thus producing a different slope from that of the calibration standards. It will not correct for additive interferences which cause a baseline shift. Refer to Section 10.5.10 for additional information on when full 4 point MSA is required as well as Appendix A for specific MSA requirements.

10. PROCEDURE

10.1. Calibration and Standardization

10.1.1. Calibration standards must be processed through the preparation procedure as described in Section 10.2, 10.3, or 10.4 along with the samples.

10.1.2. Due to the differences in preparation protocols separate calibration and calibration verification standards must be prepared for aqueous and solid matrices.

10.1.3. Calibration must be performed daily (every 24 hours). The instrument calibration date and time must be included in the raw data.

10.1.4. Set up the instrument with the operating parameters recommended by the manufacturer. Allow the instrument to become thermally stable before beginning calibration (approximately 30 minutes of warm-up is required). Refer to CVAA instrument manual for detailed setup and operation protocols.

10.1.5. Calibrate the instrument according to instrument manufacturer's instructions, using a minimum of five standards and a blank. One standard must be at or below the reporting limit. Analyze standards in ascending order beginning with the blank. Refer to Section 7.5 and Table I for additional information on preparing calibration standards and calibration levels.

10.1.6. The calibration curve is plotted by the instrument software as absorbance vs. concentration using linear regression.

- The curve must have a correlation coefficient of ≥0.995 or the instrument shall be stopped and recalibrated prior to running samples. Sample results cannot be reported from a curve with an unacceptable correlation coefficient.

- Calculate the concentration of each calibration standard from the final calibration curve. Calculate the %Error for each point. All points except the lowest must have a %Error of ≤20%. The lowest standard (RL level) must have a %Error ≤ 50%.

  - %Error is the absolute value of the difference between the calculated concentration and the true concentration divided by the true concentration x100%.

    For example, if the 0.20mg/L standard reads back as 0.17mg/L:

    [(0.20 – 0.17)/0.20] x 100% = 15%

10.1.7 Refer to Section 9.5 and 9.6 for calibration verification procedures, acceptance criteria and corrective actions. The NELAC requirement for verification of the initial calibration at varied concentrations is met daily since the ICVs, CCVs, and RLV/CRA are all at different concentrations.

10.2. Aqueous Sample Preparation - Manual

10.2.1. All calibration and calibration verification standards (ICV, ICB, CCV, CCB, RLV/CRA) are processed through the digestion procedure as well as the field samples.

10.2.2. Prepare calibration standards as in section 7.5 above and Table 1, and transfer into a series of sample digestion containers.

10.2.3. Transfer 50 mL of well-mixed sample or standard to a clean sample digestion bottle. Refer to PT-QA-024 for subsampling procedures.

Note: Reduced sample volumes can be used as long as a representative sample can be obtained and the reagent levels are adjusted to maintain the same sample to reagent ratio. All samples and standards must be processed in the same manner.

10.2.4. To the LCS container, add 1.25 mL of the 0.1 ppm working standard (7.4) to 50 mL of reagent water. To the MS and MSD containers, add 0.50 mL of the 0.1 ppm working standard to 50 mL of parent sample.

10.2.5. Add 2.5 mL of concentrated H_2SO_4 and 1.25 mL of concentrated HNO_3 mixing after each addition.

10.2.6. Add 7.5 mL of potassium permanganate solution. For samples high in organic materials or chlorides, this amount of permanganate may not be sufficient. Check samples after 15 minutes. If the purple color has disappeared, dilute the sample and begin the digestion again.

10.2.7. Add 4 mL of potassium persulfate solution for a 50 mL sample.

10.2.8. Confirm that the hot block is within the proper temperature range by checking the liquid-in-glass thermometer in the block. Place the sample rack in the block and heat the samples for two hours in a hot block at 95 ± 3℃. Record both the observed and corrected temperature reading in the batch information in TALS,

10.2.9. Cool samples for approximately 15 minutes.

10.2.10. Add 3 mL of sodium chloride-hydroxylamine hydrochloride “clearing solution” to the samples to reduce the excess permanganate (the permanganate has been reduced when no purple color remains). Add this solution in 3 mL increments until the permanganate is completely reduced (i.e. solution is colorless).

10.2.11. Once the samples are clear, adjust the final digestate volume to 50 ml with reagent water.

10.2.12. **NOTE:** All known difficult water matrices (ie. Safety Kleen Total and TCLP waters) are prepared at a 10 fold dilution, 5 mls of sample diluted to a final volume of 50 mLs with reagent water. A difficult matrix is one that contains a large amount of debris, sediment or has an extremely high pH (caustic in nature).

10.3. Aqueous Sample Preparation - Autoblock

10.3.1. Power on the Autoblock and open Autoblock Plus software. The software will not open unless the instrument is powered on.

- A Warning message about going to home position will appear – click OK

10.3.2. Expired service items list will appear (waste drum) – click OK. After addressing the necessary service items (empty waste drum), Click **Service Interface** → go to **Maintenance Screen** → and click “press” next to the service item(s) to be updated.

- A pop-up will appear asking if you want to update the service date, click OK.

10.3.3. Exit the Maintenance Screen and go to **Manual Interface**.

- A Warning message about going to home position will appear – click OK

10.3.3.1. All reagent tubes need to be primed. Check that all reagents are full enough to complete the batch. Click on the desired reagent, then click "move to drain". Move the rack position to "fill". Change the injection amount to "30.0mL" and press "inject". Repeat for all reagents.

10.3.4. Exit the Manual Interface and go to **Operator Test**. Click on "select method" and choose *245.1 and 7470A PIT.mtd*.

10.3.4.1. Type in the "batch number" and "operator name". (Initials may be used.)

10.3.4.2. Select all cell locations where samples will be placed.

- All samples and QC have an initial volume of 25mL

10.3.5. All calibration and calibration verification standards (ICV, ICB, CCV, CCB, RLV/CRA) are processed through the digestion procedure as well as the field samples. Prepare calibration standards as in section 7.5 above and Table 1.

10.3.6. Transfer 25 mL of well-mixed sample, standard, or QC to a clean sample digestion tube. Refer to PT-QA-024 for subsampling procedures.

Note: Reduced sample volumes can be used as long as a representative sample can be obtained and the reagent levels are adjusted to maintain the same sample to reagent ratio. All samples and standards must be processed similarly.

10.3.7. To the LCS container, add 0.625 mL of the 0.1 ppm working standard (7.4) to 25 mL of reagent water. To the MS and MSD containers, add 0.25 mL of the 0.1 ppm working standard to 25 mL of parent sample.

10.3.8. Make sure that the following have green rectangles next to them:

- Pump area leak
- Drain area leak
- Cabinet pressurized – the compressor should stay on always
- Blower overpressure – the fan will turn on automatically

10.3.9. Hit **Go**. The Autoblock will complete the following steps:

- Inject 0.625 mL of nitric acid in each sample
- Inject 1.25 mL of sulfuric acid in each sample
- Inject 3.75 mL of potassium permanganate in each sample
  NOTE: Check for the persistence of purple color at a practical point in the digestion before the cooling. If the color has disappeared, re-digest the samples at a dilution.
- Inject 2.0 mL of potassium persulfate in each sample
- Sample racks will rise to highest position while autoblock heats up to 108℃
- Once temperature is reached, sample racks will lower into the hotblock and cook for 2 hours.
  NOTE: Block is set at 108℃ to maintain un-capped sample temperatures are 85±3℃.
  NOTE: Temperature is maintained at 85 ±3°C rather than 95±3°C because the digestion vessels cannot be covered on the Autoblock.
- After digestion, sample racks will be brought to highest position to cool for 20 minutes.
- 1.5 mL of clearing solution will be injected after the samples are cooled.
- Analyst is responsible for bringing samples up to a final volume of 25mL.

10.3.10. After sample digestion is complete, clean the reagent tubes with laboratory reagent water using the same procedure as for priming in section 10.3.3.1.

NOTE: Use Clearing Solution first when cleaning the potassium permanganate line, followed by water rinse.

10.3.11. Turn off the Autoblock and “abort” the Autoblock Plus software.

10.4. Solid Sample Preparation

10.4.1. Prepare calibration standards as in section 7.5 above and Table 1, and transfer into a series of sample digestion containers.

10.4.2. Transfer 0.6 g of a well-mixed sample into a clean sample digestion container. Refer to PT-QA-024 for proper subsampling procedures.

10.4.2.1. For wipes, add the entire contents of the sample jar into a clean sample digestion container. For sediment samples, 1.2 g of well-mixed sample is used. TCLP leachates from waste samples are digested by this procedure using 0.6 mL initial volume.

10.4.3. To the LCS container, add 2.5 mL, and to the MS, and MSD containers, add 1.0 mL of 0.1 ppm working standard (7.4).

10.4.4. Add 5 mL of reagent water and 5 mL of Aqua Regia reagent (7.10).

10.4.5. Confirm that the hot block is within the proper temperature range by checking the liquid-in-glass thermometer in the block. Place the sample rack in the block and heat the samples for 2 minutes at 95 ± 3℃.

10.4.6. Remove the rack from the block and allow the samples to cool slightly. Add 50 mL of reagent water and 15 mL of potassium permanganate solution (7.14.1) to each sample. Let stand at least 15 minutes.

10.4.7. Check the samples for the persistence of the purple color. Add additional portions of permanganate solution, if needed, until the purple color persists for at least 15 minutes.

NOTE: If additional permanganate solution is added to samples, an equal amount must also be added to standards and blanks.

10.4.8. Add 8 mL of potassium persulfate solution (7.15.1) and mix thoroughly.

10.4.9. Return racks to the hot blocks and heat for 30 minutes after samples reach 95 ± 3℃.

10.4.10. After 30 minutes, remove the racks and allow samples to cool.

10.4.11. Add 6 mL of hydroxylamine hydrochloride solution (7.13.1) to reduce excess permanganate.

10.4.12. Bring standards, QC and samples up to a volume of 100 mL with reagent water.

10.4.13. Alternate final volumes may be used as long as the standards and samples are treated the same way and reagent volumes are adjusted proportionally.

10.5. Sample Analysis:

Note: The samples must be allowed to cool to room temperature prior to analysis or a decrease in the response signal can occur.

Note: If digestates are not free from particulates after cooling and settling, and require filtration prior to being introduced on the instrument, the associated MB and LCS must also be filtered.

10.5.1. Quick Trace Mercury Analyzer M-7600 Instrument set up:

10.5.1.1. Go to the **Reporting Options** tab and deselect **replicates** (Hit “X” and then “yes” to save.)

10.5.1.2. Go to **Sequence Parameters**, select **smart rinse**, change the µAbs to 75000 and select **abort on over-range**, then save.

10.5.1.3 Go to **Method Editor**, click on the **standards** tab, select **Coeff. of Determination (r^2) limit**, and under **Calibration Parameters**, select **pause analysis after first calibration pending confirmation to continue**. Save.

10.5.1.4 Place the rinse tube in 10% HCl rinse solution and the Stannous Chloride line in DI water.

10.5.1.5 Go to the **Instrument Control/Maintenance Log**

- Click on the **Autosampler** tab and turn on the pump.
- Click on the **Analyzer** tab and turn on the pump.

NOTE: Set speed to 50% to reduce unnecessary wear on peristaltic tubing.

10.5.1.6 Clamp down the Stannous Chloride line on the analyzer’s pump and watch for a steady flow of DI water into the analyzer (no air bubbles).

10.5.1.7 Go to the **Instrument Control/ Maintenance Log**, click the **Autosampler** tab. Under **move sample probe** select **down**.

10.5.1.8 Clamp sample line (probe line) down on the pump and check for a steady flow (no air bubbles).
Note: the post in the gas-liquid separator (GLS) should start to moisten.
10.5.1.9 Select **wet the post** after about a half inch of water accumulates in the GLS.
10.5.1.10 Once the post is lubricated, clamp the two rinse tubes down on the analyzer pump, and release both the stannous line and sample line to drain the water.
10.5.1.11 Re-clamp all four tubes after the water has drained sufficiently.
10.5.1.12 Place the Stannous Chloride line into the Stannous Chloride solution.
10.5.1.13 Attach the gas line to the GLS and let the instrument run for a few minutes.
10.5.1.14 Go to the **Instrument Control/ Maintenance Log**, click the **Analyzer** tab, select **zero instrument**, then click the graph button to observe the graph of the instrument readings.
10.5.1.15 Allow the instrument to warm up for at least 30 minutes.

10.5.2. Creating a Worksheet:
10.5.2.1 Open Leeman QuickTrace Mercury Analysis System on desktop.
10.5.2.2 Create a new file:
- Open a worksheet, select **new form**, click the search button, select **TestAmerica Template**, and name the run.
- If **TestAmerica Template** does not automatically open go to OS (C:) → users → public → public documents → Teledyne CETAC → Quicktrace → templates → TestAmerica.
- Name the run as follows: HGZ81012A.
  HGZ = instrument name
  8 = year
  10 = month
  12 = day
  A = first run that day (B = 2nd, C = 3rd, etc)

10.5.2.3 Scan the ICV, ICB, CRA, CCV, and CCB from the curve batch sheet into the worksheet and use the drop down under **sample type** to select the correlating QC option.
- CRA = CRDL Standard under sample type

NOTE: The six point curve is included in the template, however, the six points will need to be scanned from the curve batch sheet after you upload your analytical run.
10.5.2.4 Scan samples into worksheet
- Sample type is left **unknown** except for MB, LCS, and any prep blanks
  - Use the drop down tab under **sample type** and select the corresponding option.

10.5.2.5 Copy and paste the CCV & CCB every 10 samples.
- Make sure the correct rack placement is also copied.
- The closing CCV & CCB should also include the closing CRA.

10.5.2.6 Save worksheet before hitting **Go** to begin the analysis.

10.5.3. Saving and Exporting Analytical Run

10.5.3.1 Go to **print preview**. Click the print icon, select **print to file**, select the search box next to **where,** go to the **RawDataPDF** folder, name file the same as analytical run, and save.

NOTE: hit "close" not "X", "X" will close the program.

10.5.3.2 Go to "File" → "Export" → the "HGZ" folder → Name file the same as the analytical run → save.

- Once file is exported, "export complete" will appear.

10.5.4. Post Analytical Run

10.5.4.1 Disconnect the gas line from the GLS.

10.5.4.2 Place Stannous Chloride line into 10% HNO3 solution and allow the instrument to rinse for a few minutes.

10.5.4.3 Place Stannous Chloride line into DI water and allow instrument to rinse for a few minutes.

10.5.4.4 Go to the **Instrument Control/Maintenance Log**, click on the **Autosampler** tab, and under **move sample probe**, select **up.**

10.5.4.5 Place rinse line and Stannous Chloride line into empty beaker.

10.5.4.6 Once the post in the GLS is dry (white in color), turn off both pumps, unclamp the tubes, loosen the tension, and close the program.

10.5.5 The instrument software performs a linear regression analysis of the calibration standards by plotting maximum response of the standards vs. ug of mercury. Mercury concentration in the samples and QC are calculated by comparing response to the calibration curve plot. A linear correlation coefficient of $\geq$0.995 is required for an acceptable calibration curve. The instrument will not proceed with analysis if the curve does not meet this criterion.

10.5.6 All measurements must fall within the defined calibration range to be valid. When sample concentrations exceed the upper limit of the calibration curve, the samples will be diluted and reanalyzed to bring them within the calibration range. When reported sample concentrations either exceed the upper limit of the curve, and the sample cannot be rerun, an NCM will be written and the result will be qualified in the report.

10.5.7 If the sample results are negative and the absolute value of the negative result is greater than the reporting limit, the sample must be diluted and reanalyzed.

10.5.8 Baseline correction is acceptable as long as it is performed after every sample or after the CCV and CCB; resloping is acceptable as long as it is immediately preceded and followed by a compliant CCV and CCB.

10.5.9 The following example run sequence is recommended:

Instrument Calibration
ICV
ICB
RLV or CRA
CCV
CCB
10 samples
CCV
CCB
Repeat sequence of 10 samples between CCV/CCB pairs as required to complete run.
CCV
CCB

10.5.10 For TCLP samples, full four point MSA is required if all of the following conditions are met:

1) recovery of the analyte in the matrix spike is < 50%,
2) the concentration of the analyte does not exceed the regulatory level, and,
3) the concentration of the analyte is within 20% of the regulatory level.

- An alternative to performing MSA is to re-digest and reanalyze the affected sample. If the above criteria are not repeated in the reanalysis, then MSA is not required and reanalysis results shall be reported.
- Appendix A provides guidance on performing MSA analyses. For TCLP mercury determinations, MSA spikes must be added prior to sample preparation.

10.6 To facilitate the early identification of QC failures and samples requiring rerun it is strongly recommended that sample data be reviewed periodically throughout the run.

10.7 Guidelines are provided in the appendices on procedures to minimize contamination of samples and standards, preventive maintenance and parts maintenance. For instrument troubleshooting, use the auto diagnostics software. If the problem cannot be determined using the software, place a call to service personnel.

10.8 One time procedural variations are allowed only if deemed necessary in the professional judgment of supervision to accommodate variation in sample matrix, radioactivity, chemistry, sample size, or other parameters. Any variation in procedure shall be completely documented using a Nonconformance Memo and is approved by a Technical Specialist and

QA Manager. If contractually required, the client shall be notified. The Nonconformance Memo shall be filed in the project file.

10.9 Any unauthorized deviations from this procedure must also be documented as a nonconformance, with a cause and corrective action described.

11 DATA ANALYSIS AND CALCULATIONS

11.1 ICV percent recoveries are calculated according to the equation:

$$\%R = 100\left(\frac{Found(ICV)}{True(ICV)}\right)$$

11.2 CCV percent recoveries are calculated according to the equation:

$$\%R = 100\left(\frac{Found(CCV)}{True(CCV)}\right)$$

11.3 RLV percent recoveries are calculated using the same equation as the ICV or CCV (replace ICV or CCV with RLV in the above equations).

11.4 Matrix spike recoveries are calculated according to the following equation:

$$\%\ R\ = 100\left(\frac{SSR - SR}{SA}\right)$$

Where:

SSR = Spike Sample Result

SR = Sample Result

SA = Spike Added

11.5 The relative percent difference (RPD) of matrix spike/matrix spike duplicates or sample duplicates are calculated according to the following equations:

$$RPD = 100\left[\frac{|MSD - MS|}{\left(\frac{MSD + MS}{2}\right)}\right]$$

Where:

MS = determined spiked sample concentration

MSD = determined matrix spike duplicate concentration

$$RPD = 100\left[\frac{|DU1 - DU2|}{\left(\frac{DU1 + DU2}{2}\right)}\right]$$

Where:

DU1 = Sample result

DU2 = Sample duplicate result

11.6 The final concentration for an aqueous sample is calculated as follows:

mg/L = C x D

Where:

C = Concentration (mg/L) from instrument readout

D = Instrument dilution factor

11.7 For automated determinations, the final concentration determined in solid samples when reported on a dry weight basis is calculated as follows:

mg/kg, dry weight = (C x V x D)/(W x S)

Where:

C = Concentration (ug/L) from instrument readout
V = Volume of digestate (L)
D = Instrument dilution factor
W = Weight in g of wet sample digested
S = Percent solids/100

Note: A Percent Solids determination must be performed on a separate aliquot when dry weight concentrations are to be reported. If the results are to be reported on a wet weight basis, the “S” factor should be omitted from the above equation.

11.8 For automated determinations, the final concentration determined in wipe samples is calculated as follows:

ug/wipe = (C x V x D)

Where:

C = Concentration (ug/L) from instrument readout
V = Volume of digestate (L)
D = Instrument dilution factor

11.8 The LCS percent recovery is calculated according to the following equation:

$$\%R = 100\left(\frac{Found(LCS)}{True(LCS)}\right)$$

11.9 Appropriate factors must be applied to sample values if dilutions are performed.

11.10 Sample results should be reported with up to three significant figures in accordance with the TestAmerica significant figure policy.

12 METHOD PERFORMANCE

12.1 The supervisor has responsibility to ensure that an analyst who performs this procedure is properly trained in its use and has the required experience. Performance is monitored through internal QC and outside performance evaluation samples. Please refer to the QA Manual for additional information concerning Precision and Accuracy.

12.2 Demonstration of Capabilities – Prior to the analysis of samples, a Demonstration of Capabilities (DOC) as described in the QA Manual, must be performed initially, annually and any time a significant change is made to the analytical system.

12.3 Method Detection Limit Study – A Method Detection Limit (MDL) study, as described in Detection Limit SOP, PT-QA-007, must be performed initially and whenever a significant change is made to the analytical system. The MDL must be re-evaluated from quarterly MDL points at least every 12 months.

12.4 Linear Dynamic Range Verification - The linear range is the concentration above which results cannot be reported without dilution of the sample. The standards used to define the linear range limit must be analyzed under routine analytical conditions. LDRs are instrument specific and must be determined on each instrument annually, or whenever an instrument is adjusted in any way that would affect the range.

12.4.1 For the determination/verification of the upper limit of the linear dynamic range (LDR), determine the signal responses from a minimum of three different concentration standards across the estimated upper range. At least one standard must be above the current highest calibration standard concentration. The determined concentration of the linear range verification standards must be within 10% of the true value. The upper limit of the LDR is set at 90% of the concentration of the highest standard that meets the ±10% recovery limit.

13 POLLUTION PREVENTION

13.1 It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage and reagent stability). Employees must abide by the policies in Section 13 of the Corporate Environmental Health and Safety Manual (CW-E-M-001) for "Waste Management and Pollution Prevention" and the Pittsburgh Facility Addendum EH&S Manual (PT-HS-001).

13.2 This method allows for the proportional reduction of sample and reagent volumes to decrease waste generation.

14 WASTE MANAGEMENT

14.1 Waste management practices are conducted consistent with all applicable rules and regulations. Excess reagents, samples and method process wastes are disposed of in an accepted manner. Waste description rules and land disposal restrictions are followed. Waste disposal procedures are incorporated by reference to PT-HS-001. The following waste streams are produced when this method is carried out.

14.1.1 Extracted sample containing less than 1 ppb Hg. This waste is collected in waste containers identified as "Acid Waste", Waste #33. It is neutralized to a pH between 6 and 9 and is disposed down a lab sink.

14.1.2 Unused Standards. This waste collected in containers identified as "Mercury Standards Waste", Waste #4.

14.1.3 Extracted sample containing greater than 1 ppb Hg. This waste collected in containers identified as "Mercury Standards Waste", Waste #4.

14.1.4 Mercury Analyzer Waste. Waste discharged from mercury analyzer is collected in containers identified as "Mercury Standards Waste", Waste #4.

15 REFERENCES

15.1 Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, 3rd Edition, Final Update II, Revision I, September 1994, Method 7470A (Mercury)

15.2 Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, 3rd Edition, Final Update II, Revision I, September 1994, Method 7471A (Mercury)

15.3 Mercury In Solid Or Semisolid Waste (Manual Cold-Vapor Technique), Test Methods for Evaluating Solid Waste , Physical/Chemical Methods, SW-846, 3rd Edition, Final Update IV, Revision 2, February 2007, Method 7471B

15.4 Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846 Method 7000B, Flame Atomic Absorption Spectrophotometry, Revision 2, February, 2007

15.5 “Methods for the Chemical Analysis of Water and Wastes”, EPA-600/4-79-020, U.S.EPA, 1994, Method 245.1, Revision 3.0

15.6 PT-QA-006, Procurement of Standards and Materials; Labeling and Traceability

15.7 PT-QA-007, Detection Limits

15.8 PT-QA-009, Rounding and Significant Figures

15.9 PT-QA-016, Nonconformance & Corrective Action System

15.10 PT-QA-018, Technical Data Review Requirements

15.11 PT-QA-021, Quality Assurance Program

15.12 PT-QA-022, Equipment Maintenance

15.13 PT-QA-024, Subsampling

15.14 PT-QA-M-001- Pittsburgh Laboratory Quality Assurance Manual

15.15 PT-QA-031, Internal Chain-of-Custody Procedure

15.16 SOP PT-HS-001, Pittsburgh Facility Addendum Environmental Health and Safety Manual

16 METHOD MODIFICATIONS

16.1 Modifications from 7470A and 245.1

16.1.1 The 200 series methods and Chapter 1 of SW846 specify the use of reagent water with a purity equivalent to ASTM Type II water. This SOP specifies the use of a Millipore DI system or equivalent to produce reagent water. This SOP requires that reagent water must be free of the analytes of interest as demonstrated through the analysis of method blanks.

16.1.2 This SOP allows for the use of reduced sample volumes to decrease waste generation. Reagent levels are adjusted to maintain the same ratios as stated in the source methods. According to a letter from Robert Booth of EPA EMSL-Cincinnati to David Payne of EPA Region V, "Reduction in sample size and appropriate corresponding reduction in sample volume is not considered a significant change in the methodology."

16.1.3 The alternate run sequence presented in Section 10.3.7 is consistent with method requirements.

16.2 Modifications from Methods 7470A/7471B

16.2.1 Chapter 1 of SW-846 states that the method blank should not contain any analyte of interest at or above the MDL. This SOP states that the method blank must not contain any analyte of interest at or above the reporting limit.

16.2.2 Chapter 1 of SW846 specifies the use of reagent water with a purity equivalent to ASTM Type II water. This SOP specifies the use of a Millipore DI system or equivalent to produce reagent water. This SOP requires that reagent water must be free of the analytes of interest as demonstrated through the analysis of method blanks.

16.3 Modifications from 245.1

16.3.1 Method 245.1, Section 9.3 states concentrations should be reported as follows: Between 1 and 10 ug/L, one decimal; above 10 ug/L, to the nearest whole number. TestAmerica reports all Hg results under this SOP to two significant figures.

16.4 Modifications from SW846 7471A/B

16.4.1 A potassium persulfate oxidation step has been included to facilitate the breakdown of organic mercurials which are not completely oxidized by potassium permanganate. Use of potassium persulfate in combination with the permanganate improves the recovery of mercury from organo-mercury compounds. The use of persulfate has been incorporated in several recent EPA mercury protocols.

16.5 Autoblock digestion is performed with proportional reduction of samples, standards, and all reagent volumes.

17 ATTACHMENTS

17.1 Figure 1 – Aqueous Sample Preparation
17.2 Figure 2 – Solid Sample Preparation

17.3 Figure 3 – CVAA Instrumental Analysis Flowchart
17.4 Table 1 – Mercury Standard and Spike Preparation
17.5 Appendix A - MSA Guidance
17.6 Appendix B – Parts and Maintenance Guide
17.7 Appendix C - Contamination Control Guidelines
17.8 Appendix D - Preventative Maintenance Schedule
17.9 Appendix E – Metals Dilution Calculation Table

18 REVISION HISTORY

18.1 Revision 8, 9/2/2008
18.2 Revision 9, 10/8/2009
18.3 Revision 10, 6/23/2010
18.4 Revision 11, 9/7/2010
18.5 Revision 12, 12/6/2010
18.6 Revision 13, 1/31/2012
18.7 Revision 14, 4/24/2013
18.8 Revision 15, 4/28/2015
18.9 Revision 16, 7/6/2017
18.10 Revision 17, 11/27/2018
18.11 Revision 18, 5/13/2019
18.12 Revision 19, 10/8/2019
18.13 Revision 19.1, 12/13/2019
18.11 Changes to current revision:

SOP section	Change from	Change to	Reason
5.6.1	0.2M KMnO4 and 10% H2SO4	Carrier gas passes through solid KMnO4 trap	Correction for current set-up
5.6.2	deleted		Option not used
5.11		Added note See 5.6.1	Clarification
6.9		Added disposable watch glasses	Correction
6.10		Added filter paper	Correction
6.12		Added Balance	Correction
7.2	10% HNO3	2% HNO3	Correction
7.15.1		Added gently heat solution to dissolve	Clarification
9.7		Added that CRA may also be analyzed at the end of the	Clarification

		sequence	
10.1.1		Added 10.4	Correction
10.1.6		Added calibration is linear regression and added Read-back Error requirement	Clarification/Correction
10.2.6 & Figure 1	Removed additional KMnO4	Added if purple does not persist for 15 minutes, dilute and re-digest	Correction for practice
10.3.9	1.5 2	1.25	Correction
10.3.9 & figure 2		Added check for purple, dilute and re-digest	Correction for practice
10.3.9		Added note under temperature setting that maintained at 85 because uncovered	Clarification
10.5		Added note from _A1 for filtration	Clarification
12.3 and 15.7	CA-Q-S-006	PT-QA-007	Correction

Figure 1 - Aqueous Sample Preparation

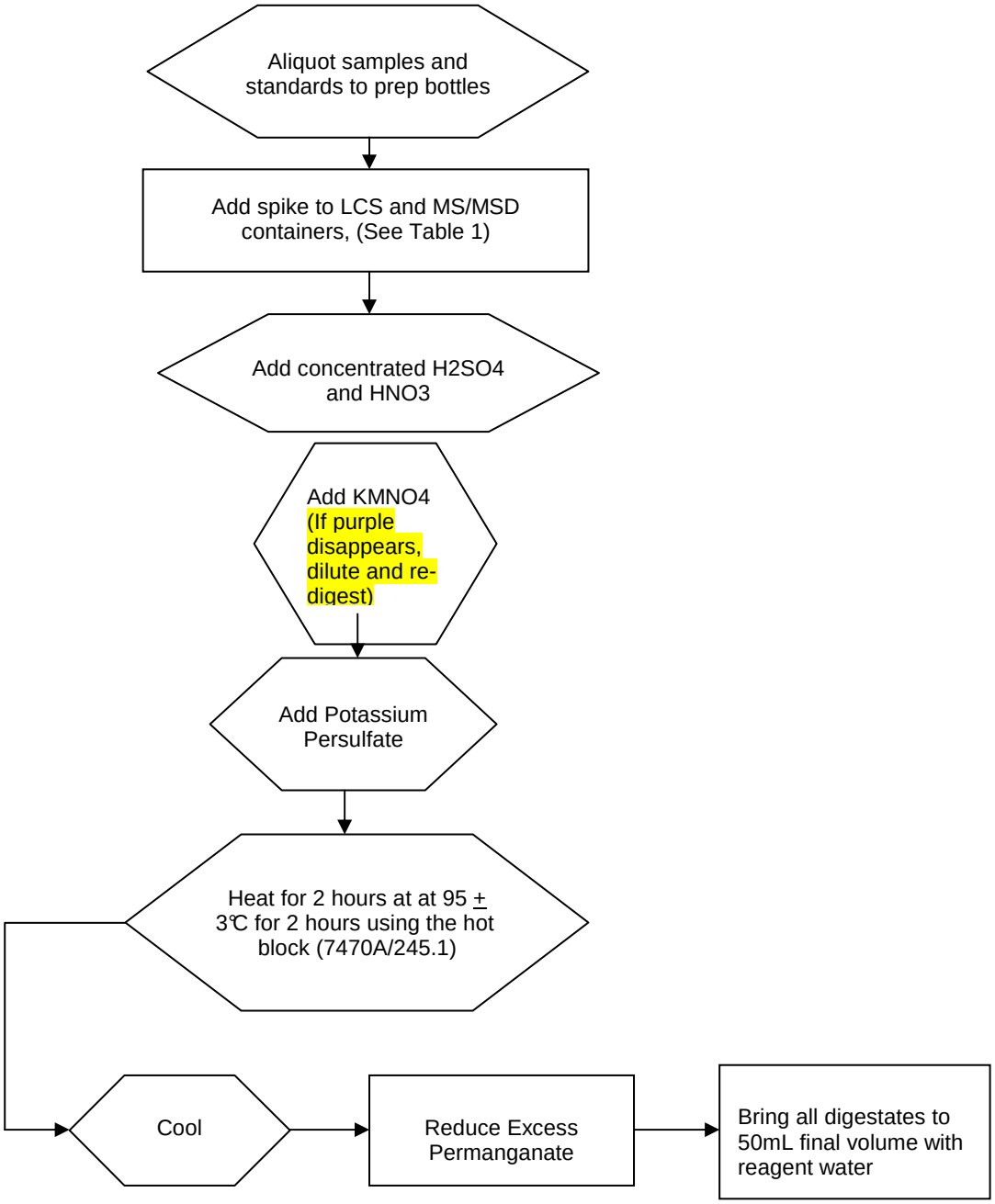


Figure 2 - Solid Sample Preparation

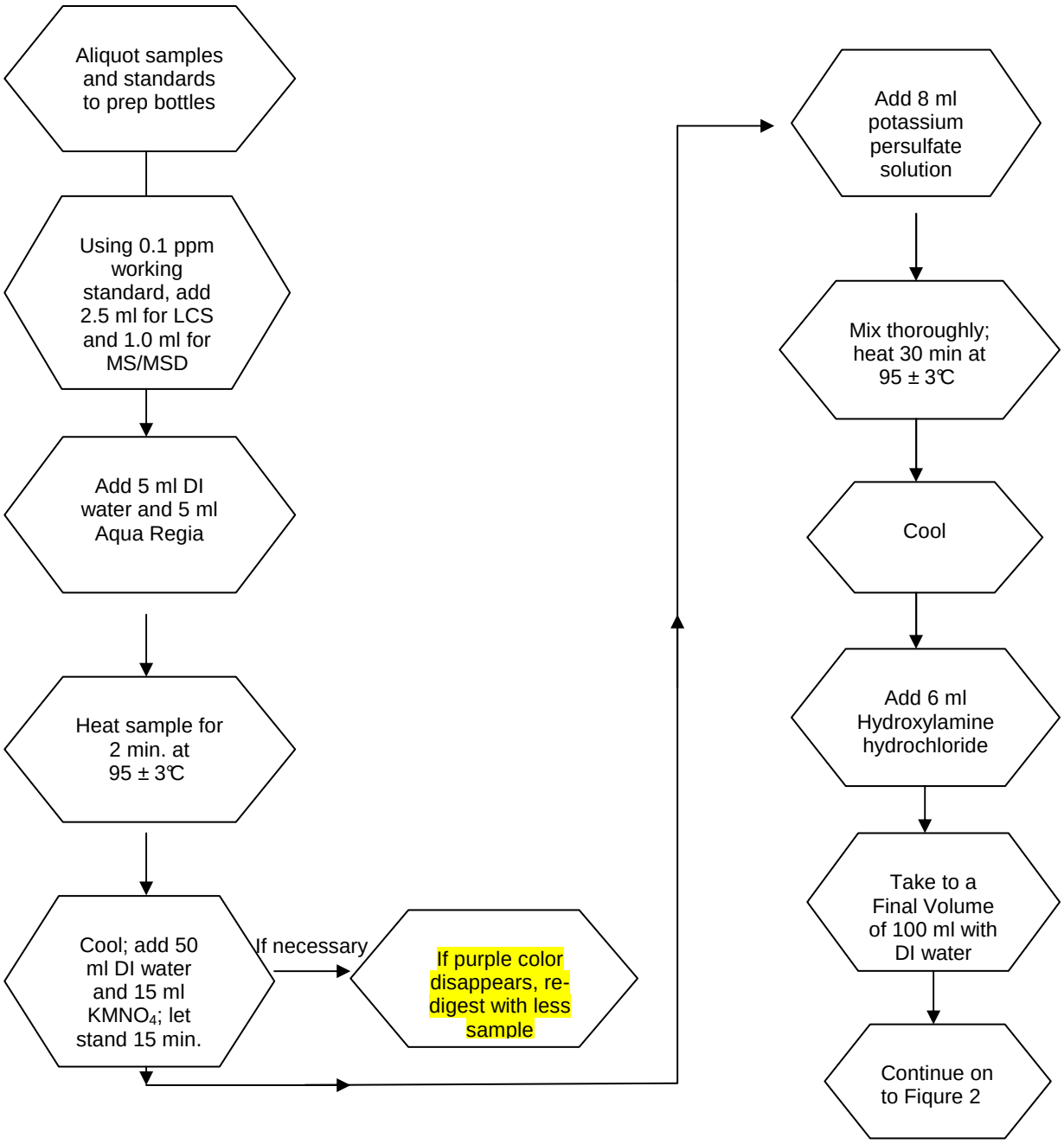


Figure 3 – CVAA Instrumental Analysis Flowchart

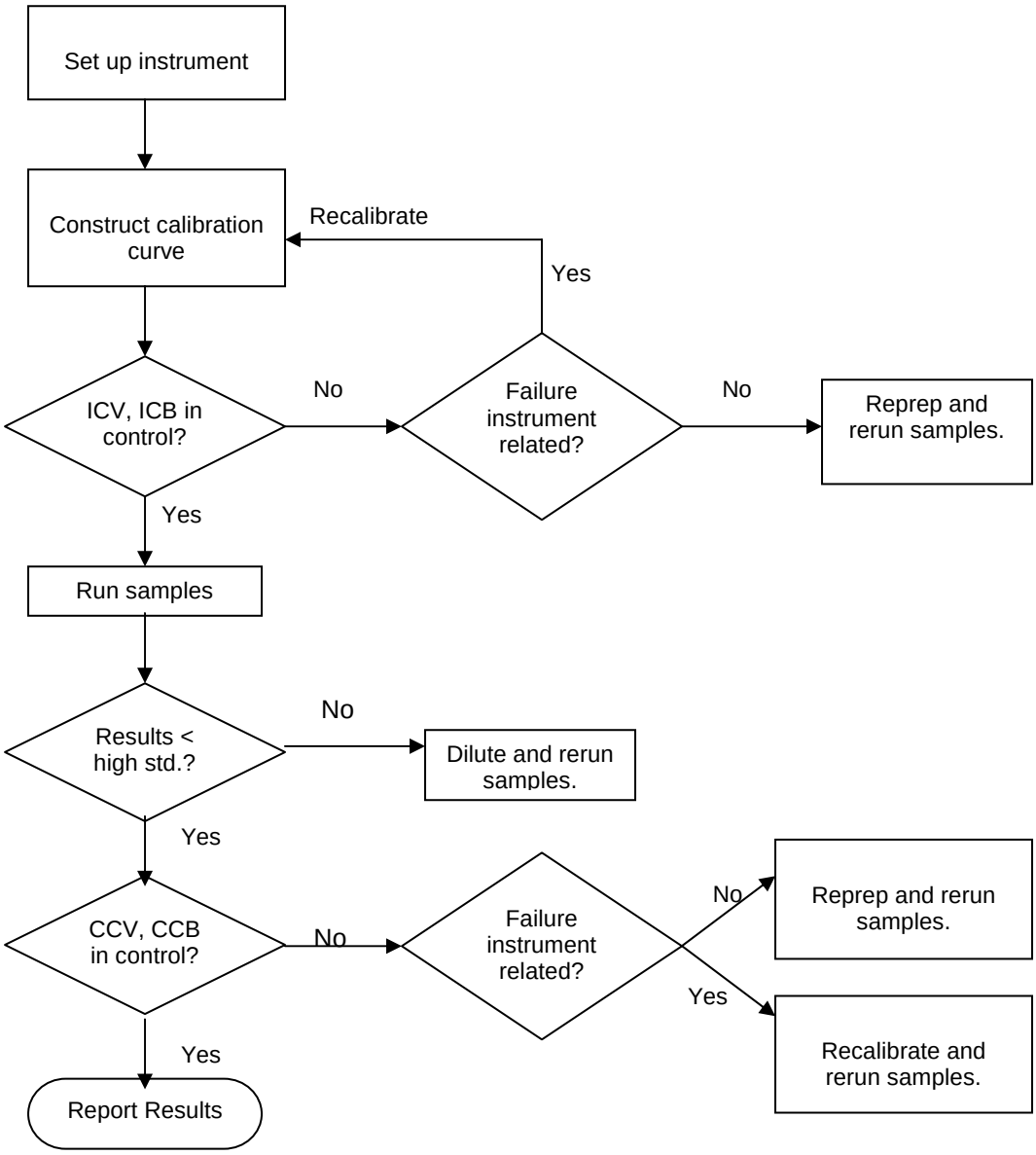


TABLE 1

CALIBRATION STANDARD*, QC STANDARD AND SPIKING LEVELS (MG/L)

Standard or QC	mL of 0.10 ppm Working Standard for manual digestion (50mL)	Final Volume (mL)	Concentration (mg/L)
Std 0	0	100	0
Std 1	0.20	100	0.0002
Std 2	0.50	100	0.0005
Std 3	1.0	100	0.001
Std 4	5.0	100	0.005
Std 5	10	100	0.010
ICV	2.5**	100	0.0025
CCV	5.0	100	0.005
RLV/CRA	0.20	100	0.0002
LCS (manual)	1.25	50	0.0025
LCS (Autoblock)	0.625	25	0.0025
MS (manual)	0.50	50	0.001
MS (Autoblock)	0.25	25	0.001
TCLP MS (manual)	0.25	50	0.005
TCLP MS (Autoblock)	0.125	25	0.005

SOP specified calibration levels must be used unless prevented by the instrument configuration or client specific requirements.

** Prepared from a second source 0.1 ppm working standard.

NOTE: TCLP initial sample volume is 5mL for manual digestion and 2.5mL for Autoblock digestion

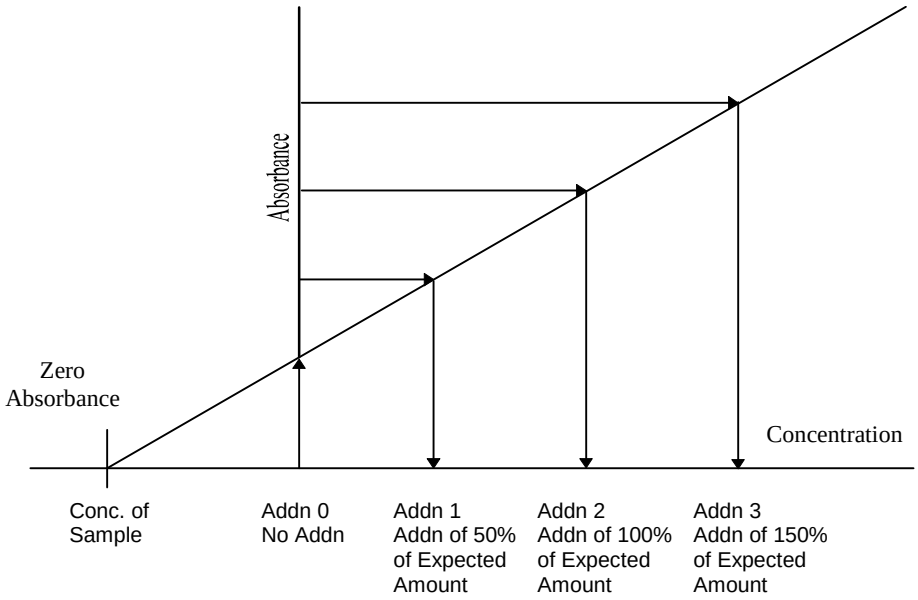
APPENDIX A - MSA GUIDANCE

Method of Standard Addition

Four equal volume aliquots of sample are measured and known amounts of standards are added to three aliquots. The fourth aliquot is the unknown and no standard is added to it. The concentration of standard added to the first aliquot should be 50% of the expected concentration. The concentration of standard added to the second aliquot should be 100% of the expected concentration and the concentration of standard added to the third aliquot should be 150% of the expected concentration. The volume of the unspiked and spiked aliquots should be the same (i.e., the volume of the spike added should be negligible in relation to the volume of sample).

To determine the concentration of analyte in the sample, the absorbance (or response) of each solution is determined and a linear regression performed. On the vertical axis the absorbance (or response) is plotted versus the concentrations of the standards on the horizontal axis using 0 as the concentration of the unspiked aliquot. An example plot is shown in Figure 1. When the resulting line is extrapolated back to zero absorbance, the point of interception of the horizontal axis is the concentration of the unknown. Calculate the correlation coefficient (r) and the x-intercept (where y=0) of the curve. The concentration in the digestate is equal to the negative x-intercept.

Figure 1



- For the method of standard additions to be correctly applied, the following limitations must be taken into consideration.
- The plot of the sample and standards must be linear over the concentration range of concern. For best results, the slope of the curve should be similar to that of a plot of the aqueous standard curve.
- The effect of the interference should not vary as the ratio of the standard added to the sample matrix changes.

APPENDIX B - MAINTENANCE GUIDE

Cold Vapor Atomic Absorption Mercury Instrument Maintenance Schedule

Daily	As Needed	Annually
Check aperture reading.	Check Hg lamp intensity.	Change Hg lamp every 50,000 hours of operation
Check argon flow/pressure.	Clean optical cell	Check liquid/gas separator.
Check tubing and replace every 50 hours of operation.	Lubricate pump	
Check drain.	Clean lens windows with methanol.	
	Replace drying tube every 300 hours of operation	
	Change permanganate trap every 1000 hours of operation	

APPENDIX C - CONTAMINATION CONTROL GUIDELINES

The following procedures are strongly recommended to prevent contamination:

All work areas used to prepare standards and spikes should be cleaned before and after each use.

All glassware should be washed with detergent and tap water and rinsed with 20% nitric acid followed by deionized water.

Proper laboratory housekeeping is essential in the reduction of contamination in the metals laboratory. All work areas must be kept scrupulously clean.

Powdered or Latex Gloves must not be used in the metals laboratory since the powder contains silica and zinc, as well as other metallic analytes. Only vinyl or nitrile gloves should be used in the metals laboratory.

Glassware should be periodically checked for cracks and etches and discarded if found. Etched glassware can cause cross contamination of any metallic analytes.

Autosampler trays should be covered to reduce the possibility of contamination. Trace levels of elements being analyzed in the samples can be easily contaminated by dust particles in the laboratory.

The following are helpful hints in the identification of the source of contaminants:

Reagents or standards can contain contaminants or be contaminated with the improper use of a pipette.

Improper cleaning of glassware can cause contamination.

Separate glassware if an unusually high sample is analyzed and soak with sulfuric acid prior to routine cleaning.

APPENDIX D - PREVENTIVE MAINTENANCE

A maintenance log is used to record when maintenance is performed on instruments. When an instrument problem occurs indicate the date, time and instrument number, then identify the problem and corrective action in the maintenance log.

The following procedures are required to ensure that that the instrument is fully operational.

Cold Vapor Atomic Absorption (Hydra AA)

Daily	As Needed	Annually
Check Hg lamp intensity.	Clean lens windows with methanol.	Change Hg lamp.
Check aperture reading.	Clean optical cell	Check liquid/gas separator.
Check pump tubing/drain tubing.	Lubricate pump	
Check argon flow/pressure.		
Check tubing and replace, if needed.		
Check drain.		
Replace drying tube.		

APPENDIX E

TESTAMERICA PITTSBURGH METALS DILUTION CALCULATION TABLE

Dilution Required	Dilution Formula
2	5 mL sample : 5 mL Matrix Blank
5	2 mL sample : 8 mL Matrix Blank
10	1 mL sample : 9 mL Matrix Blank
25	0.4 mL sample : 9.6 mL Matrix Blank
50	0.2 mL sample : 9.8 mL Matrix Blank
100	0.1 mL sample : 9.9 mL Matrix Blank

2340B Hardness by Calculation, using Ca and Mg from ICP or ICPMS analysis

1. Total Hardness, mg/L as $CaCO_3$ = 2.497[Ca, mg/L] + 4.118 [Mg, mg/L]

   Note: Calcium Hardness may be calculated using only the Ca portion of the above equation

Title: Extraction of Organic Compounds from Waters

Method(s): SW846 3500 Series and EPA 600 Series Methods

Approvals (Signature/Date):

6/25/2020	6/30/2020
Sharon Bacha Date	Steve Jackson Date
Organics Department Manager	Regional Safety Coordinator
6/25/2020	10/28/2020
Virginia Zusman Date	Deborah L. Lowe Date
Quality Assurance Manager	Laboratory Director

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1.0 SCOPE AND APPLICATION

1.1 This SOP describes extraction procedures for the preparation of semivolatile organic analytes in aqueous and TCLP leachate matrices for analysis by Gas Chromatography (GC) and Gas Chromatography / Mass Spectrometry (GC/MS). The procedures are based on SW-846 and 600 series methodology and are applicable for measurements made to comply with the Resource Conservation and Recovery Act (RCRA) and for wastewater testing.

1.2 Extraction procedures 608/608.3, 625/625.1, 3510C, and 3520C for the following determinative methods are covered: 608, 608.3, 625, 625.1, 8081A, 8081B, 8082, 8082A 8141A, 8141B, 8270C, 8270D, and 8270E.

NOTE: Some, but not all, methods are available for the Low Volume Initiative (LVI) extraction with proportionally reduced volumes to reduce waste.

1.3 The extraction procedures discussed here may be appropriate for other determinative methods when appropriate spiking mixtures are used.

1.4 On occasion clients may request slight modifications to this SOP. Any modifications to this procedure must be approved by the QA department or the Laboratory or Technical Director and documented fully in an NCM that accompanies the analytical batch.

1.5 Different levels of concentration and volumes are available to meet client specific needs. Regular level analyses are normally performed on 1 liter initial sample volumes. Low-level analyses are concentrated to a smaller final volume and offer reporting limits that are 10-40 times lower than the regular level analysis. Low Volume Initiative (LVI) extractions are performed on 100 to 250mL initial sample volumes with proportional reduction of reagents, spikes and final volumes. See Table 2 for initial and final volume details. LVI extraction procedures are performed with initial volumes of 250 mL.

NOTE: LVI extractions are not to be used for samples from South Carolina.

2.0 SUMMARY OF METHOD

2.1 Separatory Funnel Extraction

A measured volume of sample, typically 1 liter (250 mL for LVI), is adjusted, if necessary, to a specified pH and serially extracted with methylene chloride using a separatory funnel.

2.2 Continuous Liquid/Liquid Extraction (CLLE)

A measured volume of sample, typically 1 liter (250 mL for LVI), is placed into a continuous liquid/liquid extractor, adjusted, if necessary, to a specific pH and extracted with methylene chloride for 18-24 hours.

2.3 Concentration

Extracts are concentrated to specific final volumes by evaporation of the solvent under controlled conditions.

3.0 DEFINITIONS

3.1 Definitions of terms used in this SOP may be found in the glossary of the Pittsburgh Quality Assurance Manual (PT-QA-M-001).

3.2 TALS – TestAmerica Laboratory Information Management System

3.3 NCM – Non-Conformance Memo – a system within TALS for the lab to communicate to project management and others when there is an anomaly seen with the samples or batch, or a QC failure.

4.0 INTERFERENCES

4.1 Method interferences may be caused by contaminants in solvents, reagents, glassware, and other processing apparatus. All these materials must be routinely demonstrated to be free from interferences under conditions of the analysis by running laboratory method blanks as described in the Quality Control section. Specific selection of reagents may be required to avoid introduction of contaminants.

4.2 Visual interferences or anomalies (such as foaming, emulsions, odor, etc.) must be documented.

4.3 Residual Chlorine from water treatment must be removed from samples prior to extraction for 625 analysis and for 608 analysis when Aldrin is included in the compound list.

5.0 SAFETY

5.1 Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual (CW-E-M-001), the Pittsburgh Facility Addendum EH&S Manual (PT-HS-001), and this document. This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

5.2 Samples containing or suspected to contain cyanide or sulfide concentrations at or greater than 250 ppm or 500 ppm, respectively, shall be processed in a fume hood.

5.3 The use of separatory funnels to extract aqueous samples with Methylene Chloride creates excessive pressure very rapidly. Initial venting should be done immediately after the sample container has been sealed and inverted. Vent the funnel into the hood away from people and other samples. This is considered a high-risk activity, and a face shield must be worn over safety glasses or goggles when it is performed.

5.4 Nitrile gloves should be used when performing this extraction. Latex and vinyl gloves provide no significant protection against the organic solvents used in this SOP, and should not be used.

5.5 During Kuderna-Danish (KD) concentration, do not allow the extract to boil to dryness. The solvent vapors remaining in the KD apparatus may superheat and create an explosion or fire hazard. The KD apparatus and glass separatory funnels have ground glass joints, which can become stuck. Technicians must use Kevlar or other cut/puncture resistant gloves when separating stuck joints.

5.6 Care must be used when separating glass parts of extraction and concentration setups. Protective gloves must be used when separating stuck glass joints.

5.7 The following is a list of the materials used in this method, which have a serious or significant hazard rating. NOTE: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the SDS for each of the materials listed in the table. A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the SDS for each material before using it for the first time or when there are major changes to the SDS.

Material	Hazards	Exposure Limit[i]	Signs and symptoms of exposure
Acetone	Flammable	1000 ppm-TWA	Inhalation of vapors irritates the respiratory tract. May cause coughing, dizziness, dullness, and headache.
Hexane	Flammable Irritant	500 ppm-TWA	Inhalation of vapors irritates the respiratory tract. Overexposure may cause lightheadedness, nausea, headache, and blurred vision. Vapors may cause irritation to the skin and eyes.
Methylene Chloride	Carcinogen Irritant	25 ppm-TWA 125 ppm-STEL	Causes irritation to respiratory tract. Has a strong narcotic effect with symptoms of mental confusion, light-headedness, fatigue, nausea, vomiting and headache. Causes irritation, redness and pain to the skin and eyes. Prolonged contact can cause burns. Liquid degreases the skin. May be absorbed through skin.
Sodium Hydroxide	Corrosive Poison	2 ppm, 5 mg/m^3	This material will cause burns if comes into contact with the skin or eyes. Inhalation of Sodium Hydroxide dust will cause irritation of the nasal and respiratory system.
Sulfuric Acid[ii]	Corrosive Oxidizer Dehydrator	1 mg/m^3	This material will cause burns if comes into contact with the skin or eyes. Inhalation of vapors will cause irritation of the nasal and respiratory system.

[i] Exposure limit refers to the OSHA regulatory exposure limit.
[ii] Always add acid to water to prevent violent reactions.

5.8 Eye protection that protects against splash, laboratory coat and appropriate gloves must be worn while samples, standards, solvents and reagents are being handled. Cut resistant gloves must be worn doing any other task that presents a strong possibility of getting cut. Disposable gloves that have become contaminated will be removed and discarded; other gloves will be cleaned immediately.

5.9 The preparation of standards and reagents and glassware cleaning procedures that involve solvents such as methylene chloride will be conducted in a fume hood with the sash closed as far as the operations will permit. Use of methylene chloride for glassware cleaning should be avoided as far as possible.

5.10 All work must be stopped in the event of a known or potential compromise to the health and safety of a TestAmerica associate. The situation must be reported immediately to a laboratory supervisor or EH&S coordinator.

6.0 EQUIPMENT AND SUPPLIES

The following items are recommended for performing this procedure. Equivalent items should only be used when they result in an improvement in quality, efficiency, productivity, or cost. An item can be considered equivalent if with its use, the analytical and QA/QC requirements in this SOP can be met.

6.1 Glassware should be cleaned with soap and water, rinsed with water and dried in an oven at 400°C for at least 2 hours. Alternatively the glassware can be solvent rinsed with acetone or methanol followed by methylene chloride after the water rinse.

6.2 Equipment and supplies for extraction procedures

EQUIPMENT AND SUPPLIES	Separatory funnel	CLLE	Conc
Separatory Funnel: 2 L, 500 mL	X		
Separatory Funnel Rack	X		
Balance: >1400 g capacity, accurate ±1 g	X	X	
pH indicator paper, wide-range: covers extraction pH	X	X	
Graduated cylinder: 1 liter. (other sizes may be used)	X	X	
Erlenmeyer Flask or Fleaker: 125 & 300 mL (other sizes optional)	X		
Solvent Dispenser Pump or 100 mL Graduated Cylinder	X		
Continuous Liquid/Liquid Extractor		X	
Round or flat Bottom: 250, 500 mL or 1 L		X	
Boiling Chips: Contaminant free, approximately 10/40 mesh (Teflon® PTFE, carbide or equivalent).		X	X
Cooling Condensers		X	
Heating Mantle: Rheostat controlled		X	
Auto-timer for heating mantle		X	
Kuderna-Danish (K-D) Apparatus: 500 mL, 250 mL			X
Concentrator Tube: 10 mL, attached to K-D with clips			X
Snyder Column: Three-ball macro			X

EQUIPMENT AND SUPPLIES	Separatory funnel	CLLE	Conc
Water Bath: Heated, with concentric ring cover, capable of temperature control (± 5°C) up to 95°C. The bath must be used in a hood or with a solvent recovery system.			X
Vials: Glass, 2 mL, 4 mL, and 10 mL capacity with Teflon®-lined screw-cap			X
Nitrogen Blowdown Apparatus			X
Nitrogen: reagent grade.			X
Culture tubes: 10 mL, 16 mmx100 mm			X
Syringe: 1 mL	X	X	
Phase Separation Paper	X	X	
Glass Wool	X	X	
Glass Funnel: 75 X 75 mm	X	X	X
Disposable Pipettes	X	X	X
Potassium Iodide-Starch Indicator Paper	X	X	
Aluminum foil	X	X	X
Paper Towels	X	X	X

7.0 REAGENTS AND STANDARDS

The following items are recommended for performing this procedure. Equivalent items should only be used when they result in an improvement in quality, efficiency, productivity, or cost. An item can be considered equivalent if with its use, the analytical and QA/QC requirements in this SOP can be met. Please refer to the MSDS prior to the use of any reagent or standard.

7.1 Reagents for Extraction Procedures

All reagents must be ACS reagent grade or better unless otherwise specified.

REAGENTS	Separatory funnel	CLLE	Conc.
Sodium hydroxide (NaOH), Pellets: Reagent Grade	X	X	
Sodium hydroxide solution, 10 N: Dissolve 40 g of NaOH in reagent water and dilute to 100 mL.	X	X	
Sulfuric acid (H_2SO_4), Concentrated: Reagent Grade	X	X	
Sulfuric acid (1:1): Carefully add 500 mL of H_2SO_4 to 500 mL of reagent water. Mix well.	X	X	
Organic free reagent water.	X	X	
Sodium sulfate (Na_2SO_4), Granular, Anhydrous: Purify by heating at 400°C a minimum of four hours.	X	X	
Extraction/Exchange Solvents: Methylene chloride, hexane, acetone, pesticide quality or equivalent	X	X	X
Acetone: Used for cleaning	X	X	X
Sodium Thiosulfate, granular	X	X	

7.2 Standards

7.2.1 Stock Standards

Stock standards are purchased as certified solutions or prepared from neat. Semivolatile stock standards are stored at > 0.0ºC but ≤ 6.0ºC. All

stock standards must be protected from light. Stock standard solutions must be replaced after one year (from the time of preparation, if prepared in house, or from the time the ampule is opened if purchased.) Standards must be allowed to come to room temperature before use.

7.2.2 Surrogate Spiking Standards

Prepare or purchase surrogate spiking standards at the concentrations listed in Work Instruction PT-OP-WI-001. Surrogate spiking standards are prepared as dilutions of the stock standards in methanol or other water miscible solvent. Surrogate spiking solutions must be refrigerated and protected from light. The standards must be replaced at least every six months or sooner if there is reason to believe that the standard has degraded or concentrated.

7.2.3 Matrix Spiking and Laboratory Control Spiking Standards

The same spiking solution is used for the matrix spike and the Laboratory Control Sample. Prepare MS/LCS spiking standards at the concentrations listed in Work Instruction PT-OP-WI-001. Spiking standards are purchased or prepared as dilutions of the stock standards in methanol or other water miscible solvent. Spiking solutions must be refrigerated and protected from light. The standards must be replaced at least every six months or sooner if there is reason to believe that the standard has degraded or concentrated.

8.0 SAMPLE COLLECTION PRESERVATION, SHIPMENT AND STORAGE

Matrix	Sample Container	Min. Sample Size	Preservation	Leachate Extraction Holding Time	Extraction Holding Time
Waters	1 liter amber	1 Liter	Cool, > 0.0ºC but ≤ 6.0ºC	NA	7 Days
LVI Waters	250 mL amber	250 mL	Cool, > 0.0ºC but ≤ 6.0ºC	NA	7 Days
Waters for PCB Analysis (608 or 8082)	1 liter amber	1 Liter	Cool, > 0.0ºC but ≤ 6.0ºC	NA	1 year
Leachates	4oz Jar	100 grams	Cool, > 0.0ºC but ≤ 6.0ºC	14 Days	7 Days

9.0 QUALITY CONTROL

9.1 Extraction batches are composed of twenty or fewer samples, of similar matrix, plus the required batch QC. Laboratory generated QC samples (method blanks, LCS, MS/MSD) are not included in the sample count.

9.2 If insufficient sample is available to process a MS/MSD, then an LCS Duplicate may be processed to evaluate batch extraction precision. Use of a LCS/LCSD

pair in place of a MS/MSD due to insufficient sample volume must be documented using the "Insufficient Volume" NCM. Because subsamples cannot be taken from a wipe sample for MS/MSD analyses, wipe samples should be processed with a LCS/LCSD.

9.3 The extraction of at least a Matrix Spike is required for all methods. If double sample volume is not provided for any samples in the batch, document this with the "Insufficient Volume" NCM.

9.4 The following quality control samples are prepared with each batch of samples. For QC control limits, refer to the individual analytical method SOPs and TALS.

Quality Controls	Frequency
Method Blank (MB)	1 per preparation batch[1]
Laboratory Control Sample (LCS)	1 per preparation batch[1]
Matrix Spike (MS)[2,3]	1 per preparation batch[1]
Matrix Spike Duplicate (MSD)[2,3]	1 per preparation batch[1]
Surrogates	All samples and QC

[1]A preparation batch is limited to 20 samples, except for 608 and a batch is limited 10 samples.
[2]The sample used for MS/MSD is randomly selected, unless specifically requested by a client.
[3]An MS should be performed without an MSD if there is insufficient volume for both.

9.5 Method Blank

A method blank with all reagents added the same as to the samples must be prepared and analyzed with each batch of samples. Surrogates are spiked into the method blank at the same level as the samples. The method blank is used to identify any background interference or contamination of the analytical system, which may lead to the reporting of elevated concentration levels or false positive data.

9.5.1 Aqueous Method Blanks use 1000 mL (250 mL for LVI batches) of reagent water spiked with the surrogates. The method blank goes through the entire analytical procedure, including any cleanup steps.

9.5.2 TCLP Leachate Blanks (LB) use 200 mL of leachate fluid for GC/MS Semivolatiles and 100 mL for organochlorine pesticides. The leachate blank may optionally be diluted to 1000 mL with reagent water. Once the organic prep group receives the TCLP samples and leachate blank they will extract all using the appropriate method and QC, including an extracted method blank. The method blank and leachate blank go through the entire analytical procedure, including any cleanup steps.

9.6 Laboratory Control Sample (LCS)

Laboratory Control Samples are clean matrix, known concentration, laboratory-generated samples used to monitor the laboratory's day-to-day performance of routine analytical methods. The LCS, spiked with the list of target compounds

representative of the method analytes, is used to monitor the accuracy of the analytical process, independent of matrix effects. On-going monitoring of the LCS results provides evidence that the laboratory is performing the method within accepted QC guidelines for accuracy and precision. The LCS goes through the entire analytical procedure, including any cleanup steps.

9.6.1 The LCS is made up in the same way as the method blank (See sections 9.5.1 - 9.5.2) but spiked with the LCS standard as well as the surrogates.

9.6.2 For the 600 series methods (608 and 625), the LCS is equivalent to the QC Check Sample specified in the reference methods.

9.7 Surrogates

9.7.1 Surrogates are organic compounds which are similar to the target analyte(s) in chemical composition and behavior in the analytical process, but which are not normally found in environmental samples.

9.7.2 Each sample, blank, LCS and MS/MSD is spiked with surrogate standards prior to the extraction process. Surrogate spike recoveries must be evaluated by determining whether the concentration (measured as percent recovery) falls within the required recovery limits.

9.8 Matrix Spike/Matrix Spike Duplicate (MS/MSD)

9.8.1 A matrix spike is an environmental sample to which known concentrations of target analytes have been added. A matrix spike duplicate is a second spiked aliquot of the same sample, which is spiked prior to the beginning of the extraction process and prepared and analyzed along with the sample and matrix spike. Matrix spike analysis is required for every 20 samples, except for method 608, which is every 10 samples.

9.8.2 If sufficient volume is not received for both and MS and MSD, at least an MS should be extracted along with the batch to meet method QC requirements.

10.0 PROCEDURE

Procedures for separatory funnel liquid/liquid extraction (10.1), continuous liquid/liquid extraction (10.2), and extract concentration (10.3) are presented in this section.

10.1 Separatory Funnel Liquid/Liquid Extraction of Water Samples:

Refer to Figure 1 – Separatory Funnel Extraction flowchart.

10.1.1 Remove surrogate and matrix spiking solutions from refrigerator and allow them to warm to room temperature.

10.1.2 Mark the meniscus of the sample volume on the sample bottle.

10.1.3 For 625 samples, and 608 samples when the compound list includes Aldrin, check samples for residual chlorine by adding a few drops of

sample from a pipette or glass stirring rod onto a piece of potassium iodide (KI) - starch paper that has been moistened with reagent water.

10.1.3.1 If the paper turns blue, treat the entire sample volume by adding a small scoop of granular sodium thiosulfate. Check the sample again with KI-starch paper. Continue treating and checking the sample until the paper no longer turns blue.

10.1.3.2 If the residual chlorine cannot be removed after 4 additions of sodium thiosulfate, contact the PM to obtain the client's instructions on whether to proceed with the extraction.

10.1.3.3 Document any samples that have been treated to remove residual chlorine using the "Residual Chlorine" NCM.

10.1.4 Spike all samples in the bottle with surrogate solution. Also spike the MS and MSD aliquots with Matrix Spike solution (Refer to Work Instruction PT-OP-WI-001). Replace the cap and mix well. Record the spike volumes and standard numbers on the TALS worksheet. Return spiking solutions to the refrigerator as soon as possible.

Note: If the sample bottle is completely full, it may be difficult to add the spike solutions to the bottle. In this case, transfer the sample to the separatory funnel and then add the spike prior to adding any methylene chloride.

10.1.5 Measure the initial sample pH with wide-range pH paper and record on the TALS worksheet. If sample is a leachate (e.g. TCLP), compare the current pH against leachate log, Note on the benchsheet if there is any discrepancy.

10.1.5.1 Refer to Table 1 for the required extraction pH for each method. If required, sample pH is adjusted in the sample bottle if possible, to the pH range as indicated in Table 1. Use the minimum amount of 1:1 H_2SO_4 or 10 N NaOH necessary to adjust the pH into the required range. Recheck the sample with pH paper by dipping a disposable pipette into the sample and wetting the pH paper. Record the adjusted pH on the TALS worksheet.

Note: If the sample bottle is completely full, it may be difficult to adjust the pH in the bottle. In this case, transfer the sample to the separatory funnel and then adjust the pH prior to adding any methylene chloride.

10.1.5.2

10.1.6 The normal sample volume is approximately 1 liter (250 mL for LVI). Clients may send bottles that have not been completely filled. The entire volume in the bottle is extracted, and the exact volume used is documented in the TALS batch.

10.1.6.1 Other sample volumes may be used to obtain specific reporting limits, and reduced sample volumes, diluted to 1 liter with

reagent water, may be used for very dirty samples. Unusual volumes, other than LVI, must be documented in an NCM.

10.1.7 Transfer the entire sample to the separatory funnel. Add 60 mL of methylene chloride to the bottle. Cap and shake for approximately 30 seconds to rinse all inner surfaces. Transfer the methylene chloride to the separatory funnel.

10.1.8 The sample volume is determined by filling the sample bottle with reagent water up to the marked meniscus or to the top of the sample bottle if the bottle is completely full and measuring that volume in a graduated cylinder. Record the volume to the nearest 10 mLs.

10.1.8.1 If the entire sample bottle will not be used (i.e., for smaller sample aliquots such as TCLP), mix the sample in the bottle and measure out the desired volume in a graduated cylinder. Spike the surrogate, and MS solution, where appropriate, and adjust initial sample pH in the cylinder. Transfer the aliquot to the separatory funnel.

10.1.8.2 Rinse the cylinder with 60 mL (15 mL for LVI) of methylene chloride and transfer to the separatory funnel.

10.1.9 Prepare a method blank, LCS, MS and MSD for each batch as specified in section 9 of this SOP. The LCS and MS/MSD are spiked with the surrogate and matrix spike solutions, the method blank only with the surrogates (see Work Instruction PT-OP-WI-001 for spike volumes).

10.1.10 Seal and shake or rotate the separatory funnel vigorously for 2 minutes with periodic venting to release excess pressure.

Warning: Methylene chloride creates excessive pressure very rapidly. Therefore, initial venting should be done immediately after the separatory funnel has been sealed and inverted. Vent into hood away from analysts and other samples.

10.1.11 Allow the organic layer to separate from the water phase until complete visible separation has been achieved (approximately 10 minutes). If the emulsion interface between layers is more than one-third the size of the solvent layer, the analyst must employ mechanical techniques to complete the phase separation.

- The optimum technique depends upon the sample and may include stirring, filtration of the emulsion through glass wool, centrifugation, or other physical methods.
- If the emulsion cannot be broken (recovery of <80% of the methylene chloride*), transfer the sample, solvent, and emulsion into the extraction chamber of a continuous liquid-liquid extractor (CLLE) and proceed as described in continuous liquid-liquid extraction (Section 10.2). If this is done, the sample must be extracted as part of a valid CLLE batch, with all associated QC.

Note: 15 - 20 mL of methylene chloride is expected to dissolve in 1 L of water. Thus, solvent recovery could be as low as 35 mL from the first shake and still be acceptable. Subsequent shakes should recover at least 50 mL of solvent.

10.1.12 Fill a funnel with 10-20 g of anhydrous sodium sulfate. The funnel can be plugged with glass wool or filter paper may be used to hold the sodium sulfate. Drain the solvent extract from the separatory funnel through the prepared filtration funnel into a clean glass container. The extract may be drained directly into the KD flask. Close the stopcock just before the water level begins draining out of the separatory funnel. If the sodium sulfate becomes saturated with water add more to the funnel or replace the existing sodium sulfate with fresh drying agent.

10.1.13 Use a second 60 mL (15 mL for LVI) portion of methylene chloride to rinse the bottle and transfer it to the separatory funnel. Repeat the extraction process, filtering the extract through the same filter funnel into the same collection vessel. Repeat the extraction process a third time using a fresh 60 mL portion of solvent. Combine all three solvent extractions in the collection container.

10.1.14 If extraction at a secondary pH is required, adjust the pH of the sample in the separatory funnel to the pH indicated in Table 1 with a minimum amount of 10 N NaOH or 1:1 H_2SO_4. Measure with pH paper and record the adjusted pH on the TALS worksheet. Serially extract with three 60 mL (15 mL for LVI) portions of methylene chloride, as outlined in Steps 10.1.10 to 10.1.12. Collect these three extracts in the same container used for the initial pH fraction.

Note: Alternatively, the acid and base fractions may be kept separate. Separate analysis of the acid and base fractions may be required for method 625. Individual client requirements must be checked before starting the extraction.

10.1.15 Rinse the extract residue from the sodium sulfate by pouring 20-30 mL (5-10mL for LVI) of clean methylene chloride through the funnel and into the collection container.

10.1.16 Dispose of water remaining in the separatory funnel into the appropriate waste container.

10.1.17 Cover the collection container with aluminum foil if the extract is not concentrated immediately. Refer to Section 10.3 for concentration.

10.2 Continuous Liquid/Liquid Extraction from Water Samples:

Refer to Figure 2 – Continuous Liquid/Liquid Extraction flowchart.

10.2.1 Remove surrogate and matrix spiking solutions from refrigerator and allow them to warm to room temperature.

10.2.2 Assemble the apparatus. Add 200-300 mL (100 mL for LVI) of methylene chloride to the extractor body. Add 3 to 5 boiling chips to the round-bottom distilling flask.

10.2.3 Mark the meniscus of the sample volume on the sample bottle.

10.2.4 See section 10.1.3 above for checking and treating residual chlorine in 625 and 608 samples.

10.2.5 Spike all samples in the bottle with surrogate solution. Also spike the MS and MSD aliquots with Matrix Spike solution (see Work Instruction PT-OP-WI-001 for spike volumes). Mix well. Record the spike volumes and standard numbers on the TALS worksheet. Return spiking solutions to the refrigerator as soon as possible.

Note: If the sample bottle is completely full, it may be difficult to add the spike solutions to the bottle. In this case, transfer the sample to the extractor and then add the spike prior to adding any methylene chloride.

10.2.6 Measure the initial sample pH with wide-range pH paper and record on the extraction TALS worksheet. If sample is a leachate (e.g. TCLP), compare the current pH against the leachate log. Note on the benchsheet if there is any discrepancy.

10.2.6.1 Refer to Table 1 for the required extraction pH for each method. If required, sample pH is adjusted in the sample bottle if possible, to the pH range as indicated in Table 1, for the initial extraction. Use the minimum amount of 1:1 H_2SO_4 or 10 N NaOH necessary to adjust the pH into the required range. Recheck the sample with pH paper by dipping a disposable pipette into the sample and wetting the pH paper. Record the adjusted pH on the TALS worksheet.

Note: If the sample bottle is completely full, it may be difficult to adjust the pH in the bottle. In this case, transfer the sample to the extractor and then adjust the pH prior to adding any methylene chloride.

10.2.7 Transfer the entire sample to the liquid-liquid extractor. Add 60 mL (15 mL for LVI) of methylene chloride to the bottle. Cap and shake for approximately 30 seconds to rinse all inner surfaces. Transfer the methylene chloride to the liquid-liquid extractor.

10.2.7.1 Repeat the sample bottle rinse with an additional 50-100 mL (15-20 mL for LVI) portion of methylene chloride and add the rinse to the extractor.

10.2.8 The sample volume is determined by filling the sample bottle with reagent water up to the marked meniscus or to the top of the sample bottle if the bottle is completely full and measuring that volume in a graduated cylinder. Record the volume to the nearest 10 mLs.

10.2.8.1 If the entire sample bottle will not be used (i.e., for smaller sample aliquots such as TCLP), mix the sample in the bottle and

measure out the desired volume in a graduated cylinder. Spike the surrogate, and MS solution, where appropriate, and adjust initial sample pH in the cylinder. Transfer the aliquot to the liquid-liquid extractor.

10.2.8.2 Rinse the cylinder with 60 mL (15 mL for LVI) of methylene chloride and transfer to the liquid-liquid extractor.

10.2.9 Prepare a method blank and LCS for each batch as specified in section 9 of this SOP. Use 1 L (250 mL for LVI) of reagent water for method blanks and LCS. The method blank is spiked with the surrogates, the LCS and matrix spikes with the surrogates and matrix spiking solutions. Note that different spiking solutions are used for methods 625, 8270 and TCLP (see Tables 3 and 4 for spike volumes).

10.2.10 Use 200 mL of leachate for TCLP semivolatiles, measured in a graduated cylinder. The leachate may be made up to 1 L in volume with reagent water.

10.2.11 For a TCLP method blank, 200 mL (semivolatiles) of the buffer solution used in the leaching procedure and transfer to the separatory funnel. Dilute to about 1 liter with reagent water.

10.2.12 Add reagent water to the extractor body until approximately 125 mL (35 mL for LVI) of methylene chloride is pushed over into the round-bottomed flask to ensure proper operation and solvent cycling. Attach cold condenser (about 10°C). Turn on heating mantle. Inspect joints for leaks once solvent has begun cycling. Extract for 18-24 hours for both the 3500 and 600 series methods.

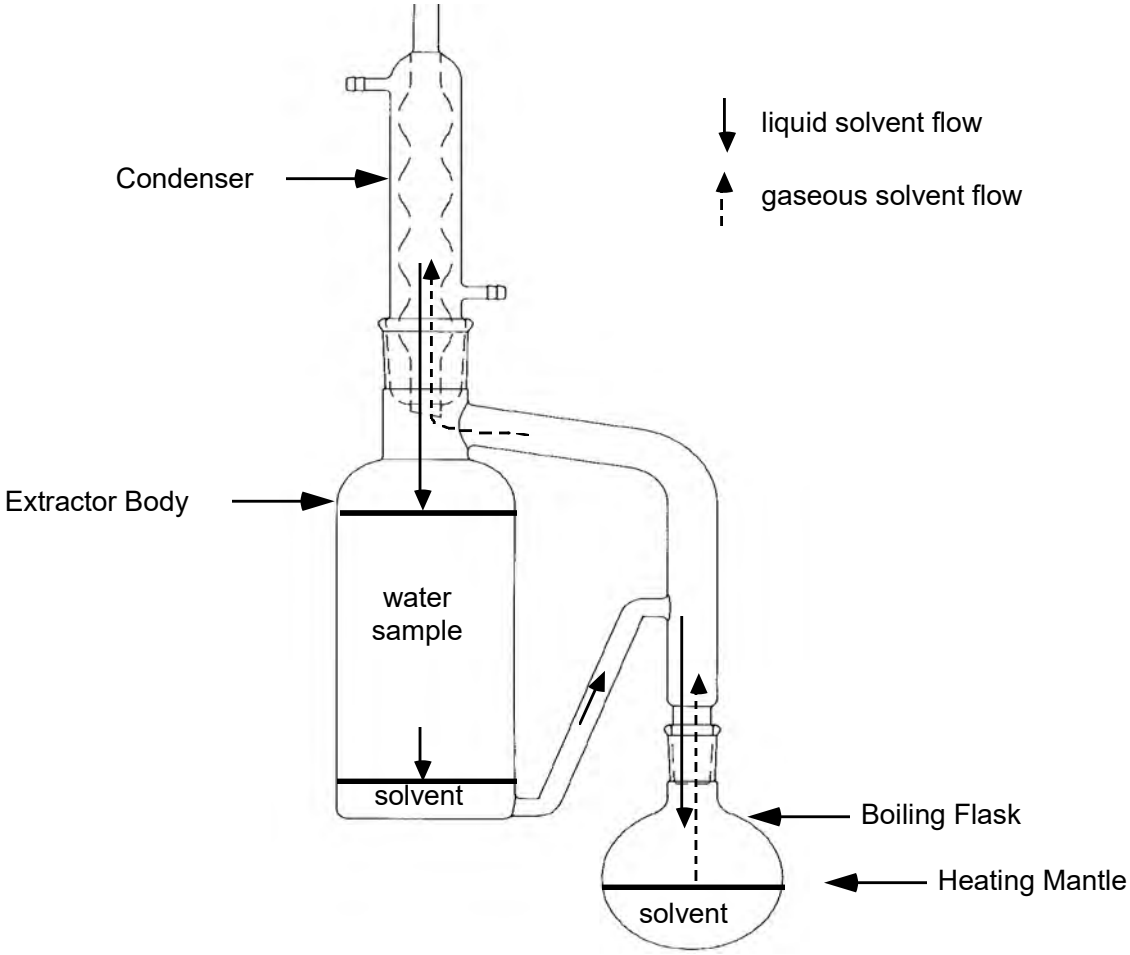
- For method 625.1, the sample pH must be checked after 2 hours of the extraction.
  - After 2 hours of extracting, turn the heating off on the samples and allow set-ups to cool for at least 20 minutes.
  - Using a disposable glass pipette, reach down to the water in the extractor body and collect a drop of sample. Drip this onto a strip of pH paper.
  - If the pH of the sample is still within the required range for that step of the extraction, continue the extraction for another 16-22 hours. Record the pH on the extraction worksheet.
  - If the pH of the sample is outside the range required, adjust the pH again to the required range within the extractor, and then continue the extraction for another 16-22 hours. Record both the observed and adjust-to pH's on the extraction worksheet.

10.2.13 If extraction at a secondary pH is required, (see Table 1) turn off the heating mantle and allow the extractor to cool. Detach the condenser and adjust the pH of the sample in the extractor body to the pH indicated in Table 1 with a minimum amount of 10 N NaOH or 1:1 H_2SO_4. Measure with pH paper and record the adjusted pH on the benchsheet. If desired, the acid and base fractions may be kept separate by replacing the boiling

flask with a clean flask and fresh solvent. Reattach the condenser and turn on heating mantle. Extract for 18-24 hours for both the 3500 and 600 series methods.

- For method 625.1, follow the procedure in section 10.2.12 above for checking the pH after 2 hours of extracting.

Note: Alternatively, the acid and base fractions may be kept separate. This may be required for method 625. Separate analysis of the acid and base fractions may also be required for method 625. Individual client requirements must be checked before starting the extraction.



10.2.14 Turn off the heating mantle and allow the extractor to cool.

10.2.15 Place a funnel containing 10-20 g of anhydrous sodium sulfate on the Kuderna-Danish (K-D) apparatus or other glass container. The funnel can be plugged with glass wool enabling it to hold the granular anhydrous sodium sulfate or phase separation filter paper may be used.

10.2.16 Dry the extract in the round bottom flask by filtering it through the sodium sulfate filled funnel. Note that it is not necessary or advisable to

attempt to add the solvent remaining in the continuous extractor body to the extract.

10.2.17 Collect the dried extract in a K-D or other glass container. Rinse the flask that contained the solvent extract with 20-30 mL (5-10 mL for LVI) of methylene chloride and add it to the funnel to complete the quantitative transfer. Dispose of solvent and water remaining in the extractor in the appropriate waste container.

Note: Some types of CLLE apparatus have built in drying columns. If this type of apparatus is used then a drying step subsequent to the extraction may not be necessary.

10.2.18 Cover with aluminum foil if the extract is not concentrated immediately. Refer to Section 10.3 for concentration.

10.3 Concentration

According to the method, type of sample and any cleanup procedures needed, different final solvents and volumes will be required. Refer to Table 2 for the appropriate final volumes and concentrations.

Refer to Figure 3 – Concentration and Cleanup flowchart.

10.3.1 Kuderna-Danish (KD) Method:

10.3.1.1 Assemble a Kuderna-Danish concentrator by attaching a 10 mL concentrator tube to the 500 mL KD flask. For procedures where the final volume is 10 mL or less, a 250 mL Erlenmyer flask may be used as an alternative to the KD flask.

10.3.1.2 Add one or two clean boiling chips and the dried extract to be concentrated to the KD flask and attach a three ball Snyder Column. Add approximately 1 mL of clean methylene chloride to the top of the Snyder column (this is important to ensure that the balls are not stuck and that the column will work properly).

10.3.1.3 Place the KD apparatus on a water bath maintained at 60-65 °C so that the tip of the concentrator tube is submerged. The water level should not reach the joint between the concentrator and the KD flask. At the proper rate of distillation, the balls will actively chatter but the chambers should not flood.

10.3.1.4 Concentrate to 5-15 mL. If the determinative method requires a solvent exchange add the appropriate exchange solvent (see Table 2) to the top of the Snyder Column, and then continue the water bath concentration back down to 1-4 mL. The Snyder column may be insulated if necessary to maintain the correct rate of distillation.

Note: Add an additional boiling chip with the addition of exchange solvent. An alternative technique for solvent exchange is to replace the macro

Snyder column and KD flask with a micro Snyder column, concentrate to approximately 1 mL, add 10 mL of exchange solvent, and concentrate back down to 1 mL. The extract must be cool before the macro Snyder assembly is removed.

Note: It is very important not to concentrate to dryness as analytes will be lost. If this happens, the extract must be discarded and the extraction started over with fresh sample.

10.3.1.5 Remove the KD apparatus from the water bath and allow it to cool for a minimum of 10 minutes. If the level of the extract is above the level of the concentrator tube joint, continue to distill the solvent as necessary. Again, allow the KD flask to cool for a minimum of 10 minutes.

10.3.1.6 If the final volume is 5 or 10 mL, the extract may be made up to volume in the graduated KD tube or transferred to a 12 mL vial previously marked at the appropriate volume level. Document the final volume. Otherwise proceed to section 10.3.2.

10.3.2 Nitrogen Evaporation to Final Concentration

10.3.2.1 Transfer the entire extract to a calibrated evaporation tube. Rinse the concentrator tube with 1-2 mL of the appropriate solvent and transfer the solvent rinsate to the evaporation tube.

10.3.2.2 Place the tube in a warm water bath that is at approximately 35°C and evaporate the solvent using a gentle stream of nitrogen. The nitrogen flow will form a slight depression on the surface of the solvent, but should not create splattering of the extract.

Note: Extracts for method 608.3 may be concentrated at temperature 50-55°C.

10.3.2.3 During the course of the evaporation rinse the sides of the evaporation tube twice with approximately 1 mL of clean solvent. The first rinse should be about half way through the process, with the second rinse when the solvent volume gets close to 1 mL. Concentrate the solvent accurately to the calibrated volume line and transfer the extract to the appropriate storage vial.

Note: It is very important not to concentrate to dryness as analytes will be lost. If this happens, the extract must be discarded and the extraction started over with fresh sample.

Vial Calibration Note: Using a Class A volumetric pipette dispense the appropriate volume of solvent into the vial being calibrated, mark the meniscus and use this vial when marking the final volume line on subsequent sample vials of that type and lot. See SOP PT-QA-026 and work instruction PT-QA-WI-008 for details.

10.3.2.4 An alternative technique is to follow the previous steps concentrating the solvent to slightly below the required final volume and then drawing the extract into a syringe. Rinse the evaporation tube with a small amount of solvent and draw additional solvent into the syringe to make up the accurate final volume.

Note: The final concentration and volume measurement steps are critical. Use care when concentrating and make certain that the final volume measurement is accurate.

10.4 If sample extract cleanup is required please refer to SOP PT-OP-028 for the appropriate cleanup technique.

11.0 CALCULATIONS / DATA REDUCTION

11.1 Not applicable – refer to analytical method SOPs.

12.0 METHOD PERFORMANCE

12.1 The supervisor has responsibility to ensure that an analyst who performs this procedure is properly trained in its use and has the required experience. Performance is monitored through internal QC and outside performance evaluation samples. Please refer to the QA Manual for additional information concerning Precision and Accuracy.

12.2 Demonstration of Capabilities – Prior to the analysis of samples, a Demonstration of Capabilities (DOC) as described in the QA Manual, must be performed initially, annually and any time a significant change is made to the analytical system.

12.3 Method Detection Limit Study – A Method Detection Limit (MDL) study, as described in the QA Manual, must be performed initially, annually and any time a significant change is made to the analytical system.

13.0 POLLUTION CONTROL

13.1 It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage and reagent stability). Employees must abide by the policies in Section 13 of the Corporate Environmental Health and Safety Manual (CW-E-M-001) for "Waste Management and Pollution Prevention" and the Pittsburgh Facility Addendum EH&S Manual (PT-HS-001).

13.2 Within the constraints of following the methodology in this SOP, use of organic solvents should be minimized.

14.0 WASTE MANAGEMENT

14.1 Waste management practices are conducted consistent with all applicable rules and regulations. Excess reagents, samples and method process wastes are disposed of in an accepted manner. Waste description rules and land disposal restrictions are followed. Waste disposal procedures are incorporated by reference to PT-HS-001. The following waste streams are produced when this method is carried out.

14.1.1 Methylene Chloride extraction waste. This waste is collected in waste containers identified as “Methylene Chloride Waste”, Waste #2.

14.1.2 Extracted water samples. This waste is collected in a waste container identified as “Extraction Water”, Waste #35. The bottom organic layer is drained into a container identified as “Methylene Chloride Waste”, Waste #2. The remaining aqueous layer is neutralized to a pH between 6 and 9 and discharged down lab sink/ drain.

14.1.3 Used sodium sulfate and glass wool or filter paper contaminated with methylene chloride from the extract drying step. This waste is collected in a container identified as “Lab Trash Waste”, Waste #12.

14.1.4 Assorted flammable solvent waste from various rinses. This waste is collected in waste containers identified as “Mixed Flammable Solvent Waste”, Waste 3.

14.1.5 Methylene chloride waste from various rinses. This waste is collected in waste containers identified as “Methylene Chloride Waste”, Waste #2.

14.1.6 Miscellaneous disposable glassware contaminated with acids, caustics, solvents and sample residue. This waste is collected in a container identified as “Lab Trash Waste”, Waste #12.

15.0 REFERENCES – CROSS-REFERENCES

15.1 Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW846, 3rd Edition, Final Update III (December 1996). Sections 3500B, 3510C, 3520C, 3540C, 3541 3550B, 3580A, 3600C, 3620B, 3640A, 3660B, and 3665A

15.2 Wisconsin Department of Natural Resources, NR 219.04, Analytical Test Methods and Procedures, April 2010

15.3 Pittsburgh Laboratory Quality Assurance Manual (PT-QA-M-001)

15.4 40 CFR Part 136, Appendix A, United States Environmental Protection Agency. 2016, Methods 608.3 and 625.1

15.5 SOP PT-QA-001, Employee Training and Orientation

15.6 SOP PT-QA-003, Glassware Clean-up for Organic/Inorganic Procedures

15.7 SOP PT-QA-006, Procurement of Standards and Materials; Labeling and Traceability

15.8 SOP PT-QA-007, Determination of Method Detection Limits (MDL)

15.9 SOP PT-QA-008, Thermometer Calibration and Temperature Monitoring

15.10 SOP PT-QA-012, Selection and Calibration of Balances and Weights

15.11 SOP PT-QA-016, Nonconformance and Corrective Action System

15.12 SOP PT-QA-021, Quality Control Program

15.13 SOP PT-QA-026, Container Accuracy Verification – Gravimetric

15.14 SOP PT-QA-031, Internal Chain of Custody

15.15 Pittsburgh Facility Addendum EH&S Manual, PT-HS-001

15.16 PT-OP-WI-001, TestAmerica Pittsburgh Organic Prep Work Instruction for Initial/Final Volumes, Exchange Solvents, LCS and Matrix Spiking Components and Concentrations

16.0 METHOD MODIFICATIONS

16.1 Some surrogate spiking concentrations are modified from those recommended in SW-846, in order to make the concentrations more consistent with the calibration levels in the determinative methods.

16.2 Spiking levels for method 608.3 have been reduced by a factor of ten to bring the levels within the normal calibration range of the instrument.

17.0 ATTACHMENTS

17.1 Table 1 – Liquid/Liquid Extraction Conditions

17.2 Table 2 – Initial Volumes, Exchange Solvents, and Final Volumes

17.3 Figure 1 – Separatory Funnel Extraction

17.4 Figure 2 – Continuous Liquid/Liquid Extraction

17.5 Figure 3 – Concentration and Cleanup

18.0 REVISION HISTORY

18.1 Revision 9, 02/01/07

18.2 Revision 9, 02/01/07

18.3 Revision 10, 09/24/07

18.4 Revision 11, 01/16/09

18.5 Revision 12, 7/1/2010

18.6 Revision 13, 3/11/2011

18.7 Revision 14, 4/22/2011

18.8 Revision 15, 12/2/2013

18.9 Revision 16, 9/10/2015

18.10 Revision 17, 10/23/2017

18.11 Revision 18, 7/26/2018

18.12 Revision 18.1, 4/29/2019

18.13 Revision 19, 2/14/2020

18.14 Revision 20, 4/28/2020

18.15 Changes to current revision

SOP section	Change from	Change to	Reason
throughout		references to and volumes for LVI procedure added	Retiring SOP PT-OP-022 and combining with this document

Table 1		
Liquid/Liquid Extraction Conditions		
Determinative Method	**Initial Ext. pH[1]**	**Secondary Ext. pH[1]**
BNA (8270)	1 - 2	11 - 12
BNA (625/625.1)	11 - 12	1 - 2
Pesticides (8081 & 608/608.3)	5 - 9	None
PCB Aroclors (8082&608/608.3)	5 - 9	None
PCB Congeners (8082)	5 - 9	None
OP Pesticides (8141)	As received	None

[1] If the laboratory has validated acid only 8270C and 8270D extraction for the target compound list required, then the base extraction step may be omitted. The required validation consists of a 4 replicate initial demonstration of capability and a method detection limit study. (See Section 12).

Table 2					
Initial Volumes, Exchange Solvents, and Final Volumes					
Group	**Method**	**Initial Water Vol. (mL)**	**Initial TCLP Vol. (mL)**	**Exchange Solvent for Analysis**	**Final Volume for Analysis (mL)**
GC/MS Semivolatiles	625 (625.1)	1000	NA	NA	10
	625 LL	1000	NA	NA	1.0
	625 LL LVI	250	NA	NA	0.25
	8270/3520C	1000	NA	NA	10
	8270 LL/3520C LL	1000	NA	NA	1.0
	8270 LVI/3520C	250	NA	NA	2.5
	8270 LL LVI/3520C	250	NA	NA	0.25
	8270 LL LVI/3510C	250	NA	NA	2.5
	8270 TCLP/3520C	NA	1000/ 200	NA	1.0/10
	8270 TCLP/3510C	NA	200	NA	10
Pesticides (8081A/B)	8081/3510C	1000	NA	Hexane	40
	8081 LL/3510C LL	1000	NA	Hexane	1.0
	8081/ 3510C LVI	250	NA	Hexane	10
	8081/3510C TCLP	NA	100	Hexane	40
	8081/3580A TCLP	NA	1.0	Hexane	40
PCB Aroclors (8082/A)	8082/3510C	1000	NA	Hexane	40
	8082 LL/3510C LL	1000	NA	Hexane	1.0
	8082/3510C LVI	250	NA	Hexane	10
	8082/3580A	NA	NA	Hexane	40
	8082/3510C TCLP	1000	NA	Hexane	40
	8082/3510C Safety Kleen	100	NA	Hexane	4.0
Pesticide and PCB Aroclors	608/608.3	1000	NA	Hexane	1.0ml
PCB Congeners (8082A)	8082A_Con/3510C	1000	NA	Hexane	2.0
OPP's (8141A/B)	8141/3510C	1000	NA	Hexane	5.0
	8141 LL/3510C LL	1000	NA	Hexane	1.0

Figure 1 - Separatory Funnel Extraction

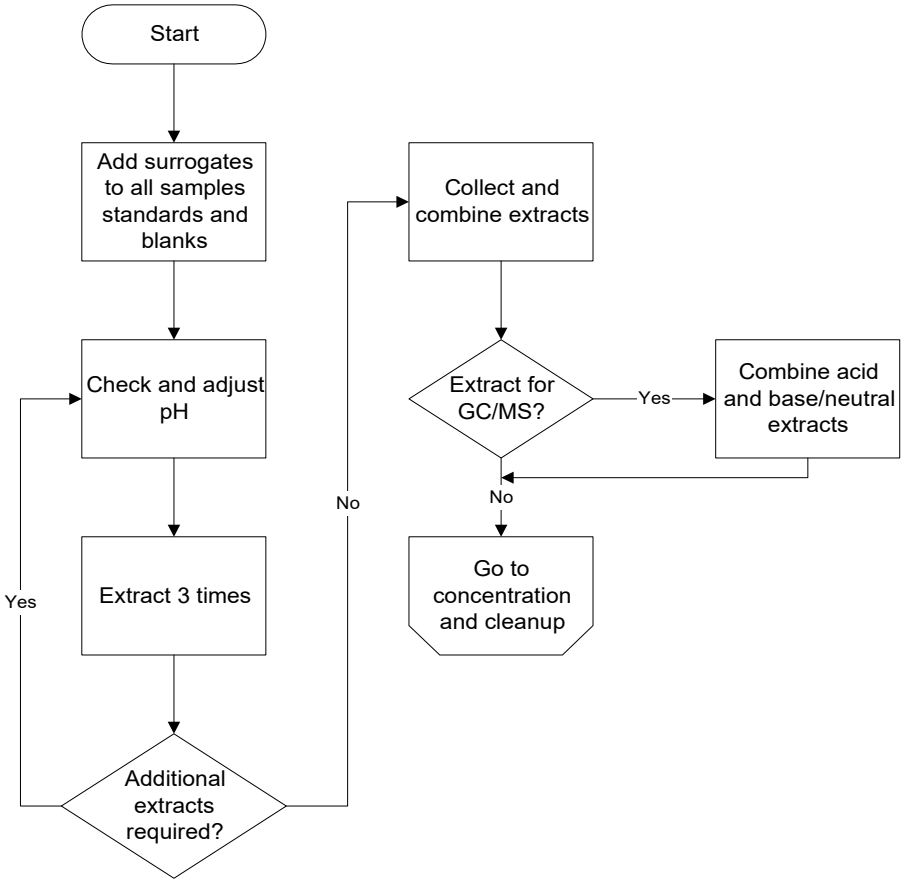


Figure 2 - Continuous Liquid/Liquid Extraction

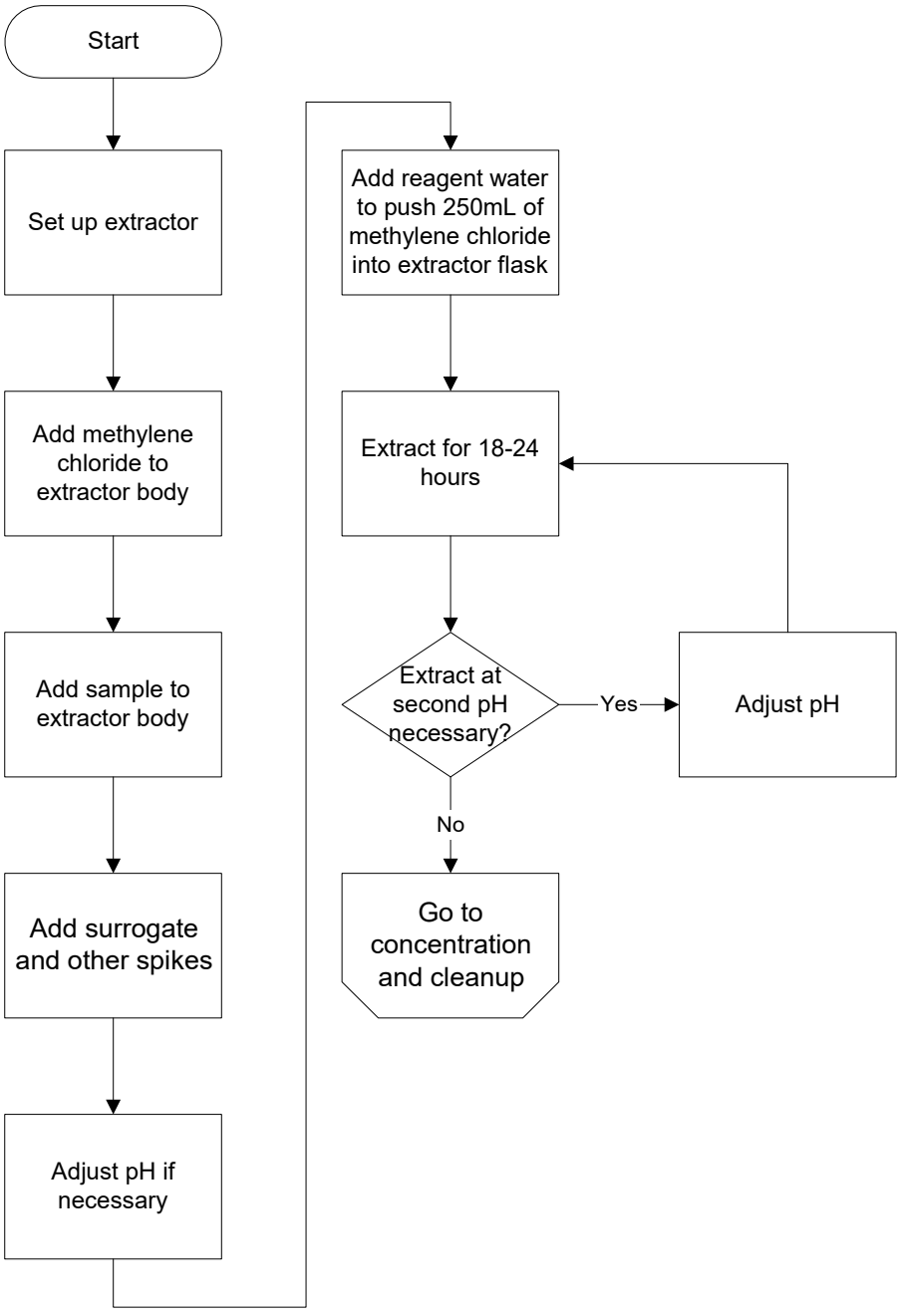
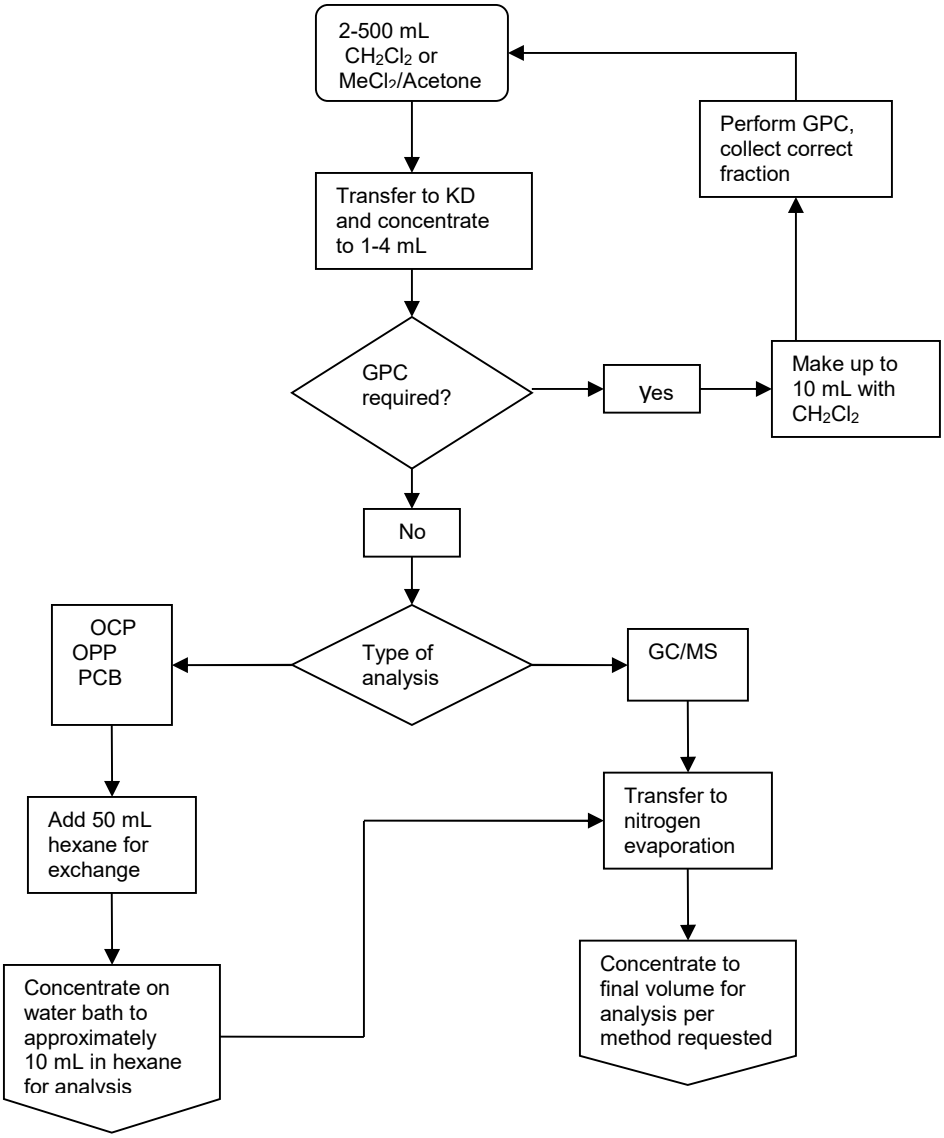


Figure 3 - Concentration and Cleanup



Quality Assurance Manual

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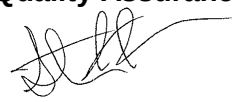
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Laboratory Director - Deborah L. Lowe — Date



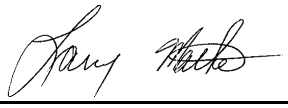
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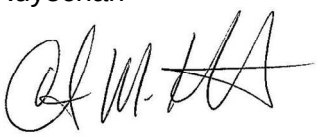
4/13/2020

Department/Technical Manager (Organics) - Sharon Bacha — Date



4/6/2020

Department/Technical Manager (Inorganics) - Roseann Ruyechan — Date



4/16/2020

Department Manager (Sample Management) - Chris Kovitch — Date

SECTION 2 - TABLE OF CONTENTS

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1.0	TITLE PAGE				2
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REFERENCED CORPORATE SOPS AND POLICIES

SOP / Policy Reference	Title
CA-C-S-001	Worksharing Program
CA-I-P-002	Electronic Reporting and Signature Policy
CA-L-P-002	Contract Compliance Policy
CA-L-S-004	Subcontracting
CA-Q-M-002	Corporate Quality Management Plan
CA-Q-QM-001	Policy on Tentatively Identified Compounds (TICs) – GC/MS Analysis
CA-Q-S-001	Acid and Solvent Lot Testing and Approval Program
CA-Q-S-002	Acceptable Manual Integration Practices
CA-Q-S-006	Detection and Quantitation Limits
CA-Q-S-009	Root Cause Analysis
CA-T-P-001	Qualified Products List
CW-E-M-001	Corporate Environmental Health & Safety Manual
CW-F-P-002	Company-Wide Authorization Matrix
CW-F-P-004	Procurement and Contracts Policy
CW-F-S-007	Fixed Asset Acquisition, Retention and Safeguarding
CW-I-M-001	IT Change Control Procedure Manual
CW-L-P-001	Records Retention Policy
CW-L-P-004	Ethics Policy
CW-L-S-002	Internal Investigation
CW-L-S-004	Subcontracting
CW-Q-S-001	Corporate Document Control and Archiving
CW-Q-S-002	Writing a Standard Operating Procedure (SOPs)
CW-Q-S-003	Internal Auditing
CW-Q-S-004	Management Systems Review
CW-Q-S-005	Data Recall Process

REFERENCED LABORATORY DOCUMENTS

SOP Reference	Title
PT-QA-001	Employee Orientation and Training (DOCs)
PT-QA-002	Internal Auditing
PT-QA-005	Measurement Uncertainty
PT-QA-006	Procurement of Standards and Materials; Labeling and Traceability
PT-QA-007	Detection Limits
PT-QA-008	Thermometer and Barometer Verification and Temperature Monitoring
PT-QA-010	Document and Spreadsheet Development & Control
PT-QA-012	Selection and Calibration of Balances and Weights
PT-QA-013	Independent QA Data Review
PT-QA-016	Nonconformance and Corrective Action System
PT-QA-017	Aqueous Pipette / Dispenser Calibration – Gravimetric Method
PT-QA-018	Technical Data Review Requirements
PT-QA-019	Records Management, Retention and Archive
PT-QA-021	Laboratory Quality Control Program
PT-QA-022	Equipment Maintenance
PT-QA-024	Subsampling
PT-QA-WI-002	Master Document List

SECTION 3
INTRODUCTION, SCOPE AND APPLICABILITY

3.1 INTRODUCTION AND COMPLIANCE REFERENCES

Eurofins TestAmerica Pittsburgh's Quality Assurance Manual (QAM) is a document prepared to define the overall policies, organization objectives and functional responsibilities for achieving Eurofins TestAmerica's data quality goals. The laboratory maintains a local perspective in its scope of services and client relations and maintains a national perspective in terms of quality.

The QAM has been prepared to assure compliance with The NELAC Institute (TNI) Standard, dated 2009, Volume 1 Modules 2 and 4, and ISO/IEC Guide 17025:2017(E). In addition, the policies and procedures outlined in this manual are compliant with Eurofins TestAmerica's Corporate Quality Management Plan (CQMP) CA-Q-M-002, and the various accreditation and certification programs listed in Appendix 3. The CQMP provides a summary of Eurofins TestAmerica's quality and data integrity system. It contains requirements and general guidelines under which all Eurofins TestAmerica facilities shall conduct their operations.

This QAM has been prepared to be consistent with requirements of the following documents:

- EPA *Requirements for Quality Management Programs* (QA/R-2), EPA/240/B-01/002, May 31, 2006
- EPA 600/4-88/039, *Methods for the Determination of Organic Compounds in Drinking Water,* EPA, Revised July 1991
- EPA 600/R-95/131, *Methods for the Determination of Organic Compounds in Drinking Water,* Supplement III, EPA, August 1995
- EPA 600/4-79-019, *Handbook for Analytical Quality Control in Water and Wastewater Laboratories,* EPA, March 1979
- *Test Methods for Evaluating Solid Waste Physical/Chemical Methods (SW846), Third Edition, September 1986, Final Update I, July 1992, Final Update IIA, August 1993, Final Update II, September 1994; Final Update IIB, January 1995; Final Update III, December 1996; Final Update IV, January 2008, Final Update V, August 2015*
- *Federal Register, 40 CFR Parts 136, 141, 172, 173, 178, 179 and 261*
- *Statement of Work for Inorganics & Organics Analysis, SOM and ISM, current versions, USEPA Contract Laboratory Program Multi-media, Multi-concentration*
- APHA, *Standard Methods for the Examination of Water and Wastewater,* 18th Edition, 19th, 20th, 21st, 22nd and on-line Editions
- *U.S. Department of Energy* Order 414.1D, *Quality Assurance*, April 25, 2011
- Nuclear Regulatory Commission (NRC) quality assurance requirements
- Marine Protection, Research, and Sanctuaries Act (MPRSA)
- Toxic Substances Control Act (TSCA)

3.2 TERMS AND DEFINITIONS

A Quality Assurance Program is a company-wide system designed to ensure that data produced by the laboratory conforms to the standards set by state and/or federal regulations. The program functions at the management level through company goals and management policies, and at the analytical level through Standard Operating Procedures (SOPs) and quality control. The Eurofins TestAmerica program is designed to minimize systematic error, encourage constructive, documented problem solving, and provide a framework for continuous improvement within the organization.

Refer to Appendix 2 for the Glossary/Acronyms.

3.3 SCOPE / FIELDS OF TESTING

The laboratory analyzes a broad range of environmental and industrial samples. Sample matrices vary among effluent water, surface water, groundwater, hazardous waste, sludge, soils, sediments, and tissue. The Quality Assurance Program contains specific procedures and methods to test samples of differing matrices for chemical, physical and biological parameters. The Program also contains guidelines on maintaining documentation of analytical processes, reviewing results, servicing clients and tracking samples through the laboratory. The technical and service requirements of all analytical requests are thoroughly evaluated before commitments are made to accept the work. Measurements are made using published reference methods or methods developed and validated by the laboratory.

The methods covered by this manual include the most frequently requested methodologies needed to provide analytical services in the United States and its territories. The specific list of test methods used by the laboratory can be found in the Statement of Qualifications (SOQ). The current list of accredited methods is maintained in Total Access. The approach of this manual is to define the minimum level of quality assurance and quality control necessary to meet these requirements. All methods performed by the laboratory shall meet these criteria as appropriate. In some instances, quality assurance project plans (QAPPs), project specific data quality objectives (DQOs) or local regulations may require criteria other than those contained in this manual. In these cases, the laboratory will abide by the requested criteria following review and acceptance of the requirements by the Laboratory Director and the Quality Assurance (QA) Manager. In some cases, QAPPs and DQOs may specify less stringent requirements. The Laboratory Director and the QA Manager must determine if it is in the lab's best interest to follow the less stringent requirements.

3.3.1 Specialty Analyses

3.3.1.1 Dredged Material Evaluations

Eurofins TestAmerica Pittsburgh offers trace level testing of waters (site-waters and elutriates), sediments, and tissues in support of Dredged Material Evaluations for in-water (ocean and inland waters) and upland (Confined Disposal Facilities (CDFs), beneficial use, etc.) disposal options. In-house capabilities for commonly requested sediment program parameters include:

- Organochlorine Pesticides
- Organophosphorus Pesticides
- PCBs (as Aroclors and Congeners)
- Volatile Organics
- Semivolatile Organics
- Metals
- Cyanide
- Total Sulfides
- Acid Volatile Sulfide (AVS) and Simultaneously Extracted Metals (SEM)
- Nitrogen, Ammonia
- Nitrogen, Nitrate + Nitrite
- Biochemical Oxygen Demand (BOD)
- Chemical Oxygen Demand (COD)
- Total Organic Carbon (combustion procedure for sediments)

- Total Solids/Moisture Content
- Total Volatile Solids
- Lipids
- With teaming arrangements with other Eurofins TestAmerica facilities, additional sediment program capabilities include:
    - Polychlorinated Dibenzo-Dioxins and Furans (PCDDs/PCDFs)
    - Butyl Tins (mono – tetra)
    - Total Kjeldahl Nitrogen
    - Total Phosphorus
    - Grain Size
    - Specific Gravity
    - Atterberg Limits

Eurofins TestAmerica Pittsburgh also generates elutriate samples following appropriate U.S. Army Corps of Engineers procedures. These include:

- Standard Elutriate Test (SET) for in-water disposal evaluations, and
- Modified Elutriate Test (MET) or Effluent Elutriate Test (EET) for CDF disposal evaluations.
- Illinois Resuspension Tests (Supernatant and Elutriate Tests).
- Dredge Elutriate Test (DRET)

Eurofins TestAmerica Pittsburgh currently supports dredge material evaluation projects following several state specific programs, as well as, under the following guidance documents:

- Ocean Testing Manual or OTM (USACE, 1991).
- New Jersey's Tidal Waters Technical Manual (NJDEP, 1997).
- Inland Testing Manual or ITM (USACE, 1998).
- Upland Testing Manual or UTM (USACE, 2003).

3.3.1.2 Tissue Analyses

Eurofins TestAmerica Pittsburgh has extensive experience in supporting projects requiring tissue analyses. These include analyses of laboratory cultured reference species from bioaccumulation tests associated with dredged material evaluations to a variety of field collected species (aquatic and terrestrial). Eurofins TestAmerica Pittsburgh has developed modifications to the standard solid methodologies (where possible) to allow for the use of smaller sample weights and achieve lower quantitation limits. In-house capabilities for commonly requested tissue parameters include:

- Organochlorine Pesticides
- PCBs (as Aroclors and Congeners)
- Semivolatile Organics
- Metals
- Lipids
- Moisture Content

- With teaming arrangements with other Eurofins TestAmerica facilities, additional tissue capabilities include:
  - Polychlorinated Dibenzo-Dioxins and Furans (PCDDs/PCDFs)
  - Butyl Tins (mono – tetra)

3.4 MANAGEMENT OF THE MANUAL

3.4.1 Review Process

The template on which this manual is based is reviewed annually by Corporate Quality Management Personnel to assure that it remains in compliance with Section 3.1. This manual itself is reviewed every two years by senior laboratory management to assure that it reflects current practices and meets the requirements of the laboratory's clients and regulators as well as the CQMP. Occasionally, the manual may need changes in order to meet new or changing regulations and operations. The QA Manager will review the changes in the normal course of business and incorporate changes into revised sections of the document. All updates will be reviewed by the senior laboratory management staff. The laboratory updates and approves such changes according to Pittsburgh laboratory SOP No. PT-QA-010, *Document Development and Control*.

SECTION 4

MANAGEMENT REQUIREMENTS

4.1 Overview

Eurofins TestAmerica Pittsburgh is a local operating unit of Eurofins TestAmerica Laboratories, Inc.. The organizational structure, responsibilities and authorities of the Corporate staff of TestAmerica Laboratories, Inc. are presented in the CQMP. The laboratory has day-to-day independent operational authority overseen by corporate officers (e.g., President and Chief Executive Officer (CEO), Chief Operating Officer (COO), Executive Vice President (VP) Operations, Corporate Quality etc.). The laboratory operational and support staff work under the direction of the Laboratory Director. The organizational structure for both Corporate & Eurofins TestAmerica Pittsburgh is presented in Figure 4-1.

4.2 Roles And Responsibilities

In order for the Quality Assurance Program to function properly, all members of the staff must clearly understand and meet their individual responsibilities as they relate to the quality program. The following descriptions briefly define each role in its relationship to the Quality Assurance Program.

4.2.1 Additional Requirements for Laboratories

The responsibility for quality resides with every employee of the laboratory. All employees have access to the QAM, are trained to this manual, and are responsible for upholding the standards therein. Each person carries out his/her daily tasks impartially and in a manner consistent with the goals and in accordance with the procedures in this manual and the laboratory's SOPs. Role descriptions for corporate personnel are defined in the CQMP. This manual is specific to the operations of Eurofins TestAmerica's Pittsburgh laboratory.

4.2.2 President and Chief Executive Officer (CEO)

The President and CEO is a member of the Board of Directors and is ultimately responsible for the quality and performance of all Eurofins TestAmerica facilities. The President and CEO establishes the overall quality standard and data integrity program for the Analytical Business, providing the necessary leadership and resources to assure that the quality standard and integrity program are met.

4.2.3 Chief Operation Officer (COO)

The COO reports directly to the President and CEO of Eurofins TestAmerica. The COO is responsible for the operations of Eurofins TestAmerica's subsidiary companies and the company's strategic growth.

4.2.4 Senior Vice President of Operations and Client Services

The SVP of Operations and Client Services leads the Client Services Organization (CSO) and oversees the operations of all Eurofins TestAmerica Laboratories, the Corporate Technical Services group and the Sales Opportunity Optimization efforts. The SVP provides direction to the VPs of Operations, Client Services Directors, Manager or Project Managers, Director of Technical Services and a Director of Sales. The SVP of Operations and Client Services reports directly to the President and CEO of Eurofins TestAmerica.

4.2.5 Vice President of Operations (VPO)

Each VP of Operations (VPO) reports directly to the SVP of Operations and Client Services. Each VPO is responsible for the overall administrative and operational management of their respective laboratories. The VPO's responsibilities include allocation of personnel and resources, long-term planning, goal setting, and achieving the financial, business, and quality objectives of Eurofins TestAmerica. The VPOs ensure timely compliance with Corporate Management directives, policies, and management systems reviews. The VPOs are also responsible for restricting any laboratory from performing analyses that cannot be consistently and successfully performed to meet the standards set forth in this manual.

4.2.6 Vice President of Quality and Environmental Health and Safety (VP-QA/EHS)

The Vice President (VP) of QA/EHS reports directly to the President and CEO. With the aid of the Executive Committee, Laboratory Directors, Quality Directors, Safety Managers, EH&S Coordinators and QA Managers, the VP-QA/EHS has the responsibility for the establishment, general overview and Corporate maintenance of the Quality Assurance and EH&S Programs within Eurofins TestAmerica. Additional responsibilities include:

- Review of QA/QC and EHS aspects of Corporate SOPs & Policies, national projects and expansions or changes in services.
- Work with various organizations outside of Eurofins TestAmerica to further the development of quality standards and represent Eurofins TestAmerica at various trade meetings.
- Prepare monthly reports for quality and EH&S metrics across the analytical laboratories and a summary of any quality related initiatives and issues.
- With the assistance of the Corporate Senior Management Teams and the EHS Managers, development and implementation of the Eurofins TestAmerica Environmental, Health and Safety Program.

4.2.7 Quality Assessment Director

The Quality Assessment Director reports to the VP-QA/EHS. The Quality Assessment Director has QA oversight of laboratories; is responsible for the internal audit system, schedule and procedure; monitors laboratory internal audit findings; identifies common laboratory weaknesses; and monitors corrective action closures. Together with the Quality Compliance Director, the Quality Systems Director, and the VP-QA/EHS, the Quality Assessment Director has the responsibility for the establishment, general overview and maintenance of the Analytical Quality Assurance Program within Eurofins TestAmerica.

4.2.8 Quality Compliance Director

The Quality Compliance Director reports to the VP-QA/EHS. The Quality Compliance Director has QA oversight of laboratories; monitors and communicates DoD / DoE requirements; develops Corporate tools for ensuring and improving compliance; develops Corporate assessment tools; identifies common laboratory weaknesses; and monitors corrective action closures. Together with the Quality Assessment Director, Quality Systems Director and the VP-QA/EHS, the Quality Compliance Director has the responsibility for the establishment, general overview and maintenance of the Analytical Quality Assurance Program within Eurofins TestAmerica.

4.2.9 Quality Systems Director

The Quality Systems Director reports to the VP-QA/EHS. The Quality Systems Director has QA oversight of laboratories; develops quality policies, procedures and management tools; monitors and communicates regulatory and certification requirements; identifies common laboratory weaknesses; and monitors corrective action closures. Together with the Quality Assessment Director, Quality Compliance Director and the VP-QA/EHS, the Quality Systems Director has the responsibility for the establishment, general overview and maintenance of the Analytical Quality Assurance Program within Eurofins TestAmerica.

4.2.10 Quality Information Manager

The Quality Information Manager is responsible for managing all company official documents (e.g., Policies, Procedures, Work Instructions), the company's accreditation database, intranet websites, external laboratory subcontracting, regulatory limits for clients on the company's TotalAccess website; internal and external client support for various company groups (e.g., Client Services, EH&S, Legal, IT, Sales) for both quality and operational functions. The Quality Information Manager reports to the VP-QA/EHS; and works alongside the Quality Assessment, Quality Compliance and Quality System Directors and EHS Managers to support both the Analytical Quality Assurance and EHS Programs within Eurofins TestAmerica.

4.2.11 Technical Services Director

The Technical Services Director is responsible for establishing, implementing and communicating Eurofins TestAmerica's Analytical Business's Technical Policies, SOPs, and Manuals. Other responsibilities include conducting technical assessments as required, acting as a technical resource in national contracts review, coordinating new technologies, establishing best practices, advising staff on technology advances, innovations, and applications.

4.2.12 Ethics and Compliance Officers (ECOs)

Eurofins TestAmerica has designated two senior members of the Corporate staff to fulfill the role of Ethics and Compliance Officer (ECO) – i.e., the Corporate Counsel & VP of Human Resources and the VP-QA/EHS. Each ECO acts as a back-up to the other ECO and both are involved when data investigations occur. Each ECO has a direct line of communication to the entire senior Corporate and lab management staff.

The ECOs ensure that the organization distributes the data integrity and ethical practices policies to all employees and ensures annual trainings and orientation of new hires to the ethics program and its policies. The ECO is responsible for establishing a mechanism to foster employee reporting of incidents of illegal, unethical, or improper practices in a safe and confidential environment.

The ECOs monitor and audit procedures to determine compliance with policies and to make recommendations for policy enhancements to the President and CEO, VPOs, Laboratory Director or other appropriate individuals within the laboratory. The ECO will assist the laboratory QA Manager in the coordination of internal auditing of ethical policy related activities and processes within the laboratory, in conjunction with the laboratory's regular internal auditing function.

The ECOs will also participate in investigations of alleged violations of policies and work with the appropriate internal departments to investigate misconduct, remedy the situation, and prevent recurrence of any such activity.

4.2.13 Chief Information Officer (CIO)

The CIO is responsible for establishing, implementing and communicating Eurofins TestAmerica's Information Technology (IT) Policies, SOPs and Manuals. Other responsibilities include coordinating new technologies, development of electronic communication tools such as Eurofins TestAmerica's intranet and internet sites, ensuring data security and documentation of software, ensuring compliance with the NELAC standard, and assistance in establishing, updating, and maintaining Laboratory Information Management Systems (LIMS) at the various Eurofins TestAmerica facilities.

4.2.14 Environmental Health and Safety Managers (Corporate)

The EHS Managers report directly to the VP-QA/EHS. The EHS Managers are responsible for the development and implementation of the Eurofins TestAmerica Environmental, Health and Safety program. Responsibilities include:

- Consolidation and tracking all safety and health-related information and reports for the company, and managing compliance activities for Eurofins TestAmerica locations
- Coordination/preparation of the Corporate Environmental, Health and Safety Manual Template that is used by each laboratory to prepare its own laboratory-specific Safety Manual/ CHP
- Preparation of information and training materials for laboratory EHS Coordinators
- Assistance in the coordination of employee exposure and medical monitoring programs to insure compliance with applicable safety and health regulations

- Serving as Department of Transportation (D.O.T.) focal point and providing technical assistance to location management
- Serving as Hazardous Waste Management main contact and providing technical assistance to location management

4.2.15 Laboratory Director (Business Unit Manager or BUMA)

Pittsburgh's Laboratory Director is responsible for the overall quality, safety, financial, technical, human resource and service performance of the whole laboratory and reports to their respective GM. The Laboratory Director is also responsible for any service centers connected with their laboratory that perform any tests, such as short holding time analysis for pH. The Laboratory Director provides the resources necessary to implement and maintain an effective and comprehensive Quality Assurance and Data Integrity Program. The Laboratory Director can also serve as the Technical Manager.

Specific responsibilities include, but are not limited to:

- Providing one or more technical managers for the appropriate fields of testing. If the Technical Manager is absent for a period of time exceeding 15 consecutive calendar days, the Laboratory Director must designate another full time staff member meeting the qualifications of the Technical Manager to temporarily perform this function. If the absence exceeds 20 consecutive calendar days, the primary accrediting authority must be notified in writing.
- Ensuring that all analysts and supervisors have the appropriate education and training to properly carry out the duties assigned to them and ensures that this training has been documented.
- Ensuring that personnel are free from any commercial, financial and other undue pressures which might adversely affect the quality of their work.
- Ensuring Eurofins TestAmerica's human resource policies are adhered to and maintained.
- Ensuring that sufficient numbers of qualified personnel are employed to supervise and perform the work of the laboratory.
- Ensuring that appropriate corrective actions are taken to address analyses identified as requiring such actions by internal and external performance or procedural audits. Procedures that do not meet the standards set forth in the QAM or laboratory SOPs may be temporarily suspended by the Laboratory Director.
- Reviewing and approving all SOPs prior to their implementation and ensures all approved SOPs are implemented and adhered to.
- Pursuing and maintaining appropriate laboratory certification and contract approvals. Supports ISO 17025 requirements.
- Ensuring client specific reporting and quality control requirements are met.
- Captaining the management team, consisting of the QA Manager, the Technical Managers and the Department Managers.
- Monitoring the validity of the analyses performed and data generated in the laboratory.
- Providing training and development programs to applicable laboratory staff as new hires and, on a scheduled basis. Training includes instruction on calculations, instrumentation management to include troubleshooting and preventive maintenance.
- The Technical Manager meets the requirements specified in the Section 5.2.6.1 of the TNI standards.

4.2.16 Quality Assurance (QA) Manager or Designee

The QA Manager has responsibility and authority to ensure the continuous implementation of the quality system at the laboratory where they work. The QA Manager is also responsible for any service centers connected with their laboratory that perform any tests, such as short holding time analysis for pH. The QA Manager reports directly to the Laboratory Director and their Corporate Quality Director. This position is able to evaluate data objectively and perform assessments without outside (e.g., managerial) influence. Corporate QA may be used as a resource in dealing with regulatory requirements, certifications and other quality assurance related items. The QA Manager directs the activities of the QA Specialists to accomplish specific responsibilities, which include, but are not limited to:

- Serving as the focal point for QA/QC in the laboratory.
- Having functions independent from laboratory operations for which he/she has quality assurance oversight.
- Maintaining and updating the QAM.
- Monitoring and evaluating laboratory certifications; scheduling proficiency testing samples.
- Monitoring and communicating regulatory changes that may affect the laboratory to management.
- Training and advising the laboratory staff on quality assurance/quality control procedures that are pertinent to their daily activities.
- Having documented training and/or experience in QA/QC procedures and the laboratory's Quality System.
- Having a general knowledge of the analytical test methods for which data audit/review is performed (and/or having the means of getting this information when needed).
- Arranging for or conducting internal audits on quality systems and the technical operation.
- Maintaining records of all ethics-related training, including the type and proof of attendance.
- Maintaining, improving, and evaluating the corrective action database and the corrective and preventive action systems.
- Notifying laboratory management of deficiencies in the quality system and ensuring corrective action is taken. Procedures that do not meet the standards set forth in the QAM or laboratory SOPs shall be investigated following procedures outlined in Section 12 and if deemed necessary may be temporarily suspended during the investigation.
- Objectively monitoring standards of performance in quality control and quality assurance without outside (e.g., managerial) influence.
- Having the responsibility and final authority to accept or reject data and to stop work in progress in the event that procedures and practices compromise the validity and integrity of analytical data.
- Coordinating of document control of SOPs, MDLs, control limits, and miscellaneous forms and information. Controlling distribution of controlled documents.
- Reviewing a percentage of all final data reports for internal consistency, including Chain of Custody (COC), correspondence with the analytical request, batch QC status, completeness

of any corrective action statements, 5% of calculations, format, holding time, sensibility and completeness of the project file contents.

- Reviewing of external audit reports and data validation requests.
- Following-up with audits to ensure client QAPP requirements are met.
- Establishing reporting schedule and preparation of various quality reports for the Laboratory Director, clients and/or Corporate QA.
- Developing suggestions and recommendations to improve quality systems.
- Researching current state and federal requirements and guidelines.
- Captaining the QA team to enable communication and to distribute duties and responsibilities.
- Ensuring Communication & monitoring standards of performance to ensure that systems are in place to produce the level of quality as defined in this document.
- Evaluating the thoroughness and effectiveness of training.
- Ensuring Compliance with ISO 17025, and other national and state programs, as applicable
- Notifying the accrediting authorities within 30 days of a change in the legal name of the laboratory or a change in any information provided on the application submitted for accreditation.
- Notifying the primary accrediting authority of any change in the laboratories ability to produce valid analytical results that persists for more than 90 calendar days for any analyte/method/matrix combination for which the laboratory holds accreditation.

4.2.17 Technical Director

The Technical Director reports directly to the Laboratory Director. He/she is accountable for all analyses and analysts under their experienced supervision and for compliance with the ISO 17025:2017 standard. He/she serves as a technical resource for Eurofins TestAmerica's personnel and clients in their field of expertise. The scope of responsibility ranges from the new-hire process and existing technology through on-going training and development programs for existing analysts and new instrumentation, Specific responsibilities include, but are not limited to:

- Managing technical projects and evaluating technologies, reviewing technical data
- Solving technical problems in the laboratory including troubleshooting instruments and developing or modifying methods as needed to meet customer requirements.
- Maintaining and repairing analytical instruments to reduce downtime.
- Consulting with clients, regulators, and others regarding technical aspects of analyses.
- Suggesting and implementing process improvements to maximize productivity, save costs, and decrease turn-around time.
- Participating in Eurofins TestAmerica's best practice process to spread best technical practices and developing Eurofins TestAmerica Standard Operating Procedures (SOPs). Leads the implementation and follow-up of the best practices and SOPs in the laboratory.
- Evaluating and adapting new technologies and methodologies. Performs non-routine analysis as required to meet the needs of current long-term clients or as a means to capture new clients in support of business development efforts.
- Training analysts and technicians in area of expertise.
- Assisting with the development of health and safety protocols.

- Consulting with Project Managers and sales staff regarding analytical techniques and capabilities.
- Investigating issues raised by clients, QA, sales, and other departments to find root cause and implement corrective action and proper response.
- Contributing technical information and evaluation for deciding major new equipment purchases and capital expenditures.
- Ensuring compliance with ISO 17025, and other national and state programs.

4.2.18 Quality Assurance Specialist

The QA Specialist is responsible for QA documentation and involvement in the following activities:

- Assisting the QA Manager in performing the annual internal laboratory audits, compiling the evaluation, and coordinating the development of an action plan to address any deficiency identified.
- Facilitating external audits, coordinating with the QA Manager and Laboratory Staff to address any deficiencies noted at the time of the audit and subsequently presented in the final audit report.
- Assisting the QA Manager in the preparation of new SOPs and in the maintenance of existing SOPs, coordinating annual reviews and updates.
- Managing the performance testing (PT) studies, coordinates follow up studies for failed analytes and works with QA Manager and Laboratory Staff to complete needed corrective action reports.
- Assisting with review and maintenance of training records.
- Assisting the Quality Manager and Project Management Group in the review of program plans for consistency with organizational and contractual requirements. Summarize and convey to appropriate personnel anomalies or inconsistencies observed in the review process.
- Assisting with management of and applications for certifications and accreditations.
- Monitoring for compliance the following QA Metrics: temperature monitoring of refrigeration units and incubators; thermometer calibrations; balance calibrations; Eppendorf/pipette calibrations; and proper standard/reagent storage.
- Performing Technical Data Audits and the Audit Miner data file review process for organic instrumentation. Maintain tracking of reviews.
- Assisting with technical review of data packages which require QA review.

4.2.19 Technical Manager / Department Manager

The Technical Manager reports directly to the Laboratory Director. The scope of responsibility ranges from the new-hire training and existing technology through the ongoing training and development programs for existing analysts and new instrumentation and for compliance with the ISO 17025 Standard. Specific responsibilities include, but are not limited to:

- Exercising day-to-day supervision of laboratory operations for the appropriate field of accreditation and reporting of results. Coordinating, writing, and reviewing preparation of all test methods, i. e., SOPs, with regard to quality, integrity, regulatory and optimum and efficient production techniques, and subsequent analyst training and interpretation of the SOPs for implementation and unusual project samples. He/she insures that the SOPs are properly managed and adhered to at the bench. He/she develops standard costing of SOPs

to include supplies, labor, overhead, and capacity (design vs. demonstrated versus first-run yield) utilization.

- Reviewing and approving, with input from the QA Manager, proposals from marketing, in accordance with an established procedure for the review of requests and contracts. This procedure addresses the adequate definition of methods to be used for analysis and any limitations, the laboratory's capability and resources, the client's expectations. Differences are resolved before the contract is signed and work begins. A system documenting any significant changes is maintained, as well as pertinent discussions with the client regarding their requirements or the results of the analyses during the performance of the contract. All work subcontracted by the laboratory must be approved by the client. Any deviations from the contract must be disclosed to the client. Once the work has begun, any amendments to the contract must be discussed with the client and so documented.
- Monitoring the validity of the analyses performed and data generated in the laboratory. This activity begins with reviewing and supporting all new business contracts, insuring data quality, analyzing internal and external non-conformances to identify root cause issues and implementing the resulting corrective and preventive actions, facilitating the data review process (training, development, and accountability at the bench), and providing technical and troubleshooting expertise on routine and unusual or complex problems.
- Providing training and development programs to applicable laboratory staff as new hires and, subsequently, on a scheduled basis. Training includes instruction on calculations, instrumentation management to include troubleshooting and preventive maintenance.
- Enhancing efficiency and improving quality through technical advances and improved LIMS utilization. Capital forecasting and instrument life cycle planning for second generation methods and instruments as well as asset inventory management.
- Coordinating sample management from "cradle to grave," insuring that no time is lost in locating samples.
- Scheduling all QA/QC-related requirements for compliance, e.g., MDLs, etc..
- Captaining department personnel to communicate quality, technical, personnel, and instrumental issues for a consistent team approach.
- Coordinating audit responses with the QA Manager.
- Ensuring compliance with ISO 17025, and other national and state programs

4.2.20 Manger of Project Management

The Manager of Project Management reports directly to the Client Services Director with dotted line reporting to the Laboratory Director. There is an entire staff of Project Managers that makes up the Project Management team. With the overall goal of total client satisfaction. In addition to the responsibilities of the Project Manager, listed in section 4.2.21, the MPM's responsibilities include, but are not limited to:

- Training project managers in technical procedures and promoting the growth of the Project Management Team
- Acting as liaison between laboratory management and the Project Management Team
- Managing human resources for the Project Management Team

4.2.21 Project Manager

The PM reports to the Manager of Project Management and serves as the interface between the laboratory's technical departments and the laboratory's clients. The responsibilities of this position include, but are not limited to:

- Ensuring that clients receive the proper sampling supplies
- Responding to client inquiries concerning sample status
- Assisting clients with the resolution of problems concerning COC
- Ensuring that client specifications, when known, are met by communicating project and quality assurance requirements to the laboratory
- Notifying the supervisors of incoming projects and sample delivery schedules
- Maintaining communication with clients on sample progress from daily status meeting with agreed-upon due dates
- Discussing with client any project-related problems, resolving service issues, and coordinating technical details with the laboratory staff
- Familiarizing laboratory staff with specific quotes, sample log-in review, and final report completeness
- Informing QA Manager of special client requests that are outside of standard operating procedure
- Monitoring the status of all data package projects in-house to ensure timely and accurate delivery of reports
- Informing clients of data package-related problems and resolve service issues
- Coordinating requests for sample containers and other services
- Verifying that subcontract laboratories hold the required certification for all analytes, methods, and matrices to be sent.

4.2.22 Project Manager Assistant (PMA)

The PMA reports to the Manager of Project Management and serves as the interface between the laboratory's technical departments and the laboratory's clients. The responsibilities of this position include, but are not limited to:

- Collating data reports, expanded deliverables and CLP data packages for delivery to clients and reviews for accuracy
- Assisting the CSMs and PMs in the reporting process
- Printing reports as needed for Project Managers
- Monitoring report due dates for timely delivery
- Providing clerical support to the CSMs, PMs and other laboratory staff as needed
- Generating credit or debit invoices to ensure proper payment in compliance with client requirements as established and communicated
- Sending final data to clients via email or courier

4.2.23 Team Leader/Supervisor

The Team Leader/Supervisor reports directly to the Organics or Inorganics Manager and/or Laboratory Director or designee. The responsibilities of this position include, but are not limited to:

- Ensuring that analysts in their department adhere to applicable SOPs and the QA Manual. He/she performs frequent SOP review to determine if analysts are in compliance and if new, modified, and optimized measures are feasible and should be added to these documents.
- Overseeing training, development of performance objectives and standards of performance, appraisal (measurement of objectives), scheduling, counseling, discipline, and motivation of analysts and documents these activities in accordance with systems developed by the QA and Personnel Departments.
- Providing guidance to analysts in resolving problems encountered daily during sample prep/analysis in conjunction with the Technical Manager(s) and/or QA Manager. Each is responsible ensuring 100% implementation of the data review and documentation, non-conformance and corrective action issues, the timely and accurate completion of performance evaluation samples and MDLs, for his/her department.
- Ensuring that all logbooks are maintained, current, and properly labeled or archived.
- Ensuring that all data is properly entered into the LIMS system and is reviewed and approved as required by laboratory documentation policy.
- Reporting all non-conformance conditions to the QA Manager and Department Manager.
- Ensuring that preventive maintenance is performed on instrumentation as detailed in the QA Manual or SOPs. He/she is responsible for developing and implementing a system for preventive maintenance, troubleshooting, and repairing or arranging for repair of instruments.
- Maintaining adequate and valid inventory of reagents, standards, spare parts, and other relevant resources required to perform daily analysis.
- Achieving optimum turnaround time on analyses and compliance with holding times.
- Assisting QA department with root cause investigations and corrective action proposals for responses to external and internal audit issues, system failures and client complaints.

4.2.24 Laboratory Analyst

Laboratory analysts are responsible for conducting analysis and performing all tasks assigned to them by the team leader or supervisor. The responsibilities of the analysts include, but are not limited to:

- Performing analyses by adhering to analytical and quality control protocols prescribed by current SOPs, this QA Manual, and project-specific plans honestly, accurately, timely, safely, and in the most cost-effective manner.
- Ensuring sample and data integrity by adhering to internal chain-of-custody procedures.
- Documenting standard and sample preparation, instrument calibration and maintenance, data calculations, sample matrix effects, and any observed non-conformance on bench sheets, lab notebooks, run logs, and/or the Non-Conformance Database.
- Reporting all non-conformance situations, instrument problems, matrix problems and QC failures, which might affect the reliability of the data, to their supervisor, Department Manager, and/or the QA Manager or member of QA staff.
- Performing 100% review of the data generated prior to entering and submitting for secondary level review. Performs data processing using available tools/software.
- Suggesting method improvements to their supervisor, the Technical Manager (s), and the QA Manager. These improvements, if approved, will be incorporated. Ideas for the

optimum performance of their assigned area, for example, through the proper cleaning and maintenance of the assigned instruments and equipment, are encouraged.

- Working cohesively as a team in their department to achieve the goals of accurate results, optimum turnaround time, cost effectiveness, cleanliness, complete documentation, and personal knowledge of environmental analysis.
    - A "work cell" is considered to be all those individuals who see a sample through the complete process of preparation, extraction, and analysis. To ensure that the entire preparation, extraction, and analysis process is completed by a group of capable individuals, the laboratory shall ensure that each member of the work cell (including a new member entering an already existing work cell) demonstrates capability in his/her area of responsibility in the sequence. Even though the work cell operates as a "team," the demonstration of capability at each individual step in the sequence, as performed by each individual analyst/team member, remains of utmost importance. A work cell may NOT be defined as a group of analysts who perform the same step in the same process (for example, extractions for Method 8270), represented by one analyst who has demonstrated capability for that step.

4.2.25 Sample Management Manager

The Sample Receiving Manager reports to the Laboratory Director and Client Services Manager. The responsibilities of this position include, but are not limited to:

- Ensuring implementation of proper sample receipt procedures, including maintenance of chain-of-custody.
- Reporting nonconformances associated with condition-upon-receipt of samples.
- Ensuring accurate login of samples into TALS.
- Ensuring that all samples are stored in the proper environment.
- Assisting Environmental Health and Safety staff with sample disposal.

4.2.26 Field Service Technician

The Field Service Technicians report to the Sample Management Department Manager. The responsibilities of the Field Service Technician include, but are not limited to:

- Performing sample collection and sample pick-up
- Ensuring sample containers are prepared for sampling
- Performing field tests and measurements and operating and maintaining equipment used for those purposes.

4.2.27 Environmental Health and Safety Coordinator

The Health and Safety Coordinator reports to the Laboratory Director and ensures that systems are maintained for the safe operation of the laboratory. The EH&S Coordinator :

- Conduct ongoing, necessary safety training and conduct new employee safety orientation.
- Assist in developing and maintaining the Chemical Hygiene/Safety Manual.
- Administer dispersal of all Safety Data Sheet (SDS) information.
- Perform regular chemical hygiene and housekeeping instruction.
- Give instruction on proper labeling and practice.
- Serve as chairman of the laboratory safety committee.
- Provide and train personnel on protective equipment.
- Oversee the inspection and maintenance of general safety equipment – fire extinguishers, safety showers, eyewash fountains, etc. and ensure prompt repairs as needed.

- Supervise and schedule fire drills and emergency evacuation drills.
- Determine what initial and subsequent exposure monitoring, if necessary to determine potential employee exposure to chemicals used in the laboratory.
- When determined necessary, conduct exposure monitoring assessments.
- Determine when a complaint of possible over-exposure is "reasonable" and should be referred for medical consultation.
- Assist in the internal and external coordination of the medical consultation/monitoring program conducted by Eurofins TestAmerica's medical consultants.

4.2.28 Hazardous Waste Coordinator

The Hazardous Waste Coordinator reports directly to the Laboratory Director. The duties include, but are not limited to:

- Staying current with the hazardous waste regulations
- Continuing training on hazardous waste issues
- Reviewing and updating annually the Hazardous Waste Contingency Plan in the Environmental Health & Safety Manual
- Auditing the staff with regard to compliance with the Hazardous Waste Contingency Plan
- Contacting the hazardous waste subcontractors for review of procedures and opportunities for minimization of waste

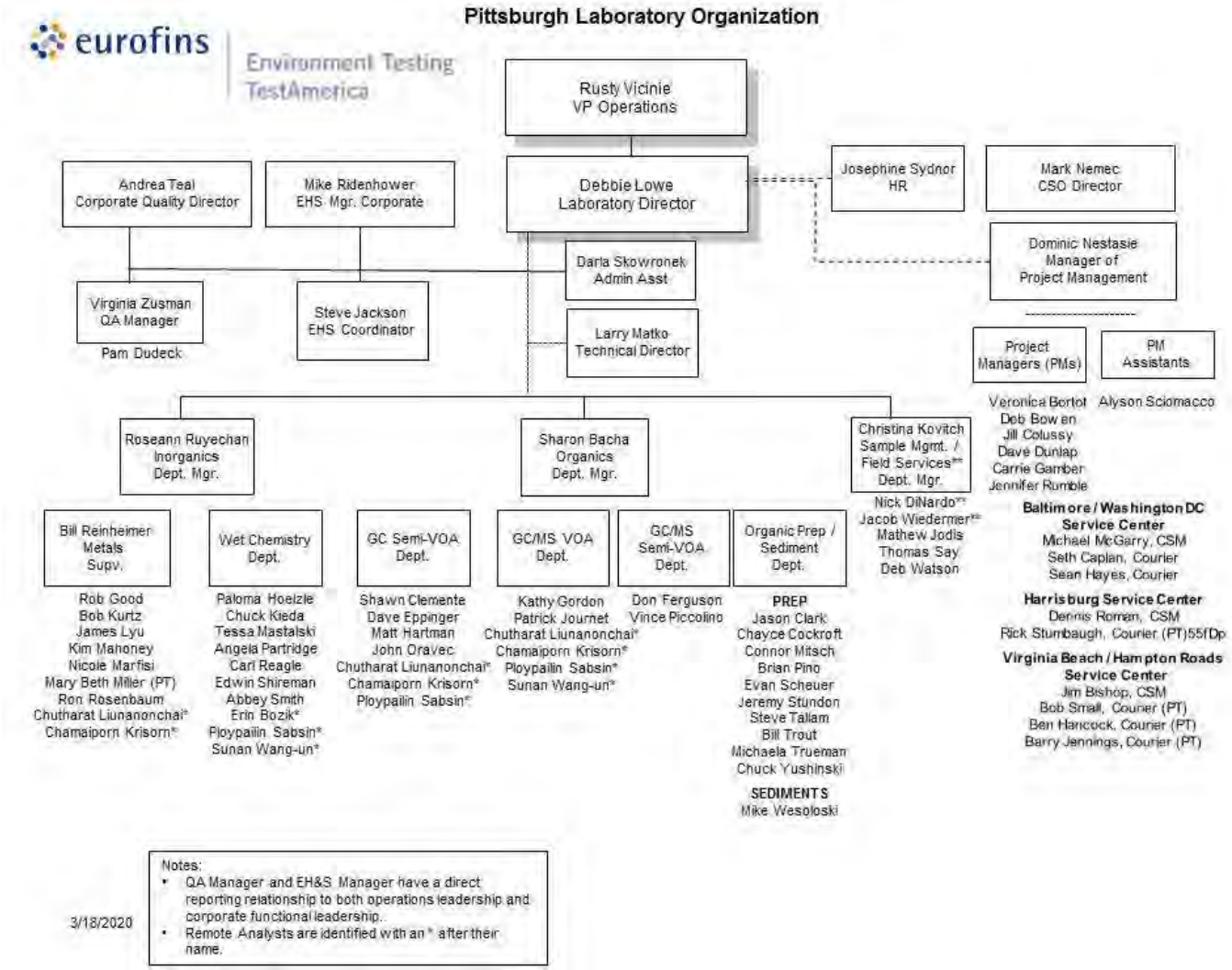
4.3 DEPUTIES

The following table defines who assumes the responsibilities of key personnel in their absence:

Key Personnel	Deputy	Comment
Laboratory Director: Deborah Lowe	Project Technical Manager – Dave Dunlap	NELAP Technical Manager (entire laboratory)
Quality Assurance Manager: Virginia Zusman	Quality Assurance Specialist: Pam Dudeck	
Technical Director: Larry Matko	Laboratory Director: Deborah Lowe	NELAP Technical Manager (Lipids & 8141)
Organics Department Manager: Sharon Bacha	Designated Senior GC and GCMS Analyst	NELAP Technical Manager (Organics)
Inorganics Department Manager: Roseann Ruyechan	Designated Metals and Wet Chemistry Supervisors / Senior Analyst	NELAP Technical Manager (Inorganics, Inorganics-Non-metals)
Organic Prep Team Leader: Sharon Bacha/Larry Matko	Designated Senior Organic Prep Analyst	
Sample Management Department Manager: Christina Kovitch	Lab Director or Designated person in the Sample Management group	

If the NELAP Technical Manager is absent for a period of time exceeding 15 consecutive calendar days, the Laboratory Director must designate another full time staff member meeting the qualifications of the Technical Manager to temporarily perform this function. If the absence exceeds 30 consecutive calendar days, the primary accrediting authority must be notified in writing.

Figure 4-1 - Laboratory Organization Chart



Note: Organization Charts are subject to change - contact the laboratory for the most recent version

SECTION 5

QUALITY SYSTEM

5.1 Quality Policy Statement

It is Eurofins TestAmerica's Policy to provide the highest quality data achievable by:

- Reading and understanding all of the quality documents applicable to each position and implementing the process in our work.
- Following all recordkeeping requirements; describing clearly and accurately all activities performed; recording "real time" as the task is carried out; understanding that it is never acceptable to "back date" entries and should additional information be required at a later date, the actual date and by whom the notation is made must be documented.
- Ensuring data integrity through the completeness, consistency, impartiality and accuracy of the data generated. Data is attributable, legible, contemporaneously recorded, original or a true copy, and accurate (ALCOA). This applies to manual paper documentation and electronic records.
- Providing accountability and traceability for each sample analyzed through proper sample handling, labeling, preparation, instrument calibration/qualification/validation, analysis, and reporting; establishing an audit trail (the who, what, when, and why) that identifies date, time, analyst, instrument used, instrument conditions, quality control samples (where appropriate and/or required by the method), and associated standard material.
- Emphasizing a total quality management process which provides impartiality, accuracy, and strict compliance with agency regulations and client requirements, giving the highest degree of confidence; understanding that meeting the requirements of the next employee in the work flow process is just as important as meeting the needs of the external client.
- Providing thorough documentation and explanation to qualify reported data that may not meet all requirements and specifications, but is still of use to the client; understanding this occurs only after discussion with the client on the data limitations and acceptability of this approach.
- Responding immediately to indications of questionable data, out-of-specification occurrences, equipment malfunctions, and other types of laboratory problems, with investigation and applicable corrective action; documenting these activities completely, including the reasons for the decisions made.
- Providing a work environment that ensures accessibility to all levels of management and encourages questions and expression of concerns on quality issues to management. Eurofins recognizes that the implementation of a quality assurance program requires management's commitment and support as well as the involvement of the entire staff
- Continually improve systems and manage risk to support quality improvement efforts in laboratory, administrative and managerial activities
- We each take personal responsibility to provide this quality product while meeting the company's high standards of integrity and ethics, understanding that improprieties, such as failure to conduct the required test, manipulation of test procedures or data, or inaccurate documentation will not be tolerated. Intentional misrepresentation of the activities performed is considered fraud and is grounds for termination.
- Provide data of known quality to its clients by adhering to approved methodologies, regulatory requirements and the QA/QC protocols.

❖ To comply with the ISO/IEC 17025:2017(E) International Standard, the 2009 and 2016 TNI Standards and to continually improve the effectiveness of the management system.

Every staff member at the laboratory plays an integral part in quality assurance and is held responsible and accountable for the quality of their work. It is, therefore, required that all laboratory personnel are trained and agree to comply with applicable procedures and requirements established by this document.

5.2 Ethics And Data Integrity

Eurofins TestAmerica is committed to ensuring the integrity of its data and meeting the quality needs of its clients. The elements of Eurofins TestAmerica's Ethics and Data Integrity Program include:

- An Ethics Policy (Corporate Policy No. CW-L-P-004) and Employee Ethics Statements
- Ethics and Compliance Officers (ECOs)
- A Training Program
- Self-governance through disciplinary action for violations
- A Confidential mechanism for anonymously reporting alleged misconduct and a means for conducting internal investigations of all alleged misconduct. (Corporate SOP No. CW-L-S-002)
- Procedures and guidance for recalling data if necessary (Corporate SOP No. CW-Q-S-005)
- Effective external and internal monitoring system that includes procedures for internal audits (Section 15 and laboratory SOP PT-QA-002 on Internal Auditing)
- Produce results, which are accurate and include QA/QC information that meets client pre-defined Data Quality Objectives (DQOs)
- Present services in a confidential, honest and forthright manner
- Provide employees with guidelines and an understanding of the Ethical and Quality Standards of our Industry
- Provide procedures and guidance to ensure the impartiality and confidentiality of all data and customer information.
- Operate our facilities in a manner that protects the environment and the health and safety of employees and the public
- Obey all pertinent federal, state and local laws and regulations and encourage other members of our industry to do the same
- Educate clients as to the extent and kinds of services available
- Assert competency only for work for which adequate personnel and equipment are available and for which adequate preparation has been made
- Promote the status of environmental laboratories, their employees, and the value of services rendered by them

5.3 Quality System Documentation

The laboratory's Quality System is communicated through a variety of documents.

- Quality Assurance Manual – Each laboratory has a lab specific quality assurance manual.
- Corporate SOPs and Policies - Corporate SOPs and Policies are developed for use by all relevant laboratories. They are incorporated into the laboratory's normal SOP distribution, training and tracking system. Corporate SOPs may be general or technical.

- Work Instructions - A subset of procedural steps, tasks or forms associated with an operation of a management system (e.g., checklists, preformatted bench sheets, forms).
- Laboratory SOPs – General and Technical
- Laboratory QA/QC Policy Memorandums

5.3.1 Order of Precedence

In the event of a conflict or discrepancy between policies, the order of precedence is as follows:

- Corporate Quality Management Plan (CQMP)
- Corporate SOPs and Policies
- Laboratory QA/QC Policy Memorandum
- Laboratory Quality Assurance Manual (QAM)
- Laboratory SOPs and Policies
- Other (Work Instructions (WI), memos, flow charts, etc.)

Note: The laboratory has the responsibility and authority to operate in compliance with regulatory requirements of the jurisdiction in which the work is performed. Where the CQMP conflicts with those regulatory requirements, the regulatory requirements of the jurisdiction shall hold primacy. The laboratory's QAM shall take precedence over the CQMP in those cases.

5.4 QA/QC Objectives For The Measurement Of Data

Quality Assurance (QA) and Quality Control (QC) are activities undertaken to achieve the goal of producing data that accurately characterize the sites or materials that have been sampled. Quality Assurance is generally understood to be more comprehensive than Quality Control. Quality Assurance can be defined as the integrated system of activities that ensures that a product or service meets defined standards.

Quality Control is generally understood to be limited to the analyses of samples and to be synonymous with the term *"analytical quality control"*. QC refers to the routine application of statistically based procedures to evaluate and control the accuracy of results from analytical measurements. The QC program includes procedures for estimating and controlling precision and bias and for determining reporting limits.

Request for Proposals (RFPs) and Quality Assurance Project Plans (QAPP) provide a mechanism for the client and the laboratory to discuss the data quality objectives in order to ensure that analytical services closely correspond to client needs. In order to ensure the ability of the laboratory to meet the Data Quality Objectives (DQOs) specified in the QAPP, clients are advised to allow time for the laboratory to review the QAPP before being finalized. The client is responsible for developing the QAPP, however, the laboratory will provide support to the client for developing the sections of the QAPP that concern laboratory activities.

Historically, laboratories have described their QC objectives in terms of precision, accuracy, representativeness, comparability, completeness, selectivity and sensitivity.

5.4.1 Precision

The laboratory objective for precision is to meet the performance for precision demonstrated for the methods on similar samples and to meet data quality objectives of the EPA and/or other

regulatory programs. Precision is defined as the degree of reproducibility of measurements under a given set of analytical conditions (exclusive of field sampling variability). Precision is documented on the basis of replicate analysis, usually duplicate or matrix spike (MS) duplicate samples.

5.4.2 Accuracy

The laboratory objective for accuracy is to meet the performance for accuracy demonstrated for the methods on similar samples and to meet data quality objectives of the EPA and/or other regulatory programs. Accuracy is defined as the degree of bias in a measurement system. Accuracy may be documented through the use of laboratory control samples (LCS) and/or MS. A statement of accuracy is expressed as an interval of acceptance recovery about the mean recovery.

5.4.3 Representativeness

The laboratory objective for representativeness is to provide data which is representative of the sampled medium. Representativeness is defined as the degree to which data represent a characteristic of a population or set of samples and is a measurement of both analytical and field sampling precision. The representativeness of the analytical data is a function of the procedures used in procuring and processing the samples. The representativeness can be documented by the relative percent difference between separately procured, but otherwise identical samples or sample aliquots.

The representativeness of the data from the sampling sites depends on both the sampling procedures and the analytical procedures. The laboratory may provide guidance to the client regarding proper sampling and handling methods in order to assure the integrity of the samples.

5.4.4 Comparability

The comparability objective is to provide analytical data for which the accuracy, precision, representativeness and reporting limit statistics are similar to these quality indicators generated by other laboratories for similar samples, and data generated by the laboratory over time.

The comparability objective is documented by inter-laboratory studies carried out by regulatory agencies or carried out for specific projects or contracts, by comparison of periodically generated statements of accuracy, precision and reporting limits with those of other laboratories.

5.4.5 Completeness

The completeness objective for data is 90% (or as specified by a particular project), expressed as the ratio of the valid data to the total data over the course of the project. Data will be considered valid if they are adequate for their intended use. Data usability will be defined in a QAPP, project scope or regulatory requirement. Data validation is the process for reviewing data to determine its usability and completeness. If the completeness objective is not met, actions will be taken internally and with the data user to improve performance. This may take the form of an audit to evaluate the methodology and procedures as possible sources for the difficulty or may result in a recommendation to use a different method.

5.4.6 Selectivity

Selectivity is defined as: The capability of a test method or instrument to respond to a target substance or constituent in the presence of non-target substances. Target analytes are separated from non-target constituents and subsequently identified/detected through one or more of the following, depending on the analytical method: extractions (separation), digestions (separation), interelement corrections (separation), use of matrix modifiers (separation), specific retention times (separation and identification), confirmations with different columns or detectors (separation and identification), specific wavelengths (identification), specific mass spectra (identification), specific electrodes (separation and identification), etc..

5.4.7 Sensitivity

Sensitivity refers to the amount of analyte necessary to produce a detector response that can be reliably detected (above the Method Detection Limit) or quantified (above the Reporting Limit).

5.5 Criteria For Quality Indicators

The laboratory maintains tables, housed in TALS, that summarizes the precision and accuracy acceptability limits for analyses performed at Eurofins TestAmerica Pittsburgh. This summary includes an effective date, is updated each time new limits are generated and is managed by the laboratory's QA department. Limits are archived within the LIMS when replaced. Unless otherwise noted, limits within these tables are laboratory generated. Some acceptability limits are derived from US EPA methods when they are required. Where US EPA method limits are not required, the laboratory has developed limits from evaluation of data from similar matrices. Criteria for development of control limits are contained in laboratory SOP PT-QA-021, *Laboratory Quality Control Program*.

5.6 Statistical Quality Control

Statistically-derived precision and accuracy limits are required by selected methods (such as SW-846) and programs. The laboratory routinely utilizes statistically-derived limits to evaluate method performance and determine when corrective action is appropriate. Analysts are restricted to using only the current limits within TALS, controlled as discussed above. The Quality Assurance department maintains an archive of all limits used within the laboratory. These limits are maintained in TALS as part of the analytical historical record. If a method defines the QC limits, the method limits are used. For further details refer to laboratory SOP PT-QA-021.

If a method, or program, defines the QC limits, the required limits are used, unless laboratory developed limits are tighter.

If a method requires the generation of historical limits, the lab develops such limits from recent data in the QC database of TALS following the guidelines described in Section 24 and laboratory SOP PT-QA-021. All calculations and limits are documented and dated when approved and effective. On occasion, a client may request contract-specified limits for a specific project.

Current QC limits are entered and maintained in TALS analyte database. As sample results and the related QC are entered into TALS the sample QC values are compared with the limits in TALS to determine if they are within the acceptable range. The analyst then evaluates if the

sample needs to be rerun or re-extracted/rerun or if a comment should be added to the report explaining the reason for the QC outlier.

5.6.1 QC Charts

The generation and use of QC Charts (Control Charts) are described in laboratory SOP PT-QA-021. The QA department evaluates control charts to determine if adjustments need to be made to existing limits or corrective actions are necessary. All findings are documented and kept on file.

5.7 Quality System Metrics

In addition to the QC parameters discussed above, the entire Quality System is evaluated on a monthly basis through the use of specific metrics (refer to Section 16). These metrics are used to drive continuous improvement in the laboratory's Quality System.

5.8 Laboratory Certification/Accreditation

The Laboratory Quality System is designed to meet the requirements of all governing bodies through which it holds certification / accreditation.

- A list of certifications and accredited scopes is maintained by the QA Department, and current certificates are posted in the laboratory lobby. Expired certificates are maintained in the QA archive.
- Certification renewal is completed on an annual basis, or within the time frame required by each accrediting agency.
- The laboratory shall read and follow all accrediting agencies' accreditation requirements before applying and when renewing accreditations.
- The laboratory indicates clearly in its reports which certifications it holds,
- The laboratory does not use their NELAP or any state certificate of accreditation, accreditation status, or accrediting agency logo to imply endorsement by any accrediting body.
- The laboratory distinguishes on data reports between testing for which the laboratory is accredited and testing for which the laboratory is not accredited. The laboratory's primary AB accreditation number is also included on all data reports.
- Upon expiration, suspension, revocation or voluntary relinquishment of accreditation, a laboratory shall:
  - Discontinue use of all catalogs, advertising, business solicitations, proposals, quotations, laboratory analytical results or other materials that contain reference to the laboratory's past accreditation status.
  - Discontinue use or display of the Department's logo.
  - Return unexpired certificates of accreditation to the Department within 48 hours.
  - Discontinuing all testing for the affected fields of accreditation.
  - Notify all customers affected by the loss or suspension in writing within 72 hours of receiving notice of the change in accreditation status.

- If there is a change in laboratory ownership, the laboratory must notify all accreditors within 10 days. Some accrediting agencies require additional change applications be submitted within 30 days.

SECTION 6

DOCUMENT CONTROL

6.1 Overview

The QA Department is responsible for the control of documents used in the laboratory to ensure that approved, up-to-date documents are in circulation and out-of-date (obsolete) documents are archived or destroyed. The following documents, at a minimum, must be controlled:

- Laboratory Quality Assurance Manual
- Laboratory Standard Operating Procedures (SOP)
- Laboratory Policies
- Work Instructions and Forms
- Logbooks and Calculation Spreadsheets
- Corporate Policies and Procedures distributed outside the intranet
- External documents that are used as part of the laboratory's Quality System

The Corporate QA Department posts Corporate Manuals, SOPs, Policies, Work Instructions, White Papers and Training Materials on the company intranet site. These Corporate documents are only considered controlled when they are read on the intranet site. Printed copies are considered uncontrolled unless the laboratory physically distributes them as controlled documents. A detailed description of the procedure for issuing, authorizing, controlling, distributing, and archiving Corporate documents is found in Corporate SOP No. CW-Q-S-001, *Corporate Document Control and Archiving*. The laboratory's internal document control procedure is defined in laboratory SOP PT-QA-010, *Document Development and Control*.

The laboratory QA Department also maintains access to various references and document sources integral to the operation of the laboratory. This includes reference methods and regulations. Instrument manuals (hard or electronic copies) are also maintained by the laboratory.

The laboratory maintains control of records for raw analytical data and supporting records such as audit reports and responses, logbooks, standard logs, training files, MDL studies, Proficiency Testing (PT) studies, certifications and related correspondence, and corrective action reports and Nonconformance Memos (NCMs). Raw analytical data consists of bound logbooks, instrument printouts, any other notes, magnetic media, electronic data and final reports.

6.2 Document Approval And Issue

The pertinent elements of a document control system for each document include a unique document title and number, pagination, the total number of pages of the item or an 'end of document' page, the effective date, revision number and the laboratory's name. The QA personnel are responsible for the maintenance of this system.

Controlled documents are authorized by the QA Department. In order to develop a new document, a responsible manager submits an electronic or paper draft to the QA Department for suggestions and approval before use. Upon approval, QA personnel add the identifying version information to the document and retain that document as the official document on file. That document is then provided to all applicable operational units. Controlled documents are

identified as such and records of their distribution are kept by the QA Department. Document control may be achieved by either electronic or hardcopy distribution.

The QA Department maintains a list of the official versions of controlled documents.

Quality System Policies and Procedures will be reviewed at a minimum of every two years, or more often as required, and revised as appropriate. Changes to documents occur when a procedural change warrants.

6.3 Procedures For Document Control Policy

For creation of or changes to SOPs and QA manual, refer to Corporate SOP No. CW-Q-S-002, *Writing a Standard Operating Procedure (SOP)* and laboratory SOP No. PT-QA-010.

Uncontrolled copies must not be used within the laboratory. Controlled documents are marked as such, and posted to a controlled laboratory access drive by the QA department. Controlled distribution is achieved electronically. Controlled hardcopies must be obtained through the QA Department. Previous revisions and back-up data are stored on a restricted access drive by the QA department. Details of the numbering system, required format, and controlled distribution of documents are described in laboratory SOP No. PT-QA-010. Editable copies are stored on a restricted access drive.

Forms, worksheets, work instructions and information are organized by department by the QA office. Controlled electronic versions are distributed through the intranet and hard copies can be printed out as needed. Editable versions are stored on a restricted access drive. All forms used in the laboratory are tracked in the controlled documents database which can be accessed by the QA department and the IT group. The procedure for the care of these documents is in laboratory SOP No. PT-QA-010.

6.4 Obsolete Documents

All invalid or obsolete documents are removed from general laboratory access, or otherwise prevented from unintended use. The laboratory has specific procedures as described above to accomplish this. Obsolete hardcopy documents are collected from employees according to distribution lists and are destroyed. At least one electronic copy of the obsolete document is archived according to laboratory SOP No. PT-QA-019, *Records Information Management*.

SECTION 7

SERVICE TO THE CLIENT

7.1 Overview

The laboratory has established procedures for the review of work requests and contracts, oral or written. The procedures include evaluation of the laboratory's capability and resources to meet the contract's requirements within the requested time period. All requirements, including the methods to be used, must be adequately defined, documented and understood. For many environmental sampling and analysis programs, testing design is site or program specific and does not necessarily fit into a standard laboratory service or product. It is the laboratory's intent to provide both standard and customized environmental laboratory services to our clients.

A thorough review of technical and QC requirements contained in contracts is performed to ensure project success. The appropriateness of requested methods, and the lab's capability to perform them must be established. Projects, proposals and contracts are reviewed for adequately defined requirements and the laboratory's capability to meet those requirements. Alternate test methods that are capable of meeting the clients' requirements may be proposed by the lab. A review of the lab's capability to analyze non-routine analytes is also part of this review process.

All projects, proposals and contracts are reviewed for the client's requirements in terms of compound lists, test methodology requested, sensitivity (detection and reporting levels), accuracy, and precision requirements (%Recovery and RPD). The reviewer ensures that the laboratory's test methods are suitable to achieve these requirements and that the laboratory holds the appropriate certifications and approvals to perform the work. The laboratory and any potential subcontract laboratories must be certified, as required, for all proposed tests.

The laboratory must determine if it has the necessary physical, personnel and information resources to meet the contract, and if the personnel have the expertise needed to perform the testing requested. Each proposal is checked for its impact on the capacity of the laboratory's equipment and personnel. As part of the review, the proposed turnaround time will be checked for feasibility.

Electronic or hard copy deliverable requirements are evaluated against the laboratory's capacity for production of the documentation.

If the laboratory cannot provide all services but intends to subcontract such services, whether to another Eurofins TestAmerica facility or to an outside firm, this will be documented and discussed with the client prior to contract approval. (Refer to Section 8 for Subcontracting Procedures.)

The laboratory informs the client of the results of the review if it indicates any potential conflict, deficiency, lack of accreditation, or inability of the lab to complete the work satisfactorily. Any discrepancy between the client's requirements and the laboratory's capability to meet those requirements is resolved in writing before acceptance of the contract. It is necessary that the contract be acceptable to both the laboratory and the client. Amendments initiated by the client and/or Eurofins TestAmerica, are documented in writing.

All contracts, QAPPs, Sampling and Analysis Plans (SAPs), contract amendments, and documented communications become part of the project record.

The same contract review process used for the initial review is repeated when there are amendments to the original contract by the client, and the participating personnel are informed of the changes.

7.2 Review Sequence And Key Personnel

Appropriate personnel will review the work request at each stage of evaluation.

For routine projects and other simple tasks, a review by the Project Manager (PM) is considered adequate. The PM confirms that the laboratory has any required certifications, that it can meet the clients' data quality and reporting requirements and that the lab has the capacity to meet the clients turn around needs. It is recommended that, where there is a sales person assigned to the account, an attempt should be made to contact that sales person to inform them of the incoming samples.

For new, complex or large projects, the proposed contract is given to the Client Relationship Manager or Proposal Team, who will decide which lab will receive the work based on the scope of work and other requirements, including certification, testing methodology, and available capacity to perform the work. The contract review process is outlined in Eurofins TestAmerica's Corporate Policy No. CA-L-P-002**,** *Contract Compliance Policy*.

This review encompasses all facets of the operation. The scope of work is distributed to the appropriate personnel, as needed based on scope of contract, to evaluate all of the requirements shown above (not necessarily in the order below):

- Contract Administrator
- VP of Operations
- Laboratory Project Management
- Laboratory Director / Technical Manager
- Laboratory and/or Corporate Quality
- Laboratory and/or Corporate EH&S
- The laboratory Director reviews the formal laboratory quote and makes final acceptance for their facility.

The Sales Director, Contract Administrator, Account Executive or Proposal Coordinator then submits the final proposal to the client.

In the event that one of the above personnel is not available to review the contract, his or her back-up will fulfill the review requirements.

7.3 Documentation

The Contracts Department maintains copies of all signed contracts. In the Pittsburgh laboratory, copies of contracts are maintained in the laboratory network public drive by the sales/marketing personnel.

Appropriate records are maintained for every contract or work request. All stages of the contract review process are documented and include records of any significant changes. Contracts review documentation and is maintained in the network public drive.

The contract will be distributed to and maintained by the appropriate sales/marketing personnel and the Account Manager. A copy of the contract and formal quote will be filed with the laboratory PM and the Lab Director.

Records are maintained of pertinent discussions with a client relating to the client's requirements or the results of the work during the period of execution of the contract. The PM keeps a phone log or an electronic record of conversations with the client.

7.3.1 Project-Specific Quality Planning

Communication of contract specific technical and QC criteria is an essential activity in ensuring the success of site specific testing programs. To achieve this goal, a PM is assigned to each client. It is the PM's responsibility to ensure that project-specific technical and QC requirements are effectively evaluated and communicated to the laboratory personnel before and during the project. QA department involvement may be needed to assist in the evaluation of custom QC requirements.

PM's are the primary client contact and they ensure resources are available to meet project requirements. Although PM's do not have direct reports or staff in production, they coordinate opportunities and work with laboratory management and supervisory staff to ensure available resources are sufficient to perform work for the client's project. Project management is positioned between the client and laboratory resources.

Prior to work on a new project, the dissemination of project information and/or project opening meetings may occur to discuss schedules and unique aspects of the project. Items to be discussed may include the project technical profile, turnaround times, holding times, methods, analyte lists, reporting limits, deliverables, sample hazards, or other special requirements. The PM introduces new projects to the laboratory staff through project kick-off meetings or to the supervisory staff during production meetings. These meetings provide direction to the laboratory staff in order to maximize production and client satisfaction, while maintaining quality. In addition, project notes may be associated with each sample batch as a reminder upon sample receipt and analytical processing.

During the project, any change that may occur within an active project is agreed upon between the client/regulatory agency and the PM/laboratory. These changes (e.g., use of a non-standard method or modification of a method) and approvals must be documented prior to implementation. Documentation pertains to any document, e.g., letter, e-mail, variance, contract addendum, which has been signed by both parties.

Such changes are also communicated to the laboratory during production and operations meetings. Such changes are updated to the project notes and are introduced to the managers at

these meetings. The laboratory staff is then introduced to the modified requirements via the PM or the individual laboratory Technical Manager. After the modification is implemented into the laboratory process, documentation of the modification is made in the case narrative of the data report(s).

The laboratory strongly encourages client visits to the laboratory and for formal/informal information sharing session with employees in order to effectively communicate ongoing client needs as well as project specific details for customized testing programs.

7.4 Special Services

The laboratory cooperates with clients and their representatives to monitor the laboratory's performance in relation to work performed for the client. It is the laboratory's goal to meet all client requirements in addition to statutory and regulatory requirements. The laboratory has procedures to ensure confidentiality to clients (Section 25).

The laboratory's standard procedures for reporting data are described in Section 25. Special services are also available and provided upon request. These services include:

- Reasonable access for our clients or their representatives to the relevant areas of the laboratory for the witnessing of tests performed for the client
- Assist client-specified third party data validators as specified in the client's contract
- Supplemental information pertaining to the analysis of their samples

  Note: An additional charge may apply for additional data/information that was not requested prior to the time of sample analysis or previously agreed upon.

When the client requests a statement of conformity to a specification or standard based on the analysis performed by the laboratory (e.g., pass/fail, in-tolerance/out-of-tolerance), the decision rule shall be clearly defined. Unless inherent in the requested specification or standard, the decision rule selected shall be communicated to the client. Associated reporting requirements are addressed in Section 25.2.18.

7.5 Client Communication

Project managers are the primary communication link to the clients. They shall inform their clients of any delays in project completion as well as any non-conformances in either sample receipt or sample analysis. Project management will maintain ongoing client communication throughout the entire client project.

Technical Managers, or their designees, are available to discuss any technical questions or concerns that the client may have.

7.6 Reporting

The laboratory works with our clients to produce any special communication reports required by the contract.

To the extent possible, results of testing or analysis of environmental samples shall be reported only if all quality control, analytical testing and sample acceptance measures are acceptable. If a quality control, analytical testing or sample acceptance measure is

found to be out of control and the results of the testing or analysis of environmental samples are to be reported, all environmental samples associated with the failed quality control measure shall be documented and the results flagged in an unambiguous manner on the sample analysis report with the appropriate data qualifiers.

7.7 Client Surveys

The laboratory assesses both positive and negative client feedback. The results are used to improve overall laboratory quality and client service. Eurofins TestAmerica's Sales and Marketing teams periodically develops lab and client specific surveys to assess client satisfaction. Survey results and both complaints and compliments are tracked in the monthly quality metrics report.

SECTION 8

SUBCONTRACTING OF TESTS

8.1 Overview

For the purpose of this quality manual, the phrase subcontract laboratory refers to a laboratory external to the Eurofins TestAmerica laboratories. To distinguish between an external laboratory and a Eurofins TestAmerica laboratory at a different location, the phrase "work sharing" or "work share lab" refers to internal subcontracting between Eurofins TestAmerica laboratories. The term outsourcing refers to the act of subcontracting tests.

When contracting with our clients, the laboratory makes commitments regarding the services to be performed and the data quality for the results to be generated. When the need arises to outsource testing for our clients because project scope, changes in laboratory capabilities, capacity or unforeseen circumstances, we must be assured that the subcontractors or work sharing laboratories understand the requirements and will meet the same commitments we have made to the client. Refer to Corporate SOP No. CA-L-S-004, *Subcontracting*, and CA-C-S-001, *Worksharing Process*.

When outsourcing analytical services, the laboratory will assure, that the subcontract or work sharing laboratory maintains a program consistent with the requirements of this document, the requirements specified in TNI/ISO 17025 and/or the client's Quality Assurance Project Plan (QAPP). All QC guidelines specific to the client's analytical program are transmitted to the subcontractor or work share lab and agreed upon before sending the samples to the subcontract facility. Additionally, work requiring accreditation will be placed with an appropriately accredited laboratory. The laboratory performing the subcontracted work will be identified in the final report, as will non-TNI accredited work where required.

Project Managers (PMs) and Account Executives (AE) for the Export Lab (i.e. the Eurofins TestAmerica laboratory that transfers samples to another laboratory) are responsible for obtaining client approval prior to subcontracting or work sharing any samples. The laboratory will advise the client of a subcontract arrangement in writing and when possible approval from the client shall be obtained and retained in the project folder. These approvals may be granted by the client in written contractual agreements between the client and Eurofins TestAmerica.

Note: In addition to the client, some regulating agencies, (e.g, USDA) or contracts may require notification prior to placing such work**.** Client notification and approval must be documented.

8.2 Qualifying And Monitoring Subcontractors

Whenever a PM, Account Executive (AE) or Customer Service Manager (CSM) becomes aware of a client requirement or laboratory need where samples must be outsourced to another laboratory, the other laboratory(s) shall be selected based on the following:

- Subcontractors specified by the client – In these circumstances, the client assumes responsibility for the quality of the data generated from the use of a subcontractor.
- Subcontractors reviewed by Eurofins TestAmerica – Firms which have been reviewed by the company and are known to meet standards for accreditations (e.g. State, TNI); technical specifications; legal and financial information.

A list of approved vendors is available on the Eurofins TestAmerica intranet site. Approved vendors' capabilities and certifications are found in the individual folders maintained on that site.

All Eurofins TestAmerica laboratories are pre-qualified for work sharing provided they hold the appropriate accreditations and can adhere to the project/program requirements. The originating laboratory is responsible for communicating all technical, quality, and deliverable requirements as well as other contract needs. (See Corporate SOP No. CA-C-S-001, *Work Sharing Process*, for details).

8.2.1 When the potential sub-contract laboratory has not been previously approved, Account Executives or PMs may nominate a laboratory as a subcontractor based on need. The decision to nominate a laboratory must be approved by the Client Relations Manager (CRM) or Laboratory Director. The CRM or Laboratory Director requests that the QA Manager or Project Manager begin the process of approving the subcontract laboratory as outlined in Corporate SOP No. CA-L-S-004, Subcontracting. The client must provide acknowledgement that the samples can be sent to that facility (an e-mail is sufficient documentation or if acknowledgement is verbal, the date, time, and name of person providing acknowledgement must be documented).

Once the appropriate accreditation and legal information is received by the laboratory, it is evaluated for acceptability and forwarded to the Corporate Quality Information Manager (QIM) for review. After the Corporate QIM reviews the documents for completeness, the information is forwarded to the Finance Department for formal signature and contracting with the laboratory. The approved vendor will be added to the approved subcontractor list on the intranet site and the finance group is concurrently notified.

The client will assume responsibility for the quality of the data generated from the use of a subcontractor they have requested the lab to use. The qualified subcontractors on the intranet site are known to meet minimal standards. Eurofins TestAmerica does not certify laboratories. The subcontractor is on our approved list and can only be recommended to the extent that we would use them.

8.3 Oversight and Reporting

8.3.1 The status and performance of qualified subcontractors will be monitored periodically by the Corporate Quality department and includes an annual review process (see Subcontracting SOP CW-L-S-004). Any problems identified will be brought to the attention of Eurofins TestAmerica's Corporate Finance, Legal or Corporate Quality personnel.

- Complaints shall be investigated. Documentation of the complaint, investigation and

corrective action will be maintained in the subcontractor's file on the intranet site. Complaints are posted using the Vendor Performance Report.

- Information shall be updated on the intranet when new information is received from the subcontracted laboratories.
- Subcontractors in good standing will be retained on the intranet listing. CSO personnel will notify all Eurofins TestAmerica laboratories, Corporate Quality and Corporate Contracts if any laboratory requires removal from the intranet site. This notification will be posted on the intranet site and e-mailed to all CSO personnel, Laboratory Directors, QA Managers, and Sales Personnel.

Prior to initially sending samples to the subcontracted laboratory, the PM confirms their certification status to determine if it's current and scope-inclusive. The information is documented within the project records.

8.3.2 For continued use of an approved external subcontractor, current certifications are viewed by the responsible Project Manager on the intranet site. For Eurofins TestAmerica laboratories, certifications can be viewed on through TALS.

8.3.3 All subcontracted samples must be accompanied by a Eurofins TestAmerica Chain of Custody (COC). A copy of the original COC sent by the client must be available in TALS for all samples workshared within Eurofins TestAmerica. Client COCs are only forwarded to external subcontractors when samples are shipped directly from the project site to the subcontractor lab. Under routine circumstances, client COCs are not provided to external subcontractors.

Through communication with the subcontracted laboratory, the PM monitors the status of the subcontracted analyses, facilitates successful execution of the work, and ensures the timeliness and completeness of the analytical report.

Non-TNI accredited work must be identified in the subcontractor's report as appropriate. If TNI accreditation is not required, the report does not need to include this information.

Reports submitted from subcontractor laboratories are not altered and are included in their original form in the final project report. This clearly identifies the data as being produced by a subcontractor facility. If subcontract laboratory data is incorporated into the laboratories EDD (i.e., imported), the report must explicitly indicate which lab produced the data for which methods and samples.

Note: The results submitted by a Eurofins TestAmerica work sharing laboratory may be transferred electronically and the results reported by the Eurofins TestAmerica work sharing lab are identified on the final report. The report must explicitly indicate which lab produced the data for which methods and samples. The final report must include a copy of the completed COC for all work sharing reports.

If a report including workshare data is sent to West Virginia clients, then individual reports per laboratory must be generated.

Note: If samples are subcontracted for SDWA compliance testing, the following information must be provided to the subcontract laboratory along with all other required chain-of-custody information: Public Water System ID Number and name of the system providing the sample, sample location ID number, and name and contact number of the Public Water System.

8.4 Contingency Planning

The full qualification of a subcontractor may be waived to meet emergency needs. This decision & justification must be documented in the project files, and the 'Purchase Order Terms And Conditions For Subcontracted Laboratory Services' must be sent with the samples and COC.

In the event this provision is utilized, the laboratory (e.g., PM) will be required to verify and document the applicable accreditations of the subcontractor. All other quality and accreditation requirements will still be applicable, but the subcontractor need not have signed a subcontract agreement with Eurofins TestAmerica at this time.

The use of any emergency subcontractor will require the PM to complete a JDE New Vendor Add Form in order to process payment to the vendor and add them to TALS. This form requires the user to define the subcontractor's category/s of testing and the reason for testing.

SECTION 9

PURCHASING SERVICES AND SUPPLIES

9.1 Overview

Evaluation and selection of suppliers and vendors is performed, in part, on the basis of the quality of their products, their ability to meet the demand for their products on a continuous and short term basis, the overall quality of their services, their past history, and competitive pricing. This is achieved through evaluation of objective evidence of quality furnished by the supplier, which can include certificates of analysis, recommendations, and proof of historical compliance with similar programs for other clients. To ensure that quality critical consumables and equipment conform to specified requirements, which may affect quality, all purchases from specific vendors are approved by a member of the supervisory or management staff. Capital expenditures are made in accordance with Eurofins TestAmerica's *Fixed Asset Acquisitions, Retention and Safeguarding* SOP No. CW-F-S-007.

Contracts will be signed in accordance with Eurofins TestAmerica's Corporate Policy No. CW-F-P-002, *Company-Wide Authorization Matrix*. Request for Proposals (RFP's) will be issued where more information is required from the potential vendors than just price. Process details are available in Eurofins TestAmerica's Corporate Policy No. CW-F-P-004, *Procurement and Contracts Policy*. RFP's allow Eurofins TestAmerica to determine if a vendor is capable of meeting requirements such as supplying all of the Eurofins TestAmerica facilities, meeting required quality standards and adhering to necessary ethical and environmental standards. The RFP process also allows potential vendors to outline any additional capabilities they may offer.

9.2 Glassware

Glassware used for volumetric measurements must be Class A or verified for accuracy according to laboratory procedure. Pyrex (or equivalent) glass should be used where possible. For safety purposes, thick-wall glassware should be used where available.

9.3 Reagents, Standards & Supplies

Purchasing guidelines for equipment and reagents must meet the requirements of the specific method and testing procedures for which they are being purchased. Solvents and acids are pre-

tested in accordance with Eurofins TestAmerica's Corporate SOP No. CA-Q-S-001, *Solvent & Acid Lot Testing and Approval Program*. Approval information for the solvents and acids tested under Corporate SOP No. CA-Q-S-001 is stored on the Eurofins TestAmerica intranet SharePoint, under Solvent Approvals. A master list of all tested materials, as well as the certificates of analysis for the materials, is stored in the same location. Additional approval and traceability instructions are found in laboratory SOP PT-QA-006, *Procurement of Standards and Materials; Labeling and Traceability*.

9.3.1 Purchasing

Chemical reagents, solvents, glassware, and general supplies are ordered as needed to maintain sufficient quantities on hand. Materials used in the analytical process must be of a known quality. The wide variety of materials and reagents available makes it advisable to specify recommendations for the name, brand, and grade of materials to be used in any determination. This information is contained in the method SOP. The analyst completes the Material Request Sheet when requesting reagents, standards, or supplies, or they may check the item out of the on-site consignment system that contains items approved for laboratory use.

If an item is not in the consignment system, the analyst must obtain approval from the area team leader/supervisor and Laboratory Director prior to the order being placed by the purchasing department. The responsible analyst places the order.

9.3.2 Receiving

It is the responsibility of the Sample Receiving department to receive the shipment. It is the responsibility of the analyst or manager who ordered the materials to document the date the materials were received. Once the ordered reagents or materials are received, the analyst or designee compares the information on the label or packaging to the original order to ensure that the purchase meets the quality level specified. This is documented through the addition of the received date and reviewers initials to the packing slip.

Lot numbers of solvents and acids are checked against the approved solvent/acid list. If the material is listed as unapproved, or is not listed, it is sequestered and returned to the vendor. Alternatively, the laboratory may test the material for the intended use, and if it is acceptable, document the approval on the approval list. Records of any testing performed locally are maintained by the QA department, and lots approvals and certificates of analysis are stored on the Eurofins TestAmerica intranet Pittsburgh SharePoint site.

Materials may not be released for use in the laboratory until they have been inspected, verified as suitable for use, and the inspection/verification has been documented.

Safety Data Sheets (SDSs) are available online through the Company's intranet website. Anyone may review these for relevant information on the safe handling and emergency precautions of on-site chemicals.

9.3.3 Specifications

Methods in use in the laboratory specify the grade of reagent that must be used in the procedure. If the quality of the reagent is not specified, analytical reagent grade will be used. It is the responsibility of the analyst to check the procedure carefully for the suitability of grade of reagent.

Chemicals must not be used past the manufacturer's expiration date and must not be used past the expiration time noted in a method SOP. If expiration dates are not provided, the laboratory may contact the manufacturer to determine an expiration date.

The laboratory assumes a five year expiration date on inorganic dry chemicals and solvents unless noted otherwise by the manufacturer or by the reference source method. Chemicals/solvents should not be used past the manufacturer's or SOPs expiration date unless verified as outlined below.

- An expiration date **cannot** be extended if the dry chemical/solvent is discolored or appears otherwise physically degraded. In this case, the dry chemical/solvent must be discarded.

- Expiration dates can be extended if the dry chemical/solvent is found to be satisfactory based on acceptable performance of quality control samples (Continuing Calibration Verification (CCV), Blanks, Laboratory Control Sample (LCS), etc.).

- If the dry chemical/solvent is used for the preparation of standards, the expiration dates can be extended 6 months if the dry chemical/solvent is compared to an unexpired independent source in performing the method and the performance of the dry chemical/solvent is found to be satisfactory. The comparison must show that the dry chemical/solvent meets CCV limits. The comparison studies are maintained on-file and available for review with each lab department and copy forwarded to QA department. Recertification is documented in TALS.

Wherever possible, standards must be traceable to national or international standards of measurement or to national or international reference materials. Records to that effect are available to the user.

Compressed gases in use are checked for pressure and secure positioning daily. To prevent a tank from going to dryness, or introducing potential impurities, the pressure should be closely watched as it decreases to approximately 15% of the original reading, at which point it should be replaced. For example, a standard sized laboratory gas cylinder containing 3,000 psig of gas should be replaced when it drops to approximately 500 psig. The quality of the gases must meet method or manufacturer specifications or be of a grade that does not cuase any analytical interference.

Water used in the preparation of samples, standards or reagents must have a specific conductivity of less than 1- µmho /cm (or specific resistivity of greater than 1.0 megohm-cm) at 25^{o}C. The specific conductivity is checked and recorded daily. If the water's specific conductivity is greater than the specified limit, the Facility Manager and appropriate Technical Managers/Supervisors must be notified immediately in order to notify all departments, decide on cessation (based on intended use) of activities, and make arrangements for correction.

The laboratory may purchase reagent grade (or other similar quality) water for use in the laboratory. This water must be certified clean by the supplier for all target analytes or otherwise verified by the laboratory prior to use. This verification is documented.

Standard lots are verified before first time use if the laboratory switches manufacturers or has historically had a problem with the type of standard.

Purchased bottleware used for sampling must be certified clean and the certificates must be maintained. If uncertified sampling bottleware is purchased, all lots must be verified clean prior to use. This verification must be maintained.

NOTE: Each bottleware type must be documented as clean down to the laboratory MDL for all target analytes for use with samples from Wisconsin.

Records of manufacturer's certification and traceability statements are maintained in files or binders in each laboratory section. These records include date of receipt, lot number (when applicable), and expiration date (when applicable). Incorporation of the item into the record indicates that the analyst has compared the new certificate with the previous one for the same purpose and that no difference is noted, unless approved and so documented by the Department Manager or QA Manager.

9.3.4 Storage

Reagent and chemical storage is important from the aspects of both integrity and safety. Light-sensitive reagents may be stored in brown-glass containers. Storage conditions are per the Corporate Environmental Health & Safety Manual (Corp. Doc. No. CW-E-M-001), method SOPs, or manufacturer instructions.

9.4 Purchase Of Equipment / Instruments / Software

When a new piece of equipment is needed, either for additional capacity or for replacing inoperable equipment, the analyst or supervisor makes a supply request to the Department Manager and/or the Laboratory Director. If they agree with the request, the procedures outlined in Corporate Policy No. CA-T-P-001, *Qualified Products List*, are followed. A decision is made as to which piece of equipment can best satisfy the requirements. The appropriate written requests are completed and purchasing places the order.

Upon receipt of a new or used piece of equipment, an identification name is assigned and added to the equipment list. IT must also be notified so that they can synchronize the instrument for back-ups. Its capability is assessed to determine if it is adequate or not for the specific application. For instruments, a calibration curve is generated, followed by MDLs, Demonstration of Capabilities (DOCs), and other relevant criteria (refer to Section 19).

For software, its operation must be deemed reliable and evidence of instrument verification must be retained by the IT Department. Software certificates supplied by the vendors are filed with the LIMS Administrator. The manufacturer's operation manual is retained in the laboratory in a designated area or near the instrument.

9.5 Services

Service to analytical instruments (except analytical balances) is performed on an as needed basis. Routine preventative maintenance is discussed in Section 20. The need for service is determined by analysts and/or Technical Managers. The service providers that perform the services are approved by the Laboratory or Technical Director.

Analytical balances are serviced and calibrated annually in accordance with laboratory SOP No. PT-QA-012, *Selection and Calibration of Balances and Weights*. The calibration and maintenance services are performed on-site, and the balances are returned to use immediately

following successful calibration. When the calibration certificates are received (usually within 24 hours of the service), they are reviewed, and documentation of the review is filed with the certificates. If the calibration was unsuccessful, the balance is immediately removed from service and segregated pending either further maintenance or disposal.

Calibration services for support equipment such as thermometers, weight sets, autopipettors, etc, are obtained from vendors with current and valid ISO 17025 accreditation for calibration of the specific piece of equipment. Prior to utilizing the vendor's services, the vendor's accreditation status is verified. Once the equipment has been calibrated, the calibration certificates are reviewed by the QA department, and documentation of the review is filed with the calibration certificates. The equipment is then returned to service within the laboratory.

9.6 Suppliers

Eurofins TestAmerica selects vendors through a competitive proposal / bid process, strategic business alliances or negotiated vendor partnerships (contracts). This process is defined in the Corporate Policy No. CW-F-P-004. The level of control used in the selection process is dependent on the anticipated spending amount and the potential impact on Eurofins TestAmerica business. Vendors that provide test and measuring equipment, solvents, standards, certified containers, instrument related service contracts or subcontract laboratory services shall be subject to more rigorous controls than vendors that provide off-the-shelf items of defined quality that meet the end use requirements. The purchasing system includes all suppliers/vendors that have been approved for use.

Evaluation of suppliers is accomplished by ensuring the supplier ships the product or material ordered and that the material is of the appropriate quality. This is documented by signing off on packing slips or other supply receipt documents. The purchasing documents contain the data that adequately describe the services and supplies ordered.

Any issues of vendor performance are to be reported immediately by the laboratory staff to the Corporate Purchasing Group by completing a Vendor Performance Report.

The Corporate Purchasing Group will work through the appropriate channels to gather the information required to clearly identify the problem and will contact the vendor to report the problem and to make any necessary arrangements for exchange, return authorization, credit, etc.

Suppliers are subject to re-evaluation, as deemed appropriate, through the use of Vendor Performance Reports used to summarize and review to determine corrective action necessary, or service improvements required by vendors.

The laboratory has access to a listing of all approved suppliers of critical consumables, supplies and services. This information is provided through the ORACLE purchasing system.

9.6.1 New Vendor Procedure

Eurofins TestAmerica employees who wish to request the addition of a new vendor must complete a Vendor Add Request Form.

New vendors are evaluated based upon criteria appropriate to the products or services provided as well as their ability to provide those products and services at a competitive cost. Vendors are also evaluated to determine if there are ethical reasons or potential conflicts of interest with Eurofins TestAmerica employees that would make it prohibitive to do business with them as well as their financial stability. The QA Department and/or the Technical Services Director are consulted with vendor and product selection that have an impact on quality.

SECTION 10

COMPLAINTS

10.1 Overview

The laboratory considers an effective client complaint handling processes to be of significant business and strategic value. Listening to and documenting client concerns captures client knowledge that enables our operations to continually improve processes and client satisfaction. An effective client complaint handling process also provides assurance to the data user that the laboratory will stand behind its data, service obligations and products.

A client complaint is any expression of dissatisfaction with any aspect of our business services (e.g., communications, responsiveness, data, reports, invoicing and other functions) expressed by any party, whether received verbally or in written form. Client inquiries, complaints or noted discrepancies are documented, communicated to management, and addressed promptly and thoroughly.

The laboratory has procedures for addressing both external and internal complaints with the goal of providing satisfactory resolution to complaints in a timely and professional manner.

The nature of the complaint is identified, documented and investigated, and an appropriate action is determined and taken. In cases where a client complaint indicates that an established policy or procedure was not followed, the QA Department must evaluate whether a special audit must be conducted to assist in resolving the issue. A written confirmation or letter to the client, outlining the issue and response taken is recommended as part of the overall action taken.

The process of complaint resolution and documentation utilizes the procedures outlined in Section 12 (Corrective Actions) and is documented following laboratory SOP PT-QA-016, *Non-Conformance and Corrective Action System*. A copy of this procedure will be made available to any interested party on request.

10.2 External Complaints

An employee that receives a complaint initiates the complaint resolution process by first documenting the complaint in the database, according to laboratory SOP PT-QA-016.

Complaints fall into two categories: correctable and non-correctable. An example of a correctable complaint would be one where a report re-issue would resolve the complaint. An example of a non-correctable complaint would be one where a client complains that their data was repeatedly late. Non-correctable complaints should be reviewed for preventive action measures to reduce the likelihood of future occurrence and mitigation of client impact.

The general steps in the complaint handling process are:

- Receiving and Documenting Complaints
- Acknowledge receipt of complaint, whenever possible
- Complaint Investigation and Service Recovery
- Process Improvement

The laboratory shall inform the initiator of the complaint of the results of the investigation and the corrective action taken, if any.

10.3 Internal Complaints

Internal complaints include, but are not limited to: errors and non-conformances, training issues, internal audit findings, and deviations from methods. Corrective actions may be initiated by any staff member who observes a nonconformance and shall follow the procedures outlined in Section 12. In addition, Corporate Management, Sales and Marketing and IT may initiate a complaint by contacting the laboratory or through the corrective action system described in Section 12.

10.4 Management Review

The number and nature of client complaints is reported by the QA Manager to the Laboratory Director and Quality Director in the QA Monthly report. Monitoring and addressing the overall level and nature of client complaints and the effectiveness of the solutions is part of the Annual Management Systems Review (Section 16).

SECTION 11

CONTROL OF NON-CONFORMING WORK

11.1 Overview

When data discrepancies are discovered or deviations and departures from laboratory SOPs, policies and/or client requests have occurred, corrective action is taken immediately. First, the laboratory evaluates the significance of the nonconforming work. Then, a corrective action plan is initiated based on the outcome of the evaluation. If it is determined that the nonconforming work is an isolated incident, the plan could be as simple as adding a qualifier to the final results and/or making a notation in the case narrative. If it is determined that the nonconforming work is a systematic or improper practices issue, the corrective action plan could include a more in depth investigation and a possible suspension of an analytical method. In all cases, the actions taken are documented using the laboratory's corrective action system (refer to Section 12).

Due to the frequently unique nature of environmental samples, sometimes departures from documented policies and procedures are needed. When an analyst encounters such a situation, the problem is presented to their manager for advice. The manager may elect to discuss it with the Laboratory Director or QA Manager or have a PM contact the client to decide on a logical course of action. Once an approach is agreed upon, the analyst documents it using the laboratories non-conformance/corrective action system described in Section 12. This information can then be supplied to the client in the form of a case narrative with the report.

Project Management may encounter situations where a client may request that a special procedure be applied to a sample that is not standard lab practice. Based on a technical evaluation, the lab may accept or opt to reject the request based on technical or ethical merit. An example might be the need to report a compound that the lab does not normally report. The lab would not have validated the method for this compound following the procedures in Section 19. The client may request that the compound be reported based only on the calibration. Such a request would need to be approved by the Laboratory Director and QA Manager, documented and included in the project folder. Deviations must also be noted on the final report with a statement that the compound is not reported in compliance with TNI (or the analytical method) requirements and the reason. Data being reported to a non-TNI state would need to note the change made to how the method is normally run.

11.2 Responsibilities And Authorities

Under certain circumstances, the Laboratory Director, a Technical Manager, or a member of the QA team may authorize departures from documented procedures or policies. The departures may be a result of procedural changes due to the nature of the sample; a one-time procedure for a client; QC failures with insufficient sample to reanalyze, etc. In most cases, the client will be informed of the departure prior to the reporting of the data. Any departures must be well documented using the laboratory's corrective action procedures. This information is documented on a Nonconformance Memo (NCM) and may also be documented in logbooks and/or data review checklists as appropriate. Any impacted data must be referenced in a case narrative and/or flagged with an appropriate data qualifier.

Any misrepresentation or possible misrepresentation of analytical data discovered by any laboratory staff member must be reported to facility Senior Management within 24-hours. The Senior Management staff is comprised of the Laboratory Director, the QA Manager, and the Technical Managers. The reporting of issues involving alleged violations of the company's Data Integrity or Manual Integration procedures must be conveyed to an ECO (e.g., the VP-QA/EHS) and the laboratory's Quality Director within 24 hours of discovery.

Whether an inaccurate result was reported due to calculation or quantitation errors, data entry errors, improper practices, or failure to follow SOPs, the data must be evaluated to determine the possible effect.

The Laboratory Director, QA Manager, ECOs, VP of Operations, and the Quality Directors have the authority and responsibility to halt work, withhold final reports, or suspend an analysis for due cause as well as authorize the resumption of work.

11.3 Evaluation Of Significance And Actions Taken

For each nonconforming issue reported, an evaluation of its significance and the level of management involvement needed is made. This includes reviewing its impact on the final data, whether or not it is an isolated or systematic issue, and how it relates to any special client requirements.

Corporate SOP entitled Data Recalls (CW-Q-S-005) is the procedure to be followed when it is discovered that erroneous or biased data may have been reported to clients or regulatory agencies.

Corporate SOP entitled Internal Investigations (CW-L-S-002) is the procedure to be followed for investigation and correction of situations involving alleged incidents of misconduct or violation of the company's ethics policy.

Laboratory level decisions are documented and approved using the laboratory's standard nonconformance/corrective action reporting in lieu of the data recall determination form contained in Eurofins TestAmerica's Corporate SOP No. CW-Q-S-005.

11.4 Prevention Of Nonconforming Work

If it is determined that the nonconforming work could recur, further corrective actions must be made following the laboratory's corrective action system. On at least a monthly basis, the QA Department evaluates non-conformances to determine if any nonconforming work has been repeated multiple times. If so, the laboratory's corrective action process must be followed.

11.5 Method Suspension/Restriction (Stop Work Procedures)

In some cases, it may be necessary to suspend/restrict the use of a method or target analyte which constitutes significant risk and/or liability to the laboratory. Suspension/restriction procedures can be initiated by any of the persons noted in Section 11.2, Paragraph 4.

Prior to suspension/restriction, confidentiality will be respected, and the problem with the required corrective and preventive action will be stated in writing and presented to the Laboratory Director.

The Laboratory Director shall arrange for the appropriate personnel to meet with the QA Manager as needed. This meeting shall be held to confirm that there is a problem, that suspension/restriction of the method is required and will be concluded with a discussion of the steps necessary to bring the method/target or test fully back on line. In some cases, that may not be necessary if all appropriate personnel have already agreed there is a problem and there is agreement on the steps needed to bring the method, target or test fully back on line. The QA Manager will also initiate a corrective action report as described in Section 12 if one has not already been started. A copy of any meeting notes and agreed upon steps should be e-mailed by the laboratory to the appropriate VP of Operations and member of Corporate QA. This e-mail acts as notification of the incident.

After suspension/restriction, the lab will hold all reports to clients pending review. No faxing, mailing or distributing through electronic means may occur. The report must not be posted for viewing on the internet. It is the responsibility of the Laboratory Director to hold all reporting and to notify all relevant laboratory personnel regarding the suspension/restriction (e.g., Project Management, Log-in, etc.). Clients will NOT generally be notified at this time. Analysis may proceed in some instances depending on the non-conformance issue.

Within 72 hours, the QA Manager will determine if compliance is now met and reports can be released, OR determine the plan of action to bring work into compliance, and release work. A team, with all principals involved (e.g., Laboratory Director, Technical Manager, QA Manager) can devise a start-up plan to cover all steps from client notification through compliance and release of reports. Project Management and the Directors of Client Services and Sales and Marketing must be notified if clients must be notified or if the suspension/restriction affects the laboratory's ability to accept work. The QA Manager must approve start-up or elimination of any restrictions after all corrective action is complete.

SECTION 12

CORRECTIVE ACTION

12.1 Overview

A major component of Eurofins TestAmerica's Quality Assurance (QA) Program is the problem investigation and feedback mechanism designed to keep the laboratory staff informed on quality related issues and to provide insight to problem resolution. When nonconforming work or departures from policies and procedures in the quality system or technical operations are identified, the corrective action procedure provides a systematic approach to assess the issues, restore the laboratory's system integrity, and prevent reoccurrence. Eurofins TestAmerica employs two systemsto manage non-conformances. Issues suspected of being systematic in nature and for which root cause analysis and a formal Corrective Action Report (CAR) are needed are documented in the Incident Corrective Action Tracking (ICAT) database. Routine batch non-conformances, events that are understood to be isolated in nature, are documented in the TALS non-conformance memo (NCM) system. See Figure 12-1 for an example CAR.

12.2 General

Problems within the quality system or within analytical operations may be discovered in a variety of ways, such as QC sample failures, internal or external audits, proficiency testing (PT) performance, client complaints, staff observation, etc.

The purpose of a corrective action system is to:

- Identify non-conformance events and assign responsibility(s) for investigating.
- Resolve non-conformance events and assign responsibility for any required corrective action.
- Identify systematic problems before they become serious.
- Identify and track client complaints and provide resolution.

12.2.1 Non-Conformance Memo (NCM) - is used to document the following types of corrective actions:

- Isolated deviations from an established procedure or SOP
- QC outside of limits (non-matrix related)
- Isolated reporting / calculation errors
- Client Complaints
- Discrepancies in materials / goods received vs. manufacturer packing slips (Forms of documentation other than NCMs in TALS are also acceptable.)
- Anomalies that occur during sample receipt, preparation or analysis

12.2.2 Corrective Action Documented in the ICAT Database

- Internal and external audit findings
- Failed or Unacceptable PT results
- Identified poor process or method performance trends
- Systematic reporting / calculation errors.

- Data recall investigations
- Questionable trends that are found in the review of NCMs
- Client complaints
- Excessive revised reports

The ICAT database is used to document background information, track the results of corrective action investigations and root cause analysis, and to provide reports of corrective action plans.

12.3 Closed Loop Corrective Action Process

Any employee in the company can initiate a corrective action. There are four main components to a closed-loop corrective action process once an issue has been identified: Cause Analysis, Selection and Implementation of Corrective Actions (both short and long term), Monitoring of the Corrective Actions, and Follow-up.

12.3.1 Cause Analysis

- Upon discovery of a non-conformance event, the event must be defined and documented. An entry into the ICAT system must be initiated. Someone is assigned to investigate the issue and the event is investigated for root cause. Table 12-1 provides some general guidelines on determining responsibility for assessment.
- The cause analysis step is the key to the process as a long term corrective action cannot be determined until the cause is determined.
- If the root cause is not readily obvious, the Technical Director, Laboratory Director, Technical Manager, or QA Manager (or QA designee) is consulted.

12.3.2 Selection and Implementation of Corrective Actions

- Where corrective action is needed, the laboratory shall identify potential corrective actions. The action(s) most likely to eliminate the problem and prevent recurrence are selected and implemented. Responsibility for implementation is assigned.
- Corrective actions shall be to a degree appropriate to the magnitude of the problem identified through the cause analysis.
- Whatever corrective action is determined to be appropriate, the laboratory shall document and implement the changes. The ICAT record is used for this documentation.

12.3.3 Root Cause Analysis

Root Cause Analysis is a class of problem solving (investigative) methods aimed at identifying the basic or causal factor(s) that underlie variation in performance or the occurrence of a significant failure. The root cause may be buried under seemingly innocuous events, many steps preceding the perceived failure. At first glance, the immediate response is typically directed at a symptom and not the cause. Typically, root cause analysis would be best with three or more incidents to triangulate a weakness. Corporate SOP No. CA-Q-S-009, *Root Cause Analysis*, describes this procedure.

Systematically analyze and document the root causes of the more significant problems that are reported. Identify, track, and implement the corrective actions required to reduce the likelihood

of recurrence of significant incidents. Trend the root cause data from these incidents to identify root causes that, when corrected, can lead to dramatic improvements in performance by eliminating entire classes of problems.

Identify the one event associated with problem and ask why this event occurred. Brainstorm the root causes of failures; for example, by asking why events occurred or conditions existed; and then why the cause occurred 5 consecutive times until you get to the root cause. For each of these sub events or causes, ask why it occurred. Repeat the process for the other events associated with the incident.

Root cause analysis does not mean the investigation is over. Look at technique, or other systems outside the normal indicators. Often creative thinking will find root causes that ordinarily would be missed, and continue to plague the laboratory or operation.

12.3.4 Monitoring of the Corrective Actions

- The Technical Manager and QA Manager are responsible to ensure that the corrective action taken was effective.
- Ineffective actions are documented and re-evaluated until acceptable resolution is achieved. Technical Managers are accountable to the Laboratory Director to ensure final acceptable resolution is achieved and documented appropriately.
- The QA Manager reviews monthly NCMs and ICAT records for trends. Highlights are included in the QA monthly report (refer to Section 16). If a significant trend develops that adversely affects quality, an audit of the area is performed and corrective action implemented.
- Any out-of-control situations that are not addressed acceptably at the laboratory level may be reported to the Corporate Quality Director by the QA Manager, indicating the nature of the out-of-control situation and problems encountered in solving the situation.

12.3.5 Follow-up Audits

- Follow-up audits may be initiated by the QA Manager and shall be performed as soon as possible when the identification of a nonconformance casts doubt on the laboratory's compliance with its own policies and procedures, or on its compliance with state or federal requirements.
- These audits often follow the implementation of the corrective actions to verify effectiveness. An additional audit would only be necessary when a critical issue or risk to business is discovered.

(Also refer to Section 15.1.4, Special Audits.)

12.4 Technical Corrective Actions

In addition to providing acceptance criteria and specific protocols for technical corrective actions in the method SOPs, the laboratory has general procedures to be followed to determine when departures from the documented policies and procedures and quality control have occurred (refer to Section 11). The documentation of these procedures is through the use of an NCM or record in the ICAT system.

Table 12-1 includes examples of general technical corrective actions. For specific criteria and corrective actions, refer to the analytical methods or specific method SOPs. The laboratory may also maintain Work Instructions on these items that are available upon request.

Table 12-1 provides some general guidelines for identifying the individual(s) responsible for assessing each QC type and initiating corrective action. The table also provides general guidance on how a data set should be treated if associated QC measurements are unacceptable. Specific procedures are included in Method SOPs, Work Instructions, QAM Sections 19 and 20. All corrective actions are reviewed monthly, at a minimum, by the QA Manager and highlights are included in the QA monthly report.

To the extent possible, samples shall be reported only if all quality control measures are acceptable. If the deficiency does not impair the usability of the results, data will be reported with an appropriate data qualifier and/or the deficiency will be noted in the case narrative. Where sample results may be impaired, the Project Manager is notified by an NCM and appropriate corrective action (e.g., reanalysis) is taken and documented**.**

12.5 Basic Corrections

When mistakes occur in records, each mistake shall be crossed-out, [not obliterated (e.g. no white-out)], and the correct value entered alongside. All such corrections shall be initialed (or signed) and dated by the person making the correction. In the case of records stored electronically, the original uncorrected file must be maintained intact and a second corrected file is created.

This same process applies to adding additional information to a record. All additions made later than the initial must also be initialed (or signed) and dated.

When corrections are due to reasons other than obvious transcription errors, the reason for the corrections (or additions) shall also be documented.

Table 12-1

Example – General Corrective Action Procedures

QC Activity *(Individual Responsible for Initiation/Assessment)*	Acceptance Criteria	Recommended Corrective Action
Initial Instrument Blank *(Analyst)*	- Instrument response < MDL	- Prepare another blank. - If same response, determine cause of contamination: reagents, environment, instrument equipment failure, etc..
Initial Calibration Standards *(Analyst, Technical Manager(s))*	- Correlation coefficient > 0.99 or 0.995 or: standard concentration value - Read-back errors within acceptance range - % Recovery within acceptance range - See details in Method SOP	- Reanalyze standards. - If still unacceptable, remake standards and recalibrate instrument.
Independent Calibration Verification (Second Source) *(Analyst, Technical Manager(s))*	- % Recovery within control limits	- Remake and reanalyze standard. - If still unacceptable, then remake calibration standards or use new primary standards and recalibrate instrument.
Continuing Calibration Standards *(Analyst, Data Reviewer)*	% Recovery within control limits	- Reanalyze standard. - If still unacceptable, then recalibrate and rerun affected samples.
Matrix Spike / Matrix Spike Duplicate (MS/MSD) *(Analyst, Data Reviewer)*	- % Recovery within limits documented in LIMS	- If the acceptance criteria for duplicates or matrix spikes are not met because of matrix interferences, the acceptance of the analytical batch is determined by the validity of the LCS. - If the LCS is within acceptable limits the batch is acceptable. - The results of the duplicates, matrix spikes and the LCS are reported with the data set. - For matrix spike or duplicate results outside criteria the data for that sample shall be reported with qualifiers.

QC Activity *(Individual Responsible for Initiation/Assessment)*	Acceptance Criteria	Recommended Corrective Action
Laboratory Control Sample (LCS) *(Analyst, Data Reviewer)*	- % Recovery within limits specified in LIMS	- Batch must be re-prepared and re-analyzed. This includes any allowable marginal exceedance. When not using marginal exceedances, the following exceptions apply: 1) when the acceptance criteria for the positive control are exceeded high (i.e., high bias) and there are associated samples that are non-detects, then those non-detects may be reported with data qualifying codes; 2) when the acceptance criteria for the positive control are exceeded low (i.e., low bias), those sample results may be reported if they exceed a maximum regulatory limit/decision level with data qualifying codes. **Note:** If there is insufficient sample or the holding time cannot be met, contact client and report with flags.
Surrogates *(Analyst, Data Reviewer)*	- % Recovery within limits of method or within three standard deviations of the historical mean	- Individual sample must be repeated. Place comment in TALS. - Surrogate results outside criteria shall be reported with qualifiers.
Method Blank (MB) *(Analyst, Data Reviewer)*	< Reporting Limit [1] For common lab contaminants, no analytes detected at greater than and equal to RL.	- Reanalyze blank. - If still positive, determine source of contamination. If necessary, reprocess (i.e. digest or extract) entire sample batch. Report blank results. - Qualify the result(s) if the concentration of a targeted analyte in the MB is at or above the reporting limit and is > 1/10 of the amount measured in the sample.
Proficiency Testing (PT) Samples *(QA Manager, Technical Manager(s))*	- Criteria supplied by PT Supplier	- Any failures or warnings must be investigated for cause. Failures may result in the need to repeat a PT sample to show the problem is corrected.
Internal / External Audits *(QA Manager, Technical Manager(s), Laboratory Director)*	- Defined in Quality System documentation such as SOPs, QAM, etc.	- Non-conformances must be investigated through ICAT system and necessary corrections must be made.
Reporting / Calculation Errors (Depends on issue – possible individuals include: Analysts, Data Reviewers, Project Managers, *Technical Manager(s)*, QA Manager, Corporate QA, Corporate Management)	- Corporate SOP CW-Q-S-005, Data Recall	- Corrective action is determined by type of error. Follow the procedures in Corporate SOP CW-L-S-002, Internal Investigations, or laboratory SOP PT-QA-016.

QC Activity *(Individual Responsible for Initiation/Assessment)*	Acceptance Criteria	Recommended Corrective Action
Client Complaints *(Project Managers, Lab Director, Sales and Marketing)*	-	- Corrective action is determined by the type of complaint. For example, a complaint regarding an incorrect address on a report will result in the report being corrected and then follow-up must be performed on the reasons the address was incorrect (e.g., database needs to be updated).
QA Monthly Report (Refer to Section 16 for an example) (QA Manager, Lab Director, *Technical Manager(s)*)	- QAM, SOPs	- Corrective action is determined by the type of issue. For example, NCMs and ICAT records for the month are reviewed and possible trends are investigated.
Health and Safety Violation (Safety Officer, Lab Director, *Technical Manager(s)*)	- Environmental Health and Safety (EHS) Manual	- Non-conformance is investigated and corrected through ICAT system.

Note:

1. Except as noted below for certain compounds, the method blank should be below the reporting limit unless there is a client specific requirement or method requirement to be evaluated to a lower level. Concentrations up to five times the reporting limit will be allowed for the ubiquitous laboratory and reagent contaminants: methylene chloride, toluene, acetone, 2-butanone and phthalates provided that they appear in similar levels in the reagent blank and samples. This allowance presumes that the detection limit is significantly below any regulatory limit to which the data are to be compared and that blank subtraction will not occur. For benzene and ethylene dibromide (EDB) and other analytes for which regulatory limits are extremely close to the detection limit, the method blank must be below the method detection limit.

Figure 12-1

Example – Corrective Action Report (iCAT)



Figure 12-1

Example – Corrective Action Report (iCAT) Cont.

Describe the Required Action:

Investigation/Response:

Root Cause:

Corrective Action Plan:

Select for Notification

Selected	Name	Title
☐		

SECTION 13

PREVENTIVE ACTION / IMPROVEMENT

13.1 Overview

The laboratory's preventive action programs improve or eliminate potential causes of nonconforming product and/or nonconformance to the quality system. This preventive action process is a proactive and continuous process if improvement activities that can be initiated through feedback from clients, employees, business providers, and affiliates. The QA Department has the overall responsibility to ensure that the preventive action process is in place, and that relevant information on actions is submitted for management review.

Dedicating resources to an effective preventive action system emphasizes the laboratory's commitment to its Quality Program. It is beneficial to identify and address negative trends before they develop into complaints, problems and corrective actions. Additionally, the laboratory continually strives to improve customer service and client satisfaction through continuous improvements to laboratory systems.

Opportunities for improvement may be discovered through any of the following on the Preventative Action Schedule:

Preventative Action	Frequency
Management System Review	Annually
Review of QA Metrics	Monthly
Trending NCMs	Monthly
Review of Control Charts of QC Results	At least annually
Trending PT Results	Semi-annually
Trending Client Complaints	Monthly
Review of Revised Reports and Invoices	Monthly
Review of Process Operations	At least annually
Staff Observations and Suggestions	As they arise

The monthly Management Systems Metrics Report shows performance indicators in all areas of the laboratory and quality system. These areas include revised reports, corrective actions, audit findings, internal auditing and data authenticity audits, client complaints, PT samples, holding time violations, SOPs, ethics training, etc. The metrics report is reviewed monthly by laboratory management, Corporate QA and Eurofins TestAmerica's Executive Committee. These metrics are used in evaluating the management and quality system performance on an ongoing basis and provide a tool for identifying areas for improvement.

Items identified as continuous improvement opportunities to the management system may be issued as goals from the annual management systems review, recommendations from internal audits, white papers, Lesson Learned, Technical Services audit report, Technical Best Practices, or as Corporate or management initiatives.

The laboratory's corrective action process is integral to implementation of preventive actions. A critical piece of the corrective action process is the implementation of actions to prevent further occurrence of a non-compliance event. Historical review of corrective action and non-conformances provides a valuable mechanism for identifying preventive action opportunities.

13.1.1 The following elements are part of a preventive action/process improvement system:

- Identification of an opportunity for preventive action or process improvement
- Process for the preventive action or improvement
- Define the measurements of the effectiveness of the process once undertake
- Execution of the preventive action or improvement
- Evaluation of the plan using the defined measurements
- Verification of the effectiveness of the preventive action or improvement
- Close-Out by documenting any permanent changes to the Quality System as a result of the Preventive Action or Process Improvement. Documentation of Preventive Action/ Process Improvement is incorporated into the monthly QA reports, corrective action process and management review.

13.1.2 Any Preventive Actions/ Process Improvements undertaken or attempted shall be taken into account during the Annual Management Systems Review (Section 16). A highly detailed report is not required; however a summary of success and failure within the preventive action program is sufficient to provide management with a measurement for evaluation.

13.2 Management Of Change

The Management of Change process is designed to manage significant events and changes that occur within the laboratory. Through these procedures, the potential risks inherent with a new event or change are identified and evaluated. The risks are minimized or eliminated through pre-planning and the development of preventive measures. The types of indicators monitored under this collective system include:

Change Type	Examples
Facility Changes	-movement of prep or instrument groups to a new location in the laboratory -introduction of significant changes in air handling or gas and solvent delivery systems -significant room additions or renovations -significant electrical or network upgrades or changes
Major Accreditation Changes	-voluntary surrender of accreditations no longer deemed necessary to the laboratory -loss of accreditation -addition of new accreditation programs
Reagents and Waste Streams	- new chemicals/reagents not previously used in the laboratory -deletion of chemicals/reagents that will mean they are no longer used at all in the laboratory -major changes to the volume of chemicals/reagents being used - a new waste stream must be developed Note: See EHS for changes in waste streams
Addition or Deletion of Laboratory Capabilities	-implementation of new regulated methods -“retiring” of active methods -method development for “in-house” methods Note: New regulatory methods and method development require specific processes and documentation before the process can begin or the method can enter production. See QA and EHS for requirements.
LIMS changes	-large system upgrades -additions of new capabilities Note: These changes are handled on a Corporate level
Key Personnel Changes	-key personnel promotions and their effect on that individuals group (experience, productivity, leadership, manpower) -key personnel losses -impact of new personnel that may add new experience or capabilities to the laboratory
New Types of	-addition of a new instrument class/technology

Instrumentation	-significant instrument upgrades that impact sensitivity, productivity or capability Note: New instrumentation requires collection and submission of instrument IDOC information before entering production. See QA for requirements.
Changes in Quality Systems and Policies	-implementation of a new Corrective Action System -changes to the Internal Audit program -implementation of uploads for Proficiency Testing samples

SECTION 14

CONTROL OF RECORDS

The laboratory maintains a records management system appropriate to its needs and that complies with applicable standards or regulations as required. The system produces unequivocal, accurate records that document all laboratory activities. The laboratory retains all original observations, calculations and derived data, calibration records and a copy of the analytical report for a minimum of five years after it has been issued. Exceptions for programs with longer retention requirements are discussed in section 14.1.2.

14.1 Overview

The laboratory has established procedures for identification, collection, indexing, access, filing, storage, maintenance and disposal of quality and technical records. A record index is listed in Table 14-1. More detailed information on retention of specific records is provided in Corporate Policy No.CW-L-P-001, *Records Retention Policy*, and Work Instruction No. CW-L-WI-001, *Eurofins TestAmerica Records Retention/Storage Schedule*. Quality records are maintained by the Quality Assurance (QA) department electronically on the corporate designated QA network drive which is backed up as part of the regular network backup. Records are of two types; either electronic or hard copy paper formats, depending on whether the record is computer or hand generated (some records may be in both formats). Technical records are maintained by the CSO group, HR, and the QA department and as outlined in laboratory SOP PT-QA-019.

Table 14-1 Record Index[1]

	Record Types [1]:	Retention Time:
Technical Records	- Raw Data - Logbooks[2] - Standards - Certificates - Analytical Records - MDLs/IDLs/DOCs - Lab Reports	5 Years from analytical report issue*
Official Documents	- Quality Assurance Manual (QAM) - Work Instructions - Policies - SOPs - Policy Memorandums - Manuals - Published Methods	Indefinitely
QA Records	- Certifications - Method and Software Validation / Verification Data	Indefinitely

	Record Types [1]:	Retention Time:
QA Records	- Internal & External Audits/Responses - Corrective/Preventive Actions - Management Reviews - Data Investigation	5 Years from archival* **Data Investigation:** 5 years or the life of the affected raw data storage whichever is greater (beyond 5 years if ongoing project or pending investigation)
Project Records	- Sample Receipt & COC Documents - Contracts and Amendments - Correspondence - QAPP - SAP - Telephone Logbooks - Lab Reports	5 Years from analytical report issue*
Administrative Records	Financial and Business Operations	Refer to CW-L-WI-001
	EH&S Manual, Permits	Indefinitely
	Disposal Records	Indefinitely
	Employee Handbook	Indefinitely
	Personnel files, Employee Signature & Initials, Administrative Training Records (e.g., Ethics)	Refer to HR Manual
	Administrative Policies	Indefinitely
	Technical Training Records	7 years
	Legal Records	Indefinitely
	HR Records	Refer to CW-L-WI-001
	IT Records	Refer to CW-L-WI-001
	Corporate Governance Records	Refer to CW-L-WI-001
	Sales & Marketing	5 years
	Real Estate	Indefinitely

[1] Record Types encompass hardcopy and electronic records.
[2] Examples of Logbook types: Maintenance, Instrument Run, Preparation (standard and samples), Standard and Reagent Receipt, Archiving, Balance Calibration, Temperature (hardcopy or electronic records).
* Exceptions listed in Table 14-2.

14.1.1 All records are stored and retained in such a way that they are secure and readily retrievable at the laboratory facility or an offsite location that provides a suitable environment to prevent damage or deterioration and to prevent loss at the laboratory or the Iron Mountain data storage facility. All records shall be protected against fire, theft, loss, environmental deterioration, and vermin. In the case of electronic records, electronic or magnetic sources, storage media are protected from deterioration caused by magnetic fields and/or electronic deterioration.

Access to the data is limited to laboratory and company employees and shall be documented with an access log**.** Records archived off-site are stored in a secure location where a record is maintained of any entry into the storage facility. Whether on-site or off-site storage is used, logs are maintained in each storage box to note removal and return of records. Retention of records are maintained on-site at the laboratory for at least 1 year after their generation and moved offsite for the remainder of the required storage time. Records are maintained for a minimum of five years unless otherwise specified by a client or regulatory requirement.

For raw data and project records, record retention shall be calculated from the date the project report is issued. For other records, such as Controlled Documents, QA, or Administrative Records, the retention time is calculated from the date the record is formally retired. Records related to the programs listed in Table 14-2 have lengthier retention requirements and are subject to the requirements in Section 14.1.3.

14.1.2 Programs with Longer Retention Requirements

Some regulatory programs have longer record retention requirements than the standard record retention time. These are detailed in Table 14-2 with their retention requirements. In these cases, the longer retention requirement is enacted. If special instructions exist such that client data cannot be destroyed prior to notification of the client, the container or box containing that data is marked as to who to contact for authorization prior to destroying the data.

Table 14-2 Special Record Retention Requirements

Program	[1]Retention Requirement
Drinking Water – All States	10 years (lab reports and raw data)
Commonwealth of MA – All environmental data 310 CMR 42.14	10 years
FIFRA – 40 CFR Part 160	Retain for life of research or marketing permit for pesticides regulated by EPA
Housing and Urban Development (HUD) Environmental Lead Testing	10 years
Alaska	10 years
Louisiana – All	10 years
Michigan Department of Environmental Quality – all environmental data	10 years
Ohio VAP	10 years and State contact prior to disposal
OSHA	30 years

[1]Note: Extended retention requirements must be noted with the archive documents or addressed in facility-specific records retention procedures.

14.1.3 The laboratory has procedures to protect and back-up records stored electronically and to prevent unauthorized access to or amendment of these records. All analytical data is maintained as hard copy or in a secure readable electronic format. For analytical reports that are maintained as copies in PDF format, refer to Section 19.15.1 and laboratory SOP PT-QA-019, Records Management, Retention, and Archive, for more information.

14.1.4 The record keeping system allows for historical reconstruction of all laboratory activities that produced the analytical data, as well as rapid recovery of historical data (Records stored off site should be accessible within 2 days of a request for such records). The history of the sample from when the laboratory took possession of the samples must be readily understood through the documentation. This shall include inter-laboratory transfers of samples and/or extracts.

- The records include the identity of personnel involved in sampling, sample receipt, preparation, or testing. All analytical work contains the initials (at least) of the personnel

involved. The laboratory's copy of the chain of custody is stored with the invoice and the work order sheets generated by TALS. Details of the COC linking procedure are described in SOP PT-SR-001. The chain of custody would indicate the name of the sampler. Any sampling notes provided with the chain of custody are also scanned into TALS.

- All information relating to the laboratory facilities equipment, analytical test methods, and related laboratory activities, such as sample receipt, sample preparation, or data verification are documented.
- The record keeping system facilitates the retrieval of all working files and archived records for inspection and verification purposes (e.g., set format for naming electronic files, set format for what is included with a given analytical data set are described in SOP PT-QA-019. Instrument data is stored sequentially by instrument. Run logs are maintained for each instrument; a copy of each day's run long is stored in the electronic files along with the data to aid in re-constructing an analytical sequence. Where an analysis is performed without an instrument, bound logbooks or bench sheets are used to record and file data or the data is entered directly into TALS as the analysis is done. Standard and reagent information is recorded in electronic standard log in TALS, and is associated with each preparation and analytical batch for which they are used.
- Changes to hardcopy records shall follow the procedures outlined in Section 12 and 19. Changes to electronic records in TALS or instrument data are recorded in audit trails.
- The reason for a signature or initials on a document is clearly indicated in the records such as "sampled by," "prepared by," "reviewed by", or "analyzed by".
- All generated data, except those that are generated by automated data collection systems, are recorded directly, promptly and legibly in permanent dark ink.
- Hard copy data may be scanned into PDF format for record storage as long as the scanning process can be verified in order to ensure that no data is lost and the data files and storage media must be tested to verify the laboratory's ability to retrieve the information prior to the destruction of the hard copy that was scanned. The procedure for this verification can be found in SOP PT-QA-019.
- Also refer to Section 19.15.1 'Computer and Electronic Data Related Requirements'**.**

14.2 Technical And Analytical Records

14.2.1 The laboratory retains records of original observations, derived data and sufficient information to establish an audit trail, calibration records, staff records and a copy of each analytical report issued, for a minimum of five years unless otherwise specified by a client or regulatory requirement. The records for each analysis shall contain sufficient information to enable the analysis to be repeated under conditions as close as possible to the original. The records shall include the identity of laboratory personnel responsible for the sampling, performance of each analysis and reviewing results.

14.2.2 Observations, data and calculations are recorded real-time and are identifiable to the specific task.

14.2.3 Changes to hardcopy records shall follow the procedures outlined in Section 12 and 19. Changes to electronic records in TALS or instrument data are recorded in audit trails.

The essential information to be associated with analysis, such as strip charts, tabular printouts, computer data files, analytical notebooks, and run logs, include:

- Laboratory sample ID code**;**
- Date of analysis; Time of Analysis is also required if the holding time is seventy-two (72) hours or less, or when time critical steps are included in the analysis (e.g., drying times, incubations, etc.); instrumental analyses have the date and time of analysis recorded as part of their general operations. Where a time critical step exists in an analysis, location for such a time is included as part of the documentation in a specific logbook, on a benchsheet or in TALS.
- Instrumentation identification and instrument operating conditions/parameters. Operating conditions/parameters are typically recorded in method specific SOPs or in instrument maintenance logs where available.
- analysis type**;**
- all manual calculations and manual integrations**;**
- analyst's or operator's initials/signature**;**
- sample preparation including cleanup, separation protocols, incubation periods or subculture, ID codes, volumes, weights, instrument printouts, meter readings, calculations, reagents**;**
- test results**;**
- standard and reagent origin, receipt, preparation, and use**;**
- calibration criteria, frequency and acceptance criteria**;**
- data and statistical calculations, review, confirmation, interpretation, assessment and reporting conventions**;**
- quality control protocols and assessment**;**
- electronic data security, software documentation and verification, software and hardware audits, backups, and records of any changes to automated data entries; and
- Method performance criteria including expected quality control requirements. These are indicated both in the TALS and on specific analytical report formats**.**

14.2.4 All logbooks used during receipt, preparation, storage, analysis, and reporting of samples or monitoring of support equipment shall undergo a periodic, documented supervisory or peer review.

14.3 Laboratory Support Activities

In addition to documenting all the above-mentioned activities, the following are retained QA records and project records (previous discussions in this section relate where and how these data are stored):

- all original raw data, whether hard copy or electronic, for calibrations, samples and quality control measures, including analysts' work sheets and data output records (chromatograms, strip charts, and other instrument response readout records)**;**
- a written description or reference to the specific test method used which includes a description of the specific computational steps used to translate parametric observations into a reportable analytical value**;**
- copies of final reports**;**
- archived SOPs**;**
- correspondence relating to laboratory activities for a specific project**;**
- all corrective action reports, audits and audit responses**;**
- proficiency test results and raw data; and
- results of data review, verification, and crosschecking procedures

14.3.1 Sample Handling Records

Records of all procedures to which a sample is subjected while in the possession of the laboratory are maintained. These include but are not limited to records pertaining to:

- sample preservation including appropriateness of sample container and compliance with holding time requirement;
- sample identification, receipt, acceptance or rejection and login;
- sample storage and tracking including shipping receipts, sample transmittal / COC forms; and
- procedures for the receipt and retention of samples, including all provisions necessary to protect the integrity of samples.

14.4 Administrative Records

The laboratory also maintains the administrative records in either electronic or hard copy form. Refer to Table 14-1.

14.5 Records Management, Storage And Disposal

All records (including those pertaining to test equipment), certificates and reports are safely stored, held secure and in confidence to the client. Certification related records are available upon request.

All information necessary for the historical reconstruction of data is maintained by the laboratory. Records that are stored only on electronic media must be supported by the hardware and software necessary for their retrieval.

Records that are stored or generated by computers or personal computers have hard copy, write-protected backup copies, or an electronic audit trail controlling access**.**

The laboratory has a record management system (a.k.a., document control) for control of laboratory notebooks/benchsheets, instrument/equipment logbooks, and records for data reduction, validation and reporting. Laboratory notebooks are issued on a per analysis basis, as needed, and are numbered sequentially. All sample data are recorded in TALS. Bench sheets are filed sequentially. Standards are maintained in the electronic standards in TALS**.** Records are considered archived when noted as such in the records management system (a.k.a., document control).

14.5.1 Transfer of Ownership

In the event that the laboratory transfers ownership or goes out of business, the laboratory shall ensure that the records are maintained or transferred according to client's instructions. Upon ownership transfer, record retention requirements shall be addressed in the ownership transfer agreement and the responsibility for maintaining archives is clearly established. In addition, in cases of bankruptcy, appropriate regulatory and state legal requirements concerning laboratory records must be followed. In the event of the closure of the laboratory, all records will revert to the control of the Corporate headquarters. Should the entire company cease to exist, as much notice as possible will be given to clients and the accrediting bodies who have worked with the laboratory during the previous 5 years of such action.

14.5.2 Records Disposal

Records are removed from the archive and destroyed after 5 years, unless otherwise specified by a client or regulatory requirement. On a project specific or program basis, clients may need to be notified prior to record destruction. Records are destroyed in a manner that ensures their confidentiality such as shredding, mutilation or incineration. (Refer to Tables 14-1 and 14-2).

Electronic copies of records must be destroyed by erasure or physically damaging off-line storage media so no records can be read.

If a third party records management company is hired to dispose of records, a "Certificate of Destruction" is required.

SECTION 15

AUDITS

15.1 Internal Audits

Internal audits are performed to verify that laboratory operations comply with the requirements of the lab's quality system and with the external quality programs under which the laboratory operates. Audits are planned and organized by the QA staff. Personnel conducting the audits should be independent of the area being evaluated. Auditors will have sufficient authority, access to work areas, and organizational freedom necessary to observe all activities affecting quality and to report the assessments to laboratory management and when requested to Corporate management.

Audits are conducted and documented as described in the Corporate SOP No. CW-Q-S-003, *Internal Auditing*. More detail on the specific elements for internal audits and data audit is described in Pittsburgh Laboratory SOP No. PT-QA-002, *Internal Auditing*. The types and frequency of routine internal audits are shown in Table 15-1. Special or ad hoc assessments may be conducted as needed under the direction of the QA staff.

Table 15-1 Types of Internal Audits and Frequency

Description	Performed by	Frequency
Quality Systems Audits	QA Department, QA approved designee, or Corporate QA	All areas of the laboratory annually
Method Audits QA Technical Data Audits*	Joint responsibility: a) QA Manager or designee b) Technical Manager or Designee (Refer to CW-Q-S-003)	50% of methods annually

Description	Performed by	Frequency
SOP Method Compliance*	Joint responsibility: c) QA Manager or designee d) Technical Manager or Designee (Refer to CW-Q-S-003)	Every 2 years
Special	QA Department or Designee	Surveillance or spot checks performed as needed, e.g., to confirm corrective actions from other audits.
Performance Testing	Analysts with QA oversight	Two successful per year for each TNI field of testing or as dictated by regulatory requirements

*Technical Data and Method Compliance audits are in addition to the annual Quality Systems Audit

15.1.1 Annual Quality Systems Audit

An annual quality systems audit is required to ensure compliance to analytical methods and SOPs, Eurofins TestAmerica's Data Integrity and Ethics Policies, TNI quality systems, client and state requirements, and the effectiveness of the internal controls of the analytical process, including, but not limited to, data review, quality controls, preventive action and corrective action. The completeness of earlier corrective actions is assessed for effectiveness & sustainability. The audit is divided into sections for each operating or support area of the lab, and each section is comprehensive for a given area. The area audits may be performed on a rotating schedule throughout the year to ensure adequate coverage of all areas. This schedule may change as situations in the laboratory warrant. See SOP PT-QA-002 for auditing area details.

Effectiveness of training will be determined during our annual QA systems evaluation. Evidence of successful training includes:

- Audit and surveillance results, control charts, proficiency testing results, data analysis, corrective and preventive actions, customer feedback, and management reviews in efforts to monitor trends and continually improve the quality system:
- Adequate documentation of training within operational areas, including one-on-one technical training for individual technologies, and for people cross-trained.
- Analysts knowledge of QA Manual and SOPs. Analysts following SOPs, i.e., practice matches SOPs.
- Analysts regularly communicate to supervisors and QA if SOPs need revision.

15.1.2 QA Technical Data Audits

QA technical audits assess data authenticity and analyst integrity. These audits are based on client projects, associated sample delivery groups, and the methods performed. Reported results are compared to raw data to verify the authenticity of results. The validity of calibrations and QC results are compared to data qualifiers, footnotes, and case narratives. Documentation is assessed by examining run logs and records of manual integrations. Manual calculations are checked. Where possible, electronic audit miner programs (e.g., Chrom AuditMiner) are used to identify unusual manipulations of the data deserving closer scrutiny. QA technical audits will include all methods within a two-year period. All analysts and data reviewers should be reviewed over the course of a two year period through at least one QA Technical Audit.

15.1.3 SOP Method Compliance

Compliance of all SOPs with the source methods and compliance of the operational groups with the SOPs will be assessed by the QA department, Technical Manager, or qualified designee at least every two years. It is also recommended that the work of each newly hired analyst is assessed within 3 months of working independently, (e.g., completion of method IDOC). In addition, as analysts add methods to their capabilities, reviews (new IDOC) of the analyst work products will be performed within 3 months of completing the documented training.

15.1.4 Special Audits

Special audits are conducted on an as needed basis, generally as a follow up to specific issues such as client complaints, corrective actions, PT results, data audits, system audits, validation comments, regulatory audits or suspected ethical improprieties. Special audits are focused on a specific issue, and report format, distribution, and timeframes are designed to address the nature of the issue.

15.1.5 Performance Testing

The laboratory participates semi-annually in performance audits conducted through the analysis of PT samples provided by a third party. The laboratory generally participates in the following types of PT studies: Water Pollution Program, Water Supply Program, Hazardous Waste Program, client supplied PTs and Lab internal PTs.

It is Eurofins TestAmerica's policy that PT samples be treated as typical samples in the production process. Furthermore, where PT samples present special or unique problems, in the regular production process they may need to be treated differently, as would any special or unique request submitted by any client. The QA Manager must be consulted and in agreement with any decisions made to treat a PT sample differently due to some special circumstance.

Unacceptable PT results are required to be entered into the ICAT system for investigation. In some cases it may be necessary for blind QC samples to be submitted to the laboratory to show a return to control.

15.2 EXTERNAL AUDITS

External audits are performed when certifying agencies or clients conduct on-site inspections or submit performance testing samples for analysis. It is Eurofins TestAmerica's policy to cooperate fully with regulatory authorities and clients. The laboratory makes every effort to provide the auditors with access to personnel, documentation, and assistance. Laboratory supervisors are responsible for providing corrective actions to the QA Manager who coordinates the response. Audit responses are due in the time allotted by the client or agency performing the audit. When requested, a copy of the audit report and the labs corrective action plan will be forwarded to Corporate Quality.

The laboratory cooperates with clients and their representatives to monitor the laboratory's performance in relation to work performed for the client. The client may only view data and systems related directly to the client's work. All efforts are made to keep other client information confidential.

15.2.1 Confidential Business Information (CBI) Considerations

During on-site audits, auditors may come into possession of information claimed as business

confidential. A business confidentiality claim is defined as "a claim or allegation that business information is entitled to confidential treatment for reasons of business confidentiality or a request for a determination that such information is entitled to such treatment." When information is claimed as business confidential, the laboratory must place on (or attach to) the information at the time it is submitted to the auditor, a cover sheet, stamped or typed legend or other suitable form of notice, employing language such as "trade secret", "proprietary" or "company confidential". Confidential portions of documents otherwise non-confidential must be clearly identified. CBI may be purged of references to client identity by the responsible laboratory official at the time of removal from the laboratory. However, sample identifiers may not be obscured from the information. Additional information regarding CBI can be found in within the 2009 TNI standards.

15.3 Audit Findings

Audit findings are documented using the ICAT system (see Section 12). The laboratory's corrective action responses may include action plans that could not be completed within a predefined timeframe. In these instances, a completion date must be set and agreed to by operations management and the QA Manager.

Developing and implementing corrective actions to findings is the responsibility of the Technical Manager where the finding originated. Findings that are not corrected by specified due dates are reported monthly to management in the QA monthly report. When requested a copy of the audit report and the labs corrective action plan will be forwarded to Corporate Quality.

If any audit finding casts doubt on the effectiveness of the operations or on the correctness or validity of the laboratory's test results, the laboratory shall take timely corrective action, and shall notify clients in writing if the investigations show that the laboratory results have been affected. Once corrective action is implemented, a follow-up audit is scheduled to ensure that the problem has been corrected.

Clients must be notified promptly, in writing, of any event such as the identification of defective measuring or test equipment that casts doubt on the validity of results given in any test report or amendment to a test report. The investigation must begin within 24-hours of discovery of the problem and all efforts are made to notify the client within two weeks after completion of the investigation.

SECTION 16
MANAGEMENT REVIEWS

16.1 Quality Assurance Report

A comprehensive QA Report shall be prepared each month by the laboratory's QA Department and forwarded to the Laboratory Director, Technical Managers, their Quality Director as well as the VP of Operations. All aspects of the QA system are reviewed to evaluate the suitability of policies and procedures. During the course of the year, the Laboratory Director, VP of Operations, or Corporate QA may request that additional information be added to the report.

On a monthly basis, Corporate QA compiles information from all the monthly laboratory reports. The Corporate Quality Directors prepare a report that includes a compilation of all metrics and notable information and concerns regarding the QA programs within the laboratories. The report

also includes a listing of new regulations that may potentially impact the laboratories. This report is presented to the Senior Management Team and VPs of Operations.

16.2 Annual Management Review

The senior lab management team (Laboratory Director, QA Manager, and Technical Managers) conducts a review annually of its quality systems and TALS to ensure its continuing suitability and effectiveness in meeting client and regulatory requirements and to introduce any necessary changes or improvements. It will also provide a platform for defining goals & objectives and action items that feed into the laboratory planning system. Corporate Operations and Corporate QA personnel is to be included in this meeting at the discretion of the Laboratory Director. The TALS review consists of examining any audits, complaints or concerns that have been raised through the year that are related to the TALS. The laboratory will summarize any critical findings that cannot be solved by the lab and report them to Corporate IT.

The Management System Review (see Corporate SOP No. CW-Q-S-004, *Management System Review*, and Work Instruction No. CW-Q-WI-003) uses information generated during the preceding year to assess the "big picture" by ensuring that routine actions taken and reviewed on a monthly basis are not components of larger systematic concerns. The monthly review should keep the quality systems current and effective, therefore, the annual review is a formal senior management process to review specific existing documentation. Significant issues from the following documentation are compiled or summarized by the QA Manager prior to the review meeting:

- Matters arising from the previous annual review
- Prior Monthly QA Reports issues
- Laboratory QA Metrics
- Review of report reissue requests
- Review of client feedback and complaints
- Issues arising from any prior management or staff meetings
- Minutes from prior senior lab management meetings - Issues that may be raised from these meetings include:
    - Adequacy of staff, equipment and facility resources.
    - Adequacy of policies and procedures.
    - Future plans for resources and testing capability and capacity.
- The annual internal double blind PT program sample performance
- Compliance to the Ethics Policy and Data Integrity Plan, including any evidence/incidents of inappropriate actions or vulnerabilities related to data Integrity
- Review of Corrective and Preventative Actions, assessments by external bodies and recommendations for improvement
- Evaluation of overall risk, including risks to impartiality, confidentiality, reporting statements of conformity, and nonconforming work.

A report is generated by the QA Manager and management. The report is distributed to the appropriate VP of Operation and the Quality Director. The report includes, but is not limited to:

- The date of the review and the names and titles of participants
- A reference to the existing data quality related documents and topics that were reviewed

- Quality system or operational changes or improvements that will be made as a result of the review [e.g., an implementation schedule including assigned responsibilities for the changes (Action Table)]

Changes to the quality systems requiring update to the laboratory QA Manual shall be included in the next revision of the QA Manual.

16.3 Potential Integrity Related Managerial Reviews

Potential integrity issues (data or business related) must be handled and reviewed in a confidential manner until such time as a follow-up evaluation, full investigation, or other appropriate actions have been completed and issues clarified. Eurofins TestAmerica's Corporate Internal Investigation and Data Recall SOPs shall be followed (SOP No. CW-L-S-002 and CW-Q-S-005). All investigations that result in finding of inappropriate activity are documented and include any disciplinary actions involved, corrective actions taken, and all appropriate notifications of clients.

Eurofins TestAmerica's President and CEO, Executive VP of Operations, VP of Client & Technical Services, VPs of Operation, and Quality Directors receive a monthly report from the VP-QA/EHS summarizing any current data integrity or data recall investigations. The VP's of Operation are also made aware of progress on these issues for their specific labs.

SECTION 17

PERSONNEL

17.1 Overview

The laboratory's management believes that its highly qualified and professional staff is the single most important aspect in assuring a high level of data quality and service. The staff consists of professionals and support personnel as outlined in the organization chart in Figure 4-1.

All personnel must demonstrate competence in the areas where they have responsibility. Any staff that is undergoing training shall have appropriate supervision until they have demonstrated their ability to perform their job function on their own. Staff shall be qualified for their tasks based on appropriate education, training, experience and/or demonstrated skills as required.

The laboratory employs sufficient personnel with the necessary education, training, technical knowledge and experience for their assigned responsibilities.

All personnel are responsible for complying with all QA/QC requirements that pertain to the laboratory and their area of responsibility. Each staff member must have a combination of experience and education to adequately demonstrate a specific knowledge of their particular area of responsibility. Technical staff must also have a general knowledge of lab operations, test methods, QA/QC procedures and records management.

Laboratory management is responsible for formulating goals for lab staff with respect to education, training and skills and ensuring that the laboratory has a policy and procedures for

identifying training needs and providing training of personnel. The training shall be relevant to the present and anticipated responsibilities of the lab staff.

The laboratory only uses personnel that are employed by or under contract to, the laboratory. Contracted personnel, when used, must meet competency standards of the laboratory and work in accordance to the laboratory's quality system.

17.2 Education And Experience Requirements For Technical Personnel

The laboratory makes every effort to hire analytical staffs that possess a college degree (AA, BA, BS) in an applied science with some chemistry in the curriculum. Exceptions can be made based upon the individual's experience and ability to learn. For supervisory positions, the Pennsylvania DEP has education and experience requirements that must be met by the Pittsburgh laboratory personnel, and these are reflected in Table 17-1 below. Selection of qualified candidates for laboratory employment begins with documentation of minimum education, training, and experience prerequisites needed to perform the prescribed task. Minimum education and training requirements for Eurofins TestAmerica employees are outlined in job descriptions and are generally summarized for analytical staff in the table below.

The laboratory maintains job descriptions for all personnel who manage, perform or verify work affecting the quality of the environmental testing the laboratory performs. Job Descriptions are located on the Eurofins TestAmerica intranet site's Human Resources web-page (Also see Section 4 for position descriptions/responsibilities).

Experience and specialized training are occasionally accepted in lieu of a college degree (basic lab skills such as using a balance, pipette or quantitation techniques, etc., are also considered).

Table 17-1 General Requirements for Analytical Staff

Specialty	Education	Experience
Extractions, Digestions, some electrode methods (pH, DO, Redox, etc.), or Titrimetric and Gravimetric Analyses	H.S. Diploma	On the job training (OJT)
GFAA, CVAA, FLAA, Single component or short list Chromatography (e.g., Fuels, BTEX-GC, IC	A college degree in an applied science or 2 years of college and at least 1 year of college chemistry	Or 2 years prior analytical experience is required
ICP, ICPMS, Long List or complex chromatography (e.g., Pesticides, PCB, Herbicides, HPLC, etc.), GCMS	A college degree in an applied science or 2 years of college chemistry	or 5 years of prior analytical experience
Spectra Interpretation	A college degree in an applied science or 2 years of college chemistry	And 2 years relevant experience Or 5 years of prior analytical experience

Specialty	Education	Experience
Technical Manager (s) – **General**	Bachelors Degree in an applied science or engineering with 24 semester hours in chemistry An advanced (MS, PhD.) degree may substitute for one year of experience	And 2 years' experience in environmental analysis of representative analytes for which they will oversee
Technical Manager (s) – **Wet Chem** only (no advanced instrumentation)	Associates degree in an applied science or engineering or 2 years of college with 16 semester hours in chemistry	And 2 years relevant experience
Technical Managers - **Microbiology**	Bachelors degree in applied science with at least 16 semester hours in general microbiology and biology An advanced (MS, PhD.) degree may substitute for one year of experience	And 2 years of relevant experience (1 year if laboratory analysis is limited to coliforms and heterotrophic plate count)
Technical Managers – **Microbiology** limited to analysis of fecal coliforms, total coliforms or heterotrophic bacteria	Associates degree in an applied science or engineering and at least 4 credit hours in biology	And 2 years of relevant experience

When an analyst does not meet these requirements, they can perform a task under the direct supervision of a qualified analyst, peer reviewer or Technical Manager, and are considered an analyst in training. The person supervising an analyst in training is accountable for the quality of the analytical data and must review and approve data and associated corrective actions.

17.3 Training

The laboratory is committed to furthering the professional and technical development of employees at all levels.

Orientation to the laboratory's policies and procedures, in-house method training, and employee attendance at outside training courses and conferences all contribute toward employee proficiency. Below are examples of various areas of required employee training.

Table 17-2 Employee Training Examples

Required Training	Time Frame	Employee Type
Environmental Health & Safety	Prior to lab work	All
Ethics –New Hire Comprehensive	30 days of hire	All
Data Integrity	30 days of hire	Technical and PMs
Quality Assurance	30 days of hire	All
Ethics – Comprehensive Refresher	Annually (within 14 months of previous)	All
Initial Demonstration of Capability (DOC)	Prior to unsupervised method performance	Technical

The laboratory maintains records of relevant authorization/competence, education, professional qualifications, training, skills and experience of technical personnel (including contracted personnel) as well as the date that approval/authorization was given. These records are kept on file at the laboratory. Also refer to "Demonstration of Capability" in Section 19.

The training of technical staff is kept up to date by:

- Each employee must have documentation in their training file that they have read, understood and agreed to follow the most recent version of the laboratory QA Manual and SOPs in their area of responsibility. This documentation is updated as SOPs are updated.
- Documentation from any training courses or workshops on specific equipment, analytical techniques or other relevant topics
- Documentation of proficiency (refer to Section 19).
- An Ethics Agreement signed by each staff member (renewed each year) and evidence of annual ethics training.
- A Confidentiality Agreement signed by each staff member signed at the time of employment.
- Human Resources maintains documentation and attestation forms on employment status & records; benefit programs; timekeeping/payroll; and employee conduct (e.g., ethics violations). This information is maintained in the employee's secured personnel file.

Evidence of successful training could include such items as:

- Adequate documentation of training within operational areas, including one-on-one technical training for individual technologies, and particularly for people cross-trained.
- Analysts knowledge to refer to QA Manual for quality issues.
- Analysts following SOPs, i.e., practice matches SOPs.
- Analysts regularly communicate to supervisors and QA if SOPs need revision, rather than waiting for auditors to find problems.

Further details of the laboratory's training program are described in the Pittsburgh Laboratory SOP No. PT-QA-001, *Employee Orientation and Training*.

17.4 Data Integrity And Ethics Training Program

Establishing and maintaining a high ethical standard is an important element of a Quality System. Ethics and data integrity training is integral to the success of Eurofins TestAmerica and is provided for each employee at Eurofins TestAmerica. It is a formal part of the initial employee comprehensive ethics and data integrity training within 30 days of their start date, and an annual

(within at most 14 months of the previous training) refresher for all employees. Senior management at each facility performs the ethics training for their staff.

In order to ensure that all personnel understand the importance Eurofins TestAmerica places on maintaining high ethical standards at all times; Eurofins TestAmerica has established a Corporate Policy No. CW-L-P-004, *Ethics Policy*, and an Ethics Statement. All initial and annual training is documented by signature on the signed Ethics Statement demonstrating that the employee has participated in the training and understands their obligations related to ethical behavior and data integrity.

Violations of this Ethics Policy will not be tolerated. Employees who violate this policy will be subject to disciplinary actions up to and including termination. Criminal violations may also be referred to the Government for prosecution. In addition, such actions could jeopardize Eurofins TestAmerica's ability to do work on Government contracts, and for that reason, Eurofins TestAmerica has a Zero Tolerance approach to such violations.

Employees are trained as to the legal and environmental repercussions that result from data misrepresentation. Key topics covered in the presentation include:

- Organizational mission and its relationship to the critical need for honesty and full disclosure in all analytical reporting
- Ethics Policy
- How and when to report ethical/data integrity issues and confidential reporting
- Record keeping
- Discussion regarding data integrity procedures
- Specific examples of breaches of ethical behavior (e.g. peak shaving, altering data or computer clocks, improper macros, etc., accepting/offering kickbacks, illegal accounting practices, unfair competition/collusion)
- Internal monitoring, investigations, and data recalls
- Consequences for infractions, including potential for immediate termination, debarment, or criminal prosecution
- Importance of proper written narration / data qualification by the analyst and project manager with respect to those cases where the data may still be usable but are in one sense or another partially deficient

Additionally, a data integrity hotline (1-800-736-9407) is maintained by Eurofins TestAmerica and administered by the Corporate Quality Department.

SECTION 18

ACCOMMODATIONS AND ENVIRONMENTAL CONDITIONS

18.1 Overview

The laboratory is a 33,000 ft^2 secure laboratory facility with controlled access and designed to accommodate an efficient workflow and to provide a safe and comfortable work environment for employees. All visitors sign in and are escorted by laboratory personnel. Access is controlled by various measures.

The laboratory is equipped with structural safety features. Each employee is familiar with the location, use, and capabilities of general and specialized safety features associated with their workplace. The laboratory provides and requires the use of protective equipment including safety glasses, protective clothing, gloves, etc., OSHA and other regulatory agency guidelines regarding required amounts of bench and fume hood space, lighting, ventilation (temperature and humidity controlled), access, and safety equipment are met or exceeded.

Traffic flow through sample preparation and analysis areas is minimized to reduce the likelihood of contamination. Adequate floor space and bench top area is provided to allow unencumbered sample preparation and analysis space. Sufficient space is also provided for storage of reagents and media, glassware, and portable equipment. Ample space is also provided for refrigerated sample storage before analysis and archival storage of samples after analysis. Laboratory HVAC and deionized water systems are designed to minimize potential trace contaminants.

The laboratory is separated into specific areas for sample receiving, sample preparation, volatile organic sample analysis, non-volatile organic sample analysis, inorganic sample analysis, microbiological sample analysis, and administrative functions.

18.2 Environment

Laboratory accommodation, test areas, energy sources, lighting are adequate to facilitate proper performance of tests. The facility is equipped with heating, ventilation, and air conditioning (HVAC) systems appropriate to the needs of environmental testing performed at this laboratory.

The environment in which these activities are undertaken does not invalidate the results or adversely affect the required accuracy of any measurements.

The laboratory provides for the effective monitoring, control and recording of environmental conditions that may affect the results of environmental tests as required by the relevant specifications, methods, and procedures. Such environmental conditions include humidity, voltage, temperature, and vibration levels in the laboratory. Systems are controlled and monitored to assure constant and consistent test conditions.

When any of the method or regulatory required environmental conditions change to a point where they may adversely affect test results, analytical testing will be discontinued until the environmental conditions are returned to the required levels.

Environmental conditions of the facility housing the computer network and TALS are regulated to protect against raw data loss.

When a laboratory performs laboratory activities at sites of facilities outside its permanent control, it shall ensure that the requirements related to facilities and environmental conditions of this document are met.

Specific requirements for facility and environmental conditions, as well as periodic monitoring of conditions, are given in the Environmental Health & Safety Manual plus each laboratory's Facility Addendum.

18.3 Work Areas

There is effective separation between neighboring areas when the activities therein are incompatible with each other. Examples include:

- Microbiological culture handling and sample incubation areas
- Volatile organic chemical handling areas, including sample preparation and waste disposal, and volatile organic chemical analysis areas

Access to and use of all areas affecting the quality of analytical testing is defined and controlled by secure access to the laboratory building as described below in the Building Security section.

Adequate measures are taken to ensure good housekeeping in the laboratory and to ensure that any contamination does not adversely affect data quality. These measures include regular cleaning to control dirt and dust within the laboratory. Work areas are available to ensure an unencumbered work area. Work areas include:

- Access and entryways to the laboratory
- Sample receipt areas
- Sample storage areas
- Chemical and waste storage areas
- Data handling and storage areas
- Sample processing areas
- Sample analysis areas

Refer to the following documents and procedures for specific requirements for microbiological laboratory facilities:

- Standard Methods, 9020B, Sec. 2
- TNI V1M5, 1.7.3.7.a

18.4 Floor Plan

A floor plan can be found in Appendix 1.

18.5 Building Security

Building keys and alarm codes are distributed to employees as necessary.

Visitors to the laboratory sign in and out in a visitor's logbook. A visitor is defined as any person who visits the laboratory who is not an employee of the laboratory. In addition to signing into the laboratory, the Environmental, Health and Safety Manual contains requirements for visitors

and vendors. There are specific safety forms that must be reviewed and signed. Visitors (with the exception of company employees) are escorted by laboratory personnel at all times, or the location of the visitor is noted in the visitor's logbook.

SECTION 19

TEST METHODS AND METHOD VALIDATION

19.1 Overview

The laboratory uses methods that are appropriate to meet our clients' requirements and that are within the scope of the laboratory's capabilities. These include sampling, handling, transport, storage and preparation of samples, and, where appropriate, an estimation of the measurement of uncertainty as well as statistical techniques for analysis of environmental data.

Instructions are available in the laboratory for the operation of equipment as well as for the handling and preparation of samples. All instructions, Standard Operating Procedures (SOPs), reference methods and manuals relevant to the working of the laboratory are readily available to all staff. Deviations from published methods are documented (with justification) in the laboratory's approved SOPs. SOPs are submitted to clients for review at their request. Significant deviations from published methods require client approval and regulatory approval where applicable.

19.2 Standard Operating Procedures (SOPs)

The laboratory maintains SOPs that accurately reflect all phases of the laboratory such as assessing data integrity, corrective actions, handling customer complaints as well as all analytical methods and sampling procedures. The method SOPs are derived from the most recently promulgated/approved, published methods and are specifically adapted to the laboratory facility. Modifications or clarifications to published methods are clearly noted in the SOPs. All SOPs are controlled in the laboratory. A SOP list is included in Appendix 4. The most current list of SOPs is maintained in the QA SOP directory, Work Instruction PT-QA-WI-002.

- All SOPs contain a revision number, effective date, and appropriate approval signatures. Controlled copies are available to all staff.
- Procedures for writing an SOP are incorporated by reference to Eurofins TestAmerica's Corporate SOP No. CW-Q-S-002, *Writing a Standard Operating Procedure*, or Pittsburgh SOP PT-QA-010, *Document Development and Control*.
- SOPs are reviewed at a minimum of every 2 years, and where necessary, revised to ensure continuing suitability and compliance with applicable requirements. SOPs related to drinking water testing are reviewed every year.

19.3 Laboratory Methods Manual

For each test method, the laboratory shall have available the published referenced method as well as the laboratory developed SOP.

Note: If more stringent standards or requirements are included in a mandated test method or regulation than those specified in this manual, the laboratory shall demonstrate that such

requirements are met. If it is not clear which requirements are more stringent, the standard from the method or regulation is to be followed. Any exceptions or deviations from the referenced methods or regulations are noted in the specific analytical SOP.

The laboratory maintains an SOP Index (PT-QA-WI-002) for both technical and non-technical SOPs. Technical SOPs are maintained to describe a specific test method. Non-technical SOPs are maintained to describe functions and processes not related to a specific test method.

19.4 Selection Of Methods

Since numerous methods and analytical techniques are available, continued communication between the client and laboratory is imperative to assure the correct methods are utilized. Once client methodology requirements are established, this and other pertinent information is summarized by the Project Manager. These mechanisms ensure that the proper analytical methods are applied when the samples arrive for log-in. For non-routine analytical services (e.g., special matrices, non-routine compound lists), the method of choice is selected based on client needs and available technology. The methods selected should be capable of measuring the specific parameter of interest, in the concentration range of interest, and with the required precision and accuracy.

19.4.1 Sources of Methods

Routine analytical services are performed using standard EPA-approved methodology. In some cases, modification of standard approved methods may be necessary to provide accurate analyses of particularly complex matrices. When the use of specific methods for sample analysis is mandated through project or regulatory requirements, only those methods shall be used.

When clients do not specify the method to be used or methods are not required, the methods used will be clearly validated and documented in an SOP and available to clients and/or the end user of the data.

The analytical methods used by the laboratory are those currently accepted and approved by the U. S. EPA and the state or territory from which the samples were collected. Reference methods include:

- *Guidelines Establishing Test Procedures for the Analysis of Pollutants Under the Clean Water Act; Analysis and Sampling Procedures; 40CFR Part 136 as amended by Method Update Rule, August 28, 2017*
- *Methods for Chemical Analysis of Water and Wastes, EPA 600 (4-79-020), 1983*
- *Methods for the Determination of Inorganic Substances in Environmental Samples, EPA-600/R-93/100, August 1993*
- *Methods for the Determination of Metals in Environmental Samples, EPA/600/4-91/010, June 1991. Supplement I: EPA-600/R-94/111, May 1994*
- *Standard Methods for the Examination of Water and Wastewater, 18th/19th /20th /on-line edition; Eaton, A.D. Clesceri, L.S. Greenberg, A.E. Eds; American Water Works Association, Water Pollution Control Federation, American Public Health Association: Washington, D.C.*
- *Test Methods for Evaluating Solid Waste Physical/Chemical Methods (SW846), Third Edition, September 1986, Final Update I, July 1992, Final Update IIA, August 1993, Final Update II,*

September 1994; Final Update IIB, January 1995; Final Update III, December 1996; Final Update IV, January 2008, Final Update V, August 2015

- *Annual Book of ASTM Standards, American Society for Testing & Materials (ASTM), Philadelphia, PA.*
- *Code of Federal Regulations (CFR) 40, Parts 136, 141, 172, 173, 178, 179 and 261*
- *Evaluation of Dredged Material Proposed for Disposal at Island, Nearshore, or Upland Confined Disposal Facilities – Testing Manual, U.S. Army Corps of Engineers, January, 2003*

The laboratory reviews updated versions to all the aforementioned references for adaptation based upon capabilities, instrumentation, etc., and implements them as appropriate. As such, the laboratory strives to perform only the latest versions of each approved method as regulations allow or require.

Other reference procedures for non-routine analyses may include methods established by specific states (e.g., Underground Storage Tank methods), ASTM or equipment manufacturers. Sample type, source, and the governing regulatory agency requiring the analysis will determine the method utilized.

The laboratory shall inform the client when a method proposed by the client may be inappropriate or out of date. After the client has been informed, and they wish to proceed contrary to the laboratory's recommendation, it will be documented.

19.4.2 Demonstration of Capability

Before the laboratory may institute a new method and begin reporting results, the laboratory shall confirm that it can properly operate the method. In general, this demonstration does not test the performance of the method in real world samples, but in an applicable and available clean matrix sample. If the method is for the testing of analytes that are not conducive to spiking, demonstration of capability may be performed on quality control samples.

A demonstration of capability (DOC) is performed whenever there is a change in instrument type (e.g., new instrumentation), matrix, method or personnel (e.g., analyst hasn't performed the test within the last 12 months). The IDOC must meet the control limits specified in the reference method, if any, or meet method LCS criteria if no IDOC specific controls are provided. (see laboratory SOP No. PT-QA-001, *Employee Orientation and Training*, for details)

Note: The laboratory shall have a DOC for all analytes included in the methods that the laboratory performs, and proficiency DOCs for each analyst shall include all analytes that the laboratory routinely performs. Addition of non-routine analytes does not require new DOCs for all analysts if those analysts are already qualified for routine analytes tested using identical chemistry and instrument conditions.

The initial demonstration of capability must be thoroughly documented and approved by the Technical Manager and QA Manager prior to independently analyzing client samples. All associated documentation must be retained in accordance with the laboratories archiving procedures.

The laboratory must have an approved SOP, demonstrate satisfactory performance, and conduct an MDL study (when applicable). There may be other requirements as stated within the published method or regulations (i.e., retention time window study).

Note: In some instances, a situation may arise where a client requests that an unusual analyte be reported using a method where this analyte is not normally reported. If the analyte is being reported for regulatory purposes, the method must meet all procedures outlined within this QA Manual (SOP, MDL, and DOC). If the client states that the information is not for regulatory purposes, the result may be reported as long as the following criteria are met:

- The instrument is calibrated for the analyte to be reported using the criteria for the method and ICV/CCV criteria are met (unless an ICV/CCV is not required by the method or criteria are per project DQOs).
- The laboratory's nominal or default reporting limit (RL) is equal to the quantitation limit (QL), must be at or above the lowest non-zero standard in the calibration curve and must be reliably determined. Project RLs are client specified reporting levels which may be higher than the QL. Results reported below the QL must be qualified as estimated values. Also see Section 19.6.1.3, Relationship of Limit of Detection (LOD) to Quantitation Limit (QL).
- The client request is documented and the lab informs the client of its procedure for working with unusual compounds. The final report must be footnoted: *Reporting Limit based on the low standard of the calibration curve.*

19.4.3 Initial Demonstration of Capability (IDOC) Procedures

Initial Demonstration and Capability (IDOC) procedure is described in laboratory SOP No. PT-QA-001.

19.4.3.1 The spiking standard used to prepare IDOCs must be prepared independently from those used in instrument calibration.

19.4.3.2 The analyte(s) shall be diluted in a volume of clean matrix sufficient to prepare four aliquots at the concentration specified by a method or the laboratory SOP.

19.4.3.3 At least four aliquots shall be prepared (including any applicable clean-up procedures) and analyzed according to the test method (either concurrently or over a period of days).

19.4.3.4 Using all of the results, calculate the mean recovery in the appropriate reporting units and the standard deviations for each parameter of interest.

19.4.3.5 When it is not possible to determine the mean and standard deviations, such as for presence/absence and logarithmic values, the laboratory will assess performance against criteria described in the method SOP.

19.4.3.6 Compare the information obtained above to the corresponding acceptance criteria for precision and accuracy in the test method (if applicable) or in the laboratory generated acceptance criteria (LCS or interim criteria) if there is no mandatory criteria established. If any one of the parameters do not meet the acceptance criteria, the performance is unacceptable for that parameter.

19.4.3.7 When one or more of the tested parameters fail at least one of the acceptance criteria, the analyst must proceed according to either option below:

- Locate and correct the source of the problem and repeat the test for all parameters of interest beginning with 19.4.3.3 above.
- Beginning with 19.4.3.3 above, repeat the test for all parameters that failed to meet criteria. Repeated failure, however, will confirm a general problem with the measurement system. If this occurs, locate and correct the source

of the problem and repeat the test for all parameters beginning with 19.4.3.1 above.

Note: Results of successive LCS analyses can be used to fulfill the DOC requirement.

A certification statement (Figure 19-1) shall be used to document the completion of each initial and continuing demonstration of capability. A copy of the certification is archived in the analyst's training file.

Methods on line prior to the effective date of this Section shall be updated to the procedures outlined above as new analysts perform their demonstration of capability. A copy of the new record will replace that which was used for documentation in the past. At a minimum, the precision and accuracy of four mid-level laboratory control samples must have been compared to the laboratory's quality control acceptance limits for methods for which standards exist.

19.5 Laboratory Developed Methods And Non-Standard Methods

Any new method developed by the laboratory must be fully defined in an SOP and validated by qualified personnel with adequate resources to perform the method. Method specifications and the relation to client requirements must be clearly conveyed to the client if the method is a non-standard method (not a published or routinely accepted method). The client must also be in agreement to the use of the non-standard method.

19.6 Validation Of Methods

Validation is the confirmation by examination and the provision of objective evidence that the particular requirements for a specific intended use are fulfilled. Validation of a method is a planned activity. A coordinator is designated for the process, who's responsibility it is to communicate the process and progress to all involved personnel.

All non-standard methods, laboratory designed/developed methods, standard methods used outside of their scope, and major modifications to published methods must be validated to confirm they are fit for their intended use. The validation will be as extensive as necessary to meet the needs of the given application. The results are documented with the validation procedure used and contain a statement as to the fitness for use.

19.6.1 Method Validation and Verification Activities for All New Methods

While method validation can take various courses, the following activities can be required as part of method validation. Method validation records are designated QC records and are archived accordingly.

When changes are made to any validated methods, the influence of such changes shall be documented and, if appropriate, a new validation shall be performed.

19.6.1.1 Determination of Method Selectivity

Method selectivity is the demonstrated ability to discriminate the analyte(s) of interest from other compounds in the specific matrix or matrices from other analytes or interference. In some

cases to achieve the required selectivity for an analyte, a confirmation analysis is required as part of the method.

19.6.1.2 Determination of Method Sensitivity

Sensitivity can be both estimated and demonstrated. Whether a study is required to estimate sensitivity depends on the level of method development required when applying a particular measurement system to a specific set of samples. Detection limit studies are conducted as described in Section 19.7 below. Where other protocols for estimations and/or demonstrations of sensitivity are required by regulation or client agreement, these shall be followed.

19.6.1.3 Relationship of Limit of Detection (LOD) to the Limit of Quantitation (LOQ)

An important characteristic of expression of sensitivity is the distinction between the LOD and the LOQ. The LOD is the minimum level at which the presence of an analyte can be reliably concluded. The LOQ is the minimum concentration of analyte that can be quantitatively determined with acceptable precision and bias, equivalent to the laboratory's routine reporting limit (RL). For most instrumental measurement systems, there is a region where semi-quantitative data is generated around the LOD (both above and below the estimated MDL or LOD) and below the LOQ. In this region, detection of an analyte may be confirmed but quantification of the analyte is unreliable within the accuracy and precision guidelines of the measurement system. When an analyte is detected below the LOQ, and the presence of the analyte is confirmed by meeting the qualitative identification criteria for the analyte, the analyte can be reliably reported, but the amount of the analyte can only be estimated. If data is to be reported in this region, it must be done so with a qualification that denotes the semi-quantitative nature of the result.

19.6.1.4 Determination of Interferences

A determination that the method is free from interferences in a blank matrix is performed.

19.6.1.5 Determination of Range

Where appropriate to the method, the quantitation range is determined by comparison of the response of an analyte in a curve to established or targeted criteria. Generally the upper quantitation limit is defined by highest acceptable calibration concentration. The lower quantitation limit or QL cannot be lower than the lowest non-zero calibration level, and can be constrained by required levels of bias and precision.

19.6.1.6 Determination of Accuracy and Precision

Accuracy and precision studies are generally performed using replicate analyses, with a resulting percent recovery and measure of reproducibility (standard deviation, relative standard deviation) calculated and measured against a set of target criteria.

19.6.1.7 Documentation of Method

The method is formally documented in an SOP. If the method is a minor modification of a standard laboratory method that is already documented in an SOP, an SOP Attachment describing the specific differences in the new method is acceptable in place of a separate SOP.

19.6.1.8 Continued Demonstration of Method Performance

Continued demonstration of Method Performance is addressed in the SOP. Continued demonstration of method performance is generally accomplished by batch specific QC samples such as LCS, method blanks or PT samples.

19.7 Method Detection Limits (MDL)/ Limits Of Detection (LOD)

The MDL is the minimum measured quantity of a substance that can be reported with 99% confidence that the concentration is distinguishable from method blank results, consistent with 40CFR Part 136 Appendix B, August, 2017. The MDL is equivalent to the TNI LOD, and is also equivalent to the DoD/DOE Quality Systems Manual (QSM) DL. The working or final MDL is the higher of the MDL value determined from spikes (MDLs) and the MDL value determined from blanks (MDLb). An initial MDL study shall be performed during the method validation process and when the method Is altered in a way that can reasonably be expected to change the sensitivity. On-going data are collected during each quarter in which samples are being analyzed. At least once every 13 months, the MDLs and MDLb are re-calculated and re-evaluated using data collected during the preceding period. Details of Eurofins TestAmerica's procedure for conducting MDL studies are given in SOP No. CA-Q-S-006.

19.8 Verification of Detection Limits

If it is found during the re-evaluation of detection limit results that more than 5% of the spiked samples do not return numeric results that meet the method qualitative identification criteria, then the spiking level shall be increased and the initial MDL study re-performed at the new spiking concentration.

19.9 Instrument Detection Limits (IDL)

The IDL is sometimes used to assess the reasonableness of the MDLs or in some cases required by the analytical method or program requirements. IDLs are most used in metals analyses but may be useful in demonstration of instrument performance in other areas.

IDLs are calculated to determine an instrument's sensitivity independent of any preparation method. IDLs are calculated either using 7 replicate spike analyses, like MDL but without sample preparation, or by the analysis of 10 instrument blanks and calculating 3 x the absolute value of the standard deviation.

19.10 Limit of Quantitation

The LOQ shall be at a concentration equivalent to the lowest calibration standard concentration, with the exception of methods using a single-point calibration, and shall be greater than the MSL. The LOQ is verified by preparing and analyzing spikes at or below the LOQ concentration, employing the complete analytical process.

When the laboratory establishes a quantitation limit, it must be initially verified by the analysis of two low level standards or QC samples at the reporting limit and quarterly thereafter. The laboratory will comply with any additional regulatory requirements.

19.11 Retention Time Windows

Most organic analyses and some inorganic analyses use chromatography techniques for qualitative and quantitative determinations. For every chromatography analysis or as specific in

the reference method, each analyte will have a specific time of elution from the column to the detector. This is known as the analyte's retention time. The variance in the expected time of elution is defined as the retention time window. As the key to analyte identification in chromatography, retention time windows must be established on every column for every analyte used for that method. These records are kept on-file and available for review. Complete details are available in the laboratory SOPs.

19.12 Evaluation Of Selectivity

The laboratory evaluates selectivity by following the checks within the applicable analytical methods, which include mass spectral tuning, second column confirmation, ICP interelement interference checks, chromatography retention time windows, sample blanks, spectrochemical, atomic absorption or fluorescence profiles, co-precipitation evaluations and specific electrode response factors.

19.13 Estimation Of Uncertainty Of Measurement

19.13.1 Uncertainty is "a parameter associated with the result of a measurement, that characterizes the dispersion of the values that could reasonably be attributed to the measurand" (as defined by the International Vocabulary of Basic and General Terms in Metrology, ISO Geneva, 1993, ISBN 92-67-10175-1). Knowledge of the uncertainty of a measurement provides additional confidence in a result's validity. Its value accounts for all the factors which could possibly affect the result, such as adequacy of analyte definition, sampling, matrix effects and interferences, climatic conditions, variances in weights, volumes, and standards, analytical procedure, and random variation. Some national accreditation organizations require the use of an "expanded uncertainty" defined as the range within which the value of the measurand is believed to lie within at least a 95% confidence level with the coverage factor k=2.

19.13.2 Uncertainty is not error. Error is a single value (i.e. the difference between the true result and the measured result). On environmental samples, the true result is never known. The measurement is the sum of the unknown true value and the unknown error. Unknown error is a combination of systematic error, or bias, and random error. Bias varies predictably, constantly, and independently from the number of measurements. Random error is unpredictable, assumed to be Gaussian in distribution, and reducible by increasing the number of measurements.

19.13.3 The minimum uncertainty associated with results generated by the laboratory can be determined by using the Laboratory Control Sample (LCS) accuracy range for a given analyte. The LCS limits are used to assess the performance of the measurement system since they take into consideration all of the laboratory variables associated with a given test over time (except for variability associated with the sampling and the variability due to matrix effects). The percent recovery of the LCS is compared either to the method-required LCS accuracy limits or to the statistical, historical, in-house LCS accuracy limits.

19.13.4 To calculate the uncertainty for the specific result reported, multiply the result by the decimal of the lower end of the LCS range percent value for the lower end of the uncertainty range, and multiply the result by the decimal of the upper end of the LCS range percent value for the upper end of the uncertainty range. These calculated values represent uncertainties at approximately the 99% confidence level with a coverage factor of k =3. As an example, for a reported result of 1.0 mg/L with an LCS recovery range of 50 to 150%, the estimated uncertainty

in the result would be 1.0 +/- 0.5 mg/L. Uncertainty determination is further described in laboratory SOP PT-QA-005, *Measurement Uncertainty*.

19.13.5 In the case where a well recognized test method specifies limits to the values of major sources of uncertainty of measurement and specifies the form of presentation of calculated results, no further discussion of uncertainty is required.

19.14 Sample Reanalysis Guidelines

Because there is a certain level of uncertainty with any analytical measurement, a sample re-preparation (where appropriate) and subsequent analysis (hereafter referred to as 'reanalysis') may result in either a higher or lower value from an initial sample analysis. There are also variables that may be present (e.g., sample homogeneity, analyte precipitation over time, etc.) that may affect the results of a reanalysis. Based on the above comments, the laboratory will reanalyze samples at a client's request with the following caveats. **Client specific Contractual Terms & Conditions for reanalysis protocols may supersede the following items.**

- Homogenous samples: If a reanalysis agrees with the original result to within the RPD limits for MS/MSD or Duplicate analyses, or within $\pm$ 1 reporting limit for samples $\leq$ 5x the reporting limit, the original analysis will be reported. At the client's request, both results may be reported.
- If the reanalysis does not agree (as defined above) with the original result, then the laboratory will investigate the discrepancy and reanalyze the sample a third time for confirmation if sufficient sample is available.
- Any potential charges related to reanalysis are discussed in the contract terms and conditions or discussed at the time of the request. The client will typically be charged for reanalysis unless it is determined that the lab was in error.
- Due to the potential for increased variability, reanalysis may not be applicable to Non-homogenous, Encore, and Sodium Bisulfate preserved samples. See the Area Technical Manager/Supervisor or Laboratory Director if unsure.

19.15 Control Of Data

The laboratory has policies and procedures in place to ensure the authenticity, integrity, and accuracy of the analytical data generated by the laboratory.

19.15.1 Computer and Electronic Data Related Requirements

The three basic objectives of our computer security procedures and policies are shown below. The laboratory is currently using the Eurofins TestAmerica TALS LIMS system, which has been highly customized to meet the needs of the laboratory. It is referred ti as TALS throughout this document. More setailed descriptions of computer systems and associated controls are given in the IT Change Control Procedure Manual (CW-I-M-001) and policies and procedures posted on Eurofins TestAmerica's Intranet site, Oasis.

19.15.1.1 **Maintain the Database Integrity:** Assurance that data is reliable and accurate through data verification (review) procedures, password-protecting access, anti-virus protection, data change requirements, as well as an internal LIMS permissions procedure.

- LIMS Database Integrity is achieved through data input validation, internal user controls, documentation of system failures and corrective actions taken, and data change requirements.
- Spreadsheets and other software developed in-house must be verified with documentation through hand calculations prior to use. Cells containing calculations must be lock-protected and controlled.
- Instrument hardware and software adjustments are safeguarded through maintenance logs, audit trails and controlled access.

19.15.1.2 **Ensure Information Availability:** Protection against loss of information or service is ensured through scheduled back-ups, stable file server network architecture, secure storage of media, line filter, Uninterruptible Power Supply (UPS), and maintaining older versions of software as revisions are implemented.

19.15.1.3 **Maintain Confidentiality:** Ensure data confidentiality through physical access controls such as password protection or website access approval, when electronically transmitting data.

19.15.2 Data Reduction

The complexity of the data reduction depends on the analytical method and the number of discrete operations involved (e.g., extractions, dilutions, instrument readings and concentrations). The analyst calculates the final results from the raw data or uses appropriate computer programs to assist in the calculation of final reportable values.

For manual data entry, e.g., Wet Chemistry, the data is reduced by the analyst and then verified by the Department Manager or alternate analyst prior to updating the data in TALS. The spreadsheets, or any other type of applicable documents, are signed by both the analyst and alternate reviewer to confirm the accuracy of the manual entry(s). The applicable data/spreadsheet is scanned in TALS with the batch.

Manual integration of peaks will be documented and reviewed and the raw data will be flagged in accordance with the Eurofins TestAmerica Corporate SOP No. CA-Q-S-002, *Acceptable Manual Integration Practices*.

Analytical results are reduced to appropriate concentration units specified by the analytical method, taking into account factors such as dilution, sample weight or volume, etc. Blank correction will be applied only when required by the method or per manufacturer's indication; otherwise, it should not be performed. Calculations are independently verified by appropriate laboratory staff. Calculations and data reduction steps for various methods are summarized in the respective analytical SOPs or program requirements.

19.15.2.1 All raw data must be retained in the worklist folder, computer file (if appropriate), and/or run log. All criteria pertinent to the method must be recorded. The documentation is recorded at the time observations or calculations are made and must be signed or initialed/dated (month/day/year). It must be easily identifiable who performed which tasks if multiple people were involved.

19.15.2.2 In general, concentration results are reported in milligrams per liter (mg/l) or micrograms per liter (µg/l) for liquids and milligrams per kilogram (mg/kg) or micrograms per

kilogram (µg/kg) for solids. For values greater than 10,000 mg/l, results can be reported in percent, i.e., 10,000 mg/l = 1**%.** Units are defined in each lab SOP.

19.15.2.3 In reporting, the analyst or the instrument output records the raw data result using values of known certainty plus one uncertain digit. If final calculations are performed external to TALS, the results should be entered in LIMS with at least three significant figures. In general, results are reported to 2 significant figures on the final report.

19.15.2.4 For those methods that do not have an instrument printout or an instrumental output compatible with TALS, the raw results and dilution factors are entered directly into TALS by the analyst, and the software calculates the final result for the analytical report. TALS has a defined significant figure criterion for each analyte.

19.15.2.5 The laboratory strives to import data directly from instruments or calculation spreadsheets to ensure that the reported data are free from transcription and calculation errors. For those analyses with an instrumental output compatible with the TALS, the raw results and dilution factors are transferred into TALS electronically after reviewing the quantitation report, and removing unrequested or poor spectrally-matched compounds. The analyst prints a copy of what has been entered to check for errors. This printout and the instrument's printout of calibrations, concentrations, retention times, chromatograms, and mass spectra, if applicable, are retained with the data file. The data file is stored on the server and every night backed up to a tape file.

19.15.3 Logbook / Worksheet Use Guidelines

Logbooks and worksheets are filled out 'real time' and have enough information on them to trace the events of the applicable analysis/task. (e.g. calibrations, standards, analyst, sample ID, date, time on short holding time tests, temperatures when applicable, calculations are traceable, etc.)

- Corrections are made following the procedures outlined in Section 12.
- Logbooks are controlled by the QA department. A record is maintained of all logbooks in the lab.
- Unused portions of pages must be "Z"'d out, signed and dated.
- Worksheets are created with the approval of the Technical Manager / QA Manager at the facility.
  - Any cells that perform calculations must have the calculation verified and the cell locked so that the formula cannot be changed.
  - The QA Manager controls all worksheets following the procedures in Section 6.

19.15.4 Review / Verification Procedures

Review procedures are outlined in several SOPs (e.g. laboratory SOPs PT-QA-013, *Independent QA Data Review*; PT-QA-018, *Technical Data Review*; and PT-SR-001, *Sample Receipt and Login*) to ensure that reported date are free from calculation and transcription errors, and that QC parameters have been reviewed and evaluated before data is reported. The laboratory also has an SOP discussing Manual Integrations to ensure the authenticity of the data (CA-Q-S-002). The general review concepts are discussed below. More specific information can be found in the SOPs.

19.15.4.1 Log-In Review - The data review process starts at the sample receipt stage. Sample control personnel review chain-of-custody forms and project instructions from the project management group. This is the basis of the sample information and analytical instructions entered into the TALS. The log-in instructions are reviewed by the personnel entering the information, and a second level review is conducted by the project management staff.

19.15.4.2 First Level Data Review - The next level of data review occurs with the analysts. As data are generated, analysts review their work to ensure that the results meet project and SOP requirements. First level reviews include inspection of all raw data (e.g., instrument output for continuous analyzers, chromatograms, spectra, and manual integrations), evaluation of calibration/calibration verification data in the day's analytical run, evaluation of QC data, documentation of standards and reagents, and reliability of sample results. The analyst transfers data into TALS, data qualifiers are added as needed. All first level reviews are documented.

19.15.4.3 Second Level Data Review – All analytical data are subject to review by a second qualified analyst or supervisor. Second level reviews include inspection of all raw data (e.g., instrument output, chromatograms, and spectra) including 100% of data associated with any changes made by the primary analyst, such as manual integrations or reassignment of peaks to different analytes, or elimination of false negative analytes. The second review also includes evaluation of initial calibration/calibration verification data in the day's analytical run, evaluation of QC data, documentation of standards and reagents, reliability of sample results, qualifiers and NCM narratives. Manual calculations are checked in second level review. All second level reviews are documented.

Issues that deem further review include the following:

- QC data are outside the specified control limits for accuracy and precision
- Reviewed sample data does not match with reported results
- Unusual detection limit changes are observed
- Samples having unusually high results
- Samples exceeding a known regulatory limit
- Raw data indicating some type of contamination or poor technique
- Inconsistent peak integration
- Transcription errors
- Results outside of calibration range

19.15.4.4 Unacceptable analytical results may require reanalysis of the samples. Any problems are brought to the attention of the Laboratory Director, Project Manager, Quality Assurance Manager, Technical Director, Department Manager or section Supervisor for further investigation. Corrective action is initiated whenever necessary.

19.15.4.5 The results are then entered or directly transferred into the computer database and a hard copy (or .pdf) is printed for the client.

19.15.4.6 As a final review prior to the release of the report, the Project Manager (or designee) reviews the results for appropriateness and completeness. This review and approval ensures that client requirements have been met and that the final report has been properly

completed. The process includes, but is not limited to, verifying that the COC is followed, cover letters/ narratives are present, flags are appropriate, and project specific requirements are met. The Project Manager may also evaluate the validity of results for different test methods given expected chemical relationships.

19.15.4.7 Any project that requires a data package is subject to a tertiary data review for transcription errors and acceptable quality control requirements. The Project Manager (or designee) then signs the final report. They also check the report for any clerical or invoicing errors. When complete, the report is sent out to the client.

19.15.4.8 As a further check of the system, the QA department reviews both selected and random analytical batches and final reports.

19.15.4.9 A visual summary of the flow of samples and information through the laboratory, as well as data review and validation, is presented in Figure 19-2.

19.15.5 Manual Integrations

Computerized data systems provide the analyst with the ability to re-integrate raw instrument data in order to optimize the interpretation of the data. Though manual integration of data is an invaluable tool for resolving variations in instrument performance and some sample matrix problems, when used improperly, this technique would make unacceptable data appear to meet quality control acceptance limits. Improper re-integrations lead to legally indefensible data, a poor reputation, or possible laboratory decertification. Because guidelines for re-integration of data are not provided in the methods and most methods were written prior to widespread implementation of computerized data systems, the laboratory trains all analytical staff on proper manual integration techniques using Corporate SOP No. CA-Q-S-002.

19.15.5.1 The analyst must adjust baseline or the area of a peak in some situations, for example when two compounds are not adequately resolved or when a peak shoulder needs to be separated from the peak of interest. The analyst must use professional judgment to determine when manual integrating is required. Analysts are encouraged to ask for assistance from a senior analyst or manager when in doubt.

19.15.5.2 Analysts shall not increase or decrease peak areas for the sole purpose of achieving acceptable QC recoveries that would have otherwise been unacceptable. The intentional recording or reporting of incorrect information (or the intentional omission of correct information) is against company principles and policy and is grounds for immediate termination.

19.15.5.3 Client samples, performance evaluation samples, and quality control samples are all treated equally when determining whether or not a peak area or baseline should be manually adjusted.

19.15.5.4 All manual integrations receive a second level review. Manual integrations must be indicated on an expanded scale “before” and “after” chromatograms such that the integration performed can be easily evaluated during data review. Expanded scale “before” chromatograms are also required for all manual integrations on QC parameters (calibrations, calibration verifications, laboratory control samples, internal standards, surrogates, etc.) unless the laboratory has another documented Corporate approved procedure in place that can demonstrate an active process for detection and deterrence of improper integration practices.

Figure 19-1

Example - Demonstration of Capability Documentation

DEMONSTRATION OF CAPABILITIY (DOC)

Laboratory Name:_____
Laboratory Address:_____
Method:_____ Matrix:_____
Date:_____ Analyst(s):_____
Source of Analyte(s):_____

Analytical Results

Analyst	Conc. (Units)	Rep 1	Rep 2	Rep 3	Rep 4	Avg. % Recovery	% RSD
_____	_____	_____	_____	_____	_____	_____	_____

% RSD = Percent relative standard deviation = standard deviation divided by average % Recovery

Raw data reference: _____

Certification Statement:

We, the undersigned, certify that:

1. The analyst identified above, using the cited test method with the specifications in the cited SOP, which is in use at this facility for the analysis of samples under the laboratory's Quality Assurance Plan, has completed the Demonstration of Capability (DOC).
2. The test method(s) was performed by the analyst identified on this certificate.
3. A copy of the test method and the laboratory-specific SOPs are available for all personnel on site. These documents have been reviewed by the analyst as part of this DOC.
4. The data associated with the demonstration of capability are true, accurate, complete, and self-explanatory.
5. All raw data necessary to reconstruct and validate these analyses have been retained at the facility, and the associated information is well organized and available for review.

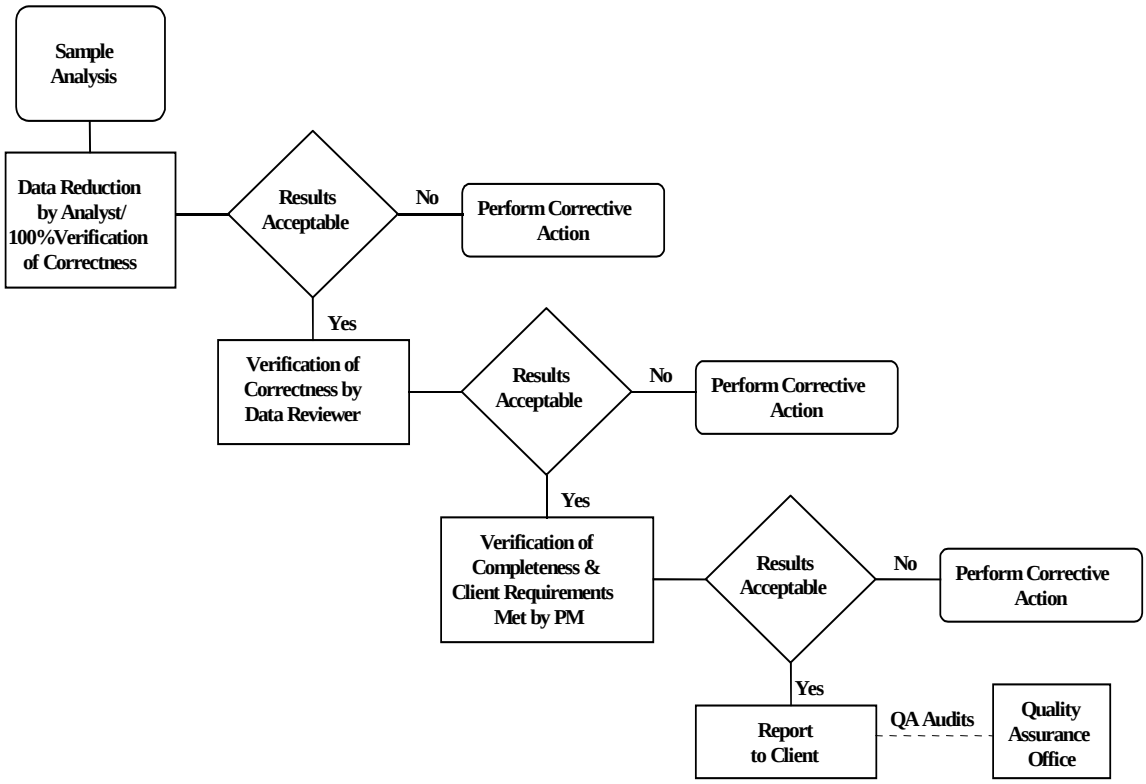
_____ _____
Analyst Signature Date

_____ _____
Technical Manager Signature Date

_____ _____
Quality Assurance Coordinator Signature Date

Figure 19-2

Example Work Flow



SECTION 20

EQUIPMENT AND CALIBRATIONS

20.1 Overview

The laboratory purchases the most technically advanced analytical instrumentation for sample analyses. Instrumentation is purchased on the basis of accuracy, dependability, efficiency and sensitivity. Each laboratory is furnished with all items of sampling, preparation, analytical testing and measurement equipment necessary to correctly perform the tests for which the laboratory has capabilities. Each piece of equipment is capable of achieving the required accuracy and complies with specifications relevant to the method being performed. Before being placed into use, the equipment (including sampling equipment) is calibrated and checked to establish that it meets its intended specification. The calibration routines for analytical instruments establish the range of quantitation. Calibration procedures are specified laboratory analytical SOPs. Also see Corporate Policy No. CA-Q-P-003, *Calibration Curves and Selection of Calibration Points*.

A list of laboratory instrumentation types is presented in Table 20-1.

Equipment is only operated by authorized and trained personnel. Manufacturer's instructions for equipment use are readily accessible to all appropriate laboratory personnel.

20.2 Preventive Maintenance

The laboratory follows a well-defined maintenance program to ensure proper equipment operation and to prevent the failure of laboratory equipment or instrumentation during use. This program of preventive maintenance helps to avoid delays due to instrument failure.

Routine preventive maintenance procedures and frequency, such as cleaning and replacements, should be performed according to the procedures outlined in the manufacturer's manual. Qualified personnel must also perform maintenance when there is evidence of degradation of peak resolution, a shift in the calibration curve, loss of sensitivity, or failure to continually meet one of the quality control criteria.

Table 20-2 lists examples of scheduled routine maintenance. It is the responsibility of each Technical Manager to ensure that instrument maintenance logs are kept for all equipment in his/her department. Preventative maintenance procedures are outlined in analytical SOPs or instrument manuals. Further detail for equipment maintenance is included in SOP No. PT-QA-022 and individual analytical SOPs. (Note: for some equipment, the log used to monitor performance is also the maintenance log. Multiple pieces of equipment may share the same log as long as it is clear as to which instrument is associated with an entry.)

Instrument maintenance logs are controlled and are used to document instrument problems, instrument repair and maintenance activities. Maintenance logs shall be kept for all major pieces of equipment. Instrument maintenance logs may also be used to specify instrument parameters.

- Documentation must include all major maintenance activities such as contracted preventive maintenance and service and in-house activities such as the replacement of electrical components, lamps, tubing, valves, columns, detectors, cleaning and adjustments.

- Each entry in the instrument log includes the Analyst's initials, the date, a detailed description of the problem (or maintenance needed/scheduled), a detailed explanation of the solution or maintenance performed, and a verification that the equipment is functioning properly (state what was used to determine a return to control. e.g. CCV run on *'date'* was acceptable, or instrument recalibrated on 'date' with acceptable verification, etc.) must also be documented in the instrument records.

- When maintenance or repair is performed by an outside agency, service receipts detailing the service performed can be affixed into the logbooks adjacent to pages describing the maintenance performed. This attached document must be signed across the page entered and the logbook so that it is clear that a page is missing if only half a signature is found in the logbook.

If an instrument requires repair (subjected to overloading or mishandling, gives suspect results, or otherwise has shown to be defective or outside of specified limits) it shall be taken out of operation and tagged as out-of-service or otherwise isolated until such a time as the repairs have been made and the instrument can be demonstrated as operational by calibration and/or verification or other test to demonstrate acceptable performance. The laboratory shall examine the effect of this defect on previous analyses.

In the event of equipment malfunction that cannot be resolved, service shall be obtained from the instrument vendor manufacturer, or qualified service technician, if such a service can be tendered. If on-site service is unavailable, arrangements shall be made to have the instrument shipped back to the manufacturer for repair. Back-up instruments, which have been approved, for the analysis shall perform the analysis normally carried out by the malfunctioning instrument. If the back-up is not available and the analysis cannot be carried out within the needed timeframe, the samples shall be subcontracted.

If an instrument is sent out for service or transferred to another facility, it must be recalibrated and the laboratory MDL verified prior to return to lab operations following the requirements in SOP PT-QA-007.

20.3 Support Equipment

This section applies to all devices that may not be the actual test instrument, but are necessary to support laboratory operations. These include but are not limited to: balances, ovens, refrigerators, freezers, incubators, water baths, field sampling devices, temperature measuring devices, thermal/pressure sample preparation devices and volumetric dispensing devices if quantitative results are dependent on their accuracy, as in standard preparation and dispensing or dilution into a specified volume. All raw data records associated with the support equipment are retained to document instrument performance.

Support equipment that provides quantitative results are calibrated or calibration verified to a recognized national metrology standard, such as NIST, where available, over the expected range of use. The acceptability for use shall be according to the needs of the analysis or application for which the equipment is being used.

Calibration and calibration verification scheduling and documentation for support equipment is maintained by the QA department. All equipment is labeled with the most recent calibration information and the next verification due date.

20.3.1 Weights and Balances

The accuracy of the balances used in the laboratory is checked every working day, before use. All balances are placed on stable counter tops.

Each balance is checked prior to initial serviceable use with at least three certified ASTM type 1 weights spanning its range of use (weights that have been calibrated to ASTM type 1 weights may also be used for daily verification). ASTM type 1 weights used only for calibration of other weights (and no other purpose) are inspected for corrosion, damage or nicks at least annually and if no damage is observed, they are calibrated at least every 5 years by an outside calibration laboratory. Any weights (including ASTM Type 1) used for daily balance checks or other purposes are recalibrated/recertified annually to NIST standards (this may be done internally if laboratory maintains "calibration only" ASTM type 1 weights).

All balances are serviced annually by a qualified service representative, who supplies the laboratory with a certificate that identifies traceability of the calibration to the NIST standards.

All of this information is recorded in logs, and the recalibration/recertification certificates are kept on file. Refer to Pittsburgh Laboratory SOP No. PT-QA-012, *Selection and Calibration of Balances and Weights*, for details.

20.3.2 pH, Conductivity, and Turbidity Meters

The pH meters used in the laboratory are accurate to ± 0.1 pH units, and have a scale readability of at least 0.05 pH units. The meters automatically compensate for the temperature, and are calibrated with at least two working range buffer solutions before each use.

Conductivity meters used in the laboratory are capable of measuring conductivity with an error not exceeding 1% or one umhos/cm, whichever is greater. The meters are also calibrated before each use with a known standard.

Turbidity meters are also calibrated before each use. All of this information is documented in logs.

Consult pH and Conductivity, and Turbidity laboratory analytical SOPs for further information.

20.3.3 Thermometers

All thermometers are calibrated on an annual basis with a NIST-traceable thermometer. IR thermometers, digital probes and thermocouples are calibrated quarterly. IR thermometers are checked daily for calibration accuracy against an NIST thermometer daily before use.

- If the temperature measuring device is used over a range of 10℃ or less, then a single point verification within the range of use is acceptable;
- If the temperature measuring device is used over a range of greater than 10℃, then the verification must bracket the range of use.

IR thermometers, digital probes and thermocouples are calibrated quarterly.

Mercury or digital NIST thermometers are recalibrated every five years (unless thermometer has been exposed to temperature extremes or apparent separation of internal liquid) by an approved outside service and the provided certificate of traceability is kept on file. The NIST

thermometer(s) have increments of at least 1 degree (0.5 degree or less increments are required for drinking water microbiological laboratories), and have ranges applicable to method and certification requirements. The NIST traceable thermometers are used for no other purpose than to calibrate other thermometers.

All of this information is documented in logbooks. Monitoring method-specific temperatures, including incubators, heating blocks, water baths, and ovens, is documented in method or device-specific logbooks. More information on this subject can be found in Pittsburgh Laboratory SOP No. PT-QA-008, *Thermometer and Barometer Verification and Temperature Monitoring*.

20.3.4 Refrigerators/Freezer Units, Waterbaths, Ovens and Incubators

The temperatures of all refrigerator units and freezers used for sample and standard storage are monitored each working day. Sample storage temperatures are monitored continuously (24/7).

Ovens, waterbaths and incubators are monitored on days of use.

All of this equipment has a unique identification number, and is assigned a thermometer for monitoring.

Sample storage refrigerator temperatures are kept between > 0ºC and ≤ 6 ºC.

Specific temperature settings/ranges for other refrigerators, ovens waterbaths, and incubators can be found in method specific SOPs.

All of this information is documented in Daily Temperature Logbooks or electronically. Refer to laboratory SOP PT-QA-008.

20.3.5 Autopipettors, Dilutors, and Syringes

Mechanical volumetric dispensing devices, including burettes, (except Class A Glassware and glass microliter syringes) are given unique identification numbers and the delivery volumes are verified gravimetrically, at a minimum, on a quarterly basis. Glass micro-syringes are considered the same as Class A glassware.

For those dispensers that are not used for analytical measurements, a label shall be applied to the device stating that it is not calibrated. Any device not regularly verified cannot be used for any quantitative measurements. Pipette calibration is described in Pittsburgh Laboratory SOP No. PT-QA-017, *Aqueous Pipette Calibration*.

Micro-syringes are purchased from Hamilton Company. Each syringe is traceable to NIST. The laboratory keeps on file an “Accuracy and Precision Statement of Conformance” from Hamilton attesting established accuracy.

20.3.6 Field Sampling Devices (Isco Auto Samplers)

Each Auto Sampler (ISCO) is assigned a unique identification number in order to keep track of the calibration. This number is also recorded on the sampling documentation.

The Auto Sampler is calibrated quarterly by setting the sample volume to 100ml and recording the volume received. The results are filed in a logbook/binder. The Auto Sampler is programmed to run three (3) cycles and each of the three cycles is measured into a graduated cylinder to verify 100ml are received.

If the RSD (Relative Standard Deviation) between the 3 cycles is greater than 10%, the procedure is repeated and if the result is still greater than 10%, then the Auto Sampler is taken out of service until it is repaired and calibration verification criteria can be met. The results of this check are kept in a logbook/binder.

20.4 Instrument Calibrations

Calibration of analytical instrumentation is essential to the production of quality data. Strict calibration procedures are followed for each method. These procedures are designed to determine and document the method detection limits, the working range of the analytical instrumentation and any fluctuations that may occur from day to day.

Sufficient raw data records are retained to allow an outside party to reconstruct all facets of the initial calibration. Records contain, but are not limited to, the following: calibration date, method, instrument, analyst(s) initials or signatures, analysis date, analytes, concentration, response, type of calibration (Avg RF, curve, or other calculations that may be used to reduce instrument responses to concentration.)

Sample results must be quantitated from the initial calibration and may not be quantitated from any continuing instrument calibration verification unless otherwise required by regulation, method or program.

If the initial calibration results are outside of the acceptance criteria, corrective action is performed and any affected samples are reanalyzed if possible. If the reanalysis is not possible, any data associated with an unacceptable initial calibration will be reported with appropriate data qualifiers (refer to Section 12).

Note: Instruments are calibrated initially and as needed after that and at least annually.

20.4.1 Calibration Standards

Calibration standards are prepared using the procedures indicated in the Reagents and Standards section of the determinative method SOP. If a reference method does not specify the number of calibration standards, a minimum of 3 calibration points (exception being ICP and ICP/MS methods) will be used.

Standards for instrument calibration are obtained from a variety of sources. All standards are traceable to national or international standards of measurement, or to national or international standard reference materials.

The lowest concentration calibration standard that is analyzed during an initial calibration must be at or below the stated reporting limit for the method based on the final volume of extract (or sample).

The other concentrations define the working range of the instrument/method or correspond to the expected range of concentrations found in actual samples that are also within the working

range of the instrument/method. Results of samples not bracketed by initial instrument calibration standards (within calibration range to at least the same number of significant figures used to report the data) must be reported as having less certainty, e.g., defined qualifiers or flags (additional information may be included in the case narrative). The exception to these rules is ICP and ICP-MS methods or other methods which define the working range with periodic linear dynamic range studies, rather than through the range of concentrations of daily calibration standards.

All initial calibrations are verified with a standard obtained from a second source and traceable to a national standard, when available (or vendor certified different lot if a second source is not available). For unique situations, such as air analysis where no other source or lot is available, a standard made by a different analyst at a different time or by a different preparation would be considered a second source. This verification occurs immediately after the calibration curve has been analyzed, and before the analysis of any samples.

20.4.1.1 Calibration Verification

The calibration relationship established during the initial calibration must be verified at least daily as specified in the laboratory method SOPs in accordance with the referenced analytical methods and in the 2009 and 2016 TNI standard. The process of calibration verification applies to both external standard and internal standard calibration techniques, as well as to linear and non-linear calibration models. Initial calibration verification (ICV) is with a standard source secondary (second source standard) to the calibration standards, but continuing calibration verifications (CCV) may use the same source standards as the calibration curve.

Note: The process of calibration verification referred to is fundamentally different from the approach called "calibration" in some methods. As described in those methods, the calibration factors or response factors calculated during calibration are used to update the calibration factors or response factors used for sample quantitation. This approach, while employed in other EPA programs, amounts to a daily single-point calibration.

All target analytes and surrogates, including those reported as non-detects, must be included in periodic calibration verifications for purposes of retention time confirmation and to demonstrate that calibration verification criteria are being met, i. e., RPD, per 2009 TNI Std. EL-V1M4 Sec. 1.7.2.

All samples must be bracketed by periodic analyses of standards that meet the QC acceptance criteria (e.g., calibration and retention time). The frequency is found in the determinative methods or SOPs.

Generally, the initial calibrations must be verified by an ICV analyzed immediately following initial calibration and before sample analysis. The ICV may be used as the first bracketing CCV if criteria for both are met.

A continuing instrument calibration verification (CCV) is generally analyzed at the beginning of each 12-hour analytical shift during which samples are analyzed. (The 12-hour analytical shift begins with the injection of the calibration verification standard (or the MS tuning standard is MS methods). The shift ends after the completion of the analysis of the last sample, QC, or standard that can be injected within 12 hours of the beginning of the shift. For methods that have quantitation by external calibration models, a CCV is analyzed at the end of each analytical sequence. Some methods have more frequent CCV requirements see specific SOPs.

Most Inorganic methods require the CCV to be analyzed after every 10 samples or injections, including matrix or batch QC samples.

Note: If an internal standard calibration is being used (e.g., GCMS and some GC methods) then bracketing standards are not required, only daily verifications are needed, except as specified by program or method requirements.

If the results of a CCV are outside the established acceptance criteria and analysis of a second consecutive (and immediate) CCV fails to produce results within acceptance criteria, corrective action shall be performed. Once corrective actions have been completed & documented, the laboratory shall demonstrate acceptable instrument / method performance by analyzing two consecutive CCVs, or a new initial instrument calibration shall be performed.

Sample analyses and reporting of data may not occur or continue until the analytical system is calibrated or calibration verified. However, data associated with a unacceptable calibration verification may be fully useable and may be reported, based upon discussion and approval of the client, under the following special conditions:

a) When the acceptance criteria for the CCV are exceeded high (i.e., high bias) and the associated samples within the batch are non-detects, then those non-detects may be reported with a footnote or case narrative comment explaining the high bias. Otherwise the samples affected by the unacceptable CCV shall be re-analyzed after a new calibration curve has been established, evaluated and accepted; or

b) When the acceptance criteria for the CCV are exceeded low (i.e., low bias), samples affected by the unacceptable CCV shall be re-analyzed after a new calibration curve has been established, evaluated and accepted.

Samples reported under one of the conditions identified above will be appropriately flagged.

20.4.1.2 Verification of Linear and Non-Linear Calibrations

Calibration verification for calibrations involves the calculation of the percent drift or the percent difference of the instrument response between the initial calibration and each subsequent analysis of the verification standard. (These calculations are available in the laboratory method SOPs.) Verification standards are evaluated based on the % Difference from the average CF or RF of the initial calibration or based on %Drift or %Recovery if a linear or quadratic curve is used.

Regardless of whether a linear or non-linear calibration model is used, if initial verification criterion is not met, then no sample analyses may take place until the calibration has been verified or a new initial calibration is performed that meets the specifications listed in the method SOPs. If the calibration cannot be verified after the analysis of a single verification standard, then adjust the instrument operating conditions and/or perform instrument maintenance, and analyze another aliquot of the verification standard. If the calibration cannot be verified with the second standard, then a new initial calibration is performed.

- When the acceptance criteria for the calibration verification are exceeded high, i.e., high bias, and there are associated samples that are non-detects, then those non-detects may be reported. Otherwise, the samples affected by the unacceptable calibration verification shall be reanalyzed after a new calibration curve has been established, evaluated and accepted.

- When the acceptance criteria for the calibration verification are exceeded low, i.e., low bias, those samples affected by the unacceptable verification shall be reanalyzed after a new calibration curve has been established, evaluated and accepted. Alternatively, a reporting limit standard may be analyzed to demonstrate that the laboratory can still support non-detects at their reporting limit.

Note: Some programs require additional verification steps for linear and quadratic calibration – i.e. reading the lowest, or all, initial calibration level standard against the curve, or verification at a low and a high concentration. See analytical SOPs and project notes for details.

20.5 Tentatively Identified Compounds (TICs) – GC/MS Analysis

For samples containing components not associated with the calibration standards, a library search may be made for the purpose of tentative identification. The necessity to perform this type of identification will be determined by the purpose of the analyses being conducted. Data system library search routines should not use normalization routines that would misrepresent the library or unknown spectra when compared to each other. Further details are given in policy memorandum CA-Q-QM-001, Policy on Tentatively Identified Compounds (TICs) – GC/MS Analysis.

Note: If the TIC compound is not part of the client target analyte list but is calibrated by the laboratory and is both qualitatively and/or quantitatively identifiable, it should not be reported as a TIC. If the compound is reported on the same form as true TICs, it should be qualified and/or narrated that the reported compound is qualitatively and quantitatively (if verification in control) reported compared to a known standard that is in control (where applicable).

20.6 GC/MS TUNING

Prior to any GCMS analytical sequence, including calibration, the instrument parameters for the tune and subsequent sample analyses within that sequence must be set.

Prior to tuning/auto-tuning the mass spectrometer the parameters may be adjusted within the specifications set by the manufacturer or the analytical method. These generally don't need any adjustment but it may be required based on the current instrument performance. If the tune verification does not pass it may be necessary to clean the source or perform additional maintenance. Any maintenance is documented in the maintenance log.

Table 20-1

Eurofins TestAmerica Pittsburgh Instrumentation Type List
(see PT-QA-WI-045 for the most current full listing of instrumentation and equipment)

GC	GC/MS	ICP	ICP/MS	IC	Carbon Analyzer	Auto-Analyzer	Variable Spectro-photometer	Electrode Meter
√	√	√	√	√	√	√	√	√

Tables 20-2

Example - Schedule of Routine Maintenance
(See SOP No. PT-QA-022 for more instrument specific information)

Daily	Weekly	Monthly	Quarterly	Annually	As Needed
Check sample waste container level.	Check peristaltic pump: proper roller pressure, sample introduction tubing, correct pump rotation, and condition of drain tubing.	Clean all filters and fans.	Replace oil in roughing pumps.	Replace oil in turbo-molecular pump.	Check electronic settings for optimum sensitivity: resolution, mass calibration, ion optics, CEM, deflector voltage.
Check quartz torch condition.	Check condition of sampler and skimmer cones.	Check recirculator water level.			
Measure quartz torch for proper alignment.	Check and drain oil mist eliminator on roughing pumps.				
Clean spray chamber and nebulizer.					
Check oil level of roughing pumps.					

SECTION 21

MEASUREMENT TRACEABILITY

21.1 Overview

Traceability of measurements shall be assured using a system of documentation, calibration, and analysis of reference standards. Laboratory equipment that are peripheral to analysis and whose calibration is not necessarily documented in a test method analysis or by analysis of a reference standard shall be subject to ongoing certifications of accuracy. At a minimum, these must include procedures for checking specifications of ancillary equipment: balances, thermometers, temperature, Deionized (DI) and Reverse Osmosis (RO) water systems, automatic pipettes and other volumetric measuring devices. (Refer to Section 20.3). With the exception of Class A Glassware and Glass microliter syringes, quarterly accuracy checks are performed for all mechanical volumetric devices. Microsyringes can be verified at least semi-annually or disposed of after 6 months of use. Wherever possible, subsidiary or peripheral equipment is checked against standard equipment or standards that are traceable to national or international standards. Class A Glassware and Glass microliter syringes should be routinely inspected for chips, acid etching or deformity (e.g., bent needle). If the Class A glassware or syringe is suspect, the accuracy of the glassware will be assessed prior to use.

21.2 NIST-Traceable Weights And Thermometers

Reference standards of measurement shall be used for calibration only and for no other purpose, unless it can be shown that their performance as reference standards would not be invalidated.
For NIST-traceable weights and thermometers, the laboratory requires that all calibrations be conducted by a calibration laboratory accredited by A2LA, NVLAP (National Voluntary Laboratory Accreditation Program) or another accreditation organization that is a signatory to a MRA (Mutual Recognition Arrangement) of one or more of the following cooperations – ILAC (International Laboratory Accreditation Cooperation) or APLAC (Asia – Pacific Laboratory Accreditation Cooperation). A certificate and scope of accreditation is kept on file at the laboratory.

Additional details can be found in laboratory SOP No's. PT-QA-008 and PT-QA-012.

21.3 Reference Standards / Materials

Reference standards/materials, where commercially available, are traceable to certified reference materials. Commercially prepared reference standards, to the extent available, are purchased from vendors accredited to ISO Guide 34 and ISO/IEC Guide 17025. All reference standards from commercial vendors shall be accompanied by a certificate that includes at least the following information:

- Manufacturer
- Analytes or parameters calibrated
- Identification or lot number
- Calibration method
- Concentration with associated uncertainties
- Purity

If a standard cannot be purchased from a vendor that supplies a Certificate of Analysis, the purity of the standard is documented by analysis. The receipt of all reference standards must be documented in the LIMS. Reference standards are labeled with a unique Standard Identification Number and expiration date. All documentation received with the reference standard is retained as a QC record and references the Standard Identification Number.

All reference, primary and working standards/materials, whether commercially purchased or laboratory prepared, must be checked regularly to ensure that the variability of the standard or material from the true value does not exceed method requirements. The accuracy of calibration standards is checked by comparison with a standard from a second source. In cases where a second standard manufacturer is not available, a vendor certified different lot is acceptable for use as a second source. For unique situations, such as air analysis where no other source or lot is available, a standard made by a different analyst would be considered a second source. The appropriate Quality Control (QC) criteria for specific standards are defined in laboratory SOPs. In most cases, the analysis of an Initial Calibration Verification (ICV) or LCS (where there is no sample preparation) is used as the second source confirmation. These checks are generally performed as an integral part of the analysis method (e.g. calibration checks, laboratory control samples).

All standards and materials must be stored and handled according to method or manufacturer's requirements in order to prevent contamination or deterioration. Refer to the Corporate Environmental Health & Safety Manual or laboratory SOPs. For safety requirements, please refer to method SOPs and the laboratory Environmental Health and Safety Manual.

Standards and reference materials shall not be used after their expiration dates unless their reliability is verified by the laboratory and their use is approved by the Quality Assurance Manager. The laboratory has contingency procedures for re-verifying expired standards. See Pittsburgh Laboratory SOP No. PT-QA-006, *Procurement of Standards and Materials, Labeling and Traceability*.

21.4 Documentation And Labeling Of Standards, Reagents, And Reference Materials

Reagents must be at a minimum the purity required in the test method. The date of reagent receipt and the expiration date are documented. The lots for most of the common solvents and acids are tested for acceptability prior to company-wide purchase. (Refer to Eurofins TestAmerica's Corporate SOP No. CA-Q-S-001, *Acid & Solvent Lot Testing and Approval*.)

All manufacturer or vendor supplied Certificate of Analysis or Purity must be retained, stored appropriately, and readily available for use and inspection. These records are maintained in the QA public drive and in the LIMS, scanned into Reagent log. Records must be kept of the date of receipt and date of expiration of standards, reagents and reference materials. In addition, records of preparation of laboratory standards, reagents, and reference materials must be retained, stored appropriately, and be readily available for use and inspection. For detailed information on documentation and labeling, please refer to laboratory analytical SOPs and SOP PT-QA-006.

Commercial materials purchased for preparation of calibration solutions, spike solutions, etc., are usually accompanied with an assay certificate or the purity is noted on the label. If the assay

purity is 96% or better, the weight provided by the vendor may be used without correction. If the assay purity is less than 96% a correction will be made to concentrations applied to solutions prepared from the stock commercial material.

21.4.1 All standards, reagents, and reference materials that may affect quality must be labeled in an unambiguous manner. Standards are logged into TALS, and are assigned a unique identification number. The following information is typically recorded within TALS.

- Standard ID
- Description of Standard
- Department
- Preparer's name
- Final volume and number of vials prepared
- Solvent type and lot number
- Preparation Date or Date opened
- Expiration Date
- Standard source type (stock or daughter)
- Standard type (spike, surrogate, other)
- Parent standard ID (if applicable)
- Parent Standard Analyte Concentration (if applicable)
- Parent Standard Amount used (if applicable)
- Component Analytes
- Final concentration of each analyte
- Comment box (text field)

Records are maintained electronically for standard and reference material preparation. These records show the traceability to purchased stocks or neat compounds. These records also include method of preparation, date of preparation, expiration date and preparer's name or initials. Preparation procedures are provided in the laboratory analytical SOPs.

21.4.2 All standards, reagents, and reference materials must be clearly labeled with a minimum of the following information:

- Date prepared or Date opened
- Expiration Date (include prep date for reagents)
- Standard ID (from electronic standard log in TALS)
- Special Health/Safety warnings if applicable

Records must also be maintained of the date of receipt for commercially purchased items or date of preparation for laboratory prepared items. Special Health/Safety warnings must also be available to the analyst. This information is maintained in standard/reagent log. Health and safety warning are in the SDS (Safety Data Sheets) which is accessed through the company intranet site.

21.4.3 In addition, the following information may be included:

- Description of standard (if different from manufacturer's label or if standard was prepared in the laboratory)
- Recommended Storage Conditions
- Concentration (if applicable)
- Initials of analyst preparing standard or opening container

All containers of prepared reagents must include, expiration date and an ID number to trace back to preparation.

Procedures for preparation of reagents can be found in the Method SOPs.

Standard ID numbers must be traceable through associated logbooks, worksheets and raw data.

All reagents and standards must be stored in accordance to the following priority: 1) with the manufacturer's recommendations; 2) with requirements in the specific analytical methods as specified in the laboratory SOP.

SECTION 22

SAMPLING

22.1 Overview

The laboratory provides sampling services for the following matrices:

- Groundwater
- Wastewater
- Potable Water
- Wastes
- Soil and Sediment

The laboratory also offers the following services:

- Flow Monitoring
- Field Parameter Analysis

Field Analyses are address in Eurofins TestAmerica Pittsburgh SOP No.'s:

- PT-FS-001 – Field Measurement of Dissolved Oxygen (DO)
- PT-FS-002 – Field Measurement of Total Residual Chlorine
- PT-FS-003 – Field Measurement of pH
- PT-FS-004 – General Sampling

22.2 Sampling Containers

The laboratory offers clean sampling containers for use by clients. These containers are obtained from reputable container manufacturers and meet EPA specifications as required. Certificates of cleanliness for bottles and preservatives are provided by the supplier and are

maintained at the laboratory. Alternatively, the certificates may be maintained by the supplier and available to the laboratory on-line.
For detailed information regarding container/bottle order, refer to laboratory SOP No. PT-SR-002, *Bottle Order Preparation and Shipping*.

22.2.1 Preservatives

Upon request, preservatives are provided to the client in pre-cleaned sampling containers. In general, containers are purchased pre-preserved from the container supplier. Whether prepared by the laboratory or bought pre-preserved, the grades of the preservatives are at a minimum:

- Hydrochloric Acid – AR Select (ACS) or equivalent
- Methanol – Purge and Trap grade
- Nitric Acid – AR Select (ACS), Trace-Metals Grade or equivalent
- Sodium Hydroxide – AR Select (ACS) or equivalent
- Sulfuric Acid – AR Select (ACS) or equivalent
- Sodium Thiosulfate – ACS Grade or equivalent
- Sodium Bisulfate – ACS Grade or equivalent

22.3 Definition Of Holding Time

The date and time of sampling documented on the COC form establishes the day and time zero. As a general rule, when the maximum allowable holding time is expressed in days (e.g., 14 days, 28 days), the holding time is based on calendar day measured. Holding times expressed in hours (e.g., 6 hours, 24 hours, etc.) are measured from date and time zero. However, there are some programs that determine holding time compliance based on the date and specific time of analysis compared to the time of sampling regardless of how long the holding time is. Holding times for analysis include any necessary reanalysis.

22.4 Sampling Containers, Preservation Requirements, Holding Times

The container type, preservation, and holding time criteria specified in the SOPs are derived from the source documents for the methods. If method required holding times as specified in the SOPs or preservation requirements are not met, the reports will be qualified using a flag, footnote or case narrative. As soon as possible, or “ASAP”, is an EPA designation for tests for which rapid analysis is advised, but for which neither EPA nor the laboratory have a basis for a holding time.

22.5 Sample Aliquots / Subsampling

Taking a representative sub-sample from a container is necessary to ensure that the analytical results are representative of the sample collected in the field. The size of the sample container, the quantity of sample fitted within the container, and the homogeneity of the sample need consideration when sub-sampling for sample preparation. It is the laboratory’s responsibility to take a representative subsample or aliquot of the sample provided for analysis.

Analysts should handle each sample as if it is potentially dangerous. At a minimum, safety glasses, gloves, and lab coats must be worn when preparing aliquots for analysis.

Guidelines for subsampling are located in laboratory SOP PT-QA-024.

SECTION 23

HANDLING OF SAMPLES

Sample management procedures at the laboratory ensure that sample integrity and custody are maintained and documented from sampling/receipt through disposal. Details can be found in laboratory SOP No. PT-SR-001, *Sample Receipt and Login*, and PT-HS-001, *Pittsburgh Facility Addendum EH&S Manual*.

23.1 Chain Of Custody (COC)

The COC form is the written documented history of any sample and is initiated when bottles are sent to the field, or at the time of sampling. This form is completed by the sampling personnel and accompanies the samples to the laboratory where it is received and stored under the laboratory's custody. The purpose of the COC form is to provide a legal written record of the handling of samples from the time of collection until they are received at the laboratory. It also serves as the primary written request for analyses from the client to the laboratory. The COC form acts as a purchase order for analytical services when no other contractual agreement is in effect. An example of a COC form may be found in Figure 23-1.

23.1.1 Field Documentation

The information the sampler needs to provide at the time of sampling on the container label is:

- Sample identification
- Date and time
- Preservative

During the sampling process, the COC form is completed and must be legible (see Figure 23-1). This form includes information such as:

- Client name, address, phone number and fax number (if available)
- Project name and/or number
- The sample identification
- Date, time and location of sampling
- Sample collectors name
- The matrix description
- The container description
- The total number of each type of container
- Preservatives used
- Analysis requested
- Requested turnaround time (TAT)
- Any special instructions
- Purchase Order number or billing information (e.g. quote number) if available
- The date and time that each person received or relinquished the sample(s), including their signed name.

When the sampling personnel deliver the samples directly to Eurofins TestAmerica personnel, the samples are stored in a cooler with ice, as applicable, and remain solely in the possession of the client's field technician until the samples are delivered to the laboratory personnel. The sample collector must assure that each container is in his/her physical possession or in his/her view at all times, or stored in such a place and manner to preclude tampering. The field technician relinquishes the samples in writing on the COC form to the sample control personnel at the laboratory or to a Eurofins TestAmerica courier. When sampling personnel deliver the samples through a common carrier (Fed-Ex, UPS), the CoC relinquished date/time is completed by the field personnel and samples are released to the carrier. Samples are only considered to be received by laboratory when personnel at the fixed laboratory facility have physical contact with the samples.

Note: Independent couriers are not required to sign the COC form. The COC is usually kept in the sealed sample cooler. The receipt from the courier is stored in login by date; it lists all receipts for each date.

23.1.2 Legal / Evidentiary Chain-of-Custody

If samples are identified for legal/evidentiary purposes on the COC, login will complete the custody seal retain the shipping record with the COC, and initiate an internal COC for laboratory use by analysts and a sample disposal record.

23.2 Sample Receipt

Samples are received at the laboratory by designated sample receiving personnel and a unique laboratory project identification number is assigned. Each sample container shall be assigned a unique sample identification number that is cross-referenced to the client identification number such that traceability of test samples is unambiguous and documented. Each sample container is affixed with a durable sample identification label. Sample acceptance, receipt, tracking and storage procedures are summarized in the following sections.

23.2.1 Laboratory Receipt

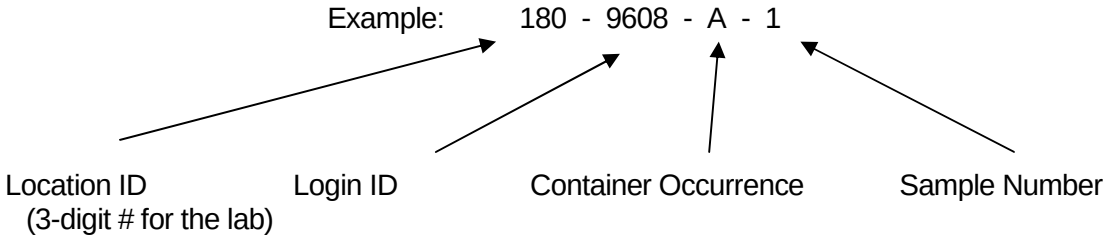
When samples arrive at the laboratory, sample receiving personnel inspect the coolers and samples. The integrity of each sample must be determined by comparing sample labels or tags with the COC and by visual checks of the container for possible damage. Samples and COC must meet the laboratory Sample Acceptance Policy (Figure 23-2). Any non-conformance, irregularity, or compromised sample receipt must be documented in TALS on the Sample Receipt checklist (Figure 23-3), and must be brought to the immediate attention of the client. The COC, shipping documents, documentation of any non-conformance, irregularity, or compromised sample receipt, record of client contact, and resulting instructions become part of the project record. This procedure is further described in laboratory SOP PT-SR-01.

23.2.1.1 Unique Sample Identification

All samples that are processed through the laboratory receive a unique sample identification to ensure that there can be no confusion regarding the identity of such samples at anytime. This

system includes identification for all samples, subsamples and subsequent extracts and/or digestates.

The laboratory assigns a unique identification (e.g., Sample ID) code to each sample container received at the laboratory. This Primary ID is made up of the following information (consisting of 4 components):



The above example states that Eurofins TestAmerica Pittsburgh Laboratory (Location 180). Login ID is 9608 (unique to a particular client/job occurrence). The container code indicates it is the first container ("A") of Sample #1.

If the primary container goes through a prep step that creates a "new" container, then the new container is considered secondary and gets another ID. An example of this being a client sample in a 1-Liter amber bottle is sent through a Liquid/Liquid Extraction and an extraction vial is created from this step. The vial would be a SECONDARY container. The secondary ID has 5 components.

Example: 180 - 9608 - A - 1 - **A** ←——**Secondary Container Occurrence**

Example: 180-9608-A-1-A, would indicate the PRIMARY container listed above that went through a step that created the 1st occurrence of a Secondary container.

With this system, a client sample can literally be tracked throughout the laboratory in every step from receipt to disposal.

23.3 Sample Acceptance Policy

The laboratory has a written sample acceptance policy (Figure 23-2) that clearly outlines the circumstances under which samples shall be accepted or rejected. These include:

- a COC must filled out completely
- samples must be properly labeled
- proper sample containers with adequate volume for the analysis and necessary QC
- samples must be preserved according to the requirements of the requested analytical method
- sample holding times must be adhered to
- Samples that require chilling must be received proper temperature
- the project manager will be notified if any sample is received in damaged condition

Data from samples which do not meet these criteria are flagged and the nature of the variation from policy is defined.

23.3.1.1 After inspecting the samples, the sample receiving personnel sign and date the COC form, make any necessary notes of the samples' conditions and store them in appropriate refrigerators or storage locations.

23.3.1.2 Any deviations from these checks that question the suitability of the sample for analysis, or incomplete documentation on the chain-of-custody will be resolved by consultation with the client. If the sample acceptance policy criteria are not met, the laboratory shall either:

- Retain all correspondence and/or records of communications with the client regarding the disposition of rejected samples, or
- Fully document any decision to proceed with sample analysis that does not meet sample acceptance criteria. Include information for the case narrative of the report.
- If the conditions listed on the Acceptance Policy are not satisfactory and when lacking direction or agreement with the client, the sample may be rejected by the laboratory.
- Any deviation that impacts sample results, such as temperature and preservation, shall be noted on sample results.

Note: North Carolina requires that they be notified when samples are processed that do not meet sample acceptance criteria.

Once sample acceptance is verified, the samples are logged into the TALS according to laboratory SOP PT-SR-001.

23.4 Sample Storage

In order to avoid deterioration, contamination or damage to a sample during storage and handling, from the time of receipt until all analyses are complete, samples are stored in refrigerators, freezers, or protected locations suitable for the sample matrix. In addition, samples to be analyzed for volatile organic parameters are stored in separate refrigerators designated for volatile organic parameters only. Samples are never to be stored with reagents, standards or materials that may create contamination.

To ensure the integrity of the samples during storage, refrigerator blanks are maintained in the volatile sample refrigerators and analyzed at least every two weeks. See laboratory SOP PT-MS-005, *Volatile Holding Blanks*.

Analysts and technicians retrieve the sample container allocated to their analysis from the designated storage location and place them on carts, analyze the sample, and return the remaining sample or empty container to the storage location from which it originally came. All unused portions of samples, including empty sample containers, are returned to the secure sample control area. Raw samples requiring cold storage are kept in the cold room for approximately 30 days after reported. Volatile samples are stored in the VOA refrigerator. All sample extracts are kept in the refrigerators for approximately two to four weeks after analysis,

which meets or exceeds most sample holding times. After the cold storage time, the samples are moved to dry room-temperature sample archive area, where they are stored for an additional four weeks before they are disposed of. This holding period allows samples to be checked if a discrepancy or question arises. Special arrangements may be made to store samples for longer periods of time. This extended holding period allows additional metal analyses to be performed on the archived sample and assists clients in dealing with legal matters or regulatory issues.

Access to the laboratory is controlled such that sample storage need not be locked at all times unless a project specifically demands it. Samples are accessible to laboratory personnel only. Visitors to the laboratory are prohibited from entering the refrigerator and laboratory areas unless accompanied by an employee of Eurofins TestAmerica.

23.5 Hazardous Samples And Foreign Soils

To minimize exposure to personnel and to avoid potential accidents, hazardous, for any sample that is known to be hazardous at the time of receipt a cautionary email communication should be sent to all applicable laboratory personnel by the project manager or designee. All hazardous samples are disposed of appropriately through a hazardous waste disposal process. Foreign soil samples are sent out for incineration by an USDA-approved waste disposal facility. Analysts will notify Sample Control of any sample determined to be hazardous after completion of analysis by sending an email. All hazardous samples are either returned to the client or disposed of appropriately through a hazardous waste disposal firm that lab-packs all hazardous samples and removes them from the laboratory. Foreign soil samples are sent out for incineration by a USDA-approved waste disposal facility.

23.6 Sample Shipping

In the event that the laboratory needs to ship samples, the samples are placed in a cooler with enough ice to ensure the samples remain just above freezing and at or below 6.0°C during transit. The samples are carefully surrounded by packing material to avoid breakage (yet maintain appropriate temperature). A trip blank is enclosed for those samples requiring water/solid volatile organic analyses. The chain-of-custody form is signed by the sample control technician and attached to the shipping paperwork. Samples are generally shipped overnight express or hand-delivered by a Eurofins TestAmerica courier to maintain sample integrity. All personnel involved with shipping and receiving samples must be trained to maintain the proper chain-of-custody documentation and to keep the samples intact and on ice. The Environmental, Health and Safety Manual contains additional shipping requirements.

Note: If a client does not request trip blank analysis on the COC or other paperwork, the laboratory will not analyze the trip blanks that were supplied. However, in the interest of good client service, the laboratory will advise the client at the time of sample receipt that it was noted that they did not request analysis of the trip blank; and that the laboratory is providing the notification to verify that they are not inadvertently omitting a key part of regulatory compliance testing.

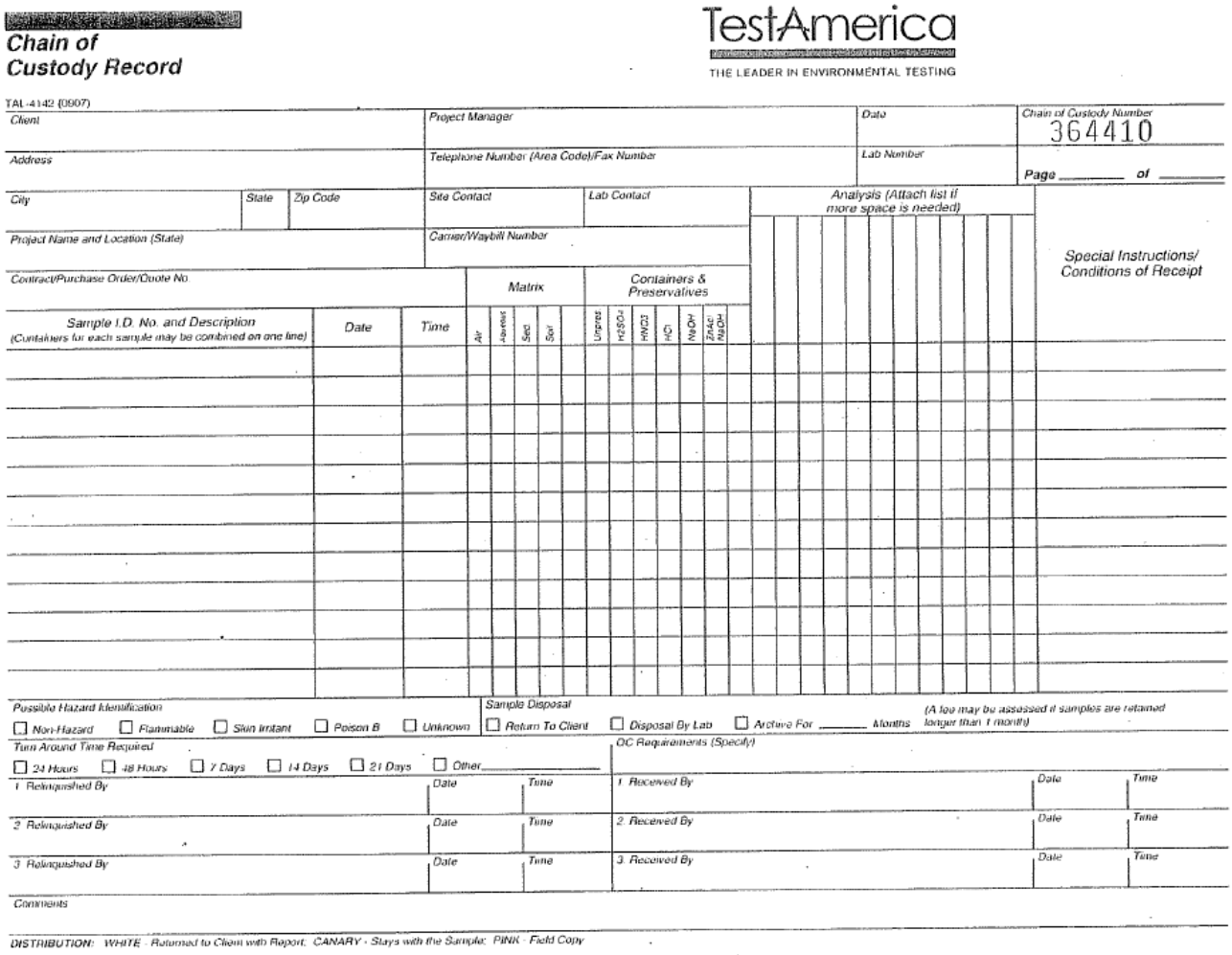
23.7 Sample Disposal

Samples should be retained for a minimum of 30 days after the project report is sent, however, provisions may be made for earlier disposal of samples once the holding time is exceeded. Some samples are required to be held for longer periods based on regulatory or client requirements (e.g., 60 days after project report is sent). The laboratory must follow the longer sample retention requirements where required by regulation or client agreement. Several possibilities for sample disposal exist: the sample may be consumed completely during analysis, the sample may be returned to the customer or location of sampling for disposal, or the sample may be disposed of in accordance with the laboratory's waste disposal procedures (SOP No. PT-HS-001). All procedures in the laboratory Environmental, Health and Safety Manual are followed during disposal. Samples are normally maintained in the laboratory no longer than two months from receipt unless otherwise requested. Unused portions of samples found or suspected to be hazardous according to state or federal guidelines may be returned to the client upon completion of the analytical work.

If a sample is part of a known litigation, the affected legal authority, sample data user, and/or submitter of the sample must participate in the decision about the sample's disposal. All documentation and correspondence concerning the disposal decision process must be kept on file. Pertinent information includes the date of disposal, nature of disposal (such as sample depletion, hazardous waste facility disposal, return to client), names of individuals who conducted the arrangements and physically completed the task. The laboratory will remove or deface sample labels prior to disposal unless this is accomplished through the disposal method (e.g., samples are incinerated). A Waste Disposal Record should be completed.

Figure 23-1

Example: Chain of Custody (COC)



Chain of Custody Record

TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

TAL-4142 (0907)

Client	Project Manager	Date	Chain of Custody Number 364410
Address	Telephone Number (Area Code)/Fax Number	Lab Number	Page _____ of _____
City / State / Zip Code	Site Contact	Lab Contact	Analysis (Attach list if more space is needed)
Project Name and Location (State)	Carrier/Waybill Number		Special Instructions/ Conditions of Receipt
Contract/Purchase Order/Quote No.	Matrix / Containers & Preservatives		

Sample I.D. No. and Description (Containers for each sample may be combined on one line)	Date	Time	Air	Aqueous	Sed.	Soil	Unpres.	H_2SO_4	HNO_3	HCl	NaOH	ZnAc/ NaOH

Possible Hazard Identification
☐ Non-Hazard ☐ Flammable ☐ Skin Irritant ☐ Poison B ☐ Unknown

Sample Disposal
☐ Return To Client ☐ Disposal By Lab ☐ Archive For _____ Months (A fee may be assessed if samples are retained longer than 1 month)

Turn Around Time Required
☐ 24 Hours ☐ 48 Hours ☐ 7 Days ☐ 14 Days ☐ 21 Days ☐ Other_____

QC Requirements (Specify)

1. Relinquished By	Date	Time	1. Received By	Date	Time
2. Relinquished By	Date	Time	2. Received By	Date	Time
3. Relinquished By	Date	Time	3. Received By	Date	Time

Comments

DISTRIBUTION: WHITE - Returned to Client with Report; CANARY - Stays with the Sample; PINK - Field Copy

Figure 23-2

Sample Acceptance Policy

All incoming work will be evaluated against the criteria listed below. Where applicable, data from any samples that do not meet the criteria listed below will be noted on the laboratory report defining the nature and substance of the variation. In addition the client will be notified either by telephone, fax or e-mail ASAP after the receipt of the samples.

1) Samples must arrive with labels intact with a Chain of Custody filled out completely. The following information must be recorded.
   - *Client name, address, phone number and fax number (if available)*
   - *Project name and/or number*
   - *Unique sample identification*
   - *Date, time and location of sampling*
   - *The collectors name*
   - *The matrix description*
   - *The container description*
   - *The total number of each type of container*
   - *Preservatives used*
   - *Analysis requested*
   - *Requested turnaround time (TAT)*
   - *Any special instructions*
   - *Purchase Order number or billing information (e.g. quote number) if available*
   - *The date and time that each person received or relinquished the sample(s), including their signed name.*
   - **Information must be legible**

2) Samples must be properly labeled.
   - Use durable labels (labels provided by Eurofins TestAmerica are preferred)
   - Include a unique identification number
   - Include sampling date and time & sampler ID
   - Include preservative used.
   - Use indelible ink
   - **Information must be legible**

3) Proper sample containers with adequate volume for the analysis and necessary QC are required for each analysis requested.

4) Samples must be preserved according to the requirements of the requested analytical method. (See Sampling Guide)

5) Most analytical methods require chilling samples to 4^{o} C (other than water samples for metals analysis). For these methods, the criteria are met if the samples are chilled to below 6^{o} C and above freezing (0^{o}C). For methods with other temperature criteria (e.g. some bacteriological methods require $\leq$ 10 ^{o}C), the samples must arrive within $\pm$ 2^{o} C of the required temperature or within the method specified range. **Note:** Samples that are hand delivered to the laboratory immediately after

collection may not have had time to cool sufficiently. In this case the samples will be considered acceptable as long as there is evidence that the chilling process has begun (arrival on ice).

5i.) Samples that are delivered to the laboratory on the same day they are collected may not meet the requirements of Section 5. In these cases, the samples shall be considered acceptable if the samples were received on ice.

5ii.) If sample analysis is begun within fifteen (15) minutes of collection, thermal preservation is not required.

5iii.) Thermal preservation is not required in the field if the laboratory receives and refrigerates the sample within fifteen (15) minutes of collection.

- Chemical preservation (pH) will be verified prior to analysis and documented, either in sample control or at the analyst's level. The project manager will be notified immediately if there is a discrepancy. If analyses will still be performed, all affected results will be flagged to indicate improper preservation.

- **FOR WATER SAMPLES TESTED FOR AVAILABLE / FREE CYANIDE (Method OIA-1677)**
  - In the Field: Samples will be collected in pre-preserved bottles for both a regular sample collection and a 1:10 diluted sample in case of the presence of sulfide.
    - The lab will test the samples for sulfide using lead acetate paper at the time of analysis and if sulfide is present in the sample above 50mg/L, the diluted sample will be analyzed.
    - If the water being collected may contain residual chlorine or other oxidizer, the sample should be treated at time of collection with sodium arsenite.

- Water samples that require ortho-phosphorus must be filtered in the field within 15 minutes of sampling. Samples received without indication of filtration in the field will have results flagged for improper preservation.

- Water samples being collected for dissolved metals analysis should be filtered in the field within 15 minutes of sampling and prior to preservation.

- Samples for coliform analysis must be in sterile containers and must be free of residual chlorine. The bottle must be filled to <u>above</u> the 100mL mark.

6) For the laboratory to meet method requirements for the analysis of *Duplicate, Matrix Spikes* and/ or *Matrix Spike Duplicates*, extra volume for at least 1 samples should be collected.
   - If *Matrix Spikes* are required on a specific sample for your project, separate sample volumes for the required QC must be collected for the requested analyses.

7) For Volatile Organic analyses: Efforts should be made to minimize any air bubbles in aqueous volatile samples. Air bubbles also the escape of volatile organics. This is especially important because air bubbles tend to form in iced samples. Volatile vials containing air bubbles larger than a pea will be treated as non-conformances.

8) All samples submitted for Volatile Organic analyses, including by method 8011, must have a Trip Blank submitted at the same time. Eurofins TestAmerica will supply a blank with the bottle order.

9) Sample Holding Times

   - Eurofins TestAmerica will make every effort to analyze samples within the regulatory holding time. Samples must be received in the laboratory with enough time to perform the sample

analysis. Except for short holding time samples (< 48hr HT) sample must be received with at least 48 hrs (working days) remaining on the holding time for us to ensure analysis.

- Analyses that are designated as "field" analyses (Odor, pH, Dissolved Oxygen, Disinfectant Residual; a.k.a. Residual Chlorine, and Redox Potential) should be analyzed ASAP by the field sampler prior to delivering to the lab (within 15 minutes). However, if the analyses are to be performed in the laboratory, Eurofins TestAmerica will make every effort to analyze the samples within 24 hours from receipt of the samples in the testing laboratory. Samples for "field" analyses received after 4:00 pm on Friday or on the weekend will be analyzed no later than the next business day after receipt (Monday unless a holiday). Samples will remain refrigerated and sealed until the time of analysis. Samples analyzed in the laboratory will be qualified on the final report to indicate holding time exceedance.

10) The project manager will be notified if any sample is received in damaged condition. Eurofins TestAmerica will request that a sample be resubmitted for analysis. The laboratory will notify the client upon sample receipt if the samples exhibit obvious signs of damage, contamination or inadequate preservation.

11) Recommendations for packing samples for shipment.

- Pack samples in Ice rather than "Blue" ice packs.

- Soil samples should be placed in plastic zip-lock bags. The containers often have dirt around the top and do not seal very well and are prone to intrusion from the water from melted ice.

- Water samples would be best if wrapped with bubble-wrap or paper (newspaper, or paper towels work) and then placed in plastic zip-lock bags.

- Fill extra cooler space with bubble wrap.

12) If the conditions listed on the Acceptance Policy are not satisfactory and when lacking direction or agreement with the client, the sample will be rejected by the laboratory.

Figure 23-3

Example: Sample Receipt Checklist

Login Sample Receipt Checklist

Client: Cardno ENTRIX

Job Number: 180-264-1

Login Number: 264
List Number: 1
Creator: Gamber, Tom

List Source: TestAmerica Pittsburgh

Question	Answer	Comment
Radioactivity either was not measured or, if measured, is at or below background	True	
The cooler's custody seal, if present, is intact.	True	
The cooler or samples do not appear to have been compromised or tampered with.	True	
Samples were received on ice.	True	
Cooler Temperature is acceptable.	True	
Cooler Temperature is recorded.	True	
COC is present.	True	
COC is filled out in ink and legible.	True	
COC is filled out with all pertinent information.	True	
Is the Field Sampler's name present on COC?	True	
There are no discrepancies between the sample IDs on the containers and the COC.	True	
Samples are received within Holding Time.	True	
Sample containers have legible labels.	True	
Containers are not broken or leaking.	True	
Sample collection date/times are provided.	True	
Appropriate sample containers are used.	True	
Sample bottles are completely filled.	True	
Sample Preservation Verified.	True	
There is sufficient vol. for all requested analyses, incl. any requested MS/MSDs	True	
VOA sample vials do not have headspace or bubble is <6mm (1/4") in diameter.	True	
Multiphasic samples are not present.	True	
Samples do not require splitting or compositing.	True	
Residual Chlorine Checked.	N/A	

SECTION 24

ASSURING THE QUALITY OF TEST RESULTS

24.1 Overview

In order to assure our clients of the validity of their data, the laboratory continuously evaluates the quality of the analytical process. The analytical process is controlled not only by instrument calibration as discussed in Section 20, but also by routine process quality control measurements (e.g. Blanks, Laboratory Control Samples (LCS), Matrix Spikes (MS), duplicates (DUP), surrogates, Internal Standards (IS)). These quality control checks are performed as required by the method or regulations to assess precision and accuracy. Quality control samples are to be treated in the exact same manner as the associated field samples being tested. In addition to the routine process quality control samples, Proficiency Testing (PT) Samples (concentrations unknown to laboratory) are analyzed to help ensure laboratory performance.

24.2 Controls

Sample preparation or pre-treatment is commonly required before analysis. Typical preparation steps include homogenization, solvent extraction, sonication, acid digestion, filtration, distillation, reflux, evaporation, drying and ashing. During these pre-treatment steps, samples are arranged into discreet manageable groups referred to as preparation (prep) batches. Prep batches provide a means to control variability in sample treatment. Control samples are added to each prep batch to monitor method performance and are processed through the entire analytical procedure with investigative/field samples.

24.3 Negative Controls

Table 24-1. Negative Controls

Control Type	Details
Method Blank (MB)	are used to assess preparation and analysis for possible contamination during the preparation and processing steps.
	The specific frequency of use for method blanks during the analytical sequence is defined in the specific standard operating procedure for each analysis. Generally it is 1 for each batch of samples; not to exceed 20 environmental samples.
	The method blank is prepared from a clean matrix similar to that of the associated samples that is free from target analytes (e.g., Reagent water, Ottawa sand, glass beads, etc.) and is processed along with and under the same conditions as the associated samples. The method blank goes through all of the steps of the process (including as necessary: filtration, clean-ups, etc.).
	Reanalyze or qualify associated sample results when the concentration of a targeted analyte in the blank is at or above the reporting limit as established by the method or by regulation, AND is greater than 1/10 of the amount measured in the sample.
Calibration Blanks	are prepared and analyzed along with calibration standards where applicable. They are prepared using the same reagents that are used to prepare the standards. In some analyses the calibration blank may be included in the calibration curve.

Table 24-1. Negative Controls

Control Type	Details
Instrument Blanks	are blank reagents or reagent water that may be processed during an analytical sequence in order to assess contamination in the analytical system. In general, instrument blanks are used to differentiate between contamination caused by the analytical system and that caused by the sample handling or sample prep process. Instrument blanks may also be inserted throughout the analytical sequence to minimize the effect of carryover from samples with high analyte content.
Trip Blank [1]	are required to be submitted by the client with each shipment of samples requiring aqueous and solid volatiles analyses (or as specified in the client's project plan). Additionally, trip blanks may be prepared and analyzed for volatile analysis of air samples, when required by the client. A trip blank may be purchased (certified clean) or is prepared by the laboratory by filling a clean container with pure deionized water that has been purged to remove any volatile compounds. Appropriate preservatives are also added to the container. The trip blank is sent with the bottle order and is intended to reflect the environment that the containers are subjected to throughout shipping and handling and help identify possible sources if contamination is found. The field sampler returns the trip blank in the cooler with the field samples.
Field Blanks [1]	are sometimes used for specific projects by the field samplers. A field blank prepared in the field by filling a clean container with pure reagent water and appropriate preservative, if any, for the specific sampling activity being undertaken. (EPA OSWER)
Equipment Blanks [1]	are also sometimes created in the field for specific projects. An equipment blank is a sample of analyte-free media which has been used to rinse common sampling equipment to check effectiveness of decontamination procedures. (TNI)
Holding Blanks	also referred to as refrigerator or freezer blanks, are used to monitor the sample storage units for volatile organic compounds during the storage of VOA samples in the laboratory

[1] When known, these field QC samples should not be selected for matrix QC as it does not provide information on the behavior of the target compounds in the field samples. Usually, the client sample ID will provide information to identify the field blanks with labels such as "FB", "EB", or "TB."

Evaluation criteria and corrective action for these controls are defined in the specific standard operating procedure for each analysis. Also further detail is provided in SOP No. PT-QA-021.

24.3.1 Negative Controls for Microbiological Methods – Microbiological Methods utilize a variety of negative controls throughout the process to ensure that false positive results are not obtained. These controls are critical to the validity of the microbiological analyses. Some of these negative controls are:

Table 24-2. Negative Controls for Microbiology

Control Type	Details
Sterility Checks (Media)	are analyzed for each lot of pre-prepared media, ready-to-use media and for each batch of medium prepared by the laboratory.
Filtration Blanks	blanks are run at the beginning and end for each sterilized filtration unit used in a filtration series. For pre-sterilized single use funnels a sterility check is performed on at least one funnel per lot.
Sterility checks (Sample Containers)	are performed on at least one container per lot of purchased, pre-sterilized containers. If containers are prepared and sterilized by the laboratory, one container per sterilization batch is checked. Container sterility checks are performed using non-selective growth media.

Sterility Checks (Dilution Water)	are performed on each batch of dilution water prepared by the laboratory and on each batch of pre-prepared dilution water. All checks are performed using non-selective growth media.
Sterility Checks (Filters)	are also performed on at least one filter from each new lot of membrane filters using non-selective growth media.

Negative culture controls demonstrate that a media does not support the growth of non-target organisms and ensures that there is not an atypical positive reaction from the target organisms. Prior to the first use of the media, each lot of pre-prepared selective media or batch of laboratory prepared selective media is analyzed with at least one known negative culture control as appropriate to the method.

24.4 Positive Controls

Control samples (e.g., QC indicators) are analyzed with each batch of samples to evaluate data based upon (1) Method Performance (Laboratory Control Sample (LCS) or Blank Spike (BS)), which entails both the preparation and measurement steps; and (2) Matrix Effects (Matrix Spike (MS) or Sample Duplicate (MD, DUP), which evaluates field sampling accuracy, precision, representativeness, interferences, and the effect of the matrix on the method performed. Each regulatory program and each method within those programs specify the control samples that are prepared and/or analyzed with a specific batch

Note that frequency of control samples vary with specific regulatory, methodology and project specific criteria. Complete details on method control samples are as listed in each analytical SOP.

24.4.1 Method Performance Control - Laboratory Control Sample (LCS)

The LCS measures the accuracy of the method in a blank matrix and assesses method performance independent of potential field sample matrix affects in a laboratory batch.

The LCS is prepared from a clean matrix similar to that of the associated samples that is free from target analytes (for example: Reagent water, Ottawa sand, glass beads, etc.) and is processed along with and under the same conditions as the associated samples. The LCS is spiked with verified known amounts of analytes or is made of a material containing known and verified amounts of analytes, taken through all preparation and analysis steps along with the field samples. Where there is no preparation taken for an analysis (such as in aqueous volatiles), or when all samples and standards undergo the same preparation and analysis process (such as Phosphorus), a calibration verification standard may be reported as the LCS. In some instances where there is no practical clean solid matrix available, aqueous LCS's may be processed for solid matrices; final results may be calculated as mg/kg or ug/kg, assuming 100% solids and a weight equivalent to the aliquot used for the corresponding field samples, to facilitate comparison with the field samples.

Certified pre-made reference material purchased from a NIST/A2LA accredited vendor may also be used for the LCS when the material represents the sample matrix or the analyte is not easily spiked (e.g. solid matrix LCS for metals, TDS, etc.).

The specific frequency of use for LCS during the analytical sequence is defined in the specific standard operating procedure for each analysis. It is generally 1 for each batch of samples; not to exceed 20 environmental samples.

If the mandated or requested test method, or project requirements, do not specify the spiking components, the laboratory shall spike all reportable components to be reported in the Laboratory Control Sample (and Matrix Spike) where applicable (e.g. no spike of pH). However, in cases where the components interfere with accurate assessment (such as simultaneously spiking chlordane, toxaphene and PCBs in Method 608), the test method has an extremely long list of components or components are incompatible, at a minimum, a representative number of the listed components (see below) shall be used to control the test method. The selected components of each spiking mix shall represent all chemistries, elution patterns and masses, permit specified analytes and other client requested components. However, the laboratory shall ensure that all reported components are used in the spike mixture within a two-year time period.

- For methods that have 1-10 target analytes, spike all components.
- For methods that include 11-20 target analytes, spike at least 10 or 80%, whichever is greater.
- For methods with more than 20 target analytes, spike at least 16 components.
- Exception: Due to analyte incompatibility in pesticides, Toxaphene and Chlordane are only spiked at client request based on specific project needs.
- Exception: Due to analyte incompatibility between the various PCB aroclors, aroclors 1016 and 1260 are used for spiking as they cover the range of all of the aroclors. Specific aroclors may be used by request on a project specific basis.

24.4.2 Positive Controls for Microbiological Methods

- Each lot of pre-prepared media (including chromofluorogenic reagent) and each batch of laboratory prepared media is tested with a pure culture of known positive reaction.

- In addition, every analytical batch also contains a pure culture of known positive reaction.

  A pure culture of known negative reaction is also tested with each analytical batch to ensure specificity of the procedure.

24.5 Sample Matrix Controls

Table 24-3 Sample Matrix Control

Control Type	Details	
Matrix Spikes (MS)	Use	used to assess the effect sample matrix of the spiked sample has on the precision and accuracy of the results generated by the method used;
	Typical Frequency [1]	At a minimum, with each matrix-specific batch of samples processed, an MS is carried through the complete analytical procedure. Unless specified by the client, samples used for spiking are randomly selected and rotated between different client projects. If the mandated or requested test method does not specify the spiking components, the laboratory shall spike all reportable components to be reported in the Laboratory Control Sample and Matrix Spike. Refer to the method SOP for complete details
	Description	essentially a sample fortified with a known amount of the test analyte(s).
Surrogate	Use	Measures method performance to sample matrix (organics only).
	Typical Frequency [1]	Are added to all samples, standards, and blanks, for all organic chromatography methods except when the matrix precludes its use or when a surrogate is not available. The recovery of the surrogates is compared to the acceptance limits for the specific method. Poor surrogate recovery may indicate a problem with sample composition and shall be reported, with data qualifiers, to the client whose sample produced poor recovery.
	Description	Are similar to matrix spikes except the analytes are compounds with properties that mimic the analyte of interest and are unlikely to be found in environment samples.
Duplicates[2]	Use	For a measure of analytical precision, with each matrix-specific batch of samples processed, a matrix duplicate (MD or DUP) sample, matrix spike duplicate (MSD), or LCS duplicate (LCSD) is carried through the complete analytical procedure.
	Typical Frequency [1]	Duplicate samples are usually analyzed with methods that do not require matrix spike analysis.
	Description	Performed by analyzing two aliquots of the same field sample independently or an additional LCS.
Internal Standards	Use	Are spiked into all environmental and quality control samples (including the initial calibration standards) to monitor the qualitative aspect of organic and some inorganic analytical measurements.
	Typical Frequency [1]	All organic and ICP methods as required by the analytical method.
	Description	Used to correct for matrix effects and to help troubleshoot variability in analytical response and are assessed after data acquisition. Possible sources of poor internal standard response are sample matrix, poor analytical technique or instrument performance.

[1] See the specific analytical SOP for type and frequency of sample matrix control samples.

[2] LCSD's are normally not performed except when regulatory agencies or client specifications require them. The recoveries for the spiked duplicate samples must meet the same laboratory established recovery limits as the accuracy QC samples. If an LCSD is analyzed both the LCS and LCSD must meet the same recovery criteria and be included in the final report. The precision measurement is reported as "Relative Percent Difference" (RPD). Poor precision between duplicates (except LCS/LCSD) may indicate non-homogeneous matrix or sampling.

24.6 Acceptance Criteria (Control Limits)

As mandated by the test method and regulation, each individual analyte in the LCS, MS, or Surrogate Spike is evaluated against the control limits published in the test method. Where there are no established acceptance criteria, the laboratory calculates in-house control limits

with the use of control charts or, in some cases, utilizes client project specific control limits. When this occurs, the regulatory or project limits will supersede the laboratory's in-house limits.

Note: For methods, analytes and matrices with very limited data (e.g., unusual matrices not analyzed often), interim limits are established using available data or by analogy to similar methods or matrices.

Once control limits have been established, they are verified, reviewed, and updated if necessary on an annual basis unless the method requires more frequent updating. Control limits are established per method (as opposed to per instrument) regardless of the number of instruments utilized.

Laboratory generated % Recovery acceptance (control) limits are generally established by taking $\pm$ 3 Standard Deviations (99% confidence level) from the average recovery of a minimum of 20-30 data points (more points are preferred).

- Regardless of the calculated limit, the limit should be no tighter than the Calibration Verification (ICV/CCV) (Unless the analytical method specifies a tighter limit).

- In-house limits cannot be any wider than those mandated in a regulated analytical method. Client or contract required control limits are evaluated against the laboratory's statistically derived control limits to determine if the data quality objectives (DQOs) can be achieved. If laboratory control limits are not consistent with DQOs, then alternatives must be considered, such as method improvements or use of an alternate analytical method.

- The lowest acceptable recovery limit will be 10% (the analyte must be detectable and identifiable). Exception: The lowest acceptable recovery limit for Benzidine will be 5% and the analyte must be detectable and identifiable.

- The maximum acceptable recovery limit will be 150%.

- The maximum acceptable RPD limit will be 35% for waters and 40% for soils. The minimum RPD limit is 15%.

- If either the high or low end of the control limit changes by $\leq$ 5% from previous, the control chart is visually inspected and, using professional judgment, they may be left unchanged if there is no effect on laboratory ability to meet the existing limits.

24.6.1 The lab must be able to generate a current listing of their control limits and track when the updates are performed. Current and historical limits are maintained in the LIMS, along with the effective dates. In addition, the laboratory must be able to recreate historical control limits. Refer to laboratory SOP PT-QA-021, *Quality Control Program*, for details on the creation, evaluation and application of statistical control limits.

24.6.2 A LCS that is within the acceptance criteria establishes that the analytical system is in control and is used to validate the process. Samples that are analyzed with an LCS with recoveries outside of the acceptance limits may be determined as out of control and should be reanalyzed if possible. If reanalysis is not possible, then the results for all affected analytes for samples within the same batch must be qualified when reported. The internal corrective action

process (see Section 12) is also initiated if an LCS exceeds the acceptance limits. Sample results may be qualified and reported without reanalysis if:

- The analyte results are below the reporting limit and the LCS is above the upper control limit.

- If the analytical results are above the relevant regulatory limit and the LCS is below the lower control limit. For further detail refer to laboratory SOP PT-QA-021 and method specific SOPs.

- For TNI work, there are an allowable number of Marginal Exceedances (ME):

<11 analytes	0 marginal exceedances are allowed
11 – 30 Analytes	1 marginal exceedance is allowed
31-50 Analytes	2 marginal exceedances are allowed
51-70 Analytes	3 marginal exceedances are allowed
71-90 Analytes	4 marginal exceedances are allowed
> 90 Analytes	5 marginal exceedances are allowed

  - Marginal exceedances are recovery exceedances between 3 SD and 4 SD from the mean recovery limit (TNI).

  - Marginal exceedances must be random. If the same analyte exceeds the LCS control limit repeatedly, it is an indication of a systematic problem. The source of the error must be located and corrective action taken. The laboratory has a system to monitor marginal exceedances to ensure that they are random.

  - Though marginal exceedances may be allowed, the data must still be qualified to indicate it is outside of the normal limits.

24.6.3 If the MS/MSDs do not meet acceptance limits, the MS/MSD and the associated parent sample are reported with a qualifier for those analytes that do not meet limits. If obvious preparation errors are suspected, or if requested by the client, unacceptable MS/MSDs are reprocessed and reanalyzed to prove matrix interference. A more detailed discussion of acceptance criteria and corrective action can be found in SOP No. PT-QA-021 – Laboratory Quality Control Program, analytical method SOPs and in Section 12 of this document.

24.6.4 If a surrogate standard falls outside the acceptance limits, if there is not obvious chromatographic matrix interference, reanalyze the sample to confirm a possible matrix effect. If the recoveries confirm or there was obvious chromatographic interference, results are reported from the original analysis and a qualifier is added. If the reanalysis meets surrogate recovery criteria, the second run is reported (or both are reported if requested by the client). Under certain circumstances, where all of the samples are from the same location and share similar chromatography, the reanalysis may be performed on a single sample rather than all of the samples and if the surrogate meets the recovery criteria in the reanalysis, all of the affected samples would require reanalysis.

24.7 Additional Procedures to Assure Quality Control

The laboratory has written and approved method SOPs to assure the accuracy of the test method including calibration (see Section 20), use of certified reference materials (see Section 21) and use of PT samples (see Section 15).

A discussion regarding MDLs, Limit of Detection (LOD) and Limit of Quantitation (LOQ) can be found in Section 19.

Use of formulae to reduce data is discussed in the method SOPs and in Section 20.

Selection of appropriate reagents and standards is included in Section 9 and 21.

A discussion on selectivity of the test is included in Section 5.

Constant and consistent test conditions are discussed in Section 18.

The laboratories sample acceptance policy is included in Section 23.

SECTION 25

REPORTING RESULTS

25.1 Overview

The results of each test are reported accurately, clearly, unambiguously, and objectively in accordance with State and Federal regulations as well as client requirements. Analytical results are issued in a format that is intended to satisfy customer and laboratory accreditation requirements as well as provide the end user with the information needed to properly evaluate the results. Where there is conflict between client requests and laboratory ethics or regulatory requirements, the laboratory's ethical and legal requirements are paramount, and the laboratory will work with the client during project set up to develop an acceptable solution. Refer to Section 7.

A variety of report formats are available to meet specific needs.

In cases where a client asks for simplified reports, there must be a written request from the client. There still must be enough information that would show any analyses that were out of conformance (QC out of limits) and there should be a reference to a full report that is made available to the client. Review of reported data is included in Section 19.

25.2 Test Reports

Analytical results are reported in a format that is satisfactory to the client and meets all requirements of applicable accrediting authorities and agencies. A variety of report formats are available to meet specific needs. The report is printed on laboratory letterhead, reviewed, and signed by the appropriate project manager. At a minimum, the standard laboratory report shall contain the following information:

25.2.1 A report title (e.g. Analytical Report) on the cover page with a "Result" column header on the sample result page

25.2.2 The cover page shall include the laboratory name, address and telephone number

25.2.3 A unique identification of the report (e.g. Eurofins TestAmerica Job ID#) and on each page an identification in order to ensure the page is recognized as part of the report and a clear identification of the end

Note: Page numbers of report are represented as page # of ## at the bottom of the page. Where the first number is the page number and the second is the total number of pages

25.2.4 A copy of the chain of custody (COC)

- Any COCs involved with Subcontracting are included

25.2.5 The name and address of client and a project name/number, if applicable

25.2.6 Client project manager or other contact

25.2.7 Description and unambiguous identification of the tested sample(s) including the client identification code

25.2.8 Date of receipt of sample, date and time of collection, and date(s) and time of test preparation and performance, and time of preparation or analysis if the required holding time for either activity is less than or equal to 72 hours

25.2.9 Date reported or date of revision, if applicable

25.2.10 Method of analysis including method code (EPA, Standard Methods, etc)

25.2.11 Reporting Limit

25.2.12 Method detection limits (if requested)

25.2.13 Definition of Data qualifiers and reporting acronyms (e.g. ND)

25.2.14 Sample results

25.2.15 QC data consisting of method blank, surrogate, LCS, and MS/MSD recoveries and control limits are included unless the client specifies they do not require reporting the QC

25.2.16 Condition of samples at receipt including temperature. This may be accomplished in the case narrative or by attaching sample login sheets. The temperature is documented on the sample receipt checklist and noted in the report case narrative.

25.2.17 A statement expressing the validity of the results, that the source methodology was followed and all results were reviewed for error

25.2.18 A statement to the effect that the results relate only to the items tested and the sample as received by the laboratory, except when information is provided by the client. When data is provided by the client there shall be a clear indication of it, and a disclaimer shall be put in the report when the client supplied data can affect the validity of the test.

25.2.19 A statement that the report shall not be reproduced except in full, without prior express written approval by the laboratory coordinator

25.2.20 A signature and title of the person(s) accepting responsibility for the content of the report and date of issue (Signatories are appointed by the Lab Director)

25.2.21 When TNI accreditation is required, the lab shall certify that the test results meet all requirements of TNI or provide reasons and/or justification if they do not

25.2.22 If applicable, the laboratory includes a cover letter

25.2.23 Where applicable, a narrative to the report that explains the issue(s) and corrective action(s) taken in the event that a specific accreditation or certification requirement was not met

25.2.24 When soil samples are analyzed, a specific identification as to whether soils are reported on a “wet weight” or “dry weight” basis

25.2.25 Appropriate laboratory certification number for the state of origin of the sample, if applicable

25.2.26 If only part of the report is provided to the client (client requests some results before all of it is complete), it must be clearly indicated on the report (e.g., preliminary report). A complete report must be sent once all of the work has been completed.

25.2.27 Any non-Eurofins TestAmerica subcontracted analysis results are provided as a separate report on the official letterhead of the subcontractor. All Eurofins TestAmerica subcontracting is clearly identified on the report as to which laboratory performed a specific analysis.

25.2.28 A clear statement notifying the client that non-accredited tests were performed and directing the client to the laboratory’s accreditation certificates of approval shall be provided when non-accredited tests are included in the report.

25.2.29 Where the laboratory is responsible for the sampling stage, in addition to the requirements listed above, reports containing the results of sampling shall include the following, where necessary for the interpretation of test results:

- The date of sampling
- Unambiguous identification of the material sampled
- The location of sampling
- A reference to the sampling plan and procedures, and deviations, additions to or exclusions from the sampling procedures
- Details of any environmental conditions during sampling that affect the interpretation of test results
- Information required to evaluate measurement uncertainty for subsequent testing

Note: It is required by the PA DEP that non-accredited parameters be clearly identified on the sample results.

Note: Refer to the Corporate SOP on Electronic Reporting and Signature Policy (No. CA-I-P-002) for details on internally applying electronic signatures of approval.

25.3 Reporting Level Or Report Type

The laboratory offers four levels of quality control reporting. Each level, in addition to its own specific requirements, contains all the information provided in the preceding level. The packages provide the following information in addition to the information described above:

- Level I is a report with the features described in Section 25.2 above.
- Level II is a Level I report plus summary information, including results for the method blank reported to the laboratory MDL, percent recovery for laboratory control samples and matrix spike samples, and the RPD values for all MSD and sample duplicate analyses.
- Level III contains all the information supplied in Level II, but presented on the CLP-like summary forms, and relevant calibration information. A Level II report is not included, unless specifically requested. No raw data is provided.
- Level IV is the same as Level III with the addition of all raw supporting data.

In addition to the various levels of QC packaging, the laboratory also provides reports in diskette deliverable form. Initial reports may be provided to clients by facsimile. Procedures used to ensure client confidentiality are outlined in Section 25.6.

25.3.1 Electronic Data Deliverables (EDDs)

EDDs are routinely offered as part of Eurofins TestAmerica's services in addition to the test report as described in section 25.2. When NELAP accreditation is required and both a test report and EDD are provided to the client, the official version of the test report will be the combined information of the report and the EDD. Data qualifiers appearing on the test report must be included in the EDD.

Pittsburgh offers a variety of EDD formats including Environmental Restoration Information Management System (ERPIMS), Staged Electronic Data Deliverable (SEDD) Environmental Quality Information System (EQuIS), Electronic Deliverable Format (EDF), Excel and custom files as requested by the client.

EDD specifications are submitted to the IT department by the PM for review and undergo the contract review process. Once the facility has committed to providing data in a specific

electronic format, the coding of the format may need to be performed. This coding is documented and validated. The validation of the code is retained by the IT staff coding the EDD.

EDDs shall be subject to a review to ensure their accuracy and completeness. If EDD generation is automated, review may be reduced to periodic screening if the laboratory can demonstrate that it can routinely generate that EDD without errors. Any revisions to the EDD format must be reviewed until it is demonstrated that it can routinely be generated without errors. If the EDD can be reproduced accurately and if all subsequent EDDs can be produced error-free, each EDD does not necessarily require a review.

25.4 Supplemental Information For Test

The lab identifies any unacceptable QC analyses or any other unusual circumstances or observations such as environmental conditions and any non-standard conditions that may have affected the quality of a result. This is typically in the form of a footnote or a qualifier and/or a narrative explaining the discrepancy in the front of the report.

Numeric results with values outside of the calibration range, either high or low are qualified as estimated.

Where quality system requirements are not met, a statement of compliance/non-compliance with requirements and/or specifications is required, including identification of test results derived from any sample that did not meet TNI sample acceptance requirements such as improper container, holding time, or temperature.

Where applicable, a statement on the estimated uncertainty of measurements; information on uncertainty is needed when a client's instructions so require.

When, as required by the client and agreed to by Eurofins TestAmerica, the report includes a statement of conformity to specification or standard (see Special Services, Section 7.4), the report shall clearly identify:

- To which results the statement applies
- Which specifications, standard or parts thereof are met or not
- The decision rule that was applied unless the decision rule is inherent in the requested specification or standard, taking into account the level of risk (such as false accept and false reject and statistical assumptions) associated with the decision rule.

Opinions and Interpretations - The test report contains objective information, and generally does not contain subjective information such as opinions and interpretations. If such information is required by the client, the Laboratory Director will determine if a response can be prepared. If so, the Laboratory Director will designate the appropriate member of the management team to prepare a response. The response will be fully documented, and reviewed by the Laboratory Director, before release to the client. There may be additional fees charged to the client at this time, as this is a non-routine function of the laboratory.

Note: Review of data deliverable packages for submittal to regulatory authorities requires responses to non-conforming data concerning potential impact on data quality. This necessitates a limited scope of interpretation, and this work is performed by the

Manager(s)/Team Leaders or as assigned by the lab Director. This is the only form of "interpretation" of data that is routinely performed by the laboratory.

When opinions or interpretations are included in the report, the laboratory provides an explanation as to the basis upon which the opinions and interpretations have been made. Opinions and interpretations are clearly noted as such and where applicable, a comment should be added suggesting that the client verify the opinion or interpretation with their regulator.

25.5 Environmental Testing Obtained From Subcontractors

If the laboratory is not able to provide the client the requested analysis, the samples would be subcontracted following the procedures outlined in the Corporate SOP on Subcontracting (SOP # CW-L-S-004).

Data reported from analyses performed by a subcontractor laboratory are clearly identified as such on the analytical report provided to the client. Results from a subcontract laboratory outside of Eurofins TestAmerica are reported to the client on the subcontract laboratory's original report stationary and the report includes any accompanying documentation.

25.6 Client Confidentiality

Eurofins TestAmerica is responsible for maintaining in confidence all client information obtained or created. In situations involving the transmission of environmental test results by telephone, facsimile or other electronic means, client confidentiality must be maintained.

Eurofins TestAmerica will not intentionally divulge to any person (other than the Client or any other person designated by the Client in writing) any information regarding the services provided by Eurofins TestAmerica or any information disclosed to Eurofins TestAmerica by the Client. Furthermore, information known to be potentially endangering to national security or an entity's proprietary rights will not be released.

Information about the client obtained from sources other than the client (r.g, complaint, regulators) shall be confidential between client and laboratory. The source of this information shall be confidential to the laboratory and shall not be shared with the client, unless agreed to by the source.

Note: This shall not apply to the extent that the information is required to be disclosed by Eurofins TestAmerica under the compulsion of legal process. Eurofins TestAmerica will, to the extent feasible, provide reasonable notice to the client before disclosing the information.

Note: Authorized representatives of an accrediting authority are permitted to make copies of any analyses or records relevant to the accreditation process, and copies may be removed from the laboratory for purposes of assessment.

25.6.1 Report deliverable formats are discussed with each new client. If a client requests that reports be faxed or e-mailed, the reports are to meet all requirements of this document, including a cover letter.

25.7 Format Of Reports

The format of reports is designed to accommodate each type of environmental test carried out and to minimize the possibility of misunderstanding or misuse.

25.8 Amendments To Test Reports

Corrections, additions, or deletions to reports are only made when justification arises through supplemental documentation. Justification is documented using the laboratory's corrective action system (refer to Section 12).

The revised report is retained on the data server, as is the original report. The revised report is stored in the data server under the job number followed by "Rev (n)" where 'n' is the revision number. The revised report will have the words "Revision (n)" on the report cover page beneath the report date.

When the report is re-issued, a notation of "report re-issue" is placed on the cover/signature page of the report or at the top of the narrative page with a brief explanation of reason for the re-issue and a reference back to the last final report generated.

25.9 Policies On Client Requests For Amendments

25.9.1 Policy on Data Omissions or Reporting Limit Increases

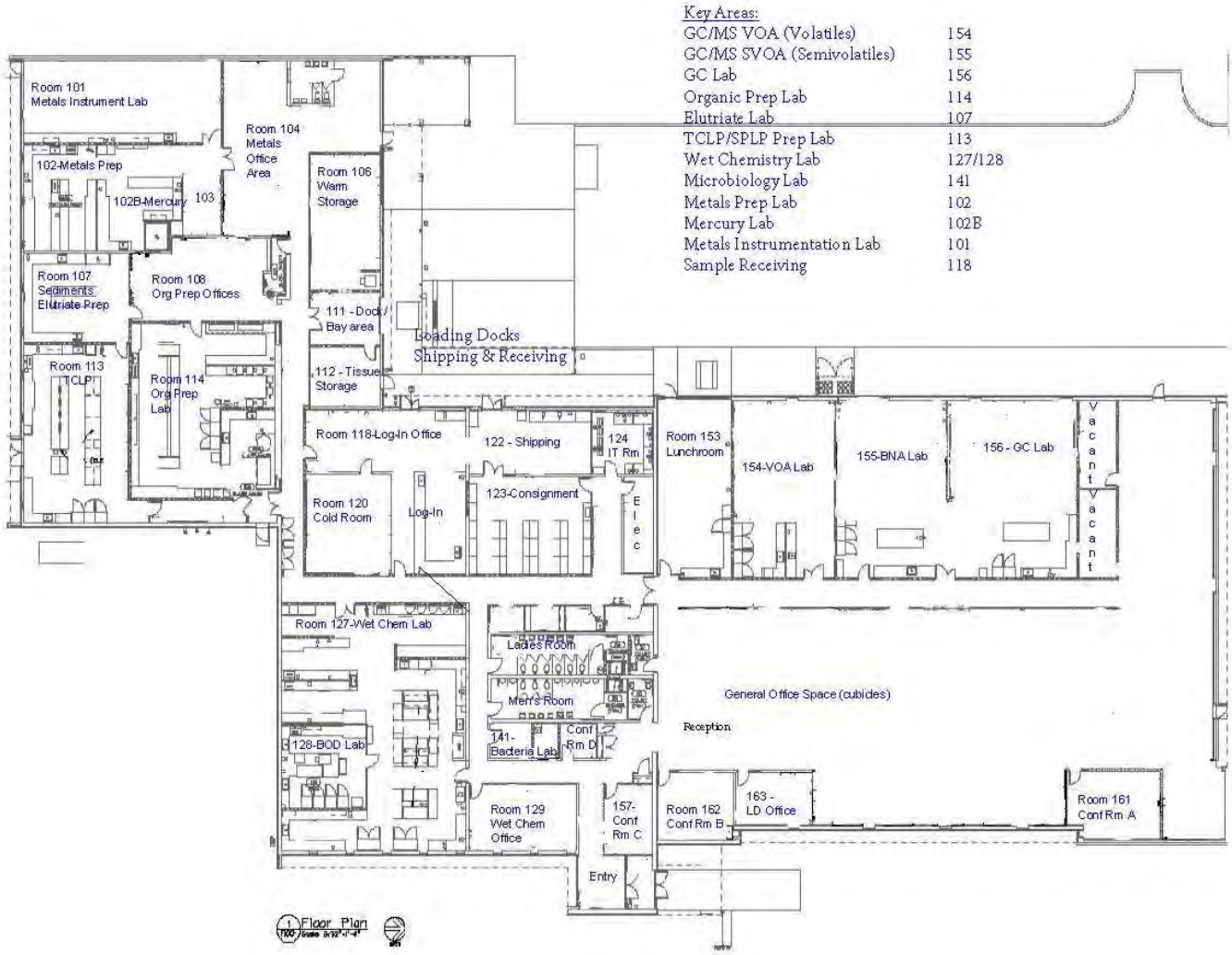
Fundamentally, our policy is simply to not omit previously reported results (including data qualifiers) or to not raise reporting limits and report sample results as ND. This policy has few exceptions. Exceptions are:

- Laboratory error
- Sample identification is indeterminate (confusion between COC and sample labels)
- An incorrect analysis (not analyte) was requested (e.g., COC lists 8315 but client wanted 8310). A written request for the change is required.
- Incorrect limits reported based on regulatory requirements
- The requested change has absolutely *no possible* impact on the interpretation of the analytical results and there is *no possibility* of the change being interpreted as misrepresentation by anyone inside or outside of our company.

25.9.2 Multiple Reports

Eurofins TestAmerica does not issue multiple reports for the same job number where there is different information on each report (this does not refer to copies of the same report) unless required to meet regulatory needs and approved by QA.

Appendix 1
Laboratory Floor Plan



Appendix 2
Glossary/Acronyms

Glossary:

Acceptance Criteria: Specified limits placed on characteristics of an item, process, or service defined in requirement documents. (ASQC)

Accreditation: The process by which an agency or organization evaluates and recognizes a laboratory as meeting certain predetermined qualifications or standards, thereby accrediting the laboratory.

Accuracy: The degree of agreement between an observed value and an accepted reference value. Accuracy includes a combination of random error (precision) and systematic error (bias) components which are due to sampling and analytical operations; a data quality indicator. (QAMS)

Analyst: The designated individual who performs the "hands-on" analytical methods and associated techniques and who is the one responsible for applying required laboratory practices and other pertinent quality controls to meet the required level of quality. (TNI)

Analytical Uncertainty: A subset of Measurement Uncertainty that includes all laboratory activities performed as part of the analysis. (TNI)

Anomaly: A condition or event, other than a deficiency, that may affect the quality of the data, whether in the laboratory's control or not.

Assessment: The evaluation process used to measure or establish the performance, effectiveness, and conformance of an organization and/or its systems to defined criteria (to the standards and requirements of laboratory accreditation). (TNI)

Audit: A systematic and independent examination of facilities, equipment, personnel, training, procedures, record-keeping, data validation, data management, and reporting aspects of a system to determine whether QA/QC and technical activities are being conducted as planned and whether these activities will effectively achieve quality objectives. (TNI)

Batch: Environmental samples which are prepared and/or analyzed together with the same process and personnel, using the same lot(s) of reagents. A **preparation batch** is composed of one to 20 environmental samples of the same matrix, meeting the above mentioned criteria and with a maximum time between the start of processing of the first and last sample in the batch to be 24 hours. An **analytical batch** is composed of prepared environmental samples (extracts, digestates or concentrates) which are analyzed together as a group. An analytical batch can include prepared samples originating from various quality system matrices and can exceed twenty (20) samples. (TNI)

Bias: The systematic or persistent distortion of a measurement process, which causes errors in one direction (i.e., the expected sample measurement is different from the sample's true value). (TNI)

Blank: A sample that has not been exposed to the analyzed sample stream in order to monitor contamination during sampling, transport, storage or analysis. The blank is subjected to the usual analytical and measurement process to establish a zero baseline or background value and is sometimes used to adjust or correct routine analytical results. (ASQC)

Calibration: A set of operations that establish, under specified conditions, the relationship between values of quantities indicated by a measuring instrument or measuring system, or values represented by a material measure or a reference material, and the corresponding values realized by standards. (TNI)

1) In calibration of support equipment the values realized by standards are established through the use of reference standards that are traceable to the International System of Units (SI).

2) In calibration according to methods, the values realized by standards are typically established through the use of Reference Materials that are either purchased by the laboratory with a certificate of analysis or purity, or prepared by the laboratory using support equipment that has been calibrated or verified to meet specifications.

Calibration Curve: The mathematical relationship between the known values, such as concentrations, of a series of calibration standards and their instrument response. (TNI)

Calibration Standard: A substance or reference material used to calibrate an instrument (QAMS)

Certified Reference Material (CRM): A reference material, accompanied by a certificate, having a value, measurement uncertainty, and stated metrological traceability chain to a national metrology institute. (TNI)

Chain of Custody (COC) Form**:** Record that documents the possession of the samples from the time of collection to receipt in the laboratory. This record generally includes: the number and types of containers; the mode of collection; the collector; time of collection; preservation; and requested analyses. (TNI)

Compromised Samples: Those samples which are improperly sampled, insufficiently documented (chain of custody and other sample records and/or labels), improperly preserved, collected in improper containers, or exceeding holding times when delivered to a laboratory. Under normal conditions, compromised samples are not analyzed. If emergency situation require analysis, the results must be appropriately qualified.

Confidential Business Information (CBI): Information that an organization designates as having the potential of providing a competitor with inappropriate insight into its management, operation or products. TNI and its representatives agree to safeguarding identified CBI and to maintain all information identified as such in full confidentiality.

Confirmation: Verification of the identity of a component through the use of an approach with a different scientific principle from the original method. These may include, but are not limited to Second Column Confirmation; Alternate wavelength; Derivatization; Mass spectral interpretation; Alternative detectors or Additional Cleanup procedures. (TNI)

Conformance: An affirmative indication or judgment that a product or service has met the requirements of the relevant specifications, contract, or regulation; also the state of meeting the requirements. (ANSI/ASQC E4-1994)

Correction*:* Actions necessary to correct or repair analysis specific non-conformances. The acceptance criteria for method specific QC and protocols as well as the associated corrective actions. The analyst will most frequently be the one to identify the need for this action as a result of calibration checks and QC sample analysis**.** No significant action is taken to change behavior, process or procedure.

Corrective Action: The action taken to eliminate the causes of an existing nonconformity, defect or other undesirable situation in order to prevent recurrence. (ISO 8402)

Data Audit: A qualitative and quantitative evaluation of the documentation and procedures associated with environmental measurements to verify that the resulting data re of acceptable quality (i.e., that they meet specified acceptance criteria). (TNI)

Data Reduction: The process of transforming the number of data items by arithmetic or statistical calculations, standard curves, and concentration factors, and collation into a more useable form. (TNI)

Data Review Checker: Automated data review feature in TALS that compares data entered to list of control and preventative review requirements and presents all findings to the first level data reviewer for response or correction.

Deficiency: An unauthorized deviation from acceptable procedures or practices, or a defect in an item. (ASQC), whether in the laboratory's control or not.

Demonstration of Capability: A procedure to establish the ability of the analyst to generate analytical results of acceptable accuracy and precision. (TNI)

Document Control: The act of ensuring that documents (and revisions thereto) are proposed, reviewed for accuracy, approved for release by authorized personnel, distributed properly, and controlled to ensure use of the correct version at the location where the prescribed activity if performed. (ASQC)

Duplicate Analyses: The analyses or measurements of the variable of interest performed identically on two subsamples of the same sample. The results from duplicate analyses are used to evaluate analytical or measurement precision but not the precision of sampling, preservation or storage internal to the laboratory. (EPA-QAD)

Equipment Blank: Sample of analyte-free media which has been used to rinse common sampling equipment to check effectiveness of decontamination procedures.

External Standard Calibration: Calibrations for methods that do not utilize internal standards to compensate for changes in instrument conditions.

Field Blank: Blank prepared in the field by filing a clean container with pure de-ionized water and appropriate preservative, if any, for the specific sampling activity being undertaken (EPA OSWER)

Field of Accreditation: Those matrix, technology/method, and analyte combinations for which the accreditation body offers accreditation.

Holding Times : The maximum times that samples may be held prior to analyses and still be considered valid or not compromised. (40 CFR Part 136)

Internal Standard: A known amount of standard added to a test portion of a sample as a reference for evaluating and controlling the precision and bias of the applied analytical test method. (TNI)

Internal Standard Calibration: Calibrations for methods that utilize internal standards to compensate for changes in instrument conditions.

Instrument Blank: A clean sample (e.g., distilled water) processed through the instrumental steps of the measurement process; used to determine instrument contamination. (EPA-QAD)

Instrument Detection Limit (IDL): The minimum amount of a substance that can be measured with a specified degree of confidence that the amount is greater than zero using a specific instrument. The IDL is associated with the instrumental portion of a specific method only, and sample preparation steps are not considered in its derivation. The IDL is a statistical estimation at a specified confidence interval of the concentration at which the relative uncertainty is ± 100%. The IDL represents a range where qualitative detection occurs on a specific instrument. Quantitative results are not produced in this range.

Laboratory Control Sample (however named, such as laboratory fortified blank, spiked blank, or QC check sample): A sample matrix, free from the analytes of interest, spiked with verified known amounts of analytes or a material containing known and verified amounts of analytes, taken through all preparation

and analysis steps of the procedure unless otherwise noted in a reference method. It is generally used to establish intra-laboratory or analyst specific precision and bias or to assess the performance of all or a portion of the measurement system.

An LCS shall be prepared at a minimum of 1 per batch of 20 or less samples per matrix type per sample extraction or preparation method, or more frequently if so required by the reference method, except for analytes for which spiking solutions are not available such as, total volatile solids, odor, temperature, or dissolved oxygen. The results of these samples shall be used to determine batch acceptance.

Least Squares Regression (1st Order Curve): The least squares regression is a mathematical calculation of a straight line over two axes. The y axis represents the instrument response (or Response ratio) of a standard or sample and the x axis represents the concentration. The regression calculation will generate a correlation coefficient (r) that is a measure of the "goodness of fit" of the regression line to the data. A value of 1.00 indicates a perfect fit. In order to be used for quantitative purposes, r must be greater than or equal to 0.99 for organics and 0.995 for inorganics.

Limit of Detection (LOD): [a.k.a., Method Detection Limit (MDL]: The MDL is the minimum measured quantity of a substance that can be reported with 99% confidence that the concentration is distinguishable from method blank results, consistent with 40CFR Part 136 Appendix B, August, 2017.

Limit(s) of Quantitation (LOQ) [a.k.a., Reporting Limit]: The minimum levels, concentrations, or quantities of a target variable (e.g., target analyte) that can be reported with a specified degree of confidence. (TNI)

(QS) Matrix: The component or substrate that contains the analyte of interest. For purposes of batch and QC requirement determinations, the following matrix distinctions shall be used:

Aqueous: Any aqueous sample excluded from the definition of Drinking Water or Saline/Estuarine. Includes surface water, groundwater, effluents, and TCLP or other extracts.

Drinking Water: any aqueous sample that has been designated as a potable or potential potable water source.

Saline/Estuarine: any aqueous sample from an ocean or estuary, or other salt water source such as the Great Salt Lake.

Non-aqueous Liquid: any organic liquid with <15% settleable solids.

Biological Tissue: any sample of a biological origin such as fish tissue, shellfish, or plant material. Such samples shall be grouped according to origin.

Solids: includes soils, sediments, sludges, and other matrices with >15% settleable solids.

Chemical Waste: a product or by-product of an industrial process that results in a matrix not previously defined.

Air *& Emissions*: whole gas or vapor samples including those contained in flexible or rigid wall containers and the extracted concentrated analytes of interest from a gas or vapor that are collected with a sorbant tube, impinger solution, filter, or other device. (TNI)

Matrix Spike (spiked sample or fortified sample): A sample prepared, taken through all sample preparation and analytical steps of the procedure unless otherwise noted in a referenced method, by adding a known amount of target analyte to a specified amount of sample for which an independent test result of target analyte concentration is available. Matrix spikes are used, for example, to determine the effect of the matrix on a method's recovery efficiency.

Matrix Spike Duplicate (spiked sample or fortified sample duplicate): A replicate matrix spike prepared and analyzed to obtain a measure of the precision of the recovery for each analyte.
Method Blank: A sample of a matrix similar to the batch of associated samples (when available) that is free from the analytes of interest and is processed simultaneously with and under the same conditions as samples through all steps of the analytical procedures, and in which no target analytes or interferences are present at concentrations that impact the analytical results for sample analyses.

Method Detection Limit: See Limit of Detection (LOD)

Negative Control: Measures taken to ensure that a test, its components, or the environment do not cause undesired effects, or produce incorrect test results.

Non-conformance**:** An indication, judgment, or state of not having met the requirements of the relevant specifications, contract, or regulation.

Observation: A record of phenomena that (1) may assist in evaluation of the sample data; (2) may be of importance to the project manager and/or the client, and yet not at the time of the observation have any known effect on quality.

Performance Audit: The routine comparison of independently obtained qualitative and quantitative measurement system data with routinely obtained data in order to evaluate the proficiency of an analyst or laboratory.

Positive Control: Measures taken to ensure that a test and/or its components are working properly and producing correct or expected results from positive test subjects.

Precision: The degree to which a set of observations or measurements of the same property, obtained under similar conditions, conform to themselves; a data quality indicator. Precision is usually expressed as standard deviation, variance or range, in either absolute or relative terms.

Preservation: Any conditions under which a sample must be kept in order to maintain chemical and/or biological integrity prior to analysis. (TNI)

Proficiency Testing: A means of evaluating a laboratory's performance under controlled conditions relative to a given set of criteria through analysis of unknown samples provided by an external source. (TNI)

Proficiency Testing Program: The aggregate of providing rigorously controlled and standardized environmental samples to a laboratory for analysis, reporting of results, statistical evaluation of the results and the collective demographics and results summary of all participating laboratories. (TNI)

Proficiency Test Sample (PT): A sample, the composition of which is unknown to the laboratory and is provided to test whether the analyst/laboratory can produce analytical results within specified acceptance criteria. (TNIS)

Quality Assurance: An integrated system of management activities involving planning, implementation, assessment,~~,~~ reporting and quality improvement to ensure that a process, item, ~~product~~ or service is of the type of quality needed and expected by the client. (TNI)

Quality Assurance [Project] Plan (QAPP): A formal document describing the detailed quality control procedures by which the quality requirements defined for the data and decisions pertaining to a specific project are to be achieved. (EAP-QAD)

Quality Control: The overall system of technical activities that measures the attributes and performance of a process, item, or service against defined standards to verify that they meet the stated requirements established by the customer; operational techniques and activities that are used to fulfill requirements for quality; also the system of activities and checks used to ensure that measurement systems are maintained within prescribed limits, providing protection against “out of control” conditions and ensuring that the results are of acceptable quality. (TNI)

Quality Control Sample: A sample used to assess the performance of all or a portion of the measurement system. One of any number of samples, such as Certified Reference Materials, a quality system matrix fortified by spiking, or actual samples fortified by spiking, intended to demonstrate that a measurement system or activity is in control. (TNI)

Quality Manual: A document stating the management policies, objectives, principles, organizational structure and authority, responsibilities, accountability, and implementation of an agency, organization, or laboratory, to ensure the quality of its product and the utility of its product to its users. (TNI)

Quality System: A structured and documented management system describing the policies, objectives, principles, organizational authority, responsibilities, accountability, and implementation plan of an organization for ensuring quality in its work processes, products (items), and services. The quality system provides the framework for planning, implementing, and assessing work performed by the organization and for carrying out required QA and QC activities. (TNI)

Raw Data: The documentation generated during sampling and analysis. This documentation includes, but is not limited to, field notes, electronic data, magnetic tapes, untabulated sample results, QC sample results, print outs of chromatograms, instrument outputs, and handwritten records. (TNI)

Record Retention: The systematic collection, indexing and storing of documented information under secure conditions.

Reference Material: Material or substance one or more properties of which are sufficiently homogeneous and well established to be used for the calibration of an apparatus, the assessment of a measurement method, or for assigning values to materials. (ISO Guide 30-2.1)

Reference Standard: Standard used for the calibration of working measurement standards in a given organization or a given location. (TNI)

Sampling: Activity related to obtaining a representative sample of the object of conformity assessment, according to a procedure.

Second Order Polynomial Curve (Quadratic): The 2^{nd} order curves are a mathematical calculation of a slightly curved line over two axis. The y axis represents the instrument response (or Response ratio) of a standard or sample and the x axis represents the concentration. The 2^{nd} order regression will generate a coefficient of determination (COD or r^2) that is a measure of the "goodness of fit" of the quadratic curvature the data. A value of 1.00 indicates a perfect fit. In order to be used for quantitative purposes, r^2 must be greater than or equal to 0.99.

Selectivity: The ability to analyze, distinguish, and determine a specific analyte or parameter from another component that may be a potential interferent or that may behave similarly to the target analyte or parameter within the measurement system. (TNI)

Sensitivity: The capability of a method or instrument to discriminate between measurement responses representing different levels (e.g., concentrations) of a variable of interest. (TNI)

Spike: A known mass of target analyte added to a blank, sample or sub-sample; used to determine recovery efficiency or for other quality control purposes.

Standard: The document describing the elements of laboratory accreditation that has been developed and established within the consensus principles of standard setting NELAC and meets the approval requirements of standard adoption organizations procedures and policies. (TNI)

Standard Operating Procedures (SOPs): A written document which details the method for an operation, analysis, or action, with thoroughly prescribed techniques and steps. SOPs are officially approved as the methods for performing certain routine or repetitive tasks. (TNI)

Storage Blank: A blank matrix stored with field samples of a similar matrix (volatiles only) that measures storage contribution to any source of contamination.

Surrogate: A substance with properties that mimic the analyte of interest. It is unlikely to be found in environment samples and is added to them for quality control purposes.

Surrogate compounds must be added to all samples, standards, and blanks, for all organic chromatography methods except when the matrix precludes its use or when a surrogate is not available. Poor surrogate recovery may indicate a problem with sample composition and shall be reported to the client whose sample produced poor recovery. (QAMS)

Systems Audit (also Technical Systems Audit): A thorough, systematic, qualitative on-site assessment of the facilities, equipment, personnel, training, procedures, record keeping, data validation, data management, and reporting aspects of a total measurement system. (EPA-QAD)

Technical Manager: A member of the staff of an environmental laboratory who exercises actual day-to-day supervision of laboratory operations for the appropriate fields of accreditation and reporting of results

Technology: A specific arrangement of analytical instruments, detection systems, and/or preparation techniques.

Traceability: The ability to trace the history, application, or location of an entity by means of recorded identifications. In a calibration sense, traceability relates measuring equipment to national or international standards, primary standards, basic physical constants or properties, or reference materials. In a data collection sense, it relates calculations and data generated throughout the project back to the requirements for the quality of the project. (TNI)

Trip Blank:
A blank matrix placed in a sealed container at the laboratory that is shipped, held unopened in the field, and returned to the laboratory in the shipping container with the field samples.

Uncertainty:
A parameter associated with the result of a measurement that characterizes the dispersion of the value that could reasonably be attributed to the measured value.

Acronyms:

CAR – Corrective Action Report
CCV – Continuing Calibration Verification
CF – Calibration Factor
CFR – Code of Federal Regulations
COC – Chain of Custody
DOC – Demonstration of Capability
DQO – Data Quality Objectives
DUP - Duplicate
EHS – Environment, Health and Safety
EPA – Environmental Protection Agency
GC - Gas Chromatography
GC/MS - Gas Chromatography/Mass Spectrometry
HPLC - High Performance Liquid Chromatography
ICP - Inductively Coupled Plasma Atomic Emission Spectroscopy
ICP/MS – ICP/Mass Spectrometry
ICV – Initial Calibration Verification
IDL – Instrument Detection Limit
IH – Industrial Hygiene
IS – Internal Standard
LCS – Laboratory Control Sample
LCSD – Laboratory Control Sample Duplicate
LIMS – Laboratory Information Management System
LOD – Limit of Detection
LOQ – Limit of Quantitation
MDL – Method Detection Limit
MDLCK – MDL Check Standard
MDLV – MDL Verification Check Standard
MRL – Method Reporting Limit Check Standard
MS – Matrix Spike
MSD – Matrix Spike Duplicate
MSDS - Material Safety Data Sheet
NELAP - National Environmental Laboratory Accreditation Program
PT – Performance Testing
TNI – The NELAC Institute
QAM – Quality Assurance Manual
QA/QC – Quality Assurance / Quality Control
QAPP – Quality Assurance Project Plan
RF – Response Factor
RPD – Relative Percent Difference
RSD – Relative Standard Deviation
SD – Standard Deviation
SOP: Standard Operating Procedure
TAT – Turn-Around-Time
VOA – Volatiles
VOC – Volatile Organic Compound

Appendix 3

Laboratory Certifications, Accreditations, Validations

Pittsburgh maintains certifications, accreditations, certifications, and validations with numerous state and national entities. Programs vary but may include on-site audits, reciprocal agreements with another entity, performance testing evaluations, review of the QA Manual, Standard Operating Procedures, Method Detection Limits, training records, etc. At the time of this QA Manual revision, the laboratory has accreditation / certification / licensing with the following organizations:

Organization	Certificate Number Or Laboratory ID Number
Arkansas	88-0690
California ELAP	2891
Connecticut	PH-0688
Florida	E871008
Illinois	002602
Kansas	E-10350
Kentucky	KY98043
Louisiana	04041
Maine	2020007
Minnesota	042-999-482
Nevada	PA00164
New Hampshire	203010
New Jersey	PA005
New York	11182
North Carolina	434
North Dakota	R-227
Oregon	PA-2151
Pennsylvania	02-00416
Rhode Island	LAO00362
South Carolina	89014002
Texas	T104704528
Utah	STLP
USDA	P330-10-00139
USDA	P-Soil -01
Virginia VELAP	460189
West Virginia	142
Wisconsin	998027800

The certificates and parameter lists (which may differ) are available, upon request, from a laboratory representative. They may be found on the Corporate web site, the laboratory's public server and in the QA web page.

Appendix 4

Pittsburgh Laboratory SOP List

Document No.	Title
PT-FS-001	Field Measurement of Dissolved Oxygen (DO) Method: SM 4500-O G
PT-FS-002	Field Measurement of Total Residual Chlorine Method: SM 4500-Cl G
PT-FS-003	Field Measurement of pH Method: SM 4500 H+B
PT-FS-004	General Sampling Instructions
PT-GC-001	Gas Chromatographic Analysis of Herbicides, SW-846 Method 8151A
PT-GC-002	Analysis of Organochlorine Pesticides and PCBs by Method 608
PT-GC-004	1,2-Dibromoethane(EDB) and 1,2-Dibromo-3-Chloropropane(DBCP) in Water by Microextraction and Gas Chromatography, Method 8011
PT-GC-005	Polychlorinated Biphenyls (PCBs) and PCBs as Congeners by GC/ECD - Method: SW-846 8082 and 8082A
PT-GC-006	Chlorinated Pesticides - Method: SW-846 8081A/B
PT-GC-007	Organophosphorus Pesticides by Gas Chromatography - Method: SW-846 8141A and 8141B
PT-GC-009	Determination of Inorganic Anions by Ion Chromatography EPA Method 300 SW-846 Method 9056A
PT-GC-010	TOC Analysis for Solids by Lloyd Kahn Method
PT-GC-013	Determination of Particulate Organic Carbon in Sediment and Estuarine / Coastal Water Matrices
PT-GC-014	Halogenated Organic Compounds by Direct Injection GC/ECD
PT-GC-015	Analysis of Organochlorine Pesticides and PCBs by EPA Method 608.3
PT-HS-001	Pittsburgh Facility Addendum to Eurofins TestAmerica Corporate Environmental Health & Safety Manual (CW-E-M-001)
PT-HS-002	Bloodborne Pathogen Exposure Control Path
PT-IP-002	Acid Digestion of Soils, SW-846 Method 3050B
PT-IP-003	Acid Digestion of Aqueous Samples by SW-846 Methods 3005A, 3010A and EPA Methods 200.7 and 200.8
PT-IT-001	Data Backup Procedures
PT-MI-001	Total Coliforms and E. coli – Presence/Absence Test Using Colilert
PT-MS-001	Semivolatile Organic Anaysis by GC/MS, Method 625
PT-MS-002	Volatile Organics by GC/MS Based on Method 624
PT-MS-005	VOA Holding Blanks

Document No.	Title
PT-MS-008	GC/MS Analysis for Semivolatile Organics, Methods: SW-846 8270C & D
PT-MS-010	Determination of Volatile Organics by GC/MS Methods: SW-846 8260B & C
PT-MS-011	Semivolatile Organic Analysis by GCMS, EPA Method 625.1
PT-MS-012	Volatile Organic Analysis by GCMS, EPA Method 624.1
PT-MT-001	Inductively Coupled Plasma-Atomic Emission Spectroscopy, Spectrometric Method for Trace Element Analyses, SW-846 Method 6010B, 6010C, 6010D and EPA Method 200.7
PT-MT-002	Analysis of Metals by Inductively Coupled Plasma/Mass Spectrometry (ICPMS) for Methods 200.8, 6020, 6020A
PT-MT-005	Preparation and Analysis of Mercury by Cold Vapor Atomic Absorption
PT-OP-001	Extraction and Cleanup of Organic Compounds from Waters and Solids, Based on SW-846 3500 and 600 Series Methods
PT-OP-002	Simplified Laboratory Runoff Procedure (SLRP)
PT-OP-003	Standard Elutriate Test (SET)
PT-OP-004	Toxicity Characteristic Leaching Procedure and Synthetic Precipitation Leaching Procedure
PT-OP-005	Modified and Effluent Elutriate Tests (MET and EET)
PT-OP-006	Long Tube Column Settling Test
PT-OP-007	Illinois Re-suspension Tests
PT-OP-008	Dredging Elutriate Test (DRET)
PT-OP-009	Sequential Batch Leach Test (SBLT) for Freshwater Sediments
PT-OP-011	Extractable Residue (Lipids) from Animal Tissue
PT-OP-015	Modified Multiple Extraction Procedure Method: SW-846 1320
PT-OP-016	Porewater Generation
PT-OP-017	Liquid-Solid Partitioning as a Function of Extract pH in Solid Materials using a Parallel Batch Procedure SW-846 Method 1313
PT-OP-018	Liquid-Solid Partitioning as a Function of Liquid-to-Solid Ratio in Solid Materials using a Parallel Batch Procedure SW-846 Method 1316
PT-OP-019	Mass Transfer Rates of Constituents in Monolithic or Compacted Granular Materials using a Semi-Dynamic Tank Leaching Procedure SW-846 Method 1315
PT-OP-020	Standard Test Method for 24-h Batch-Type Measurement of Contaminant Sorption by Soils and Sediments ASTM D4646-03
PT-OP-021	Liquid-Solid Partitioning as a Function of Liquid-to-Solid Ratio for Constituents in Solid Materials using an Up-Flow Percolation Column Procedure SW-846 Method 1314

Document No.	Title
PT-OP-022	Low Volume Extraction and Cleanup of Organic Compounds from Waters Method: SW846 3500 Series
PT-OP-023	Measurement of the Leachability of Solidified Wastes by a Short Term Test Procedure Modified ANSI/ANS-16.1-2003
PT-OP-025	Soil/Sediment Amendment Procedure
PT-OP-026	Extraction of Organic Compounds from Solids, Sediments, Tissues, and Wipes Based on SW-846 3500 Series Methods
PT-OP-027	Extraction of Herbicides by SW846 Method 8151A
PT-OP-028	Clean-up of Organic Extracts by SW-846 3600 Series Methods
PT-OP-030	Handling and Preparation of Biota and Tissue Samples
PT-OP-031	Australian Standard Leaching Procedure - AS 4439.3-1997
PT-QA-001	Employee Orientation & Training
PT-QA-002	Internal Auditing
PT-QA-003	Glassware Clean-up for Organic/Inorganic Procedures
PT-QA-004	Quarantine Soil Procedure
PT-QA-005	Measurement Uncertainty
PT-QA-006	Procurement of Standards and Materials; Labeling and Traceability
PT-QA-007	Detection Limits
PT-QA-008	Thermometer and Barometer Calibration and Temperature Monitoring
PT-QA-009	Rounding and Significant Figures
PT-QA-010	Document Development and Control
PT-QA-012	Selection and Calibration of Balances and Weights
PT-QA-013	Independent QA Data Review
PT-QA-014	Reporting Limits
PT-QA-016	Nonconformance & Corrective Action System
PT-QA-017	Aqueous Pipette Calibration – Gravimetric Method
PT-QA-018	Technical Data Review Requirements
PT-QA-019	Records Information Management
PT-QA-021	Laboratory Quality Control Program

Document No.	Title
PT-QA-022	Equipment Maintenance
PT-QA-024	Subsampling
PT-QA-026	Container Accuracy Verification – Gravimetric
PT-QA-031	Internal Chain of Custody
PT-SR-001	Sample Receipt & Login
PT-SR-002	Bottle Order Preparation and Shipping
PT-WC-001	Determination of Total and Total Volatile Solids in Waters and Wastes (Methods EPA160.4 and SM 2540B & 2540E)
PT-WC-002	Color, Method 110.2
PT-WC-003	Alkalinity, SM Method 2320B
PT-WC-004	Total Hardness (mg/L as CaCO3) by Method SM 2340C; and Hardness by Calculation SM 2340B
PT-WC-005	Turbidity by Method 180.1
PT-WC-007	Nitrate/Nitrite-N EPA Method 353.2
PT-WC-008	Acid Volatile Sulfides (AVS) and Simultaneously Extracted Metals (SEM) in Sediment
PT-WC-009	Performance Checks on Spectronic 21 and Model 1001 Spectro-Photometers
PT-WC-010	Total Sulfide as Acid Soluble Sulfide, Method 9030B/9034, Standard Method 20th Ed. 4500S-2-F
PT-WC-013	Specific Conductance by 120.1, 2510B, and 9050A
PT-WC-014	Nitrogen, Ammonia (Automated), Method 350.1
PT-WC-015	Chromium, Hexavalent (Colorimetric) by SM3500-Cr-B, SW846 3060A/7196
PT-WC-016	Biochemical Oxygen Demand (BOD) and Carbonaceous Biochemical Oxygen Demand (CBOD) by Dissolved Oxygen Probe - SM5210B
PT-WC-017	Total Organic Carbon (TOC) and Total Inorganic Carbon (TIC), Methods SM 5310C and SW-846 9060/9060A
PT-WC-018	Cyanide – Semi-Automated, Pyridine-Barbituric Acid For Total and Amenable, Cyanide in Water (Methods 335.4) and Soil Analyses (Method 9012A/9012B)
PT-WC-020	Percent Moisture, Ash, Organic Matter and Total Solids in Soil Samples - SM 2540G and ASTM D297-84
PT-WC-021	Flash Point by Pensky-Martens Closed Tester, SW-846 Method 1010A and ASTM D93-08
PT-WC-022	Ignitability of Solids for Waste Characterization EPA SW-846 Chapter 7, Section 7.1
PT-WC-023	Chemical Oxygen Demand, Low Level, Method 410.4
PT-WC-025	n-hexane extractable material (HEM) in Sludge, Sediment and Soil samples - 9071B

Document No.	Title
PT-WC-026	PH Electrometric by SM 4500 H+B and SW-846 Methods: 9045C/D and 9040B/C
PT-WC-027	Salinity by Calculation, Electrical Conductivity Method SM 2520B
PT-WC-028	Hexane Extractable Material (HEM; Oil and Grease) and Silica Gel Treated Hexane Extractable Material (SGT-HEM; TPH), Method 1664A/1664B and 9070A
PT-WC-029	Available Cyanide by Ligand Exchange and Flow Injection Analysis (FIA) Method 1677
PT-WC-031	Cyanide Extraction Procedure for Solids and Oils, SW-846 Method 9013
PT-WC-032	Total Organic Carbon Analysis for Solid Matrices by Walkley Black
PT-WC-033	DI-Leachate Procedure for Solids (1 Hour Routine DI Leachate Procedure)
PT-WC-034	Paint Filter Liquids Test, SW-846 Method 9095B
PT-WC-035	Acidity of Water and Waste Water, SM Method 2310B
PT-WC-036	Flash Point of Liquids by Setaflash (Small Scale) Closed-Cup Apparatus, SW-846 Method 1020B and ASTM Standard D 3278-96
PT-WC-037	Oxidation Reduction Potential, SM 2580B (20th Ed)
PT-WC-038	Phenolics (Automated), Method 420.1/420.2, SW-846 9065/9066
PT-WC-039	Screening Apparent Specific Gravity and Bulk Density of Waste - Method: ASTM D 5057-90
PT-WC-040	Anion Surfactants as MBAS, Standard Methods 5540C
PT-WC-041	Compatibility of Screening Analysis of Waste, Method: ASTM D5058 Test Method C – Water Compatibility
PT-WC-042	Acid Titration of Samples for Bechtel Bettis (NaOH Solution for Caustic and Na2CO3 Concentration), Bechtel-Bettis SOP - WAPD-MT(CAC)-2141, Revision 0
PT-WC-043	Determination of Total and Volatile Suspended Solids in Waters and Wastes - EPA 160.4 and SM 2540D & 2540E
PT-WC-044	Determination of Settleable Solids in Water SM 2540F
PT-WC-045	Determination of Total Dissolved and Volatile Dissolved Solids in Water and Wastes, SM 2540C & 2540E
PT-WC-046	ASTM 18-Hour Leaching Procedure

Note: The SOPs are subject to change, refer to PT-QA-WI-002 for current list of SOPs and revision numbers.

eurofins | Environment Testing TestAmerica

TITLE: Analysis for Sulfide

Method: SW-846 9030B/9034 and SM 4500-S^{2-} C & F

Approvals (Signature/Date):

4/27/2020	4/27/2020
Roseann Ruyechan Date Inorganics Department Manager	Steve Jackson Date Regional Safety Coordinator
4/22/2020	4/27/2020
Virginia Zusman Date Quality Assurance Manager	Deborah L. Lowe Date Laboratory Director

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1. SCOPE AND APPLICATION

1.1. The distillation procedure described in this method is designed for the determination of total sulfides in aqueous, solid waste materials, or effluents in conjunction with method 9030B/9034.

1.2. This procedure measures total sulfide, which is usually defined as the acid-soluble fraction of a waste. If, however, the client has previous knowledge of the waste and is concerned about both soluble sulfides such as H_2S, and metals sulfides, such as CuS and SnS_2, then total sulfide is defined as the combination of both acid-soluble and acid-insoluble fractions. For wastes where only metals sulfides are suspected to be present, only the acid-insoluble fraction needs to be performed.

1.3. Total Sulfide includes dissolved H_2S and HS^-, as well as acid-soluble metallic sulfides present in suspended matter. The S^{2-} is negligible, amounting to less than 0.5% of the dissolved sulfide at pH 12, less than 0.05% at pH 11 etc. Copper and silver sulfides are so insoluble that they do not respond in ordinary sulfide determinations; they can be ignored for most practical purposes.

1.4. This method is not applicable to oil or multiphasic sample or samples not amenable to the distillation procedure. Such samples must be forwarded to a lab that can perform the analysis by Method 9031.

1.5. Methods 9030B and 9034 are suitable for measuring sulfide concentrations in samples, which contain between 0.2 mg/kg to 50 mg/kg of sulfide.

1.6. Method 9030B is not applicable for distilling reactive sulfide. Method 9034 may be used to quantify the concentration of sulfide from the reactivity test.

1.7. The titration procedure is suitable for the measurement of Sulfide at concentrations in waters above 3 mg/L. This concentration is used as the reporting limit.

1.8. Unionized Hydrogen Sulfide (SM 4500-S^{2-}F) can be calculated from the concentration of dissolved sulfide, the sample pH and the practical ionization constant of H_2S.

1.9. Any modifications to this procedure must be approved by the QA department or the Laboratory or Technical Director and documented fully in an NCM that accompanies the analytical batch.

2. SUMMARY OF METHOD

2.1. For acid-soluble sulfide samples by method 9030B/9034, separation of sulfide from the sample matrix is accomplished by the addition of sulfuric acid to the sample. The sample is heated to 70℃ in a sealed distillation a pparatus for 90 minutes. The distillation apparatus is continuously purged with nitrogen gas. Any hydrogen sulfide (HS) that is formed is carried by the nitrogen stream into gas scrubbing bottles containing a solution of zinc acetate where the hydrogen sulfide precipitates as zinc sulfide.

2.2. For acid-insoluble sulfide samples, separation of sulfide from the sample matrix is accomplished by suspending the sample in hydrochloric acid by vigorous agitation. The acidified sample is distilled at 100^{o}C under a stream of nitrogen. Hydrogen sulfide gas is released from the sample and collected in gas scrubbing bottles containing zinc(II) and a strong buffer. Zinc sulfide precipitates.

2.3. The sulfide in the zinc sulfide precipitate is quantified titrimetrically by Method 9034. Excess iodine is added to the sample, which oxidizes the sulfide to sulfur under acidic conditions. The excess iodine is back titrated with sodium thiosulfate. Samples are distilled according to method 9030B to remove interferences.

2.4. For method SM 4500-S^{2-}F, excess iodine is added to a sample that is treated with zinc acetate to produce zinc sulfide. The iodine oxidizes the sulfide to sulfur under acidic conditions. The excess iodine is back titrated with sodium thiosulfate. Samples are pre-treated according to method SM 4500-S^{2-}C to remove interferences.

3. DEFINITIONS

3.1. TALS – TestAmerica Laboratory Information Management System

3.2. NCM - Non-Conformance Memo – a system within TALS for the lab to communicate to project management and others when there is an anomaly seen with the samples or batch, or a QC failure.

3.3. Refer to the glossary in the Laboratory Quality Manual (PT-QA-M-001), current version for additional definitions.

4. INTERFERENCES

4.1. Aqueous samples must be taken with minimum aeration to avoid volatilization of sulfide, which oxidizes sulfide to sulfur compounds that are not detected. Use deoxygenated, deionized water and reagents. Avoid aerating the sample to minimize the exposure of the sample to atmospheric oxygen.

4.2. Carbonates in the sample may affect the distillation by causing excessive gassing when acid is added.

4.3. Reduced sulfur compounds, such as sulfite and hydrosulfite, will decompose in acid and form sulfur dioxide. This gas may carry over to the zinc acetate solutions and subsequently react with iodine during the titration, thus causing a positive bias to the

results. This method shows no sensitivity to sulfite up to 10 mg/kg of sulfite in the sample.

4.4. The iodometric method suffers interference from reducing substances that react with iodine, including thiosulfate, sulfite, and various organic compounds.

5. SAFETY

5.1. Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual (CW-E-M-001), the Pittsburgh Facility Addendum EH&S Manual (PT-HS-001), and this document. This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

5.2. Safety Note: If you forget, and the samples have been heated once on the distillation block, DO NOT add reagents, let the samples completely cool first and redistill.

5.3. Sodium Sulfide will form Hydrogen Sulfide (HS) gas if combined with water moisture or strong acids. Inhalation of H_2S gas can cause headache, dizziness, nausea, unconsciousness and potentially death.

5.4. The following is a list of the materials used in this method, which have a serious or significant hazard rating. NOTE: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the SDS for each of the materials listed in the table. A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the SDS for each material before using it for the first time or when there are major changes to the SDS.

Material (1)	Hazards	Exposure Limit (2)	Signs and Symptoms of Exposure
Hydrochloric Acid	Corrosive Poison	5 ppm - Ceiling	Inhalation of vapors can cause coughing, choking, inflammation of the nose, throat, and upper respiratory tract, and in severe cases, pulmonary edema, circulatory failures, and death. Can cause redness, pain, and severe skin burns. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
Iodine	Poison Corrosive Oxidizer	0.1 ppm - Ceiling	Vapors severely irritating and can burn the mucous membranes and respiratory tract. Liquid contact may cause blistering burns, irritation, and pain. Vapors may be severely irritating to the skin. Vapors are severely irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
Sodium Sulfide	Corrosive	10ppm-TWA 15ppm-STEL	Will form Hydrogen Sulfide (HS) gas if combined with strong acids. Inhalation of HS gas may be fatal. Symptoms include

			painful conjunctivitis, headache, nausea, dizziness, coughing, and, in extreme cases, pulmonary edema and possible death. Irritant. Contact with skin can produce serious caustic burns and painful inflammation and possible destruction of tissue. Eye contact can cause inflammation, tearing and pain. Severe contact can cause destruction of tissue.
Material (1)	**Hazards**	**Exposure Limit (2)**	**Signs and Symptoms of Exposure**
Sulfuric Acid	Corrosive Oxidizer Dehydrator Poison Carcinogen	1 mg/m^3 - TWA	Inhalation produces damaging effects on the mucous membranes and upper respiratory tract. Symptoms may include irritation of the nose and throat and labored breathing. Symptoms of redness, pain, and severe burns can occur. Contact can cause blurred vision, redness, pain, and severe tissue burns. Can cause blindness.
(1) – Always add acid to water to prevent violent reactions.			
(2) – Exposure limit refers to the OSHA regulatory exposure limit.			

5.5. Eye protection that protects against splash, laboratory coat, and appropriate gloves must be worn while samples, standards, solvents, and reagents are being handled. Cut resistant gloves must be worn doing any other task that presents a strong possibility of getting cut. Disposable gloves that have been contaminated will be removed and discarded; other gloves will be cleaned immediately.

5.6. Exposure to chemicals must be maintained as low as reasonably achievable, therefore, unless they are known to be non-hazardous, all samples must be opened, transferred and prepared in a fume hood, or under other means of mechanical ventilation. Solvent and waste containers will be kept closed unless transfers are being made.

5.7. VITON gloves may be worn when halogenated solvents are used for extraction or sample preparation, nitrile gloves may be used when other solvents are handled. [Note: VITON is readily degraded by acetone; all solvents will readily pass through disposable latex rubber gloves.]

5.8. The preparation of standards and reagents will be conducted in a fume hood with the sash closed as far as the operation will permit.

5.9. All work must be stopped in the event of a known or potential compromise to the health and safety of a TestAmerica associate. The situation must be reported immediately to a laboratory supervisor or the EH&S coordinator.

5.10. This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

6. EQUIPMENT AND SUPPLIES

The following items are recommended for performing this procedure. Equivalent items should only be used when they result in an improvement in quality, efficiency, productivity, or cost. An item can be considered equivalent if with its use, the analytical and QA/QC requirements in this SOP can be met.

6.1. Boiling tube
6.2. Inlet adapter
6.3. Dropping funnel
6.4. Gas inlet
6.5. Impinged bubbler
6.6. Bubbler vessels
6.7. WestClips®, size 24/40
6.8. Gas line “T” connector
6.9. Class A Volumetric flasks, pipettes, and burettes
6.10. High purity nitrogen gas
6.11. Regulator
6.12. Easy Midi distillation heating block
6.13. 100mL disposable flasks
6.14. 50mL burette
6.15. Glass microfiber filter

7. REAGENTS AND STANDARDS

Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee of Analytical Reagents of the American Chemical Society, where such specifications are available. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

7.1. Reagent water - All references to water in this method refer to laboratory reagent water or deionized water

7.2. Hydrochloric acid – concentrated, purchased

7.2.1 Hydrochloric acid (9.8N): Slowly add 817mL of concentrated HCL to 100mL reagent water in a 1L volumetric flask and dilute to volume with reagent water.

7.2.2 Hydrochloric acid (6N): Slowly add 500mL of concentrated HCl to 400mL reagent water in a 1L volumetric flask and dilute to volume with reagent water.

7.3. Sulfuric acid (H_2SO_4) – concentrated, purchased

7.3.1. 1:4 Sulfuric acid – slowly add 25mL concentrated acid to 50mL laboratory reagent water in a 100mL volumetric flask and bring to volume with reagent water. THIS SOLUTION WILL BE VERY HOT.

7.4. Zinc acetate dehydrate – $Zn(O_2CCH_3)_2 \bullet 2\ H_2O$, purchased

7.5. Sodium acetate – $Na(O_2CCH_3)$, purchased

7.6. Zinc acetate solution (0.5M) – for Acid-Soluble Sulfides: Dissolve about 110g zinc acetate dihydrate in 200mL of reagent water. Add 1mL concentrated HCL, to prevent precipitation of zinc hydroxide. Dilute to 1L. Store at room temperature. This solution expires 1 year after preparation, not to exceed the expiration of the parent salt.

7.7. Zinc acetate/sodium acetate buffer – for Acid-Insoluble Sulfides: Dissolve 100g sodium acetate and 11g zinc acetate in 800ml reagent water. Add 1mL concentrated HCL, to prevent precipitation of zinc hydroxide. Dilute to 1L. Store at room temperature. This solution expires 1 year after preparation, not to exceed the expiration of the parent salts.

7.8. UHP/zero grade nitrogen gas, gas chromatographic grade, with two-stage regulator

7.9. Starch indicator (0.5%), purchased

7.10. Sodium Thiosulfate solution (0.025N), purchased with certificate of analysis.

7.10.1. If necessary, this solution may be prepared in the lab from certified ACS $Na_2S_2O_3.5H_2O$ by weighing 24.82g, diluting to 4L in reagent water and preserving with 36 mL of 1N NaOH. (NOTE: Weights/Volumes can be adjusted as long as proportionality is maintained.)

7.11. 0.025N Iodine, commercially purchased standard. This standard can also be prepared from 1N Iodine solution commercially purchased. Pipette with a class A pipette 50mL of 1N solution and diluting to 2L with reagent water.

7.12. 1000ppm Sodium sulfide spiking solution is prepared by dissolving approximately 1.8g $Na_2S\bullet 9H_2O$ in 250mL reagent water. The exact concentration of the solution must be titrometrically determined due to loss of sodium sulfide due to atmospheric oxygen and/or water vapor. (NOTE: Weights/Volumes can be adjusted as long as proportionality is maintained.) Prepare this standard fresh weekly and determine the exact concentration every day of use. Store at $4 \pm 2^{o}C$. A new TALS Reagent must be entered each day if the concentrations changes from the previously entered standard. The mass must be adjusted to match the titration determined concentration from the worksheet. (See Figure 2)

7.12.1. Secondary 1000ppm Sulfide Spiking Solution – prepared following steps in 7.12 above, with a second source of sodium sulfide.

7.13. Potassium Biiodate powder, commercially purchased

7.13.1. Standard 0.0250N Potassium Biiodate: dissolve 0.8124g potassium biiodate (dried 2 hours) in reagent grade water and dilute to 1 L.

7.14. Stannous Chloride Crystal, Dihydrate, Purchased

7.15 Anti-foaming agent, Sigma Antifoam Y-30 or similar

8. SAMPLE COLLECTION, PRESERVATION, SHIPMENT AND STORAGE

Sample container, preservation requirements and holding times are listed below.

Matrix	Sample Container	Min. Sample Size	Preservation	Holding Time	Reference
Water	Plastic or Glass	250 mL	2N Zinc acetate and 6N NaOH to pH >9, Cool > 0 to ≤ 6.0°C	7days from collection	40 CFR Part 136
Solid	Glass	4 oz amber jar	2N zinc acetate until soil is moistened Cool >0 to ≤ 6.0°C	7days from collection	40 CFR Part 136

8.1. All aqueous samples and effluents must be preserved in the field with zinc acetate and sodium hydroxide to pH > 9. Sample bottle should be filled completely, with a minimum of aeration. The treated sample is relatively stable and can be held for up to seven days. For solid samples, the surface of the solid should be covered with 2N zinc acetate until moistened. If samples are received not properly preserved, this is noted in an NCM to be included in the narrative.

8.2. Samples must be kept cooled at >0 to ≤ 6℃ and stored headspace free.

8.3. If after distillation, the distillate can not be immediately titrated it may be stored at >0 to ≤ 6℃ for up to 24 hours before final titration pro vided the 7 day holding time from sample collection is not exceeded.

9. QUALITY CONTROL

9.1. The following quality control samples are prepared with each batch of samples.

Quality Controls	Frequency	Control Limit
ICV/CCV (not distilled)	Prior to sample analysis, after every 10 titrations and at the end of the run	4500 S^{2-} F 90-110% 9034 85-115%
ICB/CCB (not distilled)	Prior to sample analysis, after every 10 titrations and at the end of the run	< RL
Method Blank (MB)	1 per preparation batch[1]	< RL

Quality Controls	Frequency	Control Limit
Laboratory Control Sample (LCS)	1 per preparation batch[1]	85-115 %
Matrix Spike (MS)[3]/MSD	1 per preparation batch[1]	75-125[2]% and 20% RPD

[1]A batch is limited to 20 samples.
[2]Statistical control limits will be developed when there is sufficient data and updated as per SOP PT-QA-021.
[3]The MS/MSD is randomly selected, unless specifically requested by a client.

9.2. Method Blank (MB): A method blank is processed using reagent grade water and all chemical reagents and procedures as the associated samples. The MB associated with distillation of soil samples uses glass, plastic or Teflon beads to simulate a solid matrix. It is used to monitor laboratory and/or reagent contamination. As a part of the QC batch it accompanies the samples through all the steps of the preparation and analytical procedure. Any treatment step performed on a sample must also be performed on the MB.

9.2.1. The ICB and CCB are reagent water only, and are not taken through the distillation step with samples. The ICB is analyzed at the beginning of the analytical batch, prior to any samples. A CCB is analyzed after every 10, or fewer, samples, and at the end of the analytical batch. For water batches analyzed without distillation, the ICB serves as the MB.

9.2.2. All blank (ICB/CCB/MB) results must be less than the reporting limit; otherwise all samples must be re-prepared and reanalyzed, unless one of the following conditions applies:

- If all associated samples are non-detect, results may be reported with the NCM “Method Blank – Report, ND”. If associated samples have concentrations that are greater than 10 times the MB concentration, results may be reported with the NCM “Method Blank – Report, 10X”.

- If reanalysis is not possible due to limited sample quantity, the PM will be notified and results will be reported flagged with a “B” qualifier and the NCM “Method Blank – Insufficient Sample”.

- If reanalysis will be outside of holding time, the PM must be notified and client instructions obtained for reporting results or reanalyzing sample. Corrective action taken must be included in the case narrative.

9.3. The exact Sodium Sulfide Spiking Standards’ concentration must be determined each day of use by titrating 1.0mL of solutions 7.12 and 7.12.1, diluted to approximately 100mL with reagent water. Titrate each stock standard, following the analytical procedure in section 10.5. Use the Sulfide Analysis Standardization Logsheet, PT-WC-

WS-011, to calculate both the true value and the mass to enter into the TALS Reagent for this true value. (See Figure 2).

NOTE: The TALS Reagent is adjusted for the titrated true value by entering the exact weight of sodium sulfide crystals that would produce the determined concentrations.

Sodium sulfide in g = determined concentration / 534

9.4. ICV and CCV are prepared in laboratory reagent water with 1.0mL of spiking solution 7.12, and the true value is determined per section 9.3. Recovery must be within ± 85-115% for method 9034 and within 90-110% for method SM 4500 S^{2-} F.

9.4.1. The ICV and CCV are not taken through the distillation step with the samples. If samples are analyzed without distillation, the ICV may serve as the LCS, and the ICV control limits must be met. ICV/CCV control limits for method 9034 are 85-115%, for SM 4500S^{2-} F, the limits are 90-110%.

9.4.2. If the ICV does not meet recovery criteria, check calculations, re-standardize the iodine solution and titrant and repeat. If a CCV does not meet criteria, reanalyze once. If the failure repeats, re-standardize the iodine solution and titrant and repeat the analysis of all samples run after the last passing CCV.

9.5. Laboratory Control Sample (LCS): The LCS is a spiked blank matrix used to monitor the accuracy of the analytical process independent of possible interference effects due to sample matrix. The LCS associated with soil samples uses glass, plastic or Teflon beads to simulate a solid matrix. Laboratory Control Sample is processed through all preparation and analytical steps with the samples. Any treatment step performed on a sample must also be performed on the LCS.

The LCS spike is 1.0mL of the secondary spiking solution 7.12.1, and the true value is determined per section 9.3. Recovery must be within ± 15% for method 9034 and ± 10% for method SM 4500 S^{2-} F.

9.5.1. If the LCS does not meet recovery criteria, all associated samples must be redistilled and reanalyzed, unless one of the following conditions applies:

- If the LCS recovery is above the control limits, and all sample results are non-detect, results may be reported using the “LCS/LCSD - %R High” NCM.
- If re-distillation/reanalysis is not possible due to limited sample quantity, the laboratory project manager will be notified to inform the client. Results shall be reported flagged with a “*” qualifier and the NCM “LCS/LCSD – Insufficient Sample”.
- If reanalysis will be outside of holding time, the client must be notified and approval from the client must be obtained for the reanalysis.

9.6. One matrix spike and matrix spike duplicate pair is required on 1 per every 20 samples in the batch. The MS is performed on a second portion, and the MSD on a third, of a sample chosen at random in the batch, unless a client specifies a sample to be used. If there is insufficient sample to perform both an MS and MSD, an MS only may be analyzed.

9.6.1. Prepare the MS and MSD by spiking 1.0mL of the spiking solution (7.12.1) into the second and third sample portion prior to distillation (or analysis if no distillation step is required).

- If the MS fails recovery criteria of 75-125%, evaluate the other batch QC results. If the LCS is acceptable and the MS/MSD are comparable and outside of QC limits, then a matrix effect is indicated. No reanalysis is required, results are reported with the NCM “MS/MSD - %R, LCS Pass”.

9.6.2. The relative percent difference (RPD) between the MS and MSD must be within ± 20 percent. If this criteria is not met, report results using the “MS/MSD/DUP - %RPD” NCM.

10. PROCEDURE

10.1. Standardization

The working iodine solution and sodium thiosulfate titrant are standardized daily, and the information is recorded in the standardization log, which is attached to the TALS batch.

10.1.1. 0.025N Sodium thiosulfate (7.10) standardization

10.1.1.1. Place 2g KI in 250mL beaker and add 100mL Super-Q water and stir. Add 10mL biiodate (7.13.1) and then 10mL 1:4 H_2SO_4 (7.3.1).

10.1.1.2. Place in the dark for 5 minutes. Dilute to 150mL (if additional volume is need to see the color change) and add starch indicator (7.9) to turn the solution black or dark blue.

10.1.1.3. Titrate with $Na_2S_2O_3$ (section 7.10) to clear endpoint.

NOTE: Once the color changes from dark to light blue, the titration to a clear endpoint must be done slowly to avoid over titrating.

10.1.1.4. Repeat the procedure two more times and average the three results. The normality of the titrant is calculated as:

$$N\ Na_2S_2O_3 = \frac{10\ mL\ biiodate \times 0.025\ N\ biiodate}{mL\ Na_2S_2O_3\ titrant}$$

10.1.2. 0.025N Iodine (7.11) standardization:

10.1.2.1. Place 20mL 0.0250N iodine in Erlenmeyer flask and dilute to approximately 100mL with reagent water. Add 2mL of 6 N HCl (7.2.2) and several drops of starch indicator (7.9).

10.1.2.2. Titrate with $Na_2S_2O_3$ (7.10) to a clear endpoint.

NOTE: Once the color changes from dark to light blue, the titration to a clear endpoint must be done slowly to avoid over titrating.

10.1.2.3. Repeat the procedure two more times and average the three results. The normality of the iodine solution is calculated as:

$$N\ I = \frac{N\ Na_2S_2O_3 \times mL\ Na_2S_2O_3\ titrant}{mL\ Iodine\ solution}$$

(See the example Standardization worksheet in Figure 2.)

10.2. General Sample Prep

10.2.1. If the sample is aqueous, shake the sample container to suspend any solids, then quickly decant the appropriate volume (50mL) of the sample to a graduated cylinder and transfer to the distillation flask. Be sure that a representative aliquot is used, or use the entire sample.

10.2.2. If the sample is aqueous but contains soft clumps of solid, break the clumps and homogenize the sample by shaking the sample vigorously in its container. Then the mixture of sample may be aliquotted to produce a mixture that is completely homogenous.

10.2.3. If the sample contains solid objects the solids must be broken manually. Clay-like solids should be cut with a spatula or scalpel in a crystallizing dish. Non-porous harder objects, for example stones or pieces of metal, are excluded from the sample portion used for analysis unless otherwise instructed by the client.

10.3. Method 9030B/9034 – Acid-Soluble Sulfides Preparation and Analysis

10.3.1. A preliminary experiment must be done on one water and one solid sample from each job in the analytical batch. If there are multiple jobs from the same project, then one sample from each sampling site may be tested and the result applied to all the samples from the same site. This preliminary experiment determines the approximate amount of sulfuric acid required to adjust a measured amount of the sample to pH less than or equal to 1. Place 50mL of water or 10.0g of solid sample in a beaker. Add reagent water if the samples are solid until the total volume is 50mL. Stir the mixture, determine and record the initial pH. Slowly add concentrated sulfuric acid until the pH is less than or equal to 1. (pH may be checked with pH paper.) Record the final pH. Record the amount of acid needed to change the pH to $\leq$ 1. Discard this preliminary sample.

CAUTION: Toxic hydrogen sulfide may be generated from the acidified sample. This operation **must** be performed in a hood and the sample left in the hood until the sample has been made alkaline or the sulfide has been destroyed.

10.3.2. Place a sample aliquot in the boiling tube. The default maximum sample size is 50mL for water matrices and 5.0g (in 45mL deionized water) for solid and sediment matrices. Place the boiling tube containing the sample in the heater block and assemble the sulfide distillation apparatus as shown in Figure 1.

NOTE: It may be necessary to add antifoaming agent to known swelling or foaming samples to prevent carry-over into the collection vessels. When

antifoam is added to samples it MUST also be added to the Method Blank and LCS.

10.3.3. Place 2.5mL of Acid-Soluble Sulfide (Zinc Acetate) scrubber solution (7.6) + 25mL reagent water in each of two bubbler vessels.

10.3.4. Place an impinged bubbler in the first (front) vessel, a fritted bubbler in the second (back) vessel, and seal them with WestClips®. The sealed vessels and impingers function as the gas scrubbers. Connect the first scrubber to the inlet adapter and place the second bubbler vessel in the bubbler vessel rack. Connect the two impingers in series using Tygon® tubing.

10.3.5. Close stopcock of side arm funnel. **Place 10 mL plus the volume of sulfuric acid calculated in step 10.3.1 in the dropping funnel (amount of acid needed to lower pH to 1 <u>plus</u> an additional 10 mL of acid).**

For example, if the volume of sulfuric acid determined in section 10.3.1 is 3 mL, then the total volume of sulfuric acid added to the dropping funnel will be 13 mL.

10.3.6. Connect a high-purity (GC grade) nitrogen gas source to the main inlet of the gas manifold of the aluminum heater block as specified in the Heater Block Operation Manual. Use a two-stage gas tank regulator and set the pressure into the gas manifold to 20psi.

10.3.7. Connect a black gas line from each gas manifold valve to a “T” connector and a tygon gas line from the “T” to each of the two gas inlets of the apparatus. One at the top of the dropping funnel and one at the inlet adapter as shown in Figure 1.

10.3.8. Purge assembled apparatus with high-purity nitrogen for 10 minutes to remove atmospheric oxygen from the apparatus and contained solutions. During purge, adjust nitrogen flow such that approximately five bubbles per second exit the base of the inlet adapter. A proper flow rate is indicated by a steady stream at the fritted bubbler.

10.3.9. Heat the distillation apparatus to 70^{O} C. Measure and Record the temperature of the hotblock during distillation in TALS LIMS by using a digital K Type thermometer or equivalent. Rotate where the thermometer is placed in the hotblock for each digestion. Set heater block temperature controls to the following settings:

RATE1: 15^{O} C/MIN RATE2: 0
TEMP1: 70^{O} C TEMP2: 70^{O} C
TIME1: >1.5HOUR TIME2: 0
TIMING CONFIGURATION: CONF1

(Set Time 1 to a period longer than 1.5 hours to prevent the second temperature program from immediately starting.)

10.3.10. Open stopcock of dropping funnel and allow all of the sulfuric acid to drip into the boiling tube at a rate of approximately 1mL/min. Continue to observe the boiling tube contents as acid is added to monitor for excessive foaming, swelling of solids, or formation of a non-homogeneous mixture that is not suspended by the bubbling action in the tube. It may be necessary to use a smaller sample aliquot under such conditions. Once dropping funnel is empty, close the stopcock quickly to ensure sulfide evolved from the sample is not lost into the funnel.

10.3.11. Start the heating cycle AFTER the acid has been added. Samples are heated for 90 minutes once the set temperature is reached.

10.3.12. After the 90 minute heating period, discontinue heating. Remove bubbler vessels. Turn off the nitrogen flow. If possible, leave the set-up sealed until ready to analyze the distillate. If not possible, remove the collection vessels carefully so as not to disturb the settled solids, seal and label separately. Store in an area where they will not be disturbed until analysis.

10.3.13. Carefully combine the gas scrubber solutions just prior to analysis by following the steps below. Do not shake the collection solutions to avoid exposure to air and subsequent loss of sulfide. Determine the concentration of acid-soluble sulfide in the zinc acetate gas scrubber solutions by using the Titrimetric-iodine method (9034) following the steps below.

10.3.14. Pipette 10 mL of standardized 0.025N iodine solution (7.11) and 2mL of 6N HCl (7.2.2) into an Erlenmeyer flask. Add approximately 30mL reagent water.

10.3.15. Pour the collection solution from the first scrubber into the Erlenmeyer flask containing the iodine solution by using a funnel with its tip placed below the iodine solution. Pour the solution from the second scrubber into the first scrubber vessel to rinse it and add it to the Erlenmeyer flask in the same manner.

10.3.16. Pipet another 5mL of 0.025N iodine solution (7.11) and 2mL of 6N HCl (7.2.2) into the second scrubber vessel. Rinse the second scrubber and impinger with the acidified iodine solution, to collect any remaining precipitate. Pour the

acidified iodine into the first scrubber and rinse the vessel and impinger. Add this to the analysis flask. Rinse any remaining iodine solution from both scrubbers with a small amount of reagent water and add this to the analysis flask. There should be no visible traces of precipitate after rinsing. Bring the final volume in the Erlenmeyer flask to approximately 100mL with reagent water.

10.3.17. If at any point in transferring the zinc acetate solution or rinsing the bottles, the amber color of the iodine disappears or fades to yellow, additional 0.025N iodine, in 5mL increments, must be added until the dark iodine color persists. This additional volume must be added to the amount from Section 10.3.14 and 10.3.16 for calculations. Record the total volume of standardized 0.025N iodine solution used.

10.3.18. Add several drops of starch indicator to turn the solution in the analysis flask black or dark blue.

10.3.19. Titrate the solution in the flask with standard 0.025N sodium thiosulfate solution (7.10). The sample will turn from black or dark blue to light blue. Continue titrating until the blue color disappears completely. Titrate the solution slowly as the color lightens to avoid overshooting the endpoint. Record the volume of titrant used, then double check that the titrated sample color does not change further by adding one more drop of sodium thiosulfate titrant. Record the first volume of titrant if the color does not change. Record the final titrant volume if it does.

10.4. Method 9034/9030B Acid Insoluble Sulfide (AIS) Preparation and Analysis

10.4.1. As the concentration of HCl during distillation must be within a narrow range for successful distillation of H_2S, the water content must be controlled. It is imperative that the final concentration of HCl in the distillation flask be about 6.5N and that the sample is mostly suspended in the fluid by the action of swirling the sample boiling tube. This is achieved by adding 10mL of reagent water, including water in the sample, 20 mL of 9.8N HCl (7.2.1), and the sample to the boiling tube. Solids which absorb water and swell will restrict fluid motion and, therefore, lower recovery will be obtained. Samples should be limited to 5 g dry weight.

10.4.2. Add 1 gram of stannous chloride (7.14) and then place the boiling tube containing the sample in the heater block and assemble the sulfide distillation apparatus as shown in **Figure 1.**

10.4.3. Place 25mL of the sodium acetate/zinc acetate buffer (7.7) in each of two bubbler vessels. Place an impinged bubbler (front) in the first vessel, a fritted

bubbler (back) in the second vessel, and seal them with size 24/40 WestClips®. The sealed vessels and impingers function as the gas scrubbers. Connect the first scrubber to the inlet adapter and place the second bubbler vessel in the bubbler vessel rack. Connect the two impingers in series using Tygon® tubing.

10.4.4. Close stopcock of side arm funnel, then add 20 mL of 9.8 N HCl (7.2.1).

10.4.5. Connect a high-purity (GC grade) nitrogen gas source to the main inlet of the gas manifold of the aluminum heater block as specified in the Heater Block Operation Manual. Use a two-stage gas tank regulator and set the pressure into the gas manifold to 20psi.

10.4.6. Connect a black gas line from each gas manifold valve to a “T” connector and a tygon gas line from the “T” to each of the two gas inlets of the apparatus. One at the top of the dropping funnel and one at the inlet adapter as shown in Figure 1.

10.4.7. Purge assembled apparatus with high-purity nitrogen for 10 minutes to remove atmospheric oxygen from the apparatus and contained solutions. During purge, adjust nitrogen flow such that approximately five bubbles per second exit the base of the inlet adapter. A proper flow rate is indicated by a vigorous foaming action at the fritted bubbler.

10.4.8. Heat the distillation apparatus to 100℃. Measure and Record the temperature of the hotblock during distillation in TALS LIMS by using a digital K Type thermometer or equivalent. Rotate where the thermometer is placed in the hotblock for each digestion. Set heater block temperature controls to the following settings:

RATE1: 15℃/MIN	RATE2: 0
TEMP1: 100℃	TEMP2: 100℃
TIME1: 1.5 HOUR	TIME2: 0
TIMING CONFIGURATION: CONF1	

10.4.9. Open stopcock of dropping funnel and allow all of the hydrochloric acid to drip into the boiling tube at a rate of approximately 1mL/min. Continue to observe the boiling tube contents as acid is added to monitor for excessive foaming, swelling of solids, or formation of a non-homogeneous mixture that is not suspended by the bubbling action in the tube. It may be necessary to use a smaller sample aliquot under such conditions. Once dropping funnel is empty, close the stopcock to ensure sample is not lost into the funnel. It may also be necessary to add antifoam to known swelling solids and waters.

10.4.10. After the 90 minute heating period, discontinue heating. Remove bubbler vessels. Turn off the nitrogen flow. If possible, leave the set-up sealed until ready to analyze the distillate. If not possible, remove the collection vessels carefully, seal and label separately.

10.4.11. Determine the concentration of acid-soluble sulfide in the sodium acetate/zinc acetate gas scrubber solutions following steps 10.3.13 through 10.3.19 above, using 10mL of 6N HCl (7.2.2) in step 10.3.14 rather than 2mL.

10.5. Method SM 4500-S^{2-}F

10.5.1. Add 10 mL of a 0.025N iodine solution (7.11) into a 125mL Erlenmeyer flask or beaker. The iodine solution is bright orange/brown in color.

10.5.2. Add approximately 2ml of 6N HCl (7.2.2).

10.5.3. Pipette 50 mL of sample into the flask, keeping the tip of the pipette below the surface of the sample. (If necessary, add reagent water until there is sufficient solution in the flask to see the color change at the end of the titration.) Add several drops of starch indicator to turn the solution black/dark blue.

NOTE: When preparing a matrix spike, spike the sample portion first, then transfer it to the flask containing the acidified iodine solution.

10.5.4. If the iodine color disappears, re-prepare the sample with additional iodine solution in 10.5.1. If re-preparation is not possible due to limit sample volume, add more iodine solution in 10mL increments until the color remains. Record the total volume of standard iodine added.

10.5.5. Back-titrate with 0.0250 N sodium thiosulfate (7.10), Titrate slowly as the blue color fades so as not to overshoot the end point. The endpoint is reached when the blue color disappears completely. Record the volume of titrant used.

11 CALCULATIONS / DATA REDUCTION

11.1. Calculate the amount of Sulfide in the sample:

$$\text{Sulfide (mg/L)} = \frac{[(A \times B) - (C \times D)] \times 16000}{\text{mL of sample}}$$

Where:

A = mL of iodine solution

B = N of iodine solution

C = mL of $Na_2S_2O_3$ solution

D = N of $Na_2S_2O_3$ solution

11.2. Un-ionized Hydrogen Sulfide as S^{2-} (mg/L) = J x Conc. of dissolved sulfide (mg/L)

Where:

J - the proportion of dissolved sulfide present as H_2S as determined by sample pH – pK' from Figure 3

pK' - the logarithm of the practical ionization constant for hydrogen sulfide at a given conductivity and temperature from Figure 4.

11.3. Duplicate sample (Relative Percent Difference):

%RPD = 100 x | A - B | / [(A+B)/2]

Where:

A - Original sample concentration

B - Duplicate sample concentration

11.4. Spike percent recovery:

$$Theoretical\ Spike\ Conc. = \frac{\left(\begin{matrix}Spike\\Conc.\end{matrix}\right) \times \left(\begin{matrix}Vol.\ of\\Spike\ Added\end{matrix}\right)}{Final\ Vol.\ Spiked}$$

$$\%\ \mathrm{Recovery} = \frac{(Spiked\ Sample\ Conc.) - Sample\ \mathrm{Result}}{Theoretical\ Spike\ Conc.} \times 100$$

12. METHOD PERFORMANCE

12.1. The supervisor has responsibility to ensure that an analyst who performs this procedure is properly trained in its use and has the required experience. Performance is monitored through internal QC and outside performance evaluation samples. Please refer to the QA Manual for additional information concerning Precision and Accuracy.

12.2. Demonstration of Capabilities – Prior to the analysis of samples, a Demonstration of Capabilities (DOC) as described in the QA Manual, must be performed initially, annually and any time a significant change is made to the analytical system.

12.3. Method Detection Limit (MDL) for Titrimetric Procedures - The detection limit for titrimetric procedures can be defined by the smallest increment of titrant that can be measured during a titration to cause a chemical change. The detection limit is calculated based on the titrant concentration, the sample size, and the minimum volume of titrant that can be measured. Method Blank performance may be used to evaluate the validity of the quantitation limit.

13. POLLUTION CONTROL

13.1. It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage and reagent stability). Employees must abide by the policies in Section 13 of the Corporate Environmental Health and Safety Manual (CW-E-M-001) for "Waste Management and Pollution Prevention."

14. WASTE MANAGEMENT

14.1. Waste management practices are conducted consistent with all applicable rules and regulations. Excess reagents, samples and method process wastes are disposed of in an accepted manner. Waste description rules and land disposal restrictions are followed. Waste disposal procedures are found in the Pittsburgh Facility Addendum EH&S Manual (PT-HS-001).

14.2. The following waste streams are produced when this method is carried out:

- Acidic waste generated by sample titration - This waste is collected in a waste container identified as "Acid Waste", Waste #33. This waste is neutralized to a final pH between 6 and 9 and discharged down into a lab sink.
- Unused sample distillate - This waste is collected in a waste container identified as "Acid Waste", Waste #33. This waste is neutralized to a final pH between 6 and 9 and discharged down into a lab sink.

15. REFERENCES

15.1. SW-846 Method 9030B, Acid-Soluble and Acid-Insoluble Sulfides: Distillation; Revision 2, December1996

15.2. SW-846 Method 9034, Titrimetric Procedure for Acid-Soluble And Acid Insoluble Sulfides; Revision 0, December 1996

15.3. Standard Methods for the Examination of Water and Waste Water, Method 4500-S^{2-} H, Calculation of Un-ionized Hydrogen Sulfide, 2000, 2011

15.4. Standard Methods for the Examination of Water and Wastewater, Method 4500-S^{2-} C and F, Pretreatment and Iodometric Method, 2000, 2011

15.5. PT-QA-001, Employee Orientation and Training

15.6. PT-QA-006, Procurement of Standards and Materials; Labeling and Traceability

15.7. PT-QA-012, Selection and Calibration of Balances and Weights

15.8. PT-QA-016, Nonconformance & Corrective Action System

15.9. PT-QA-021, Quality Assurance Program

15.10. PT-QA-024, Subsampling

15.11. PT-QA-031, Internal Chain of Custody

15.12. PT-QA-M-001, Pittsburgh Laboratory Quality Assurance Manual

15.13. PT-HS-001, Pittsburgh Facility Addendum EH&S Manual

15.14. CW-E-M-001, TestAmerica Corporate Environmental Health & Safety Manual

16. METHOD MODIFICATIONS

16.1. Method 9030B, section 7.1.2.3 requires 37% formaldehyde solution to be added to the collection scrubber solutions as a treatment to prevent interference from sulfite or hydrosulfite in the solid sample. The addition of formaldehyde interferes with and slows the response of the reaction during the titration analysis significantly, leading to over-titration of distilled samples. Per section 3.2 of method 9030B, the sulfite/hydrosulfite does not interfere at concentrations <10mg/kg. For this reason, the formaldehyde solution has been removed from this procedure.

17. ATTACHMENTS

17.1. Figure 1 – Sulfide distillation apparatus

17.2. Figure 2 – Example Sulfide Standardization Logsheet

17.3. Figure 3 - Proportions of H_2S and HS^- in dissolved sulfide (SM18 Figure 4500-S^{2-}:2.)

17.4. Figure 4 – Values of pK', logarithm of practical ionization constant for hydrogen sulfide (SM18 Table 4500-S^{2-}:I.)

17.5. Figure 5 - Example Sulfide TALS Worksheet (9034)

17.6. Figure 5A – Example Sulfide TALS Worksheet (SM4500 S^{2-} F)

17.7. Figure 6 - Example Distillation Sheet

18. REVISION HISTORY

18.1. Revision 7, 5/30/2008

18.2. Revision 8, 7/14/2008

18.3. Revision 9, 10/13/2009
18.4. Revision 10, 4/8/2010
18.5. Revision 11, 10/3/2010
18.6. Revision 12, 12/16/2011
18.7. Revision 13, 10/15/2012
18.8. Revision 14, 2/18/2014
18.9. Revision 15, 7/8/2015
18.10. Revision 16, 11/28/2017
18.11. Revision 16.1, 5/13/2019
18.12. Revision 17, 3/2/2020
18.13. Revision 18, 4/9/2020
18.14. Changes to current revision

SOP section	Change from	Change to	Reason
Title and 15.4		Added reference to SM4500 S2-C	Correction for SC audit finding
1.1	Removed statement that water samples for analysis by method SM4500 S^{2-} F do not require distillation		Correction for SC audit finding
2.3		Added 9030B distillation to remove interferences	Correction for SC audit finding
2.4		Added SM4500 S2-C to remove interferences	Correction for SC audit finding

Figure 1 – Sulfide distillation apparatus

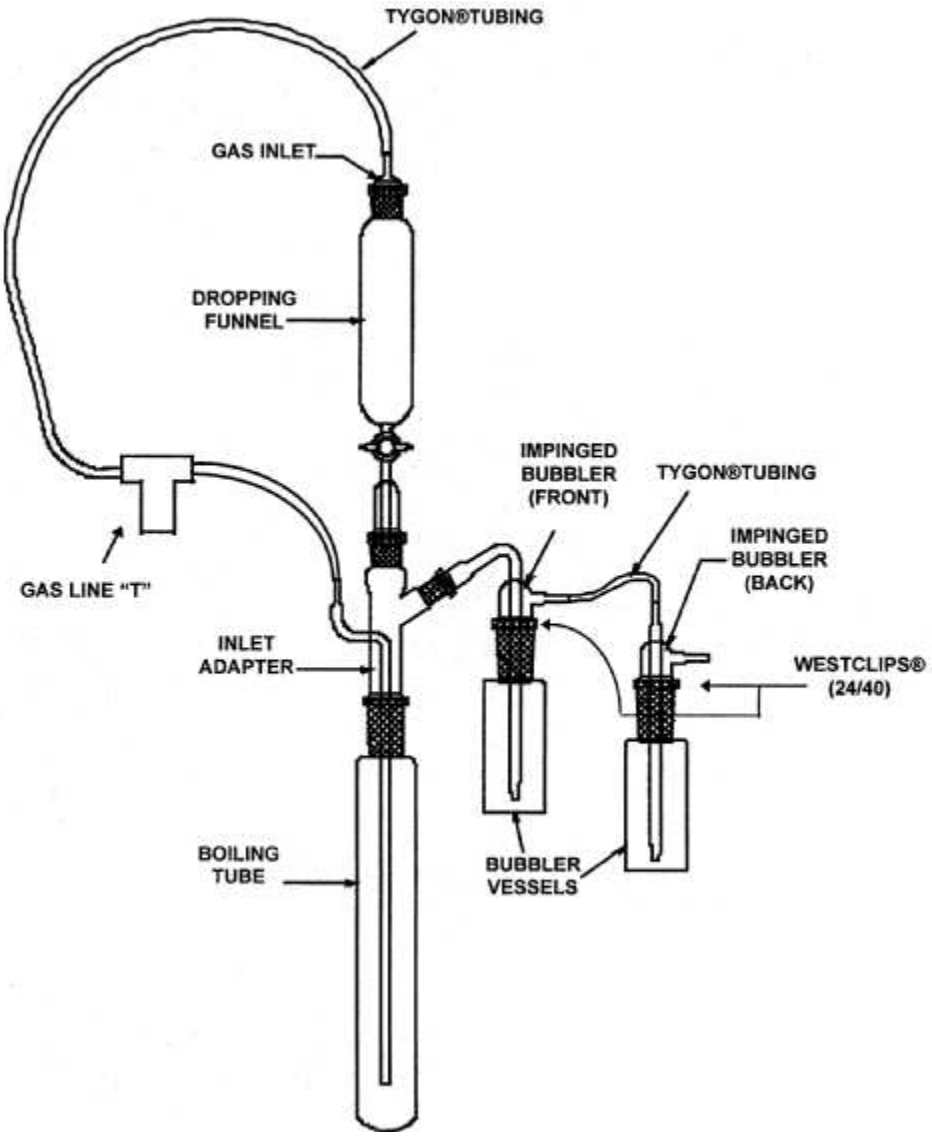


Figure 2 – Example Sulfide Standardization Logsheet

TestAmerica
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Controlled Document
Pittsburgh
Worksheet No. PT-WC-WS-011 R3
Effective Date: 9/14/2017
page 1 of 1

Sulfide Analysis Standardization Logsheet

Analyst: **Date:**

Standardization of Sodium Thiosulfate Titrant (A)			Reagent #:	
Titrate 10mL of 0.025N Potassium Biiodate to clear endpoint				
$KH(IO_3)_2$ Lot #:		**KI Lot #:**		**1:4 H_2SO_4:**
Trial 1 mL:		**Trail 2 mL:**		**Trial 3 mL:**
Average mL (X):	**0.00**	**Normality of Titrant:**	**#DIV/0!**	
N of $Na_2S_2O_3$ = (10mL $KH(IO_3)_2$)(0.025N) / XmL $Na_2S_2O_3$				

Standardization of Iodine Solution (B)			Reagent #:	
Titrate 20mL of Iodine solution to clear endpoint using the sodium thiosulfate titrant standardized above				
Trial 1 mL:		**Trail 2 mL:**		**Trial 3 mL:**
Average mL (Y):	**0.00**	**Normality of Iodine:**	**#DIV/0!**	
N of Iodine = (Y mL $Na_2S_2O_3$)(N of $Na_2S_2O_3$) / 20mL Iodine				

Sulfide Spike Concentration Determination			
Titrate 1.0mL of Standard and 10mL Iodine Solution to a clear endpoint using the standardized sodium thiosulfate titrant			
Primary Standard ID:		**Secondary Standard ID:**	
mL Titrant to endpoint:		mL Titrant to endpoint:	
Primary Stock Conc. (mg/L):	#DIV/0!	Second Stock Conc. (mg/L):	#DIV/0!

Std Conc. (mg/L) = {[(mL B*N B) - (mL A * N A)] x 16000}/mL Std
A is Sodium Thiosulfate titrant, B is Iodine Solution

TALS Reagent weight (g): Analyzed Conc / 534	
Primary Std: #DIV/0!	Secondary Std: #DIV/0!

Figure 3 – Proportions of H_2S and HS^- in dissolved sulfide (SM18 Figure 4500-S^{2-}: 2)

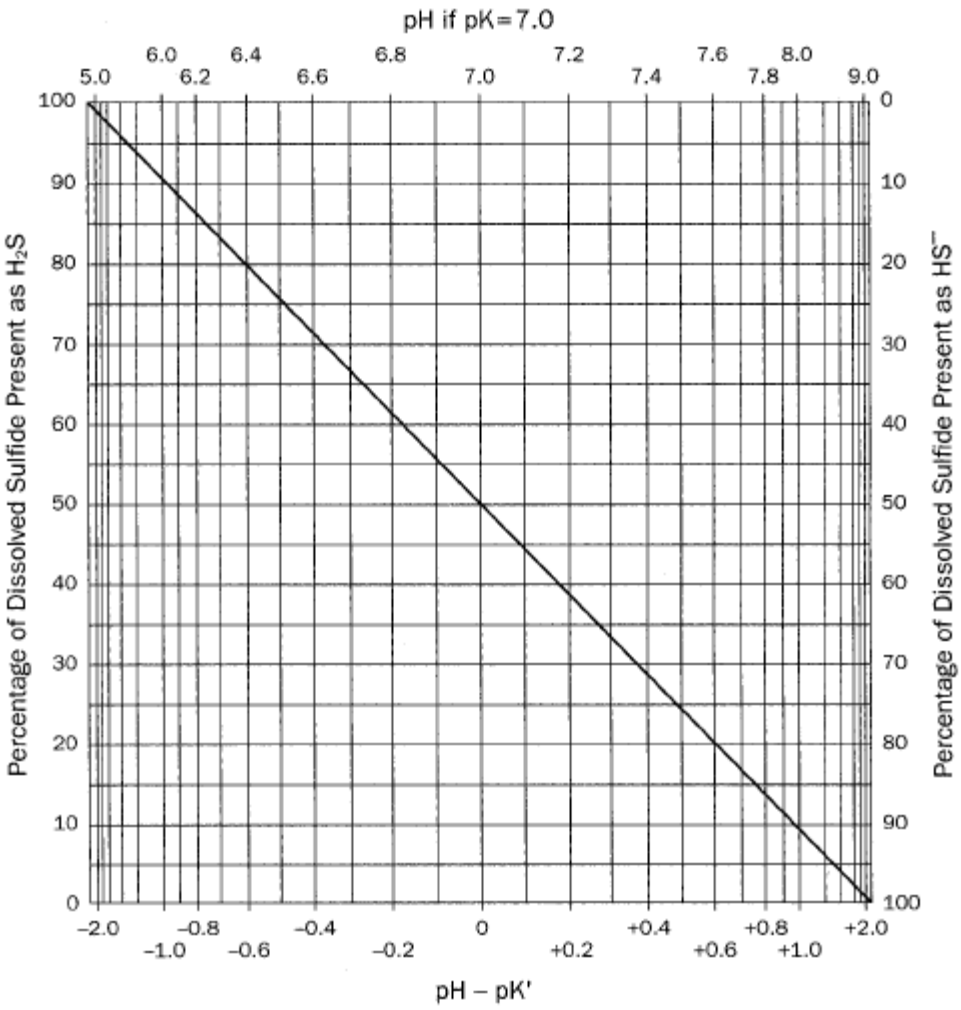


Figure 4 – Values of pK', logarithm of practical ionization constant for hydrogen sulfide (SM18 Table 4500-S^{2-}: I)

Conductivity at 25℃ µmhos/cm	PK' at Given Temperature		
	20℃	25℃	30℃
0	---	7.03*	---
100	7.08	7.01	6.94
200	7.07	7.00	6.93
400	7.06	6.99	6.92
700	7.05	6.98	6.91
1 200	7.04	6.97	6.90
2 000	7.03	6.96	6.89
3 000	7.02	6.95	6.88
4 000	7.01	6.94	6.87
5 200	7.00	6.93	6.86
7 200	6.99	6.92	6.85
10 000	6.98	6.91	6.84
14 000	6.97	6.90	6.83
22 000	6.96	6.89	6.82
50 000	6.95	6.88	6.81

*Theoretical

Figure 5 – Example Sulfide TALS Worksheet (9034)

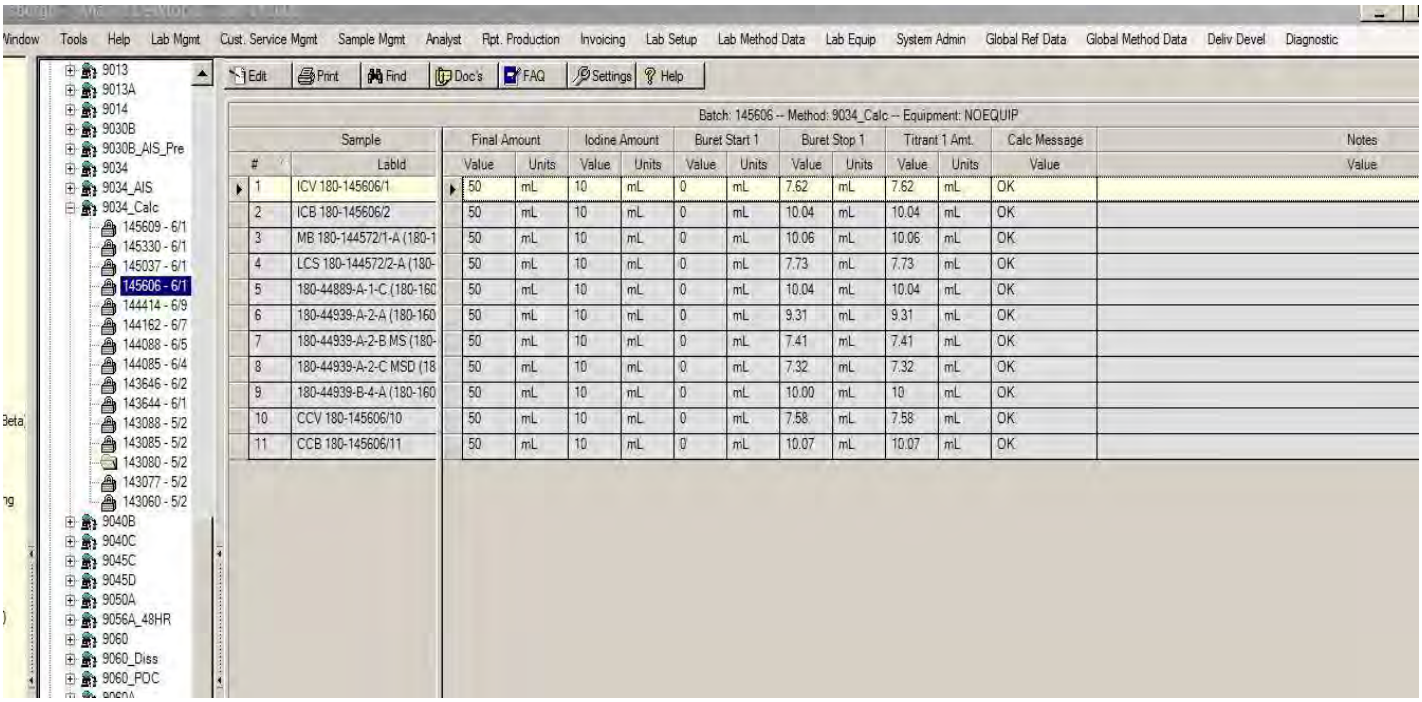


Figure 5A – Example Sulfide TALS Worksheet (SM 4500-S^{2-} F)



Edit | Print | Find | Doc's | FAQ | Settings | Help

Batch: 217061 -- Method: SM4500_S2_F -- Equipment: NOEQUIP

Sample		Buret Start 1		Buret Stop 1		Iodine Amount		Titrant 1 Amt.		Initial Amount		Final Amount		Calc Message
#	LabId	Value	Units	Value	Units	Value	Units	Value	Units	Value	Units	Value	Units	Value
1	ICV 180-217061/1	0.00	mL	7.23	mL	10.00	mL	7.23	mL	50	mL	200	mL	OK
2	ICB 180-217061/2	0.00	mL	9.93	mL	10.00	mL	9.93	mL	50	mL	200	mL	OK
3	LCS 180-217061/3	0.00	mL	7.32	mL	10.00	mL	7.32	mL	50	mL	200	mL	OK
4	MB 180-217061/4	0.00	mL	9.57	mL	10.00	mL	9.57	mL	50	mL	200	mL	OK
5	180-68035-O-1 (180-2417	0.00	mL	9.65	mL	10.00	mL	9.65	mL	50	mL	200	mL	OK
6	180-68035-O-1 MS (180-2	0.00	mL	7.09	mL	10.00	mL	7.09	mL	50	mL	200	mL	OK
7	180-68035-O-1 MSD (180-	0.00	mL	7.20	mL	10.00	mL	7.2	mL	50	mL	200	mL	OK
8	180-68035-O-2 (180-2417	0.00	mL	9.87	mL	10.00	mL	9.87	mL	50	mL	200	mL	OK
9	180-68061-J-1 (180-24185	0.00	mL	9.76	mL	10.00	mL	9.76	mL	50	mL	200	mL	OK
10	CCV 180-217061/10	0.00	mL	7.21	mL	10.00	mL	7.21	mL	50	mL	200	mL	OK
11	CCB 180-217061/11	0.00	mL	9.76	mL	10.00	mL	9.76	mL	50	mL	200	mL	OK

Figure 6- Example Sulfide Distillation Sheet

Metals/Inorganics Prep Worksheet

(Used for Collecting Prep Info)

Batch Number: 180-15839 Analyst: Gaydas, Jared Batch Open: 9/29/2011 7:55:19PM

Method Code: 180-9034_Calc-180 Batch End:

Sulfide, Acid soluble and Insoluble (Titrimetric)

	Input Sample Lab ID	Input Sample Lab ID (Analytical Method)	SDG	Matrix	Initial Amount	Final Amount	Due Date	Analytical TAT	Div Rank	Comments
1		ICV~180-15839/1 N/A	N/A			50 mL	N/A	N/A	N/A	
2		ICB~180-15839/2 N/A	N/A			50 mL	N/A	N/A	N/A	
3		MB~180-15834/1-A N/A	N/A			50 mL	N/A	N/A	N/A	
4		LCS~180-15834/2-A N/A	N/A			50 mL	N/A	N/A	N/A	
5		180-4212-F-1-A (9034_Calc)	N/A	Distillate		50 mL	10/5/11	8_Days - E	N/A	
6		180-4212-F-2-A (9034_Calc)	N/A	Distillate		50 mL	10/5/11	8_Days - E	N/A	
7		180-4212-F-3-A (9034_Calc)	N/A	Distillate		50 mL	10/5/11	8_Days - E	N/A	
8		180-4212-F-4-A (9034_Calc)	N/A	Distillate		50 mL	10/5/11	8_Days - E	N/A	
9		180-4447-F-1-A (9034_Calc)	N/A	Distillate		50 mL	10/12/11	8_Days - R	N/A	
10		180-4447-F-3-A (9034_Calc)	N/A	Distillate		50 mL	10/12/11	8_Days - R	N/A	
11		180-4268-G-1-A (9034_Calc)	N/A	Distillate		50 mL	10/12/11	19_Days - R	N/A	
12		180-4268-G-2-A (9034_Calc)	N/A	Distillate		50 mL	10/5/11	8_Days - E	N/A	
12		180-4268-G-2-A (9034_Calc)	N/A	Distillate		50 mL	10/5/11	8_Days - E	N/A	
12		180-4268-G-2-A (9034_Calc)	N/A	Distillate		50 mL	10/5/11	8_Days - E	N/A	
12		180-4268-G-2-A (9034_Calc)	N/A	Distillate		50 mL	10/5/11	8_Days - E	N/A	

Printed : 10/4/2011 Page 1 of 5 TestAmerica Pittsburgh

SOP # with Revision: PT-WC-017 Rev. 17
SOP Title: Total Organic Carbon (TOC) and Total Inorganic Carbon (TIC)
Amendment Number: 1

Effective Date of Amendment:
Append this form to the front of the SOP copy.

CONTROLLED DISTRIBUTION	
COPY # :	**001**
ISSUED TO :	**Pittsburgh SOP Directory**

The following SOP change is in effect as of the stated date. *This form will remain attached to the referenced SOP until such a time that the SOP is updated, approved, and redistributed, at which time it will become part of the historical SOP record.*

1. Briefly explain the reason for the update: 40 CFR Part 136 Update of Reference Year

2. Category for SOP Change:

☒ Typographical Corrections (Non-Technical) – Retraining Not Required.

☐ Typographical Corrections (Technical- define below) – Retraining is required.

☐ Procedural Changes (Define Below) – Retraining Required.

3. Summary of Procedure Change (circle to indicate if there are attachments to this form: No / Yes: # pages attached = ___)

15.2 Standard Methods for the Examination of Water and Wastewater, Method 5310C, 2000, editorial revision 2011, 2014

Does this change result in a Method Modification? Yes (No)
If yes, please explain below.

Any method modification will be incorporated into section 16 of the SOP at the next revision.

8/13/21
8-16-21

Approval Signature/Date: Technical

Amanda L Grelli 08/12/21

Approval Signature/Date: QA Manager or Designee

Title: Total Organic Carbon (TOC) and Total Inorganic Carbon (TIC)

Method(s): Standard Methods 5310C and SW-846 9060A

Approvals (Signature/Date):

1/23/2020	1/20/2020
Roseann Ruyechan Date	Steve Jackson Date
Inorganics Department Manager	Regional Safety Coordinator
1/17/2020	2/6/2020
Virginia Zusman Date	Deborah L. Lowe Date
Quality Assurance Manager	Laboratory Director

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1. SCOPE AND APPLICATION

1.1 The purpose of this SOP is to provide a procedure for the determination of Total Organic Carbon (TOC) or Total Inorganic Carbon (TIC) in waters and similar matrices. This SOP is based on SW-846 Method 9060A and Standard Methods 5310C.

1.2 The working linear range is 1 mg/L to 100 mg/L on the Aurora 1030 instrument. For soils the sample must undergo a leaching process prior to analysis, such as ASTM Leach. Sediment samples undergo elutriate generation prior to analysis. The reporting limit is 1 mg/L.

1.3 On occasion clients may request slight modifications to this SOP. Any modifications to this procedure must be approved by the QA department or the Laboratory or Technical Director and documented fully in an NCM that accompanies the analytical batch.

2. SUMMARY OF METHOD

2.1 Organic carbon is converted to carbon dioxide (CO_2) using chemical oxidation. The CO_2 is then measured by an infrared detector. The amount of CO_2 in a sample is directly proportional to the concentration of carbonaceous material in the sample.

3. DEFINITIONS

3.1 TALS – TestAmerica Laboratory Information Management System

3.2 NCM – Non-Conformance Memo – a system within TALS for the lab to communicate to project management and others when there is an anomaly seen with the samples or batch, or a QC failure.

3.3 Refer to the glossary in the Pittsburgh Laboratory Quality Assurance Manual, PT-QA-M-001, for other common laboratory terms.

4. INTERFERENCES

4.1 Contaminants in solvents, reagents, glassware, and other processing apparatus that lead to discrete artifacts may cause method interferences. All of these materials must be routinely demonstrated to be free from interferences under conditions of the analysis by running laboratory method blanks as described in the Quality Control section. Specific selection of reagents may be required to avoid introduction of contaminants.

4.2 Carbonate and bicarbonate interfere but are eliminated by the acidification and purging step of the instrument.

5. SAFETY

5.1 Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual (CW-E-M-001), the Pittsburgh Facility Addendum EH&S Manual (PT-HS-001) and this document. This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

5.2 The auto sampler has a probe that is sharp; use caution not to stick yourself.

5.3 The Sodium Persulfate is a strong oxidizer. Avoid contact with combustible materials, organic materials, strong reducing agents, and excess heat.

5.4 The following is a list of the materials used in this method, which have a serious or significant hazard rating. NOTE: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the SDS for each of the materials listed in the table. A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the SDS for each material before using it for the first time or when there are major changes to the SDS.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Sulfuric Acid	Corrosive Oxidizer Dehydra-dator	1 mg/m^3	This material will cause burns if comes into contact with the skin or eyes. Inhalation of vapors will cause irritation of the nasal and respiratory system.
Phosphoric Acid	Corrosive	3 mg/M^3 PEL 1ppm STEL	Inhalation is not an expected hazard unless misted or heated to high temperatures. May cause redness, pain, and severe skin burns. May cause redness, pain, blurred vision, eye burns, and permanent eye damage.
Sodium Persulfate	Oxidizer Corrosive	0.1 mg/M^3-TWA as Persulfates	Causes irritation to the respiratory tract. Symptoms may include sore throat, shortness of breath, inflammation of nasal passages, coughing, and wheezing. Causes severe irritation or burns to the skin and eyes. Symptoms include redness, itching, pain and burns. May cause allergic skin reactions. Can cause eye damage.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

5.5 Eye protection that protects against splash, laboratory coat, and appropriate gloves must be worn while samples, standards, solvents, and reagents are being handled. Cut resistant gloves must be worn doing any other task that presents a strong possibility of getting cut. Disposable gloves that have become contaminated will be removed and discarded; other gloves will be cleaned immediately.

5.6 Exposure to chemicals must be maintained as low as reasonably achievable, therefore, unless they are known to be non-hazardous, all samples must be opened, transferred, and prepared in a fume hood or under other means of mechanical ventilation. Solvent and waste containers will be kept closed unless transfers are being made.

5.7 The preparation of standards and reagents will be conducted in a fume hood with the sash closed as far as the operation will permit.

5.8 All work must be stopped in the event of a known or potential compromise to the health and safety of a TestAmerica associate. The situation must be reported immediately to a laboratory supervisor or EH&S coordinator.

6. EQUIPMENT AND SUPPLIES

The following items are recommended for performing this procedure. Equivalent items should only be used when they result in an improvement in quality, efficiency, productivity, or cost. An item can be considered equivalent if with its use, the analytical and QA/QC requirements in this SOP can be met.

6.1 OI Corporation Model 1030 TOC Analyzer, with 1088 vial multisampler
6.2 Nitrogen gas and regulator
6.3 Class A Volumetric flasks, various sizes
6.4 Class A Volumetric pipettes, various sizes
6.5 Vials: 40mL Amber glass with stir bars
6.6 Class A Graduated cylinders, various sizes
6.7 pH strips, wide range
6.8 Top loading balance capable of weighing to ± 0.01g
6.9 Automatic Re-pipetter

7. REAGENTS AND STANDARDS

The following items are recommended for performing this procedure. Equivalent items should only be used when they result in an improvement in quality, efficiency, productivity, or cost. An item can be considered equivalent if with its use, the analytical and QA/QC requirements in this SOP can be met. Please refer to the SDS prior to the use of any reagent or standard.

Reagent grade chemicals must be used, unless otherwise indicated. If a certificate of analysis is not available, or the purity of a reagent or standard is questionable, analyze the reagent/standard

prior to using for the analysis of any client samples. Document the purity check in the TALS Reagent module with the entry for the reagent/standard.

7.1 Sodium Persulfate

7.1.1 Sodium Persulfate Solution: Add 100g sodium persulfate ($Na_2S_2O_8$) to a 1L volumetric flask and dilute to 1L final volume with reagent water. This solution is given a 6 month expiration date.

7.2 Phosphoric Acid, concentrated

7.2.1 Phosphoric Acid Solution: Carefully add 59mL concentrated phosphoric acid (H_3PO_4) to 900mL of reagent water in a 1L volumetric flask. Dilute to 1L final volume with reagent water. This solution is given a 6 month expiration date.

7.3 Sulfuric Acid, concentrated

7.4 TOC Primary Stock Standard, 1000ppm: This is a commercially purchased, certified standard. Follow the manufacturer's expiration date and storage requirements.

7.5 TOC Secondary Stock Standard, 1000ppm: This certified standard is commercially purchased from a different source than the primary stock. The manufacturer's expiration date and storage requirements are used.

7.5.1 If necessary, a 1000ppm TOC stock can be prepared as follows: Dilute 1.06g KHP (potassium acid phthalate) to volume in a 500mL volumetric flask. KHP is oven dried overnight at 180°C and stored in a dessicator for at least an hour before use.

7.6 TOC Calibration Standards: Prepare the calibration standards listed below from the primary stock standard (7.4). Prepare standards fresh daily for calibration use.

Concentration (mg/L)	Stock Volume (mL)	Stock Concentration (mg/L)	Final Volume (mL)
100	10	1000	100
50	5	1000	100
25	2.5	1000	100
10	1	1000	100
1	0.1	1000	100

7.7 Initial Calibration Verification (ICV) – This is a second source standard analyzed as part of initial calibration verification. A 40 mg/L standard is prepared by transferring 4 ml of the secondary stock (7.5) to a 100 ml volumetric flask and diluting to volume with reagent grade water. Prepare this standard fresh daily.

7.8 Continuing Calibration Verification (CCV): Prepare a 10mg/L verification standard by adding 2.0mL of the secondary stock standard (7.5) to a 200mL volumetric flask and diluting to volume. Prepare this standard fresh daily.

7.9 Laboratory Control Sample (LCS): Prepare a 20 mg/L standard by adding 4 mL of the primary stock standard (7.4) to a 200 ml volumetric flask and diluting to volume with reagent grade water. Prepare this standard fresh daily.

7.10 Matrix Spike/Matrix Spike Duplicate: Add 400µL of the secondary stock standard (7.5) to 40 mL of sample for a final concentration of 10 mg/L. (Spike concentration may be varied as appropriate to the native sample concentrations expected.)

7.11 TIC stock standard, 1000 mg/L: This is purchased commercially from the same vendor with different lot numbers for the primary and secondary source standards. If necessary it may also be prepared as follows: Prepare by adding 3.5g Na_2HCO_3 (sodium bicarbonate) to a 1L flask. Dilute to volume with reagent water. Keep tightly stoppered. Do not acidify.

7.11.1 TIC Calibration Standards: Prepare in the same manner as the TOC calibration standards (7.6) using the TIC primary stock standard (7.11). Prepare standards fresh daily as needed.

7.11.2 TIC Verification Standard (CCV): Prepare in the same manner as the TOC verification standard (7.8) using the TIC secondary stock standard (7.11). Prepare standard fresh daily.

8. SAMPLE COLLECTION, PRESERVATION, SHIPMENT AND STORAGE

Listed below are the holding times and the references that include preservation requirements.

Matrix	Sample Container	Minimum Sample Size	Preservation	Holding Time
Water	Amber Glass VOA Vial	40 mL	pH < 2 with Sulfuric acid; Cool >0.0˚C but ≤ 6.0°C	28 days from collection

9. QUALITY CONTROL

The following quality control samples are prepared with each batch of samples.

Quality Controls	Frequency	Control Limit
ICV	After the ICAL	90-110%
ICB	After the ICV	< RL (9060A) <½ RL (SM5310C)
MB	1 per preparation batch[1]	< RL (9060A) <½ RL (SM5310C)
LCS	1 per preparation batch[1]	85-115%[2]
LCSD	If insufficient sample provided for an MS/MSD	85-115%[2]; 20% RSD
MS/MSD[3]	1 per preparation batch[1] (SM5310C) Every 10 samples (9060A)	75-125%; 20% RSD
CCV	Every 10 samples	90-110%
CCB	Every 10 samples	< RL (9060A) <½ RL (SM5310C)
RLV	Quarterly (SM5310C)	50-150%

[1]A batch is limited to 20 samples.
[2]Statistical control limits may be developed per SOP PT-QA-021, but must be 85-115% or tighter.
[3]The sample used for MS/MSD pair or Duplicate are randomly selected, unless specifically requested by a client.

9.1 The initial and continuing calibration requirements are listed in section 10.1 below. If the initial calibration, or initial calibration verification fails to meet criteria, prepare fresh standards and repeat calibration. If a continuing calibration check standard or blank fails to meet criteria, repeat the analysis once. If the failure repeats, prepare and analyze a fresh calibration curve and repeat the analysis of all samples analyzed after the last passing calibration check.

9.2 One Method Blank (MB) must be analyzed along with each batch of 20 or fewer samples. The method blank matrix is laboratory reagent water. If samples are filtered by the laboratory for Dissolved Organic Carbon analysis, a MB must be filtered and analyzed along with the samples.

9.2.1 The average (reported) Method Blank results must be less than the RL for method 9060A, and less than ½ the RL for method 5310C. Sample results associated with MB's that have hits above the MDL shall be reported with "B" qualifiers. If the MB has a hit above the RL (or ½ the RL for method 5310C), all associated samples shall be reanalyzed unless one of the following situations exists:

- If the target analyte is not detected above the RL in client samples, the data may be reported with qualifiers and using the "Method Blank – Report, ND" NCM.

- If the sample concentration is greater than 10 times the MB concentration, results may be reported with qualification and using the "Method Blank – Report, 10X" NCM.
- If neither of the above criteria are met and reanalysis is not possible due to limited sample volume, the sample data must be qualified. These results must be reported using the "Method Blank – Insufficient Sample" NCM.
- If reanalysis cannot occur for another reason, the client must be contacted and their response documented in the case narrative of the report.

NOTE: If method 9060A is used and the individual results as well as the average are reported, each individual injection must meet the MB criteria above.

9.3 Laboratory Control Sample (LCS) – One LCS is analyzed along with each batch of 20 or fewer samples, prepared according to section 7.9 above. The average (reported) LCS matrix is the same as for the MB. LCS recovery must be within 85-115%.

9.3.1 If recovery is outside the control limits, re-inject the standard once. If failure repeats, all associated sample must be reanalyzed unless one of the following situations exists:

- If LCS recovery is greater than 115% and the sample results are < RL, the data may be reported with qualification and using the "LCS/LCSD - %R High" NCM.
- If the above criteria are not met and reanalysis is not possible due to limited sample volume, the sample data must be qualified. These results must be reported using the "LCS/LCSD – Insufficient Sample" NCM.
- If reanalysis cannot occur for another reason, the client must be contacted and their response documented in the case narrative of the report.

NOTE: If method 9060A is used and the individual results as well as the average are reported, each individual injection must meet the LCS criteria above.

9.4 One sample per every 10 (for method 9060A) or 20 (for method 5310C) is also used for matrix spike and matrix spike duplicate analysis. The sample is spiked with a known amount of organic carbon per section 7.10 above. Percent recovery of the spike must be within 75-125%, and the %RPD for the pair must be $\leq$20%.

9.4.1 If the MS or MSD recovery is outside acceptance criteria, and the LCS recovery was within criteria, it is possible that the matrix of the sample contained an interference. Results may be reported with proper qualification using the MS/MSD - %R, LCS Pass" NCM.

- If the native analyte concentration in the MS/MSD exceeds 4x the spike level for that analyte, the recovery data are reported with a "4" qualifier in TALS LIMS.

9.4.2 If there is insufficient sample for MS/MSD, a LCS and LCSD may be prepared and analyzed. The recovery for LCS/LCSD must be ± 15 % and the RPD must be within ± 20%. If an LCSD is necessary, document the lack of sufficient volume to perform an MS/MSD using the "MS/MSD – Insufficient volume" NCM.

9.4.3 Samples identified as field blanks cannot be used for MS/MSD analysis.

9.5 Semiannually, the inorganic purging efficiency of the instrument must be checked by analyzing a second aliquot of the LCS, spiked with the Inorganic Carbon spike. Recovery of this TIC spiked sample and the batch LCS must agree within 10%RPD.

9.5.1 If inorganic carbon carry-over is evident in the spiked sample, adjust sample containers, volume, purge gas flow rate and/or purge time to obtain complete removal of inorganic carbon. Any changes made will be noted in the instrument maintenance log and will be a permanent change to the procedure.

9.6 A Reporting Limit Verification Standard must be analyzed on at least a quarterly basis for method 5310C. This standard is prepared from either the primary or secondary stock standard at a concentration 1-2 times the reporting limit and is analyzed in duplicate. The average result must be within 50-150% of the true value.

9.6.1 If recovery of the RLV is outside control limits, prepare a fresh standard and reanalyze. If the failure repeats, re-evaluate the reporting limit with a fresh calibration curve and a single day, 7-point MDL study.

10. PROCEDURE

10.1 Calibration

10.1.1 Initial setup

Constant Settings	
STD Mass	6.76ug C
Sample Volume	2.4mL
Acid Volume	1.0mL
Oxidant Volume	1.5mL

10.1.2 Adjust the nitrogen to 50-60 psi using the flow valve on the tank. The gauge should always be set at 50-60 psi when the instrument is not in use. (NOTE: The pressure reading on the instrument should read 20 psi when the tank pressure is between 50-60 psi.)

10.1.3 If the main valve reads < 200 psi, the tank should be changed. Do not allow the tank to go empty.

10.1.4 Remove the reagent bottles and fill with appropriate reagents (phosphoric acid solution and sodium persulfate). Do not fill bottles completely full; leave a small amount of air space. Loosely reconnect caps (and tubing), and replace the bottles into the instrument.

10.1.5 Calibrate the instrument at least every 6 months, or more often as needed when CCVs or CCBs fail to meet acceptance criteria, or when other problems are encountered. Calibrate using blank and minimum five calibration standards.

10.1.6 Choose “sequences” from the “databases” menu option, open up the calibration template.

10.1.7 Confirm all information.

10.1.8 Analyze an ICV/ICB immediately after the curve. ICV is from a second source, which is separate source from the curve.

10.1.9 Save the file using the current date as the filename.

10.1.10 Evaluate the data. The instrument uses the equation y = mx + b to calculate the linear 1st order calibration curve, where $r^2 \geq 0.995$. The correlation coefficient of the original curve must be $r^2 \geq 0.995$ or recalibration is required.

- Calculate the %Error for each calibration standard read back against the plotted curve. The %E for each point above the lowest concentration must be $\leq$ 20%. The %E for the lowest point must be $\leq$ 50%.

10.1.11 ICV/CCV (Initial &Continuing Calibration): The run is checked at the beginning (ICV – separate source from the curve), after every ten environmental samples (CCV – same source as ICV), and at the end of the run of the same species (TOC or TIC) using a midrange CCV made from a secondary source (Section 7.8 or 7.11.2) to verify continued linearity. A reported CCV value cannot vary from the original curve by more than ± 10% or recalibration is required.

- If CCV recovery is greater than 110% and the sample results are below the RL, the data may be reported with qualification and an NCM specifying the

CCV, its recovery and the <RL samples being reported. Repeated CCV failure will require a new calibration curve to be analyzed

NOTE: If method 9060A is used and the individual results as well as the average are reported, each individual injection must meet the CCV criteria above.

10.1.12 System cleanliness is checked every ten environmental samples and at the end of the run using a Continuing Calibration Blank (CCB). A reported CCB value cannot contain the analyte of interest above the reporting limit (9060A) or ½ reporting limit (SM5310C), or recalibration is required.

- If the target analyte is not detected above the RL in client samples, the data may be reported with qualifiers and NCM specifying the CCB above the RL (or ½ RL) and the associated non-detect samples reported.
- If the sample concentration is greater than 10 times the CCB concentration, results may be reported with qualification and an NCM specifying the CCB, its concentration, and the associated >10x samples reported.

  NOTE: If method 9060A is used and the individual results as well as the average are reported, each individual injection must meet the CCB criteria above.

10.1.13 All prepared standards are logged into the reagent module of TALS. All standards are assigned a unique number for identification.

10.2 Sample Preparation

10.2.1 The TOC samples, which come from the field in containers ready for analysis, are sparged by the instrument prior to TOC analysis to remove inorganic carbons that may be present. Samples requiring both TOC & TIC are received as two separate samples that are analyzed separately.

10.2.2 The instrument has analytical modes for TIC, TOC & TC analysis. Analysis after sparging is calculated by the instrument as TOC. Unsparged analysis is calculated as TC. The difference between the two results is calculated as TIC. All calculations are performed by the instrument software program based on calibration curves.

10.2.3 All sample preparation and analysis information will be documented in TALS. Raw data will be scanned and attached to the appropriate TALS batch for inclusion in the report.

10.2.4 Sample size is selected in the instrument set-up and is the same for all sample, calibration, and QC injections. The samples containing particulates will be stirred with a magnetic stirrer. The needle depth can be adjusted if necessary.

10.2.5 Samples and associated QC (i.e. Blanks, LCS, MS/MSD, ICV and CCV's) analyzed according to SM5310C will be analyzed in replicate until the injection of consecutive measurements obtained are reproducible to within ± 10% RPD.

10.2.5.1 For samples outside of ± 10% RPD criteria, the laboratory will perform up to 4 sample injections, taking the most consistent results and initiate a non-conformance indicating that the results are not within criteria due to the nature of the sample matrix. If for any reason the laboratory is not provided with enough sample to perform the analysis in replicate a notation will be made in the case narrative.

10.2.6 Samples and associated QC (i.e. Blanks, LCS, MS, ICV and CCVs) analyzed according to SW-846 Method 9060A will be analyzed in quadruplicate. Both the average and the range will be reported. Quadruplicate can mean 4 injections from the same vial or 2 injections each from 2 vials of the same sample or 1 injection each from 4 vials of the same sample depending on how the client performs the sampling process. If for any reason the laboratory is not provided with enough sample to perform the analysis in quadruplicate a notation will be made in the case narrative.

10.2.7 CCB/CCV's are analyzed per 10 samples, not injections, for both Method 5310C and 9060A.

10.2.8 The pH of TOC samples will be taken after analysis and documented in the appropriate field in the TALS Batch Worksheet. An NCM will be generated for any sample found to have a pH of > 2 so that this information can be pulled into the job-specific Case Narrative.

10.2.9 See the manufacturer's instructions for an instrument-troubleshooting guide and maintenance requirements.

11. CALCULATIONS / DATA REDUCTION

11.1 Total Organic Carbon, mg/L = Instrument Value x Dilution

Where replicate measurements are performed:

TOC, mg/L = average of the 4 instrument values* x dilution

* The four values will be considered to have an acceptable level of consistency, if each of the values lies within ± 35% of the mean. If analysis of any replicate exceeds the 35%, reanalysis will be performed and the findings narrated.

11.2 MS/MSD % Recovery = ((observed spike-sample conc.)/true spike)x100

11.3 Duplicate Sample (Relative Percent Difference):

$$RPD = \frac{|X_1 - X_2|}{\left(\frac{X_1 + X_2}{2}\right)} \text{ x } 100\%$$

Where:
X_1 = Original Result
X_2 = Duplicate

11.4 LCS Percent Recovery:

$$\text{LCS \% Recovery} = \left(\frac{\text{Observed Conc. in LCS}}{\text{True LCS Conc.}}\right) \text{ x } 100\%$$

11.5 Calculation of TIC

TIC = TC – TOC

Where:

TIC = Total Inorganic Carbon

TC = Total Carbon

TOC = Total Organic Carbon

Note: the calculation for TC, TOC and TIC is a function of the instrument's analytical software.

11.6 All instrument data is uploaded into TALS. Raw data is attached to the TALS Batch as a pdf.

12. METHOD PERFORMANCE

12.1 The supervisor has responsibility to ensure that an analyst who performs this procedure is properly trained in its use and has the required experience. Performance is monitored through internal QC and outside performance evaluation samples. Please refer to the QA Manual for additional information concerning Precision and Accuracy.

12.2 Demonstration of Capabilities – Prior to the analysis of samples, a Demonstration of Capabilities (DOC) as described in the QA Manual and SOP PT-QA-001, must be performed initially, annually and any time a significant change is made to the analytical system.

12.3 Method Detection Limit Study – A Method Detection Limit (MDL) study, as described in Eurofins TestAmerica corporate Detection Limits SOP (CA-Q-S-006), must be performed initially and any time a significant change is made to the analytical system. The MDL must be evaluated at least every 12 months.

13. POLLUTION CONTROL

13.1 It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage and reagent stability). Employees must abide by the policies in Section 13 of the Corporate Environmental Health and Safety Manual (CW-E-M-001) for "Waste Management and Pollution Prevention" and the Pittsburgh Facility Addendum EH&S Manual (PT-HS-001).

14. WASTE MANAGEMENT

14.1 Waste management practices are conducted consistent with all applicable rules and regulations. Excess reagents, samples and method process wastes are disposed of in an accepted manner. Waste description rules and land disposal restrictions are followed. Waste disposal procedures are incorporated by reference to PT-HS-001. The following waste streams are produced when this method is carried out.

14.1.1 Acidic waste from the auto-analyzer. (This waste is collected in a waste container identified as "Acid Waste", Waste #33. This waste is neutralized to a final pH between 6 and 9 and discharged down into a lab sink.

15. REFERENCES/CROSS-REFERENCES

15.1 SW846, Test Methods for Evaluating Solid Waste, Third Edition, Total Organic Carbon, Method 9060A, Revision 1, November 2004

15.2 Standard Methods for the Examination of Water and Wastewater, Method 5310C, 2000, editorial revision 2011

15.3 TestAmerica Pittsburgh Quality Assurance Manual, PT-QA-M-001

15.4 SOP PT-QA-001, Employee Orientation & Training

15.5 SOP PT-QA-006, Procurement of Standards and Materials; Labeling and Traceability

15.6 SOP PT-QA-007, Detection Limits

15.7 SOP PT-QA-009, Rounding and Significant Figures

15.8 SOP PT-QA-014, Reporting Limits

15.9 SOP PT-QA-016, Nonconformance & Corrective Action System
15.10 SOP PT-QA-017, Aqueous Pipette Calibration – Gravimetric Method
15.11 SOP PT-QA-021, Quality Assurance Program
15.12 SOP PT-QA-022, Equipment Maintenance
15.13 SOP PT-QA-031, Internal Chain of Custody
15.14 SOP PT-HS-001, Pittsburgh Facility Addendum EH&S Manual to the CW-E-M-001, Corporate Health and Safety Manual for "Waste Management and Pollution Prevention", current revision
15.15 Corporate SOP CA-Q-S-006, Detection Limits

16. METHOD MODIFICATIONS

16.1 If samples require dilution or smaller volumes than specified in this method, the RL will be elevated.

17. ATTACHMENTS

17.1 None

18. REVISION HISTORY

18.1 Revision 5, 8/3/07
18.2 Revision 6, 09/10/2009
18.3 Revision 7, 4/8/2010
18.4 Revision 8, 6/28/2010
18.5 Revision 9, 9/9/2010
18.6 Revision 10, 11/10/2010
18.7 Revision 11, 12/23/2011
18.8 Revision 12, 10/15/2012
18.9 Revision 13, 1/15/2013
18.10 Revision 14, 12/2/2013
18.11 Revision 15, 12/23/2015
18.12 Revision 16, 11/24/2017
18.13 Revision 16.1, 5/13/2019
18.14 Changes to current revision

SOP section	Change from	Change to	Reason
1.3	Removed text referring to QA Manual	Added SOP Checklist text on modications	SOP Checklist Format
7	Removed "Reagent grade" from individual reagents entries	Added blanket Reagent Grade chemicals statement	clarification
9.2.1, 9.3, 10.1.11, 10.1.12		Added: average(reported) to results and NOTE that individual must pass if 9060A reporting individual and average	clarification

9.5	Check for inorganic carbon removal per batch	Check for inorganic carbon to semiannual to be performed on an aliquot of LCS	Update
10.1.2	50 psi	50-60 psi Added a NOTE that the instrument should read 20 psi based on the 50-60psi reading on the tank	Clarification
10.1.11		Added if CCV fails high and samples are ND, samples may be reported	Clarification
10.1.12		Added if CCB fails and samples are ND or >10x the CCB concentration, samples may be reported	Clarification
12.3	MDLs per QA Manual	MDLs per corporate SOP CA-Q-S-006	Correction
15.15		Added reference for CA-Q-S-006	Clarification

SOP # with Revision: PT-WC-018 Rev. 21
SOP Title: Total Cyanide Distillation and Analysis, Semi-Automated Colorimetry
Amendment Number: 1

Effective Date of Amendment:
Append this form to the front of the SOP copy.

CONTROLLED DISTRIBUTION	
COPY # :	**001**
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The following SOP change is in effect as of the stated date. *This form will remain attached to the referenced SOP until such a time that the SOP is updated, approved, and redistributed, at which time it will become part of the historical SOP record.*

1. Briefly explain the reason for the update: 40 CFR Part 136 Update of Reference Year

2. Category for SOP Change:

☒ Typographical Corrections (Non-Technical) – Retraining Not Required.

☐ Typographical Corrections (Technical- define below) – Retraining is required.

☐ Procedural Changes (Define Below) – Retraining Required.

3. Summary of Procedure Change (circle to indicate if there are attachments to this form: No / Yes: # pages attached = ___)

Standard Methods for the Examination of Water and Waste Water, Method 4500-CN-C, Total Cyanide after Distillation and E, Colorimetric Method, 1999, editorial revision 2011, 2016

Does this change result in a Method Modification? Yes (No)
If yes, please explain below.

Any method modification will be incorporated into section 16 of the SOP at the next revision.

8-13-21
8-16-21

Approval Signature/Date: Technical

Amanda L Gulla 08/12/21

Approval Signature/Date: QA Manager or Designee

Title: Total Cyanide Distillation and Analysis, Semi-Automated Colorimetry

Methods: EPA Method 335.4, SM4500-CN C/E, SW-846 9010C, 9012B and 9014 - Discrete

Approvals (Signature/Date):

4/6/2020	4/6/2020
Roseann Ruyechan Date	Steve Jackson Date
Inorganics Department Manager	Regional Safety Coordinator
3/25/2020	4/1/2020
Virginia Zusman Date	Deborah L. Lowe Date
Quality Assurance Manager	Laboratory Director

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1. SCOPE AND APPLICATION

1.1 This method is applicable to the determination of Total Cyanide in waters, solids, tissues and wipes using a Seal AQ2 Discrete Autoanalyzer/Spectrophotometer. It is based on EPA Method 335.4, SW846 Methods 9010C, 9012B, and 9014, and SM4500-CN C & E.

1.2 The reporting limit is 0.010 mg/L for waters, 0.25 mg/kg for solids and tissues and 0.50 ug/wipe for wipes.

1.3 On occasion clients may request slight modifications to this SOP. Any modification to this procedure must be approved by the QA department of the Laboratory or Technical Director, and documented fully in an NCM that accompanies the analytical batch.

2. SUMMARY OF METHOD

2.1 For Total Cyanide: Cyanide, as HCN, is released from cyanide complexes by distilling/refluxing the sample with strong acid and absorbed in a sodium hydroxide solution.

2.2 The sodium hydroxide solution is analyzed colorimetrically on an autoanalyzer using the pyridine-barbituric acid method. The color is read at 575 nm.

3. DEFINITIONS

3.1 TALS – Test America Laboratory Information Management System

3.2 NCM – Non-Conformance Memo – a system within TALS for the lab to communicate to project management and others when there is an anomaly seen with the samples or batch, or a QC failure.

3.3 Refer to the glossary in the Laboratory Quality Manual (PT-QA-M-001) for additional definitions.

4. INTERFERENCES

4.1 Nitrates and nitrites may be a positive interference at concentrations greater than 10mg/L, but are eliminated by the addition of sulfamic acid to all samples and QC during distillation.

4.2 Oxidizing agents such as chlorine decompose most cyanides and can lead to low biased results. The presence of residual chlorine should be checked for and treated at

the time of collection. Samples are checked again prior to distillation. Chlorine interference can be removed by drop-wise addition of sodium arsenite or ascorbic acid crystals to the sample until there is no reaction to KI-starch paper.

4.3 Sulfide in the samples interferes with color formation during analysis and must be removed prior to distillation. Sulfides are removed by the addition of cadmium carbonate until there is no reaction to lead acetate paper.

5. SAFETY

5.1 Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual (CW-E-M-001), the Pittsburgh Facility Addendum EH&S Manual (PT-HS-001) and this document. This procedure involves hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

Note: This method is designed to generate Hydrogen Cyanide (HCN) gas. Inhalation of CN gas can cause irritation, dizziness, nausea, unconsciousness and potentially death. Always perform the distillation under a fume hood, and ensure the hood is operating properly when in use.

5.2 Do not add reagents to heated samples. If samples have been heated prior to adding distillation reagents, turn off the heating block, allow the samples to completely cool first, and begin the distillation again.

5.3 Latex, vinyl, Nitrile or similar gloves may be used when handling samples, standards and reagents.

5.4 Ensure cooling water is turned on to the distillation unit. Otherwise the samples may boil over and come into contact with the heating block.

5.5 If samples are identified with cyanide concentrations equal to or greater than 200 mg/L, immediately notify the department manager and personnel responsible for hazardous waste shipping. Those samples must be identified as extremely hazardous for other analysts and must receive special attention during disposal.

5.6 The following is a list of the materials used in this method, which have a serious or significant hazard rating. This list may not include all materials used in the method. The table contains a summary of the primary hazards listed in the SDS for each of the materials listed in the table. A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in

the SDS for each material before using it for the first time or when there are major changes to the SDS.

Material	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Potassium Cyanide	Poison Irritant	5 mg/m3, TWA	Very hazardous in case of skin contact (permeator), of ingestion, of inhalation. Hazardous in case of skin contact (irritant), of eye contact (irritant). Corrosive to eyes and skin. The amount of tissue damage depends on length of contact. Eye contact can result in corneal damage or blindness. Skin contact can produce inflammation and blistering. Inhalation of dust will produce irritation to gastro-intestinal or respiratory tract, characterized by burning, sneezing and coughing. Severe over-exposure can produce lung damage, choking, unconsciousness or death. Repeated exposure to a highly toxic material may produce general deterioration of health by an accumulation in one or many human organs.
Pyridine	Flammable Irritant	5 ppm-TWA	Inhalation causes severe irritation to the respiratory tract. Symptoms of overexposure include headache, dizziness, nausea, and shortness of breath. Causes severe irritation possibly burns, to the skin. Symptoms include redness and severe pain. Absorption through the skin may occur, resulting in toxic effects similar to inhalation. May act as a photosensitizer. Vapors cause eye irritation. Splashes cause severe irritation, possible corneal burns and eye damage.
Silver Nitrate	Poison Corrosive Oxidizer	0.01 mg/m3 TWA	Symptoms of inhalation may include burning sensation, coughing, wheezing, laryngitis, shortness of breath, headache, nausea and vomiting. Symptoms of skin contact include redness, pain, and severe burns; eye contact causes blurred vision, redness, pain, severe tissue burns and eye damage.
Sodium Hydroxide	Corrosive	2 mg/m3-Ceiling	Severe irritant. Effects from inhalation of dust or mist vary from mild irritation to serious damage of the upper respiratory tract, depending on severity of exposure. Symptoms may include sneezing, sore throat or runny nose. Contact with skin can cause irritation or severe burns and scarring with greater exposures. Causes irritation of eyes, and with greater exposures it can cause burns that may result in permanent impairment of vision, even blindness.

Material	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Sulfuric Acid (1)	Corrosive Oxidizer Dehydra-dator	1 mg/m^3	This material will cause burns if comes into contact with the skin or eyes. Inhalation of vapors will cause irritation of the nasal and respiratory system.
Zinc Acetate	Irritant	None Listed	Symptoms of skin or eye contact include redness, itching and pain.
Cadmium Carbonate	Carcinogen Poison Irritant	0.01 mg/m^3 - TWA	Toxic if inhaled or swallowed. May cause skin, eye and respiratory tract irritation.
Sulfamic Acid	Corrosive Irritant	None Listed	Extremely destructive to tissues of the mucous membranes and upper respiratory tract. Symptoms may include burning sensation, coughing, wheezing, laryngitis, shortness of breath, headache, nausea and vomiting. May cause pulmonary edema, a medical emergency. Pulmonary edema may be delayed up to 48 hours. Swallowing can cause severe burns of the mouth, throat and stomach leading to death. Can cause sore throat, vomiting and diarrhea. Skin contact can result in redness, pain and severe burns. Eye contact can cause blurred vision, redness, pain, severe tissue burns and eye damage.
Sodium arsenite	Poison Reproduc-tive Hazard Carcinogen	None listed	Symptoms of inhalation include respiratory tract irritation. Causes skin irritation, and may be fatal if absorbed through skin.
Ascorbic acid	Not classified	OSHA PEL TWA (Total Dust) 15 mg/m3	Most important symptoms and effects, both acute and delayed:Irritation, Nausea,Headache, Shortness of breath.
Chloramine-T	Corrisive	None Listed	Causes burns by all exposure routes. May cause allergy or asthma symptoms or breathing difficulties if inhaled. Product is a corrosive material. Use of gastric lavage or emesis is contra indicated. Possible perforation of stomach or esophagus should be investigated: Ingestion causes severe swelling, severe damage to the delicate tissue and danger of perforation: Symptoms of allergic reaction may include rash, itching, swelling, trouble breathing, tingling of the hands and feet, dizziness, light-headedness, chest pain, muscle pain.
Barbituric Acid		None Listed	There are no signs or symptoms of exposure.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

5.7 Eye protection that protects against splash, laboratory coat, and appropriate gloves must be worn while samples, standards, solvents, and reagents are being handled. Cut resistant gloves must be worn doing any other task that presents a strong possibility of getting cut. Disposable gloves that have been contaminated will be removed and discarded; other gloves will be cleaned immediately.

5.8 Exposure to chemicals must be maintained as low as reasonably achievable. Therefore, unless they are known to be non-hazardous, all samples must be opened, transferred and prepared in a fume hood, or under other means of mechanical ventilation. Solvent and waste containers will be kept closed unless transfers are being made. Preparation of sodium hydroxide solutions produces considerable amounts of heat. Use plastic containers to mix this solution if possible. If glass containers are used, they must be free of any cracks or irregularities.

5.9 The preparation of standards and reagents will be conducted in a fume hood with the sash closed as far as the operation will permit. Ensure cooling water is turned on to the distillation unit. Otherwise the samples may boil over and come into contact with the heating plates.

5.10 All work must be stopped in the event of a known or potential compromise to the health and safety of a TestAmerica associate. The situation must be reported immediately to a laboratory supervisor or EH&S coordinator.

6. EQUIPMENT AND SUPPLIES

The following items are recommended for performing this procedure. Equivalent items should only be used when they result in an improvement in quality, efficiency, productivity, or cost. An item can be considered equivalent if with its use, the analytical and QA/QC requirements in this SOP can be met.

6.1 Cyanide Distillation Apparatus – Midi “Easy-Dist System” Westco/Unity

6.2 SimpleDist® Micro Cyanide Distillation System, heating block and distillation tube units – Environmental Express

Note: The volume markings on the SimpleDist® collection tubes must be verified for each lot following the procedure in SOP PT-QA-026 prior to use.

6.3 Vacuum Pump with trap

6.4 Class A Graduated Cylinders: Various sizes

6.5 Class A Volumetric Flasks: Various Sizes

6.6 Automated Pipette

6.7 Balance: Accurate to 0.1 mg

6.8 Seal AQ2 Discrete Spectrophotometer/Autoanalyzer and AQ2 analytical software, detector wavelength set to 575nm

6.9 Lead Acetate Indicator Paper

6.10 Potassium Iodide (KI) – Starch Indicator Paper

6.11 pH paper

6.12 Disposable 120 mL containers

6.13 Glass/plastic/Teflon beads

7. REAGENTS AND STANDARDS

The following items are recommended for performing this procedure. Equivalent items should only be used when they result in an improvement in quality, efficiency, productivity, or cost. An item can be considered equivalent if with its use, the analytical and QA/QC requirements in this SOP can be met. Please refer to the SDS prior to the use of any reagent or standard.

Many of the reagents and standards listed below may be purchased commercially as an alternative to preparing them in-house. Purchased standards and reagents must come with a certificate of analysis. Reagents and chemicals purchased must be reagent grade, or better if specified below. Standards and reagents will use the manufacturer's assigned expiration date unless one is not provided. If not provided, the same expiration period will be assigned as listed for the lab-prepared reagent, using the received date in place of the preparation date.

NOTE: Any reagents and/or spiking solutions stored in the refrigerator should be warmed to room temperature prior to use.

7.1 Use deionized (DI) or reagent grade water (18 mohm) to prepare all solutions.

7.2 Distillation Reagents – Midi distillation (50mL sample)

7.2.1 Sodium Hydroxide (0.25 N): Purchased with certification of analysis

- Alternately, this solution may be prepared in the laboratory: In a 1L volumetric flask, dissolve 10.0g NaOH in approximately 900mL reagent-grade water. Dilute to volume with reagent grade water and mix thoroughly. Store in a plastic bottle, at room temperature.

- If purchased, follow manufacturer's storage and expiration instructions. If prepared in the lab, the solution expires 1 year from the preparation date, not to exceed the expiration of the parent solution or solid.

- Discard solution if a precipitate is seen.

7.2.2 Concentrated Sulfuric Acid, H_2SO_4

7.2.3 Magnesium Chloride hexahydrate ($MgCl_2 \bullet 6H_2O$)

7.2.4 Magnesium Chloride Solution, 510g/L - this solution is purchased

- Alternately, this solution may be prepared in the laboratory: Dissolve 510g of $MgCl\bullet 6H_2O$ in DI water and dilute to 1 L in a volumetric flask. Store at room temperature. If prepared in the laboratory, this solution expires 1 year from preparation date, not to exceed the expiration of the parent solid.

7.2.5 Sulfamic Acid, crystals

7.2.6 Glacial Acetic Acid, CH_3CO_2H

7.2.7 Powdered Cadmium Carbonate

7.2.8 Sodium Arsenite Solution (0.1N): Dissolve 3.2g $NaAsO_2$ in 250mL DI water. Store at room temperature. This solution expires 1 year from preparation date, not to exceed the expiration of the parent solid.

7.2.9 Ascorbic acid crystals

7.3 Distillation Reagents – SimpleDist® Method (6mL sample)

7.3.1 Sodium Hydroxide (0.25 N): See 7.2.1 above

7.3.2 Sulfamic Acid Solution: Dissolve 12g sulfamic acid (7.2.5) in 100 mL reagent water. Store in plastic bottle at room temperature. This solution expires 1 month after preparation.

7.3.3 Sulfuric Acid/Magnesium Chloride Solution – Add 277 mL of reagent water to a 500 mL volumetric flask. Add 80.5 g magnesium chloride hexahydrate (7.2.3) and stir until completely dissolved. Slowly add 189 mL concentrated sulfuric acid (7.2.2) with stirring. CAUTION: This addition will produce significant heat from the solution. This should be performed in a hood as toxic fumes may be produced. Allow solution to cool to room temperature and dilute to 500 mL.

Store in glass or plastic container at room temperature. This solution expires 1 year after preparation.

7.4 Analytical Reagents

7.4.1 Sodium Hydroxide (0.25N): See 7.2.1 above.

7.4.2 Phosphate Buffer 1.0 M: In a 1L volumetric flask, dissolve 138g KH_2PO_4 in approximately 800mL reagent-grade water. Dilute to volume with reagent grade water and mix thoroughly. Store at >0 to $\leq 6^oC$. This solution expires 6 months from the preparation date, not to exceed the expiration of the parent solid. Discard if solution becomes cloudy, or mold growth is seen.

7.4.3 Chloramine-T Solution: Dissolve 1.0g of chloramine-T hydrate in reagent-grade water. Dilute to 100mL volume with reagent grade water and mix thoroughly. Store at >0 to $\leq 6^oC$. Prepare fresh weekly.

7.4.4 Pyridine - Barbituric Acid Reagent: In the fume hood, place 15g barbituric acid in a 1L volumetric flask and add approximately 250 mL of reagent grade water, rinsing down the sides of the flask to wet the barbituric acid. Add 75 mL pyridine (C_5H_5N) with stirring. Add 15 mL concentrated hydrochloric acid (12 M HCl) slowly, and mix until the barbituric acid is dissolved. Dilute to volume with reagent grade water and mix thoroughly. Store at >0 to $\leq 6^oC$ in a dark bottle. This solution expires 6 months from the preparation date, not to exceed the expiration of any parent components.

Note: Barbituric Acid is difficult to obtain, and must be ordered 6 months in advance. Request a new supply before you run out. (This chemical can only be purchased from MG Scientific – Avantor, JT Baker B654-05.)

7.4.5 Silver Nitrate solution (0.0192N), purchased with certificate of analysis. Follow manufacturer's storage and expiration requirements.

7.4.6 Rhodanine Indicator, purchased - Follow manufacturer's storage and expiration requirements.

7.5 Standards Preparation:

7.5.1 Cyanide Stock Standard (1000 mg/L CN): This is purchased commercially with certificate of analysis. This standard should be standardized weekly as follows:

7.5.1.1 Dissolve approximately 0.2g NaOH in 25mL of DI water in beaker 1.

7.5.1.2 Add 5.0mL of CN Stock to beaker 2 and dilute to 50mL with DI water. Repeat with second source standard (7.5.2) in beaker 3.

7.5.1.3 Add 10 drops of Rhodanine Indicator to each beaker.

7.5.1.4 Titrate solutions with 0.0192N Silver Nitrate to a salmon-pink endpoint.

7.5.1.5 Record the titration information in the CN Stock Logbook.

7.5.1.6 ((A-B) x 1000mg/L) / C = True Value of Stock, where:

A = mL $AgNO_3$ to titrate stock
B = mL $AgNO_3$ to titrate blank
C = mL stock analyzed (5.0)

7.5.2 Second Source (Independent) Cyanide Stock (1000mg/L CN): This solution is purchased commercially from a different NIST traceable source than solution 7.5.1 and is used for verification of the calibration source (ICV).

7.5.3 Cyanide Working Solution 1 (Curve, CCV, MS/MSD) 10mg/L CN: Pipette 1mL* of the Stock Standard (7.5.1) into a 100mL volumetric flask containing 50 mL of 0.25 N NaOH. Dilute to volume with 0.25 N NaOH and mix thoroughly. Prepare fresh weekly.

7.5.3.1 *If the CN Stock (7.5.1) True Value as calculated in 7.5.1.1 – 7.5.1.5 is not 1000mg/L, adjust the volume of stock used to prepare Solution 1:

X = (10.0mg/L x 100mL) / T, where:

T = True Value of the stock as calculated (mg/L)
X = mL stock required to prepare 10mg/L solution

7.5.4 Cyanide Second Source Intermediate Solution for ICV and LCS (10mg/L CN): Prepare an intermediate standard from the second source stock (7.5.2) following the same steps as in 7.5.3 above.

7.5.5 Calibration Standards: Set of six. The standard curve may not have fewer than five standards. Calibration standards are distilled along with SimpleDist® samples. Standards are not distilled for use with the Midi distilled samples. Prepare fresh daily.

7.5.5.1 Calibration Preparation Standard: Prepare a 0.50 mg/L standard by pipetting 5.0 mL of the 10.0 mg/L working standard (7.5.3) into a 100 mL

volumetric flask and dilute to volume with 0.25N NaOH. This standard is prepared fresh at least every 3 days.

7.5.5.2 Manual Standard Curve for SimpleDist® samples: 0.5, 0.25, 0.1, 0.05, 0.025 and 0.01 mg/L. Using a calibrated pipette, add 6.0, 3.0, 1.2, 0.6, 0.3 and 0.12 mL of the 0.5 mg/L standard (7.5.5.1) into a SimpleDist® sample tube. Bring to 6mL volume with 0.25 N NaOH. 0.25 N NaOH is used for the 0 mg/L standard or calibration blank and ICB/CCB, and is also distilled.

7.5.5.3 Automated Calibration Curve for Midi-distilled samples: The instrument uses the 0.50 mg/L standard (7.5.5.1) to prepare the same calibration standards as listed above in 7.5.5.2.

7.5.5.4 To verify the efficiency of the distillation procedure low and high standards prepared daily from the same free cyanide source (7.5.1) as the calibration curve are distilled along with the sample batch. The recovery must be within ±10 percent of the response for the calibration standards.

- Low standard (0.05mg/L) = 5.0mL Calibration Std-0.5 (7.5.5.2)/50mL with 0.25N NaOH
- High standard (0.25mg/L) = 1.25mL Working Solution 1 (7.5.3)/50mL with 0.25N NaOH

7.5.6 ICV – Initial Calibration Verification (0.20 mg/L CN): Pipette 2.0mL of the second source 10mg/L working standard (7.5.4) into a 100mL volumetric flask. Bring to volume with 0.25 N NaOH and mix. This standard is not distilled for Midi-distilled samples. Prepare fresh every 3 days.

7.5.7 CCV - Continuing Calibration Verification (0.10mg/L CN): Add 1.0mL of 10mg/L Working Solution 1 (7.5.3) to a 100mL volumetric flask. Bring up to volume with 0.25N NaOH and mix thoroughly. This standard is not distilled for Midi-distilled samples. Prepare fresh every 3 days.

7.5.8 Complex Cyanide Stock Standard for complex LCS (1000 mg/L) – purchased commercially and prepared from a complex cyanide source, such as potassium ferrocyanide.

7.5.8.1 Intermediate Complex Cyanide Standard (10 mg/L): Pipette 1mL of the 1000 mg/L Complex Cyanide Stock Standard into a 100mL volumetric flask containing 50 mL of 0.25 N NaOH. Dilute to volume with 0.25 N NaOH and mix thoroughly.

7.5.9 Instrument blanks (ICB and CCBs) are 0.25N NaOH. Instrument blanks are not distilled.

8. SAMPLE COLLECTION, PRESERVATION, SHIPMENT AND STORAGE

Table 1 –Holding Time and Preservation Requirements

Matrix	Sample Container	Minimum Sample Size	Preservation	Holding Time to Analysis	Reference
Water	Amber Glass or Plastic	500 mL	pH > 12 with NaOH, Cool > 0 to ≤ 6.0°C	14 days from collection	40 CFR Part 136
Solid	Glass, 8 oz.	250 grams	Unpreserved, Cool > 0 to ≤ 6.0℃	14 days from collection	40 CFR Part 136

9. QUALITY CONTROL

Table 2 – QA/QC Requirements and Frequency

Quality Controls	Frequency	Control Limit
Initial Calibration, minimum 5 standards and a blank	Daily	Linear regression, $r^2 \geq 0.995$, y-intercept $\leq \pm$ 1/2RL
ICV (Second Source Standard)	After the ICAL	90-110%
ICB, CCBs and MB	After ICV and CCVs	< RL for method 9012/9014 < MDL for method 335.4 < ½ RL for SM4500 CN
Continuing Calibration Verification CCV	1 per every 10 samples	90-110 %
MDLV or LOQ verification (LLOQ for metho 9014)	Quarterly to validate quantitation capability at low analyte concentration levels	±35% of True Value with ≤20% RSD between MDLV's
Low and High Distilled Standard Check	1 set per prep batch (required for 9010C)	90-110%
Laboratory Control Sample (LCS) LCS may be either free CN (from 7.4.4) Or Complex CN (7.4.8)	1 per preparation batch	90-110% (335.4 and SM4500-CN E) 85-115% (9012B and 9014) waters See sect 9.4.2 for soil LCS information
Duplicate (MD)	1/20 samples Required for SM4500CNC&E	≤ 20% RPD
Matrix Spike (MS)	1/10 samples 1/20 samples	90-110% (335.4 and SM4500-CN E) 75-125% (9012B and 9014)
Matrix Spike Duplicate (MSD)	1/20 sample	90-110% Recovery(335.4/SM4500-CN) 75-125% (9012B and 9014) ≤ 20% RPD

9.1 A Preparation Batch is 20 or fewer samples of the same matrix, distilled within the same working shift using the same reagent lots.

9.2 A Method Blank is distilled and analyzed along with each batch of 20 or fewer samples of the same matrix.

9.2.1 The MB for water batches is 0.25N NaOH.

9.2.2 The MB for solid batches must include a clean solid matrix like glass or Teflon beads. For the Midi distillation, 1 g of clean solid matrix is used, 0.2 g for the SimpleDist® method.

9.2.3 If the MB has a concentration above the reporting limit for methods 9012/9014, above the MDL for method 335.4, or above ½ the RL for method SM 4500 CN, the analyst will check calculations and instrument performance and reanalyze the MB once. If the MB is confirmed to be contaminated, one of the following corrective actions is required:

- Evaluate the sample results. If all associated samples are non-detect, results may be reported with the NCM "Method Blank – Report, ND". If associated samples have concentrations that are greater than 10 times the MB concentration, results may be reported with the NCM "Method Blank – Report, 10X".

- Samples for which neither of the above criteria apply must be redistilled and reanalyzed.

- If re-distillation/reanalysis is not possible due to limited sample quantity, the laboratory project manager will be notified to inform the client. Results shall be reported flagged with a "B" qualifier and the NCM "Method Blank – Insufficient Sample".

- If reanalysis will be outside of holding time, the client must be notified and approval from the client must be obtained for the reanalysis. If reanalysis is performed past holding time, both the initial and reanalysis results must be included in the report.

9.3 A Laboratory Control Sample is distilled and analyzed along with each batch of 20 or fewer samples of the same matrix.

9.3.1 A water batch LCS may be prepared from the free Cyanide Stock Standard (7.5.4) or from a complex cyanide source (7.5.8). For SimpleDist® distillation the LCS is prepared by adding 0.12 mL of 10mg/L intermediate standard to 6mL of 0.25N NaOH.

9.3.2 The LCS for solid matrices is a commercially purchased Standard Reference Material or certified solid QC standard. 1.0g of the solid LCS is used for the Midi distillation and 0.2g for the SimpleDist®. Recovery of a solid LCS must be within the control limits supplied with the standard by the manufacturer.

9.3.3 If LCS recovery is outside the control limits, the analyst will check calculations and instrument performance and reanalyze the LCS once. If the LCS failure is confirmed, one of the following corrective actions is required:

- Evaluate the sample results. If LCS recovery is above control limits and all associated samples are non-detect, results may be reported with the NCM "LCS/LCSD - %R High".
- Samples associated with low LCS recovery, or samples with detected CN associated with high LCS recovery must be redistilled and reanalyzed.
- If redistillation/reanalysis is not possible due to limited sample quantity, the laboratory project manager will be notified to inform the client. Results shall be reported flagged with a "*" qualifier and the NCM "LCS/LCSD – Insufficient Sample".
- If reanalysis will be outside of holding time, the client must be notified and approval from the client must be obtained for the reanalysis.

9.4 Low and high concentration free cyanide standards (7.5.5.4 - 50ppb and 250ppb prepared from the same source as the calibration standards) are distilled and analyzed along with each sample batch. These standards are to verify the distilled vs undistilled method performance, and are not matrix-matched to the samples in the batch. Corrective actions are the same as for the LCS above. A fresh calibration curve may be analyzed and the sample batch reanalyzed without re-distillation once.

9.5 If the ICV does not verify the calibration curve, check the instrument conditions and if necessary perform instrument maintenance, then analyze a new calibration curve followed by a fresh ICV.

9.6 All reported data must be bracketed by an acceptable calibration curve and a CCV, or by consecutive acceptable CCVs. If a CCV %Recovery is outside control limits, one of the following corrective actions is required:

9.6.1 Evaluate the sample results. If CCV recovery is above control limits (recoveries up to 120%) and all associated samples are non-detect, results may be reported with the NCM "CCV- %D High, Sample ND". If CCV recovery is above 120%, samples not bracketed by acceptable CCV must be reanalyzed.

9.6.2 Samples associated with low CCV recovery, or samples with detected CN associated with high CCV recovery must be reanalyzed. This applies to all samples bracketed by the failing CCV.

9.7 A Continuing Calibration Blank (ICB and CCB), which is 0.25 N NaOH will be run immediately following each ICV and CCV. All reported data must be bracketed by instrument blanks (ICB and CCBs) that show continuous clean operation of the instrument. If a CCB has a concentration above the reporting limit for methods 9012/9014, above the MDL for method 335.4, or above ½ the RL for method SM 4500CN, one of the following corrective actions is required:

9.7.1 Evaluate the sample results. If all associated samples are non-detect or samples have concentrations that are greater than 10 times the CCB concentration, results may be reported with the NCM "CCB – Outside Criteria, Samples ND or 10X".

9.7.2 Samples for which neither of the above criteria apply must be reanalyzed. This applies to all samples which the failing CCB brackets.

9.8 Lower Limit of Quantitation Check Standard (LLOQ) – For Method 9014 only, the LLOQ is the lowest point of quantitation for the laboratory. The LLOQ is verified by the analysis of a standard at the LLOQ concentration that have been taken through all distillation and analytical steps. At a minimum, the LLOQ verification is analyzed quarterly basis to validate quantitation capability at low analyte concentration levels.

9.8.1 The recovery of the LLOQ should be ± 35% of the true value and the RSD between successive LLOQ's should be ≤20%.

9.8.2 If recovery or RPD is outside the acceptance range, an MDL study must be performed and the RL (LLOQ) must be reestablished.

9.9 A matrix spike is required on one sample per every 10 in a preparation batch for EPA method 335.4, and 1 per every 20 samples for all other methods addressed by this SOP. The MS is performed on a second portion of a sample chosen at random in the batch, unless a client specifies a sample to be used.

9.9.1 For the Midi Distillation, prepare the MS by spiking 0.5mL of the 10mg/L Working Standard 1 (7.5.3) into 50mL of water sample or 1 g of soil sample. For sediment samples, add 1.0 mL of Working Standard 1 to 2 g of sample. The spike concentration is 0.10 mg/L or 5 mg/Kg.

- For SimpleDist® distillation, prepare the MS by spiking 0.12mL of the 10mg/L Working Standard 1 (7.5.3) into 6.0mL of water sample or 0.5g of

soil or sediment sample. The spike concentration is 0.20 mg/L or 1.2 mg/Kg.

9.9.2 If the MS (or the MSD) fails recovery criteria, check calculations and instrument performance, and reanalyze the failed QC once. If MS (or MSD) failure is confirmed, one of the following corrective actions is required:

- Evaluate the other batch QC results. If the LCS is acceptable and the MS/MSD are comparable and outside of QC limits, then a matrix effect is indicated. No reanalysis is required, results are reported with the NCM "MS/MSD - %R, LCS Pass".

- Evaluate the parent sample result. If the parent sample required dilution because the initial concentration of the sample is excessively high, then the MS/MSD should be run at the same dilution as the unspiked sample. The MS/MSD should never be run at a dilution greater than the associated sample as this alters the matrix of the sample and thereby makes it invalid. If the parent sample concentration is greater than 4x the spiked amount, the MS recovery cannot be accurately calculated, and the NCM "MS/MSD – High Targets" should be used.

9.10 A sample duplicate is a second portion of a sample chosen at random that is taken through the distillation and analytical steps along with the parent sample.

9.10.1 Duplicate (MD), Matrix Spike (MS), and Matrix Spike Duplicate (MSD) analyses are required for method SM4500-CN C&E.

9.10.2 For methods 335.4 and 9010C & 9012/9014 either a MD and MS set or an MS and MSD set may be analyzed along with the sample batch to meet QC criteria.

9.10.3 If the %RPD limit is exceeded, and sample and duplicate results are above 5x the reporting limit, sample and duplicate should be redistilled. If sample and duplicate results are <5x the reporting limits, results should agree within a difference of the reporting limit concentration.

10. PROCEDURE

10.1 Calibration

10.1.1 A standard curve, (prepared as in section 7.5.5) is analyzed daily on the auto analyzer for cyanide.

10.1.1.1 The calibration standards are not distilled with the Midi distillation procedure. The calibration standards are distilled with the SimpleDist® distillation procedure.

10.1.1.2 The standard curve is analyzed in order of increasing concentration.

10.1.1.3 A linear regression (1st order) calibration curve of absorbance vs concentration is plotted. The correlation coefficient must be greater than or equal to 0.995 and the absolute value of the y-intercept must be less than or equal to ± ½ the reporting limit.

10.1.1.4 Calculate the %Error for each calibration standard read back against the plotted curve. The %E for each point above the lowest concentration must be ≤ 20%. The %E for the lowest point must be ≤ 50%.

Note: Temperature of reagents and spiking solution can affect the response factor of the colorimetric determination. The reagents and standards stored in the refrigerator should be warmed to room temperature prior to use.

10.2 Distillation

10.2.1 Water samples must be checked for proper preservation and the presence of matrix interferences prior to distillation.

10.2.1.1 Check the sample pH by pouring a small portion of the sample into a disposable cup and dipping a pH test strip into it. Document the sample pH directly on the TALS Worksheet. If the sample pH is <12, notify the lab project manager that the sample was improperly preserved. Correct the pH to ≥12 in the sample by adding 1mL of 1N NaOH to a 100mL aliquot of sample, mixing and checking the pH again. Document the final pH, if adjusted, on the TALS worksheet. If adjustment was needed, apply the NCM "Preservation – pH Adjusted, pH Included" to the sample.

10.2.1.2 Samples should be checked in the field for the presence of residual chlorine or other oxidizers and pre-treated. Check samples again prior to distillation to confirm using Potassium Iodide (KI) Starch paper. Drip several drops of sample onto a KI-Starch paper strip. If the paper turns blue, it is necessary to treat the sample to remove the oxidizer prior to distillation. To a 100mL portion of sample, add 0.1N Sodium Arsenite solution (7.2.8) or 0.06g Ascorbic acid (7.2.9) slowly while stirring, until a drop of sample no longer produces a reaction with the KI-Starch paper. Document the presence or absence of

chlorine/oxidizers on the TALS Worksheet. If pretreatment is needed, document the lot of treatment used in the TALS Batch Information, and apply the NCM "Preservation – Chlorine-Pre-Treatment" to the sample. NOTE: if any samples are treated, the MB and LCS must have this treatment added as well.

10.2.1.3 Samples must be checked for the presence of sulfides prior to distillation. Drip a few drops of sample onto lead acetate test paper. If the paper darkens, the sample must be treated to remove the sulfide interference. Document the presence or absence of sulfide on the TALS Worksheet. If pretreatment is needed, document the lot of treatment used in the TALS Batch Information, and apply the NCM "Preservation – Sulfide – Pre-Treatment" to the sample. NOTE: if any samples are treated, the MB and LCS must have this treatment added as well.

- Add powdered cadmium carbonate slowly to a 75mL portion of sample with stirring. Yellow cadmium sulfide will precipitate from the sample.

- When a drop of treated sample no longer reacts with the lead acetate test strip, filter the sample through a dry filter paper into a dry beaker and take from this the sample volume to be distilled. It is not necessary to distill calibration standards if this method of pre-treatment is used.

- Solid, tissue and wipe samples are not checked for sulfides until after the distillation is complete. If sulfides are detected in the distillate, it is treated with powdered cadmium carbonate until there is no reaction to the lead acetate paper.

10.2.2 Prepare batch QC according to sections 7.5 and 9 above.

10.2.3 For water samples, pour 50mL of sample into a distillation flask for Midi distillation or 6mL into the sample tube for SimpleDist® distillation.

10.2.4 Refer to PT-QA-024 for subsampling procedures. For solids, wastes and tissues, weigh 1.0g of sample and transfer to a distillation flask and bring volume to approximately 50 mL with reagent grade water for Midi and 0.2g of sample to approximately 5mL with reagent water for SimpleDist® distillation.

- For sediments, use 2.0 g of sample for Midi or 0.5g sample for SimpleDist® and proceed as above.

- For wipe samples, add the entire wipe to the distillation flask along with any liquid present in the jar and bring to 50mL with reagent water. Wipes can only be distilled by the Midi distillation method, they cannot be prepared using the SimpleDist® procedure.

10.2.5 For MIDI distillation procedure, continue to step 10.2.6. For SimpleDist® distillation instructions, skip to section 10.2.12.

10.2.6 Add 50 mL of 0.25 N NaOH solution to the receiving flasks.

10.2.7 Assemble the cyanide distillation apparatus. Turn on the vacuum source and adjust the flow such that an even stream of air bubbles from the scrubber tube in the collection flask.

10.2.8 Through the inlet tube, add approximately 0.2g of sulfamic acid to all samples and QC. Rinse the inlet tube with a small amount of reagent water and allow the samples to mix for three minutes.

10.2.9 Add 2.5mLs of concentrated sulfuric acid. Rinse the inlet tube with a small amount of reagent water and allow the samples to mix for three minutes. Add 2.0mL magnesium chloride solution and rinse the inlet tube with a small amount of reagent grade water.

10.2.10 Turn on the cooling water. Set the Westco controller to Program 1 to begin heating. Heat the sample to boiling, adjust the vacuum on each sample to keep the air flow steady without overflowing the collection flask. The samples are distilled/refluxed for 1.5 hours after boiling begins. Be sure to adjust the airflow as necessary. After the heating period, turn off the heat and allow to cool for at least fifteen minutes. Keep the vacuum on until samples are cooled and ready for transfer.

10.2.11 Disconnect the scrubber and drain the sodium hydroxide solution from the collection flask into a 120mL disposable container. Label the container with the sample ID. Proceed to section 10.3 for analysis.

10.2.12 For the SimpleDist® procedure, set the temperature of the heating block to 120^{o}C and allow the heat to stabilize for approximately 30 minutes.

10.2.13 Add 2.0 mL of 0.25N NaOH solution to the “Measure” end of the collection tube for each sample and QC. Cap the tube with 1 membrane and cap. Ensure that the membrane is not torn and shows a visible edge all the way around the tube before adding the cap.

10.2.14 Add 6 mL of sample or QC to each sample tube.

NOTE: An ID may be marked on the tube with permanent ink, but labels cannot be placed on the sample tubes, as this will interfere with loading them in the block.

10.2.15 Add 0.10mL of sulfamic acid solution (7.3.2) and swirl gently to mix.

10.2.16 Add 0.75 mL of sulfuric acid/magnesium chloride solution (7.3.3) to THE FIRST sample tube. Immediately place the "Distill" end of a filled and capped collection tube (10.2.13) over the open end of the sample tube. Press the 2 pieces together to form a rough fit. You will not be able to hand press a tight seal.

10.2.17 Place the distillation unit in the tube press. Hold the collection tube by the break joint (indentation in the center) to give support during the sealing procedure. Firmly press the two pieces together with one motion. The collection tube should be flush with the ring around the edge of the sample tube. If the pieces are not flush, or at a visible angle, repeat the procedure with a fresh portion of sample starting at step 10.2.14.

10.2.18 Repeat until all samples and collection tubes are connected.

10.2.19 Place each assembled and sealed distillation units into the pre-heated block. Ensure that the unit is completely inserted in the block by having the ring on the "Distill" side of the tube flush against the surface of the block.

10.2.20 Distill samples for 45 minutes.

10.2.21 Heat and chemical resistant gloves must be worn for the following steps.

10.2.22 Remove ONE unit from the heating block. Immediately hold it over a disposal container and pull the sample tube off of the collection tube. This must be done quickly, or the distillate could suck back into the sample tube. If this occurs, the distillation process must be started over.

10.2.23 Dispose of the remaining sample liquid in an acid waste container.

10.2.24 Turn the collection tube so that the capped side is down, roll the collected solution around the sides of the vial to collect any condensate. Allow the tubes to cool for approximately 30 minutes, then snap the tube at the break joint.

10.2.25 Bring the distillate up to 6mL final volume with 0.25N NaOH solution. The tube may be capped with parafilm if the analysis will not be done immediately.

10.3 Automated Analysis by Seal AQ2

10.3.1 Open ASQ2 shortcut.

10.3.2 Turn instrument on using the large switch located on the back. Let the instrument warm up for at least 1 hour before analyzing samples. Make sure that the waste container is empty and the DI water container is full.

10.3.3 Add reagents to the reagent wells. Make sure the reagent wedges are full, but DO NOT over-fill. (The autosampler will stop if reagents are over-filled. In the event that the reagents are not in the reagent wheel, reagent positions can be checked in the software using the Maintenance and Setup tab, and selecting Reagent Sets.

10.3.3.1 Position 10 – 0.50 mg/L Standard (7.5.5.1) – position used for Midi distilled sample analysis only

10.3.3.2 Position 5 – Phosphate Buffer (7.4.2)

10.3.3.3 Position 6 - Chloramine-T Solution (7.4.3)

10.3.3.4 Position 7 – Pyridine-Barbituric Acid Reagent (7.4.4)

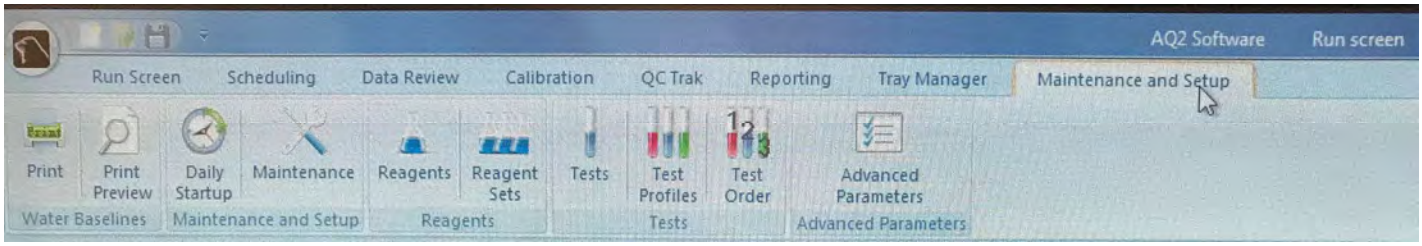
10.3.3.5 Position 8 – 0.25N NaOH solution (7.4.1) – position only used for dilutions for SimpleDist® samples, not blanks

10.3.3.6 Position 9 – 0.10mg/L CCV Standard (7.5.7) – position used for Midi distilled sample analysis only

10.3.4 Check the gold platens. If they are not already pushed in, push them in and tighten the screw knobs (front-left and back-center).

NOTE: If the gold platens are not over-tightened, they do not need to be unscrewed after every run, although it is good practice to do so.

10.3.5 Once the SEAL has been warmed up, select “Daily Start Up” from the Maintenance and Settings tab.



10.3.6 Running the Daily Start Up procedure will wash the cuvette 3times, as well as prime the syringe 10 times and run the water baselines, ensuring the SEAL is ready for daily use.

- Print the water baselines from the Maintenance and Setup tab next t the Daily Startup button.

NOTE: One of the filters should have a value of approximately 8.9. The “Dark” filter should have a value of approximately 0.003. If these numbers are not achieved, perform maintenance or flush the system and repeat.

AQ2 Water Baselines Page 1 of 1

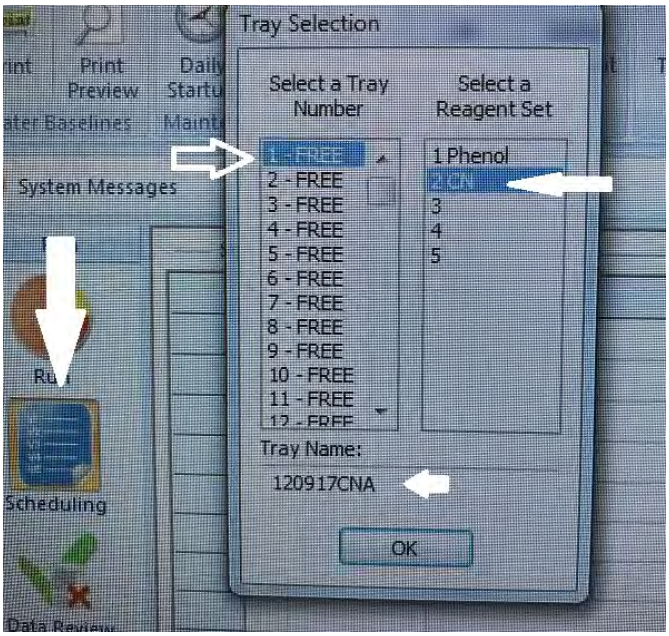
AQ2 Water Baselines

SEAL Analytical

Software Version: 2.1.4
Report Requested By: TestAmerica
Date & Time: 2017-12-09 12:43:27

Filter Number	Filter	Last Value	Current Value
1	405nm	6.2618 v	6.2444 v
2	480nm	6.2695 v	6.2573 v
3	505nm	6.4503 v	6.4424 v
4	520nm	8.4706 v	8.4430 v
5	578nm	7.4736 v	7.4597 v
6	660nm	7.3852 v	7.3753 v
7	880nm	7.1980 v	7.1901 v
8	Dark	0.0052 v	0.0049 v

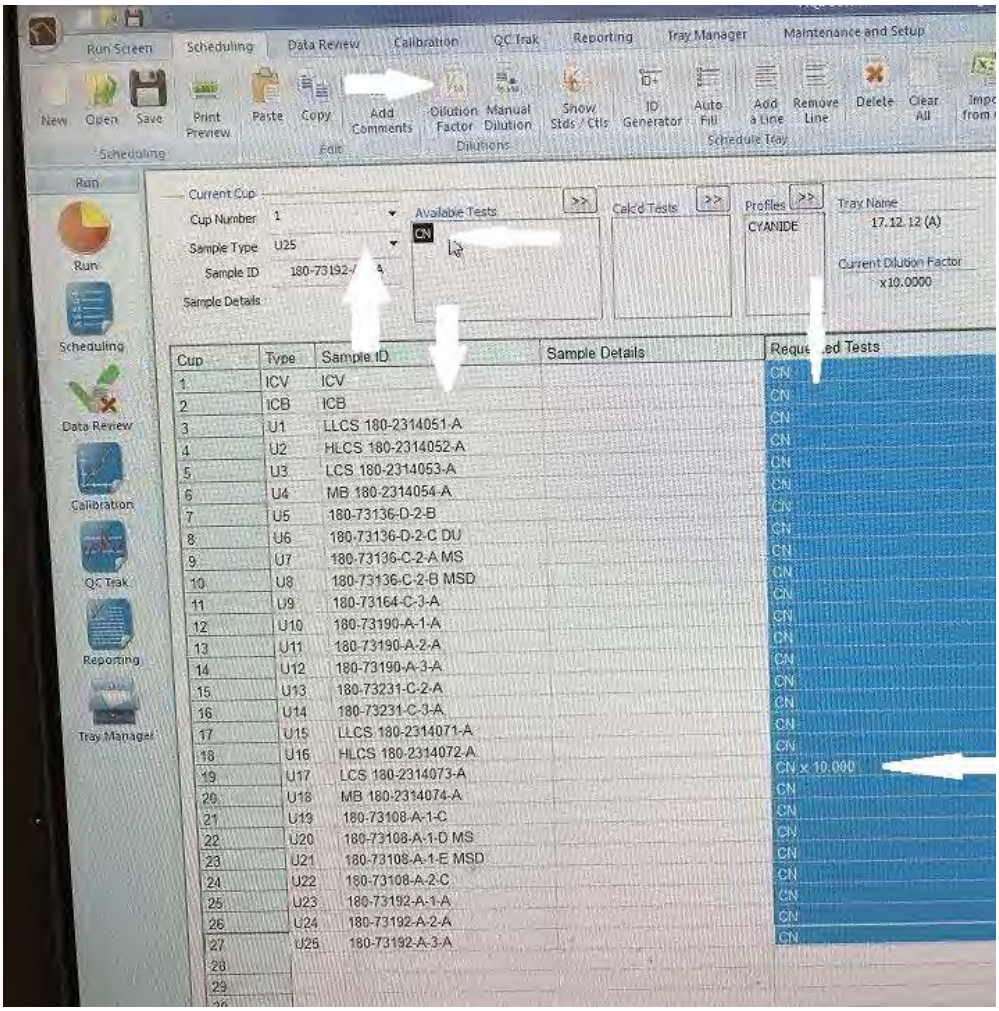
10.3.7 Click on the Scheduling icon. Choose the first "FREE" tray and select the CN reagent set. Name the schedule by using the day's date and "CAN" (or B, C, etc).



10.3.8 In the Scheduling table, highlight the first row in the Sample ID column and select ICV from the drop down menu. Move to the next row and select ICB from the drop down menu. Scan in the samples from the prep batch information sheet directly below the ICB.

10.3.9 Highlight all entry rows and click on CN in the Available Tests box.

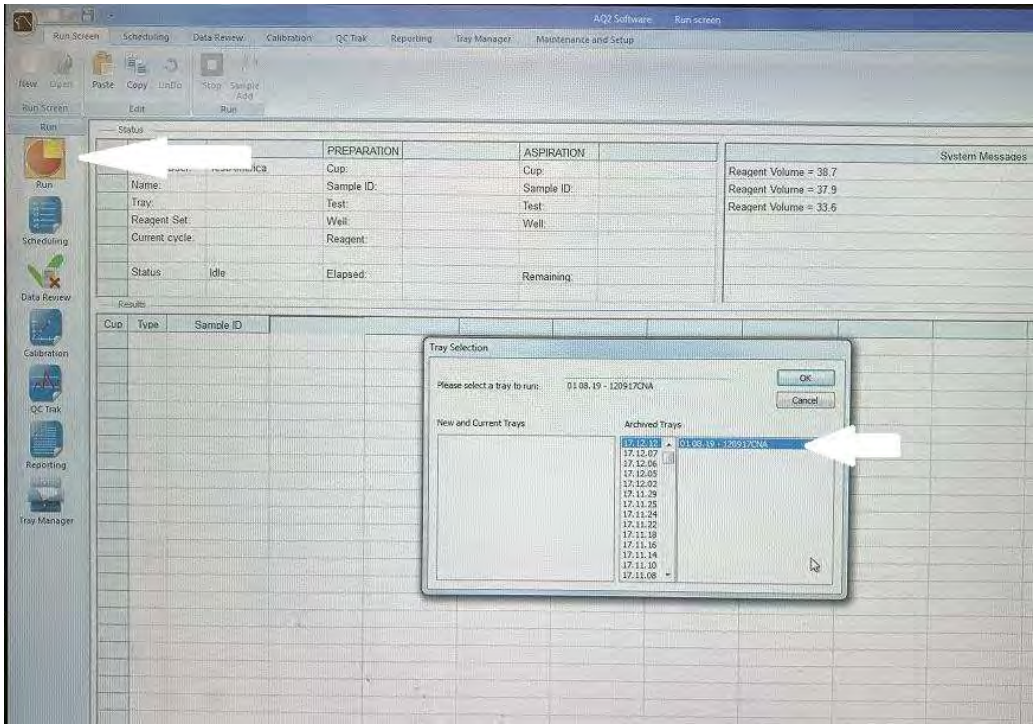
10.3.10 If a dilution is necessary, for the solid LCS for example, click on the sample in the tray. Click the Dilution Factor icon at the top of the screen and select the appropriate dilution from the menu and click OK. Click the test name under Available Tests twice to apply the dilution.



10.3.11 Save the schedule by date: <<121219CNA>>

10.3.12 Add autosampler cups to the tray according to the schedule. Using disposable transfer pipettes, add samples into the sample cups that correspond to their spot on the tray. Load the tray onto the instrument.

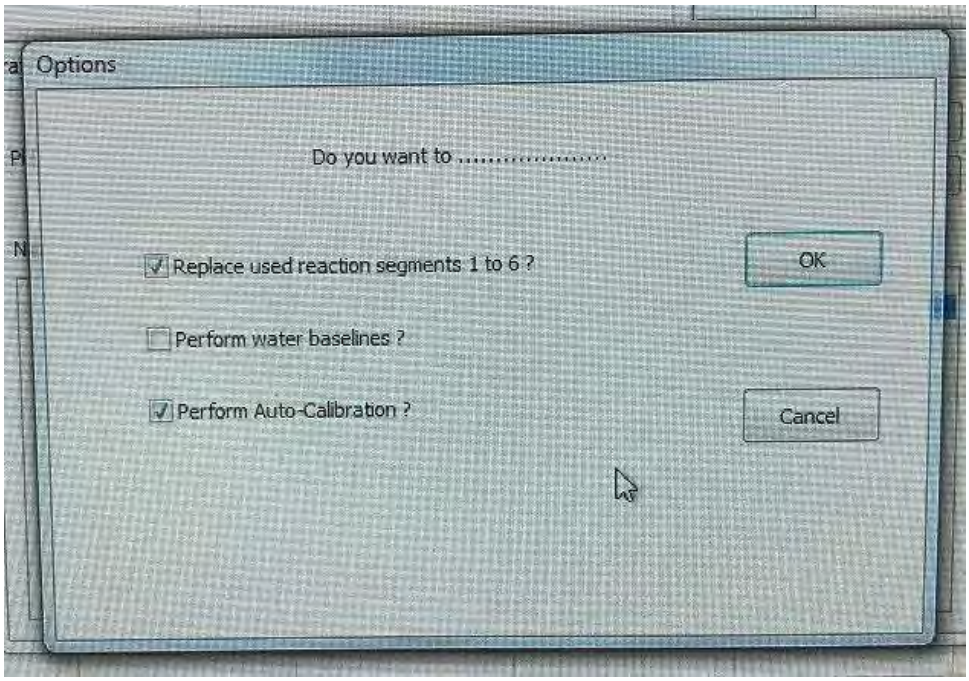
10.3.13 Click on the Run icon. Choose the schedule that was just saved, and click OK.



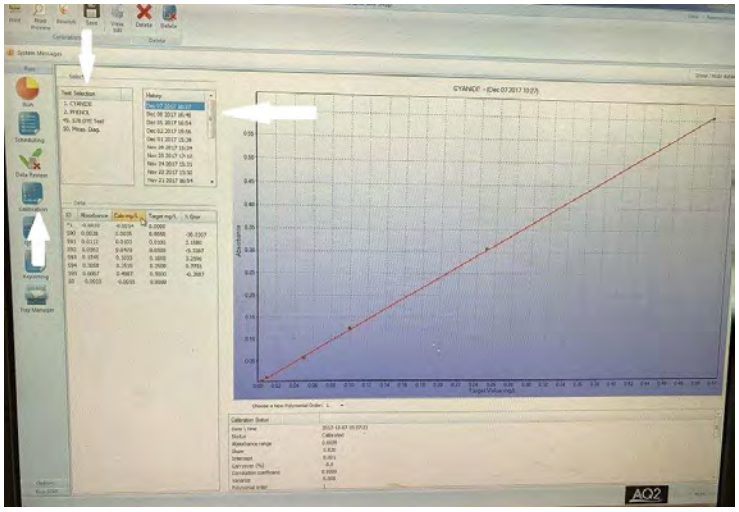
10.3.14 A pop-up box will appear with three options. Select Options 1 and 3 for Midi distilled sample analysis (Select only option 1 for SimpleDist® distilled samples) and click OK.

10.3.14.1 For SimpleDist® distilled sample analysis, the calibration standards, ICV/CCV and ICB/CCB are set up in the schedule using the distilled standards in individual sample cups, rather than a single position per section 10.3.3.

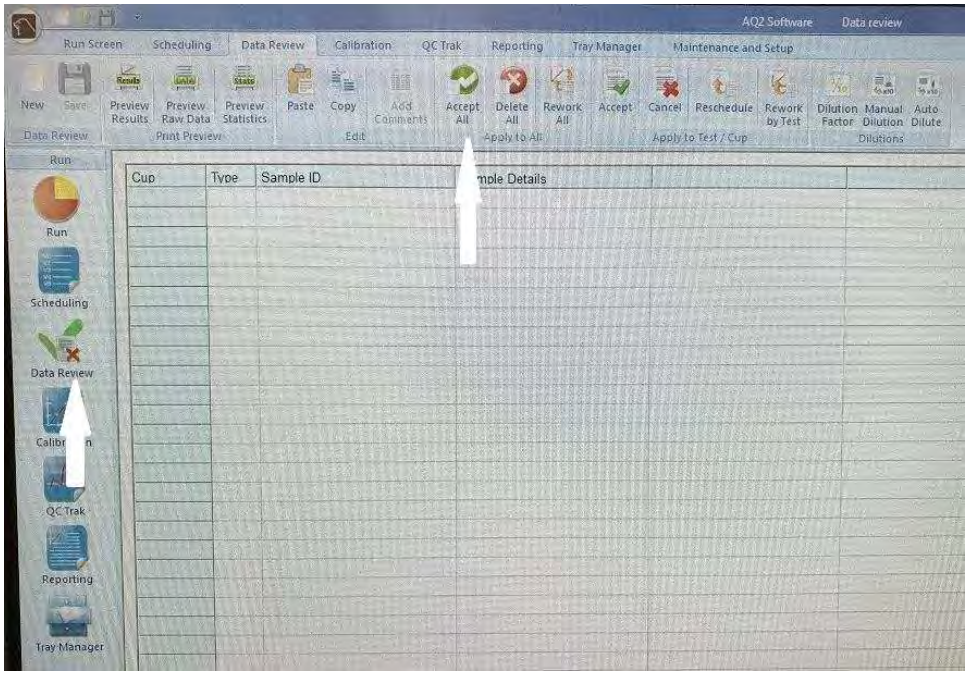
Calibration Standards are entered into the schedule as SS, S2, S3, S4, S5, S6, and S7, followed by ICV and ICB. The CCV and CCB must be put into the schedule in their required place every 10 samples.



10.3.15 Check the Calibration curve as soon as possible after the curve has completed running. Click the Calibration icon on the left hand side of the screen. Select CYANIDE and the date of the calibration. Review the curve according to the acceptance criteria.



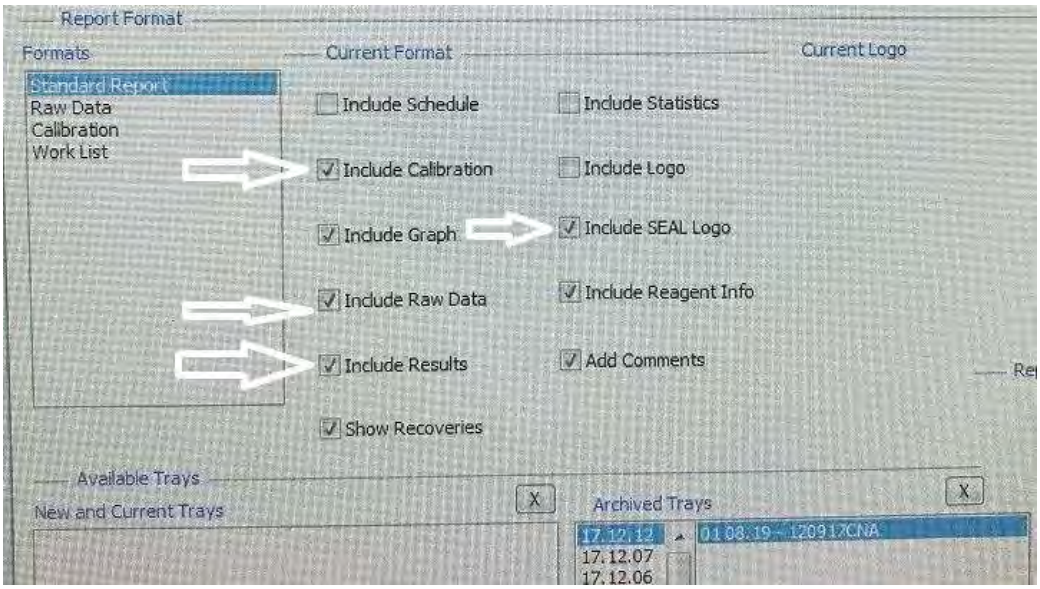
10.3.16 When the analysis of the tray is complete, select Data Review from the left hand side of the screen. Choose the tray ID from the pop-up box and select OK. Review the run results. Click the Accept All icon. Choose Yes to keep the run, rename the file, and click save.



10.3.17 Click on the Reporting icon from the left menu on the Run screen.



10.3.18 From the pop-up box, choose to include Calibration, Raw Data, Results, and Logo. Highlight the file under the New and Current Tray column and click on Chronological Order Report. When the report appears, click Print.



10.3.19 When the run is finished and the report printed, open the Maintenance window. Choose Wash the Cuvette and click on Extra Wash. Repeat once.

10.3.20 Loosen the screw knobs (front-left and back-center) for the gold platens. Remove reagents, empty the waste container and dispose of the sample left in the autosampler cups.

10.3.21 Logout of the software, and turn the instrument off – large switch and then small switch.

11. CALCULATIONS / DATA REDUCTION

11.1 Total Cyanide concentrations are calculated by the instrument software by comparing sample peak heights with the calibration curve. The calibration curve is constructed by the software by plotting the standard concentrations vs peak heights, using linear regression. Conversion of results to µg/L is accomplished, where necessary, by multiplying results in mg/L by 1000.

For water samples: $C = A \times DF \times FV/IV$

Where:

C	=	Sample concentration in mg/L
A	=	Concentration from calibration curve in mg/L
DF	=	Dilution factor
FV	=	Final volume of distillate in mL
IV	=	Initial volume used for distillation in mL

For soil samples: $C = \frac{A \times DF \times FV}{IW \times S}$

Where:

C	=	Sample concentration in mg/kg, dry weight
A	=	Concentration from calibration curve in mg/L
DF	=	Dilution factor
FV	=	Final volume of distillate in mL
IW	=	Initial weight of sample distilled in g
S	=	Percent solids/100

Note: A Percent Solids determination must be performed on a separate aliquot when dry weight concentrations are to be reported. If the results are to be reported on a wet weight basis, the "S" factor should be omitted from the above equation. The "S" factor is not used in the calculation of results for tissue samples.

For wipe samples: $C = A \times DF \times FV \times 1000$

Where

C	=	Sample concentration in ug/wipe
A	=	Concentration from calibration curve in mg/L
DF	=	Dilution factor
FV	=	Final volume of distillate in L

11.2 Percent Recovery (%R) Calculation:

$$\%R = \frac{\text{observed value}}{\text{true value}} \times 100\,\%$$

11.3 The matrix spike and analytical spike percent recovery calculation:
MS Percent Recovery:

$$\text{MS \% Recovery} = \left(\frac{\text{Observed Conc. of MS} - \text{Conc. of Smp}}{\text{True Spike Conc.}} \right) \times 100\,\%$$

11.3.1 When sample concentration is less than the instrument detection limit, use 0 for sample results only for purposes of calculating percent recovery.

11.3.2 The units for reporting spike sample results will be identical to those used for reporting sample results (µg/L for aqueous and mg/Kg dry weight basis for solid).

11.4 Duplicate Sample Relative Percent Difference calculation:

$$RPD = \frac{|X_1 - X_2|}{\left(\frac{X_1 + X_2}{2}\right)} \times 100\,\%$$

Where:

X1 = Original Result
X2 = Duplicate Result

12. METHOD PERFORMANCE

12.1 The supervisor has responsibility to ensure that an analyst who performs this procedure is properly trained in its use and has the required experience. Performance is monitored through internal QC and outside performance evaluation samples. Please refer to the QA Manual for additional information concerning Precision and Accuracy.

12.2 Demonstration of Capabilities – Prior to the analysis of samples, a Demonstration of Capabilities (DOC) as described in the QA Manual, must be performed initially, annually and any time a significant change is made to the analytical system.

12.3 Method Detection Limit Study – A Method Detection Limit (MDL) study, as described in Detection Limits SOP PT-QA-007, must be performed initially and any time a significant

change is made to the analytical system. The MDL must be evaluated at least every 12 months.

12.3.1 Two LOQ verification standards shall be analyzed on each instrument every quarter. These standards may be used as the quarterly MDL points if analyzed at or below the RL.

12.4 Method 335.4 also requires a Linear Range Confirmation to be run every 6 months.

13. POLLUTION CONTROL

13.1 It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage and reagent stability). Employees must abide by the policies in Section 13 of the Corporate Environmental Health and Safety Manual (CW-E-M-001) for "Waste Management and Pollution Prevention" and PT-HS-001, Pittsburgh's Facility Addendum EH&S Manual.

13.2 This method does not contain any specific modifications that serve to minimize or prevent pollution.

14. WASTE MANAGMENT

14.1 Waste management practices are conducted consistent with all applicable rules and regulations. Excess reagents, samples and method process wastes are disposed of in an accepted manner. Waste description rules and land disposal restrictions are followed. Waste disposal procedures are incorporated by reference to PT-HS-001. The following waste streams are produced when this method is carried out.

14.1.1 Filter paper contaminated with bismuth, lead or cadmium sulfide: This waste is collected in waste containers identified as "Soils with Metals", waste stream #29.

14.1.2 Aqueous rinsates from distillation tube clean up: This waste is placed in containers identified as "Acid Waste", Waste #33. It is neutralized to a pH between 6 and 9 and disposed down a lab sink.

14.1.3 Unused sample distillates: This waste is placed in waste containers identified as "Sodium Hydroxide / Pyridine Waste", Waste #1.

14.1.4 Miscellaneous solid waste contaminated with sample residue, acids, caustics and reagents used in this SOP: This waste is placed in a trash container and disposed with regular trash.

14.1.5 Aqueous analytical waste, neutral to slightly basic, contaminated with 3% pyridine: This waste is placed in waste containers identified as “Sodium Hydroxide / Pyridine Waste”, Waste #1.

14.1.6 Aqueous alkaline material from the auto-analyzer/titrations: This waste is placed in waste containers identified as “Sodium Hydroxide / Pyridine Waste”, Waste #1.

15. REFERENCES / CROSS-REFERENCES

15.1 SW846, Test Methods for Evaluating Solid Waste, Third Edition; Total and Amenable Cyanide Distillation; Method 9010C, Revision 3, November 2004

15.2 SW846, Test Methods for Evaluating Solid Waste, Third Edition; Total and Amenable Cyanide, Automated UV; Method 9012 B, Revision 2, November 2004

15.3 SW846, Test Methods for Evaluating Solid Waste, Third Edition: Titrimetric and Manual Spectrophotometric Determinative Methods for Cyanide; Method 9014, Revision 1, July 2014

15.4 Determination Of Total Cyanide By Semi-Automated Colorimetry, Method 335.4, Revision 1, August 1993; Environmental Monitoring Systems Laboratory, Office Of Research And Development, U.S. Environmental Protection Agency, Cincinnati, Ohio 45268

15.5 Standard Methods for the Examination of Water and Waste Water, Method 4500-CN^- C, Total Cyanide after Distillation and E, Colorimetric Method, 1999, editorial revision 2011

15.6 SimpleDist® Micro Heating Block Operating & Instruction Manual, Environmental Express, 1065EE2_M Revision 2, August 2016

15.7 PT-QA-024, Subsampling

15.8 PT-QA-006, Procurement of Standards and Materials: Labeling and Traceability

15.9 PT-QA-016, Nonconformance & Corrective Action System

15.10 PT-QA-021, Quality Assurance Program

15.11 PT-QA-022, Equipment Maintenance

15.12 PT-QA-M-001, Laboratory Quality Assurance Manual

15.13 PT-HS-001, Pittsburgh Facility Addendum to TestAmerica Corporate Environmental to TestAmerica Corporate Environmental Health & Safety Manual (CW-E-M-001)

15.14 Seal AQ2 User's Guide Revision 1.1 and AQ2 Series Software version 4

15.15 PT-QA-031, Internal Chain of Custody

15.16 PT-QA-007, Detection Limits

16. METHOD MODIFICATIONS

16.1 According to Method 335.4, section 10.5, calibration standards are to be analyzed in order of decreasing concentration. TestAmerica Pittsburgh will analyze the calibration curve in order of increasing standard concentration for all methods referenced.

16.2 The initial calibration curve concentrations given in SW846 method 9012B section 7.4.1 and 9014 section 7.3.1 are replaced with a shorter range curve in section 7.5.5 of this SOP.

16.3 Sample pre-treatment sulfide by method SM4500-CN^- C using Lead Carbonate is performed using Cadmium Carbonate by this SOP (from method 335.4).

16.4 The sample duplicate required by section 8.4 of method 9010C may be replaced by the performance of an MS and MSD, since CN is not often detected in samples and the MS/MSD provides a better chance of measuring the method precision.

16.5 A matrix spike concentration of 200 ug/L is used for all methods, instead of the 40 ug/L mentioned in section 8.5 of method 9010C/9012B.

16.6 The SimpleDist® distillation procedure uses proportional reduced volumes of the reagents required by method 335.4 and 9010C. The SimpleDist® distillation time has been reduced to 45 minutes per the reduced volume and manufacturer's instructions.

17. ATTACHMENTS

17.1 Figure 1 – SimpleDist® Set-up Procedure

18. REVISION HISTORY

18.1 Revision 11, 07/14/2008
18.2 Revision 12, 09/10/2009
18.3 Revision 13, 06/03/2010
18.4 Revision 14, 08/03/2011
18.5 Revision 15, 09/26/2012

18.6 Revision 16, 01/29/2015

18.7 Revision 17, 12/23/2015

18.8 Revision 18, 12/18/2017

18.9 Revision 19, 10/29/2018

18.10 Revision 19.1, 5/14/2019

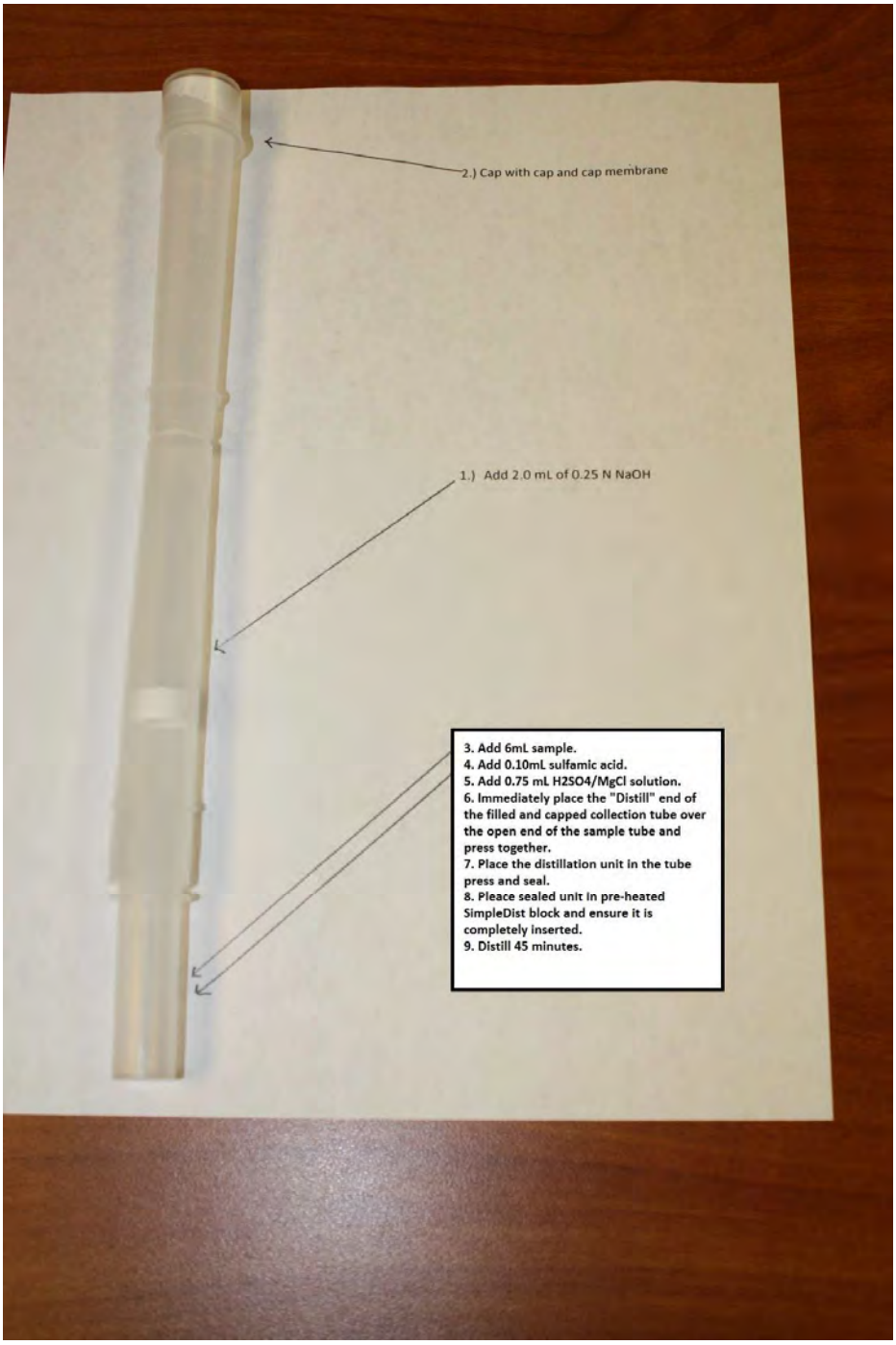
18.11 Revision 20, 10/8/2019

18.12 Changes to current revision

SOP section	Change from	Change to	Reason
1.2	RL of 0.005 mg/L	RL of 0.010 mg/L	Correction
4.2		Added or ascorbic acid	Option
4.3	Removed bismuth nitrate		Lab not using
5.6 Table	Removed bismuth nitrate	Added ascorbic acid, chloramine-T and barbituric acid	Correction
6.2		Added requirement to verify SimpleDist tube volume	Clarification
7.2.7	Removed bismuth nitrate		Correction
7.2.9		Added ascorbic acid	Option
7.5.5.1		Added Calibration Preparation Standard 0.5 mg/L	Clarification
7.5.5.2	Removed 0.005 mg/L standard	Added 0.25 mg/L standard and updated standard preparation volumes for 6mL used for SimpleDist procedure	Correction
7.5.5.3		Added “for Midi distillation”	Clarification
7.5.6 & 7.5.7		Added “Not distilled for Midi”	Clarification
9.2.2	0.5g soil for SimpleDist	0.2g	Correction
9.9.1 bullet 1	0.06mL for 0.10mg.L spike	0.12mL for 0.20mg/L spike	Correction
10.1.2	removed		Bismuth nitrate not used
10.2.1.1	Dropwise 5N NaOH	1ml of 1N NaOH	Correction
10.2.1.2		Added or 0.06g ascorbic acid	Option
10.2.1.2 & 10.2.1.3		Added if any samples treated, MB & LCS must have treatment added	Correction

10.2.4	0.5g soil for SimpleDist	0.2g	Correction
10.2.20, 16.6	90 minutes	45 minutes for SimpleDist	Correction
10.2.24		Add to let samples cool 30 minutes	Correction
10.3.3		Corrected section 7 references	Correction
10.3.3.1 & 10.3.3.6		Added positions used only for Midi distilled sample analysis	Clarification
10.3.14		Added only option 1 checked for SimpleDist distilled sample analysis	Clarification
10.3.14.1		Added for SimpleDist sample analysis standards placed in schedule	Clarification
12.3 & 15.6	CA-Q-S-006	PT-QA-007	Correction
16.2	Reference to SOP 7.4.5.1	7.5.5	Correction
16.5	Spike 0.1 mg/L	Spike 0.2 mg/L	Correction

Figure 1 – SimpleDist® Distillation Procedure



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Title: Ignitability of Solids for Waste Characterization

Methods: SW-846 Chapter 7, Section 7.1

Approvals (Signature/Date):

Roseann Ruyechan Inorganics Department Manager	12/11/2019 Date	Steve Jackson Regional Safety Coordinator	12/10/2019 Date
Virginia Zusman Quality Assurance Manager	12/10/2019 Date	Deborah L. Lowe Laboratory Director	12/30/2019 Date

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1. SCOPE AND APPLICATION

1.1. This method is applicable to the determination of ignitability of solids as it pertains to waste characterization outlined in Chapter 7 of EPA SW846. It is used to identify wastes that either present fire hazards under routine storage, disposal and transportation or are capable of "severely exacerbating" a fire that is already burning.

1.2. There is no instrument or method detection limit applicable to this test. Results are reported qualitatively as either "Yes" or "No" depending on the ignitability of the sample.

1.3. Applicable Matrices: Solids, including domestic and industrial wastes, sludges, and petroleum wastes.

1.4. The analytical time depends on the matrix and method of analysis. For solids, the analytical time is approximately 5 minutes.

1.5. On occasion clients may request slight modifications to this SOP. Any modifications to this procedure must be approved by the QA department or the Laboratory or Technical Director and documented fully in an NCM that accompanies the analytical batch.

2. SUMMARY OF METHOD

2.1. Solids are analyzed by exposure to open flame for a set length of time after which the flame is removed and the sample observed to determine its ignitability characteristic. If the material will burn, a subsequent set of tests determine ignitability according to the definitions in Chapter 7 of SW-846.

3. DEFINITIONS

3.1. Ignitability - The capability of a solid sample, under standard conditions of temperature and pressure, to cause fire through friction, moisture absorption, or spontaneous chemical changes and, when ignited, to burn so vigorously and persistently as to create a hazard.

3.2. TALS – TestAmerica Laboratory Information Management System

3.3. NCM - Non-Conformance Memo – a system within TALS for the lab to communicate to project management and others when there is an anomaly seen with the samples or batch, or a QC failure.

3.4. Refer to the glossary in the Laboratory Quality Assurance Manual (PT-QA-M-001) for general laboratory definitions.

4. INTERFERENCES

4.1. Improper storage of samples may cause loss of volatiles and lead to erroneous results.

5. SAFETY

5.1. Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual (CW-E-M-001), the Pittsburgh Facility Addendum EH&S Manual (PT-HS-001) and this document. This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

5.2. Because of the unknown reactivity of the materials being tested, the sash of the hood should be lowered to shield the operator. As an alternative, the operator may use a face shield if using a sash is not possible.

5.3. In the event a sample ignites in the test apparatus do not attempt to remove the sample. Turn off the apparatus and flame. The flame should go out when the cup is closed. If this does not happen the flame may be extinguished by covering the sample with a non-flammable material. After the apparatus has cooled the sample may be removed.

5.4. Eye protection that protects against splash and appropriate gloves must be worn while samples, standards, solvents, and reagents are being handled. Cut resistant gloves must be worn doing any other task that presents a strong possibility of getting cut. Disposable gloves that have been contaminated will be removed and discarded; other gloves will be cleaned immediately.

NOTE: In addition to safety glasses a face shield should also been worn when performing this procedure. The hood sash should also be pulled down to act as an additional barrier when performing this procedure.

5.5. There are no materials used in this method that have a serious or significant hazard rating.

5.6. Exposure to chemicals must be maintained **as low as reasonably achievable**, therefore, unless they are known to be non-hazardous, all samples must be opened, transferred and prepared in a fume hood, or under other means of mechanical ventilation. Solvent and waste containers will be kept closed unless transfers are being made.

5.7. In order to stabilize conditions during the actual test, the fume hood when the test is being conducted may be turned off.

5.8. All work must be stopped in the event of a known or potential compromise to the health and safety of a TestAmerica's associate. The situation must be reported **immediately** to a laboratory supervisor and/or the EH&S Coordinator.

6. EQUIPMENT AND SUPPLIES

The following items are recommended for performing this procedure. Equivalent items should only be used when they result in an improvement in quality, efficiency, productivity, or cost. An item can be considered equivalent if with its use, the analytical and QA/QC requirements in this SOP can be met.

6.1. Ceramic crucibles, 50 mL capacity

6.2. Source of open flame: "barbecue" butane lighter, or equivalent. **NOTE:** a butane cigarette lighter, short wooden matches, or a high temperature propane torch are **NOT** an acceptable alternative. The flame source must allow the analyst to remain away from the sample.

7. REAGENTS AND STANDARDS

7.1. There are no reagents and standards required for this method.

8. SAMPLE COLLECTION, PRESERVATION, SHIPMENT AND STORAGE

8.1. Sample container, preservation techniques and holding times may vary and are dependent on sample matrix, method of choice, regulatory compliance, and/or specific contract or client requests. Listed below are the holding times and the references that include preservation requirements.

Matrix	Sample Container	Minimum Sample Size	Preservation	Holding Time
Solid waste, sludges, petroleum waste	Glass	10 g	>0.0˚C but ≤ 6.0 ℃	None

NOTE: No chemical preservation is required or appropriate

9. QUALITY CONTROL

9.1. Sample/Sample Duplicate (DUP) - One Sample/Sample Duplicate pair must be processed for each QC batch of 20 samples. The QC batch is defined as a set of up to 20 field samples plus associated laboratory QC samples that are similar in composition (matrix) and that are processed within the same time period using the same reagent and standard lots.

NOTE: All samples that are YES for ignitability are analyzed in duplicate for confirmation purposes.

9.1.1. The results of the DUP pair are used to determine analytical variability.

9.1.2. The DUP pair is evaluated qualitatively. If both the sample and its duplicate are found to be either both ignitable (YES/YES) or both not ignitable (NO/NO), the QC is considered in control.

9.1.3. The DUP QC is out of control if the two results are different (YES/NO). In this case, the data must be qualified appropriately (e.g. sample heterogeneity) and an explanation provided in the report narrative.

9.2. There are no other QC analyses applicable to this test.

10. PROCEDURE

10.1. This method has no calibration or standardization requirements.

10.2. Any significant variation in procedure shall be completely documented using an NCM.

10.3. In a fume hood, place a small quantity of sample (approximately 1 gram) into the ceramic crucible. It is not necessary to weigh the sample, but a small quantity should be used to minimize any potential hazards.

10.4. Wear a face shield in addition to safety glasses, or pull the hood sash down to act as a shield. Turn off the hood (if necessary to maintain a stable flame), touch the ignition source (see section 6.2) to the sample. Keep the flame in contact with the sample for 5 ± 2 seconds.

10.5. Remove the ignition source and observe the sample.

10.6. Observations can be reduced to three broad categories:

Reaction of Sample	Classification
Sample does **not** ignite	Not ignitable, no further testing necessary
Sample ignites but burns only in contact with flame	Not ignitable, no further testing necessary
Sample ignites and burns continuously	Proceed to section 10.6.1

10.6.1. Wear a face shield in addition to safety glasses, or pull the hood sash down to act as a shield for the following steps. Place approximately 5g of solid into a crucible and stir for 10 seconds. If the sample ignites and burns continuously, then it is classified as ignitable. If not, proceed to section 10.6.2

10.6.2. Add 5 mL of water to the sample in the crucible. If the sample ignites and burns continuously, then it is classified as ignitable. If not, proceed to section 10.6.3.

10.6.3. Add approximately 5g of solid to a crucible. Heat the crucible from underneath with a burner for 30 seconds. If the sample ignites and burns continuously, then it is classified as ignitable. If not, the sample is classified as not ignitable.

11. DATA ANALYSIS AND CALCULATIONS

11.1. If the result is “Ignitable”, report as “Yes”.

11.2. Samples which do not meet the “Ignitability” criteria should be reported as “No.”

11.3. The sample information is directly entered into the TALS Batch Results.

12. METHOD PERFORMANCE

12.1. The supervisor has responsibility to ensure that an analyst who performs this procedure is properly trained in its use and has the required experience. Performance is monitored through internal QC.

12.2. Demonstration of Capability – Prior to the analysis of samples, a Demonstration of Capability (DOC) as described in the QA Manual must be performed.

12.3. Method Detection Limit Study does not apply to this procedure.

13. POLLUTION CONTROL

13.1. All waste will be disposed of in accordance with Federal, State and Local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this method and the policies in section 13 of the Corporate Safety Manual for "Waste Management and Pollution Prevention" and the Pittsburgh Facility Addendum EH&S Manual (PT-HS-001).

13.2. This method does not contain any specific modifications that serve to minimize or prevent pollution.

14. WASTE MANAGEMENT

14.1. The following waste streams are produced when this method is carried out.

14.2. Excess non-flammable samples. This waste is placed in waste containers identified as "Mixed Flammable Solvent Waste", Waste #3.

14.3. Excess flammable samples and waste solvents. This waste is placed in waste containers identified as "Mixed Flammable Solvent Waste", Waste #3.

15. REFERENCES

14.4. Characteristics Introduction And Regulatory Definitions, Chapter 7, Section 7.1 Ignitability, Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, EPA SW846, 3rd edition, Revision 4, November 1994

14.5. Pittsburgh Laboratory Quality Assurance Manual, PT-QA-M-001

14.6. PT-QA-008, Thermometer Calibration and Temperature Monitoring

14.7. PT-QA-016, Non-Conformance & Corrective Action System

14.8. PT-QA-021, Quality Assurance Program

14.9. PT-HS-001, Pittsburgh Facility Addendum EH&S Manual

14.10. PT-QA-031, Internal Chain of Custody

15. METHOD MODIFICATIONS

15.1. None

16. ATTACHMENTS

16.1. None

17. REVISION HISTORY

17.1. Revision 2, 7/21/08

17.2. Revision 3, 3/2/2011

17.3. Revision 4, 5/6/2013

17.4. Revision 5, 9/28/2015

17.5. Revision 6, 9/27/2017

17.6. Revision 6.1, 5/14/2019

17.7. Changes to current revision

SOP section	Change from	Change to	Reason
1.5		Added modifications must be approved and documented	Clarification
3.3		Added definition of NCM	Clarification

Title: Electrometric pH

Method(s): SW846 Methods 9040B/C and 9045C/D and Standard Method 4500-H^+B

Approvals (Signature/Date):

10/12/2020	9/18/2020
Roseann Ruyechan Date	Steve Jackson Date
Inorganics Department Manager	Regional Safety Coordinator
9/17/2020	10/21/2020
Virginia Zusman Date	Deborah Lowe Date
Quality Assurance Manager	Laboratory Director

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1. SCOPE AND APPLICATION

1.1. This method is applicable to the determination of pH in waters, wastewaters, solids and wastes. It is based on SW846 Methods 9040 B & C and 9045 C & D and Standard Method 4500-H^+B. The working range is 1 - 14 pH units.

1.2. For pH in waste, sediment, or sludge, if water is present at < 20% of the total sample volume, by visual estimate, the sample is treated as a solid, and method 9045C or D is used. If water is present at > 20% of the total volume, the sample may be analyzed using method 9040B or C.

1.3. On occasion clients may request slight modifications to this SOP. Any modifications to this procedure must be approved by the QA department or the Laboratory or Technical Director and documented fully in an NCM that accompanies the analytical batch.

2. SUMMARY OF METHOD

2.1. The pH is determined electrometrically. The pH meter is calibrated with a series of known pH buffers. The impact of the actual sample temperature is electronically compensated for using Automatic Temperature Compensation (ATC).

NOTE: The use of ATC does not adjust the pH reading at the current sample temperature to what it would be at 25°C. If the pH at a specified temperature, such as 25°C, is required, the sample must be warmed/cooled in a water bath before analysis.

2.2 The analysis may be performed by the Autotitrator for clean water samples.

3. DEFINITIONS

3.1. TALS – TestAmerica Laboratory Information Management System

3.2. NCM – Non-Conformance Memo - a system within TALS for the lab to communicate to project management and others when there is an anomaly seen with the samples or batch, or a QC failure.

3.3. Refer to the glossary in the Laboratory Quality Assurance Manual (PT-QA-M-001) for additional definitions.

4. INTERFERENCES

4.1. Samples with very low or very high pH may give incorrect readings on the meter. For samples with a true pH of >10, the measured pH may be incorrectly low. This error can be minimized by using a low-sodium-error electrode.

4.2. Extreme temperatures can affect the pH reading even with the automatic temperature compensation. Samples should be analyzed near room temperature.

4.3. Coatings of oily material or particulate matter can impair electrode response. These may be removed by gentle wiping or detergent washing the electrode followed by a deionized water rinse. Treatment with 1:10 HCl may be necessary to remove the film, followed by a deionized water rinse.

4.3.1. Samples may not be filtered to remove particles prior to analysis.

5. SAFETY

5.1. Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual (CW-E-M-001), the Pittsburgh Facility Addendum EH&S Manual (PT-HS-001) and this document. This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

The following is a list of the materials used in this method, which have a serious or significant hazard rating. This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the SDS for each of the materials listed in the table. A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the SDS for each material before using it for the first time or when there are major changes to the SDS.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Hydrochloric Acid	Corrosive Poison	5 ppm-Ceiling	Inhalation of vapors can cause coughing, choking, inflammation of the nose, throat, and upper respiratory tract, and in severe cases, pulmonary edema, circulatory failure, and death. Can cause redness, pain, and severe skin burns. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

5.2. Eye protection that protects against splash, laboratory coat, and appropriate gloves must be worn while samples, standards, solvents, and reagents are being handled. Cut resistant gloves must be worn doing any other task that presents a strong possibility of getting cut. Disposable gloves that have been contaminated will be removed and discarded; other gloves will be cleaned immediately.

5.3. All work must be stopped in the event of a known or potential compromise to the health and safety of a TestAmerica associate. The situation must be reported immediately to a laboratory supervisor or EH&S coordinator.

6. EQUIPMENT AND SUPPLIES

The following items are recommended for performing this procedure. Equivalent items should only be used when they result in an improvement in quality, efficiency, productivity, or cost. An item can be considered equivalent if with its use, the analytical and QA/QC requirements in this SOP can be met.

6.1. pH meter with combination pH electrode and automatic temperature compensation

- Accumet XL15, Accumet AR25, or equivalent

6.2. Snap Top Bottles, 2 oz and 4 oz

6.3. Top loading balance: Capable of accurately weighing ± 0.01 g

6.4. Stir plate and stir bars

6.5. Mechanical Shaker

6.6. Automated analysis option: Man-Tech MT-100 Autotitrator with PC-Titrate™ software and Automax73 autosampler with 50 mL sample tubes

7. REAGENTS AND STANDARDS

The following items are recommended for performing this procedure. Equivalent items should only be used when they result in an improvement in quality, efficiency, productivity, or cost. An item can be considered equivalent if with its use, the analytical and QA/QC requirements in this SOP can be met. Please refer to the SDS prior to the use of any reagent or standard.

7.1. Standards

7.1.1. pH 2, 4, 7, 10 and pH 13 buffers, purchased with certificate of analysis, use manufacturer's expiration date. Store buffers at room temperature.

7.1.2. Second source pH 7 buffer, purchased with certificate of analysis, use manufacturer's expiration date. Store buffer at room temperature.

7.1.3. Fresh buffers are poured from the main container into closed lid cups for daily use.

7.1.4. Concentrated Hydrochloric acid (HCl)

7.1.4.1. 1:10 HCl Cleaning Solution – slowly add 20 mL concentrated HCl to 100 mL of laboratory reagent water in a 200 mL volumetric flask. Mix well and dilute to volume.

7.1.5. Acetone may be used to remove oily substances from the probe, followed by a thorough water rinse.

8. SAMPLE COLLECTION, PRESERVATION, SHIPMENT AND STORAGE

8.1. Samples may be collected in glass or plastic containers.

8.2. This analysis should be performed in the field within 15 minutes of sampling for all matrices. If samples are analyzed in the laboratory, beyond the 15 minutes, results shall be reported with an "HF" qualifier to indicate that the technical holding time was exceeded.

8.3. Samples are to be shipped and stored at >0.0°C but ≤6.0°C. No chemical preservation is required or permitted.

9. QUALITY CONTROL

9.1. A batch is a group of 20 or fewer samples of similar matrix excluding QC samples (LCS, and sample DUP) that are processed together in the same 24 hour period.

9.2. Duplicates - Sample duplicates are performed at a minimum frequency of one per every 10 samples (or fewer) of the same matrix. The %RPD between sample and duplicate must be ≤ 2%.

9.2.1. If the %RPD exceeds 2%, two fresh portions of sample and duplicate must be analyzed until the readings stabilize within the required limit.

9.3. ICV/LCS: second source pH buffer 7 is analyzed after the initial calibration. The pH readings must be 6.95 to 7.05 pH units.

9.3.1. If the ICV reading is outside the range, repeat once with a fresh portion of second source buffer. If the failure repeats, the initial meter calibration must be repeated with fresh buffers. Sample analysis must not proceed until the meter calibration is verified successfully.

9.4. CCV: pH buffer 7, same source as the calibration, is analyzed after every 10 samples and at the end of the sequence. The pH readings must be 6.95 to 7.05 pH units.

9.4.1. If a CCV falls outside the required range, repeat once with a fresh portion of buffer. If the failure repeats, the initial meter calibration must be repeated with fresh buffers. All samples analyzed after the last passing CCV must be reanalyzed.

9.5. Any unauthorized deviations from this procedure must also be documented as a nonconformance, using the appropriate NCM.

10. PROCEDURE

10.1. Calibration

10.1.1. Initial Calibration

10.1.1.1. Refer to the manufacturer's manual for instrument specific calibration steps.

10.1.1.2. The following procedure is applied when calibrating the manual pH meter:

- Prior to taking any pH readings, check that the fill hole at the top of the pH probe is open.
- Rinse the electrodes with reagent water and place in the pH 4.0 buffer. Enter the value (4.0) of the standard into the pH meter using the touch screen. Allow the value to stabilize.
- Rinse the electrodes and place in the pH 7.0 buffer. Enter the value (7.0) of the standard into the pH meter using the touch screen. Allow the value to stabilize.
- Repeat these steps for the 2, 10 and 13 pH buffers.

10.1.1.3. The pH meter must be calibrated daily or each day it is used. The calibration is recorded on the analytical log sheet. The slope of the calibration must meet $\geq$ 95%, or the manufacturer's requirements if tighter. If the manufacturer's slope requirement is not met, the meter

will display an error message. Pour fresh buffer solutions into the cups and repeat the calibration. If this does not resolve the problem, change the KCL solution in the electrode. If still having a problem the probe may need to be changed. Note any needed maintenance in the meter maintenance log, including electrode maintenance.

10.1.1.4. The pH readings must be within 0.05 pH units of each buffer solution value. If not repeat adjustments on successive portions of the two buffer solutions until the readings are within 0.05 pH units (s.u.).

10.1.1.5. The following procedure is applied when calibrating the Autotitrator:

- Empty the waste container and fill the DI Rinse jugs with fresh reagent water daily before beginning calibration / analysis.
- Fill autosampler tubes with fresh 2, 4, 7, 10 and 13 pH buffers. Use one tube per buffer, the instrument will pull multiple aliquots.
- Click load template and select **pH X 3 CAL**
- Click **START**. The meter will proceed with the calibration and will indicate if any of the slope and individual pH readings do not meet criteria.

10.1.1.6. If the pH meter has been turned off, it must be calibrated prior to use.

10.1.1.7. After the calibration the ICV/LCS, a second source pH 7 buffer, is analyzed before sample analysis. The pH readings must be 6.95 – 7.05 pH units. This standard also serves as the LCS.

10.1.2. Calibration Check / Continuing Calibration

10.1.2.1. The CCV, which is a 7 buffer of the same source as the calibration standard, is analyzed after every ten samples to ensure the calibration remains linear.

10.1.2.2. The pH meter must be recalibrated if the buffer is not within 6.95-7.05 pH units. If this range is exceeded, reanalyze all samples analyzed since the last pH buffer that met criteria.

10.1.2.3. If samples of extreme pH are included in the batch (<4 or >10), it may be appropriate to analyze a CCV buffer close to the pH of the samples, in addition to the CCV 7 buffer.

10.2. Sample Preparation:

10.2.1. Waters (Method 4500H+ B or 9040B/C)

10.2.1.1. No preparation necessary for waters and wastewaters.

10.2.1.2. Waters should be allowed to equilibrate to near room temperature prior to analysis. Temperature is recorded on worksheet. The pH meter is equipped with a temperature probe, and calibration is verified quarterly.

10.2.2. Solids (Method 9045C/D)

10.2.2.1. Place 20 g (± 0.5 g) of sample into a beaker or other suitable container. (A sample size of less than 20 g can be used as long as the 1:1 weight to volume relationship is maintained.)

10.2.2.2. Add 20 mL of reagent water and mix for five minutes.

NOTE: In the event a sample absorbs the water, additional water will be added at 5 mL increments until sufficient liquid is obtained to cover the pH probe. An NCM will be written to document this anomaly.

10.2.2.3. Allow sample to stand for one hour to allow the solids to settle out or centrifuge off the aqueous phase for pH measurement.

10.2.2.4. Sediments, sludges and wastes: based upon the nature of the sample matrix, the analyst will use appropriate technical judgment, per section 1.2, and prepare the sample according to either 10.2.1 or 10.2.2

10.3. Sample Analysis

Prior to taking any pH readings, check that the fill hole at the top of the pH probe is open.

10.3.1. Manual Waters

10.3.1.1. Place 2 aliquots of the sample in 2 clean Snap Top bottles using a sufficient volume to cover the sensing elements of the electrode (approximately 20 mL). Allow the pH to stabilize (swirling or stirring may quicken stabilization). Record the pH on the analytical log sheet. Remove the electrodes from the sample. Rinse and gently dab off the electrodes between each measurement. Repeat measurement on successive volumes of sample until values differ by less than 0.1 pH units. Report the last reading that is within acceptance range. Store the electrodes in pH 4 buffer or manufacturer's storage solution when not in use.

10.3.2. Autotitrator Waters

10.3.2.1. Load Template **pH X 3**

10.3.2.2. Click **Add rows** and type in the number of spaces for samples and QC in the batch.

10.3.2.3. Scan in samples and type in QC IDs

10.3.2.4. Click and drag the number of vials down in the vial column.

10.3.2.5. Click and drag the schedule and order number columns down. (Hold CTRL, click on the cell and drag it down to the last cell.)

10.3.2.6. Save the Template as MMDDYYPH, for example 100120PH.

10.3.2.7. Fill autosampler tubes according to the schedule. Use one tube per sample and QC, the instrument will pull multiple aliquots.

10.3.2.8. Click **START**.

10.3.2.9. Example Sequence:

Tap
DI Rinse
LCS
Sample
Sample Duplicate
8 more samples
CCV

-CCV after every 10 samples
-Close with CCV and 2 DI Rinses

10.3.3. Solids (Supernatant)

10.3.3.1. Immerse the pH electrodes in the supernatant layer of the sample - be careful not to stir up solids. Allow pH to stabilize and record it on the analytical log sheet. Remove and rinse the electrodes between each measurement. Store electrodes in pH 4 buffer or manufacturer's storage solution when not in use.

10.3.3.2. If the sample contains oil or other substances that will coat or damage the electrodes, the pH should be analyzed using pH Paper Method if client allows this change.

10.3.4. If the pH of the sample is >12.0, the pH must be measured at 25 ± 1 ºC. Use a water bath set at this temperature to warm a portion of the sample in a closed container.

10.4. Autotitrator Data Upload

10.4.1. To begin the data export to TALS, select "Export" under the "File" option at the top left of the screen of the Water Analysis Historical Data Report. A box will pop up with options for file type and file name:

- Change the File Type by selecting "Fixed Field ASCII File"(.TXT)" from the drop down menu.
- Change the File Name by clicking the ",,," box next to the File Name box. Name the file for the date of the run and analysis type (for example: 102120PH).
- Click "OK" to save the file.

10.4.2. Open the software "ManTech FT" from the PC desktop.

10.4.3. Open the saved data file in .TXT form by clicking the "…" box located to the right of the "Raw Data File" option. Select the appropriate data file and click "OK.

10.4.4. DO NOT CHANGE THE IMPORT PATH.

10.4.5. On the bottom of the ManTech FT software [age, click "Load Preset".

10.4.6. In the box that pops up, select pH.

10.4.7. Be sure to select pH, pH2, pH3, and Temperature, temp2 and temp3 from the Available Analytes list to report all readings.

10.4.8. After verifying that the data series selected are correct, click "Transfer File". A box will pop up to let the user know that the file transfer has been completed.

10.5. Analytical Documentation

10.5.1. All analytical information, results and associated QC, are electronically recorded in TALS as the analysis is performed, including the analytical data from standards and any corrective actions or modifications to the method. The time of the individual sample readings is documented in the TALS batch. (Autotitrator will upload analysis times.)

10.5.2. All standards are logged into the TALS reagent module. All standards are assigned a unique number for identification, and this number is recorded in each analytical batch. Standards entry is reviewed by the supervisor or designee.

10.5.3. Daily meter calibration is documented in the Meter Calibration log, specific to each pH meter. The Autotitrator calibration will be included with the raw data.

11. CALCULATIONS / DATA REDUCTION

11.1. Any sample with a pH < 2 or > 13 will need to have a "cn" qualifier added plus an NCM stating "The pH is estimated because the result is outside the calibrated range of pH 2-13."

12. METHOD PERFORMANCE

12.1. The supervisor has responsibility to ensure that an analyst who performs this procedure is properly trained in its use and has the required experience. Performance is monitored through internal QC and outside performance evaluation samples.

12.2. Demonstration of Capability - Prior to analysis of any client samples, the analyst must demonstrate their ability to successfully analyze samples. This demonstration shall consist of calibrating the pH meter (slope meeting manufacturer's control limit) and analyzing 4 LCS (pH 7) standards. The results of the 4 LCSs must all be within 6.95-7.05. DOCs are repeated annually and any time a significant change Is made to the analytical procedure.

12.3. Determination and verifications of MDL and RL are not applicable to this procedure.

13. POLLUTION CONTROL

13.1. All waste will be disposed of in accordance with Federal, State and Local regulations. Where feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this method and the policies in section 13 for "Waste Management and Pollution Prevention" of the Corporate Environmental Health & Safety Manual (CW-E-M-001) and the Pittsburgh Facility Addendum EH&S Manual (PT-HS-001).

13.2. This method does not contain any specific modifications that serve to minimize or prevent pollution.

14. WASTE MANAGEMENT

14.1. Waste management practices are conducted consistent with all applicable rules and regulations. Excess reagents, samples and method process wastes are disposed of in an accepted manner. Waste description rules and land disposal restrictions are followed. Waste disposal procedures are incorporated by reference to PT-HS-001. The following waste streams are produced when this method is carried out.

14.2. The following waste streams are produced when this method is carried out.

14.2.1. Acidic sample waste generated by the analysis. This waste is checked with pH paper, neutralized to a pH between 6 and 9 and discharged down a lab sink.

14.2.2. Alkaline sample waste generated by the analysis. This waste is checked with pH paper, neutralized to a pH between 6 and 9 and discharged down a lab sink.

14.2.3. Exhausted oil samples utilized in the analysis. This waste is collected in waste containers identified as "Mixed Flammable Solvent Waste", Waste #3.

14.2.4. Solvent waste generated by the analysis. This waste is collected in waste containers identified as "Mixed Flammable Solvent Waste", Waste #3.

14.2.5. Exhausted acidic and/or alkaline buffer solutions utilized in the analysis. This waste is checked with pH paper, neutralized to a pH between 6 and 9 and discharged down a lab sink.

14.2.6. Exhausted soil samples utilized in the analysis. This is collected in containers identified as "Lab Trash", Waste #12.

15. REFERENCES

15.1. SW846, Test Methods for Evaluating Solid Waste, 3rd Edition, pH Electrometric Measurement, Method 9040B & 9040C

15.2. SW846, Test Methods for Evaluating Solid Waste, 3rd Edition, Soil pH, method 9045C and 9045D

15.3. Standard Methods for the Examination of Water and Wastewater, method 4500-H+ B, 2000, 2011 editorial revision

15.4. TestAmerica Corporate Environmental Health & Safety Manual, CW-E-M-001

15.5. Pittsburgh Facility Addendum EH&S Manual, PT-HS-001

15.6. Pittsburgh Quality Assurance Manual, PT-QA-M-001

15.7. SOP PT-QA-001, Employee Orientation and Training

15.8. SOP PT-QA-021, Quality Assurance Program

15.9. SOP PT-QA-006, Procurement of Standards and Materials; Labeling and Traceability

15.10. SOP PT-QA-016, Nonconformance & Corrective Action System

15.11. SOP PT-QA-031, Internal Chain of Custody

16. METHOD MODIFICATIONS

16.1. None

17. ATTACHMENTS

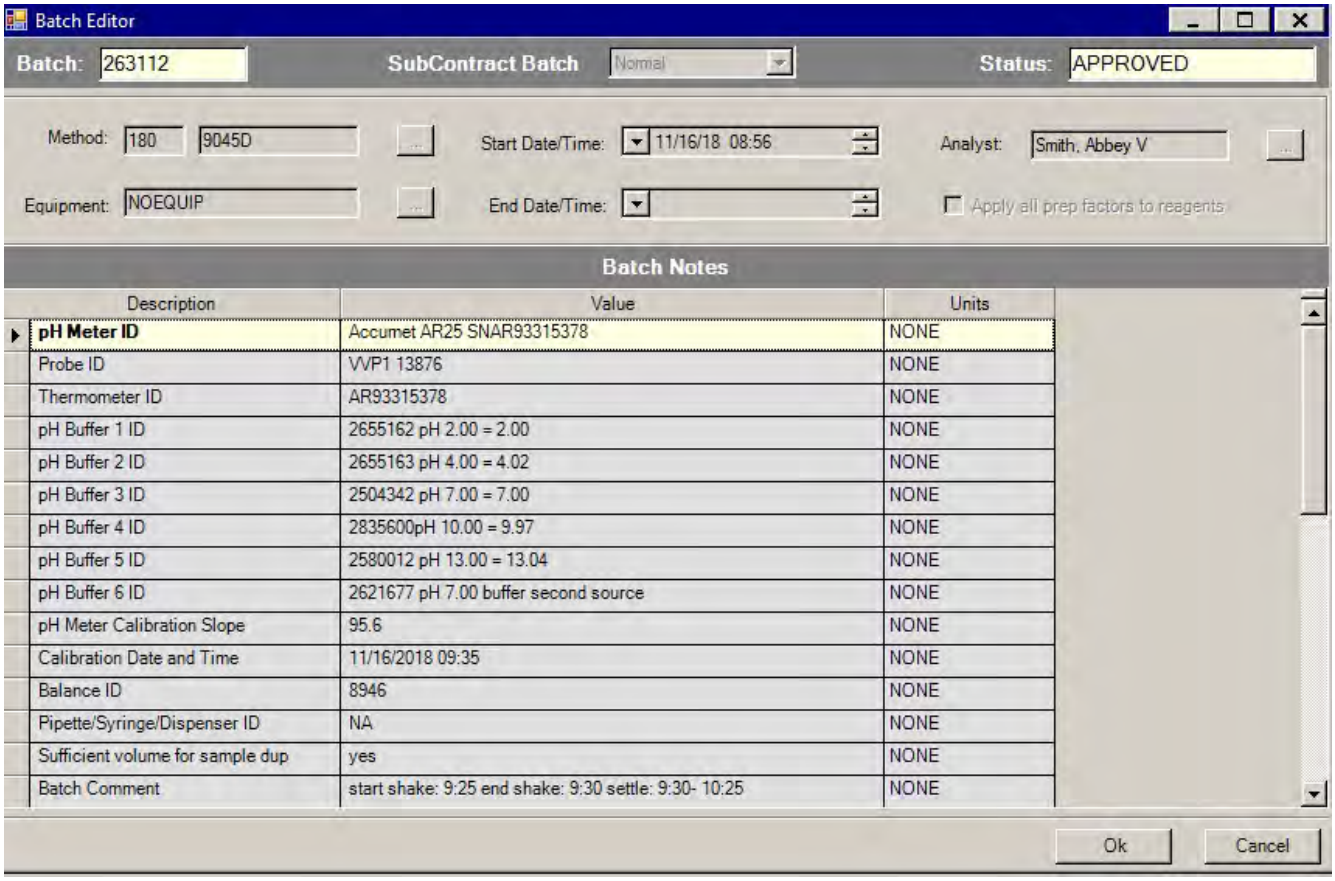
17.1. Attachment 1 – TALS Screenshots for pH Data and Reagents

18. REVISION HISTORY

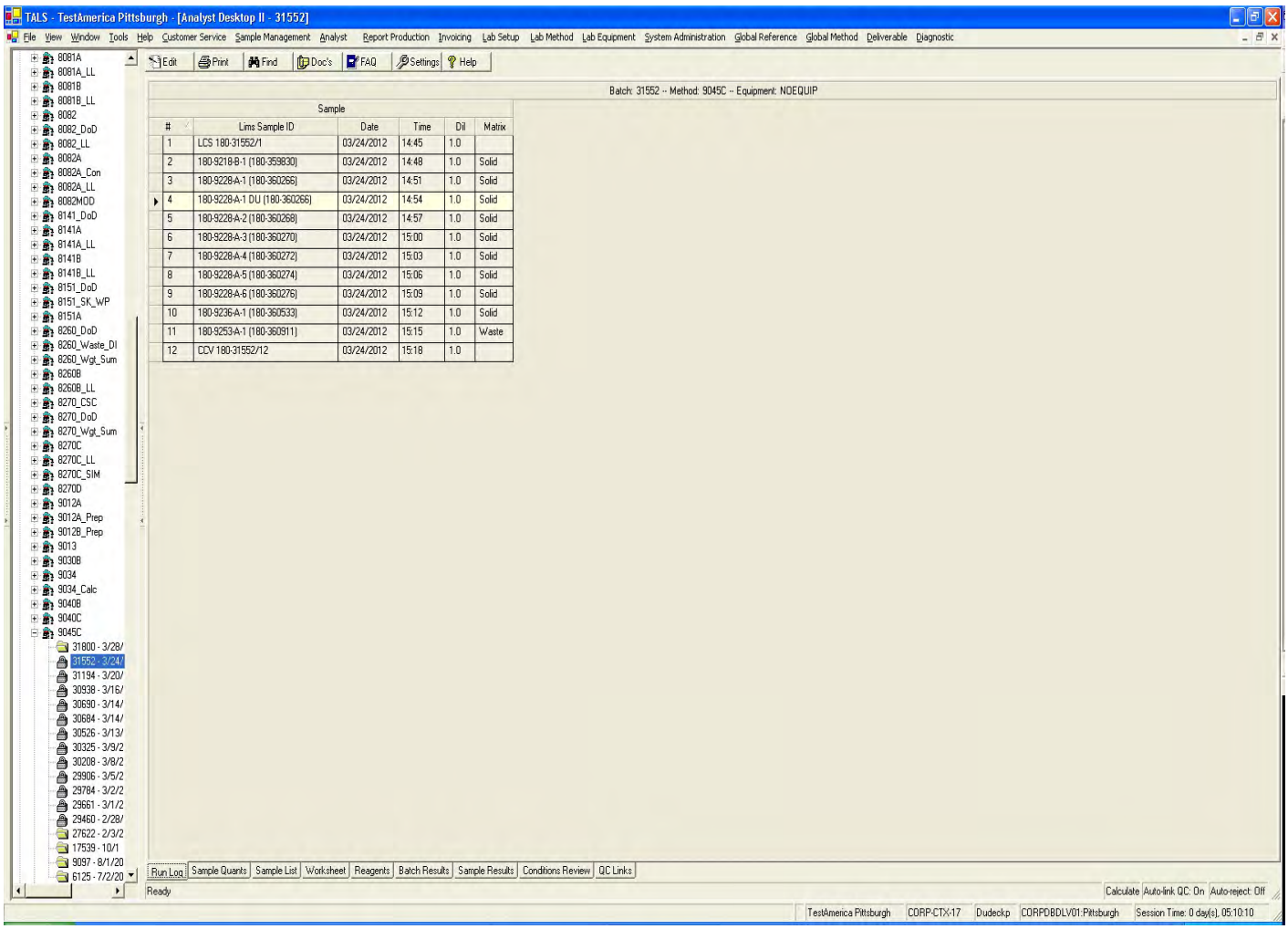
18.1. Revision 6, 09/21/07
18.2. Revision 6.1, 10/17/07
18.3. Revision 7, 10/14/2009
18.4. Revision 8, 3/2/2011
18.5. Revision 9, 5/15/2012
18.6. Revision 10, 7/3/2013
18.7. Revision 11, 8/28/2014
18.8. Revision 12, 11/29/2016
18.9. Revision 13, 11/30/2018
18.10. Revision 13.1, 5/14/2019
18.11. Revision 14, 4/9/2020
18.12. Changes to current revision

SOP item	Change from	Change to	Reason
2.2		Added reference to automated analysis for waters	New option
6.6		Added Autotitrator	New option
10.1.1.5, 10.3.2, 10.4, 10.5.1, 10.5.3		Added details for Autotitrator analysis	New option

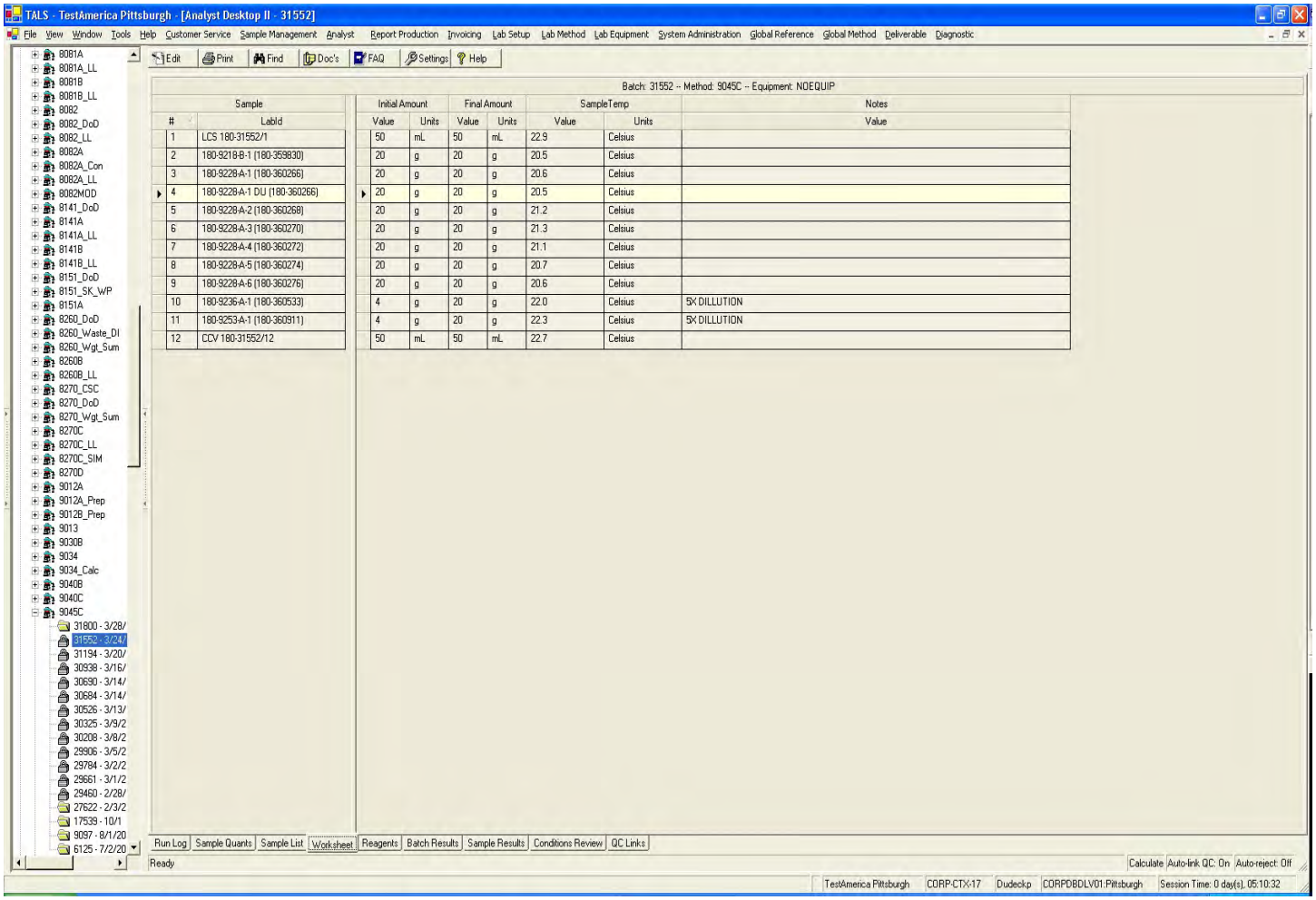
Attachment 1 – TALS Screenshots for pH Data and Reagents



Attachment 1 – TALS Screenshots for pH Data and Reagents (cont.)



Attachment 1 – TALS Screenshots for pH Data and Reagents (cont.)



Title: Hexane Extractable Material (HEM; Oil and Grease) and Silica Gel Treated Hexane Extractable Material (SGT-HEM; TPH) Method 1664A/1664B and SW-846 Method 9070A

Methods: Method 1664A/1664B and 9070A

Approvals (Signature/Date):

5/11/2020		5/13/2020	
Sharon Bacha	Date	Steve Jackson	Date
Organics Department Manager		Regional Safety Coordinator	
5/8/2020		5/18/2020	
Virginia Zusman	Date	Deborah Lowe	Date
Quality Assurance Manager		Laboratory Director	

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1.0 Scope and Application

1.1 This method quantifies the n-hexane extractable material (HEM; Oil and Grease) and n-hexane extractable material that is not adsorbed by silica gel (SGT-HEM or TPH (non-polar material)) in surface and saline waters and aqueous wastes. Generally, the extractables consist of non-volatile hydrocarbons, vegetable oils, animal fats, waxes, soaps, greases, and similar materials.

1.2 This method is for use in the Environmental Protection Agency's (EPA's) survey and monitoring programs under the Clean Water Act; the Resource Conservation and Recovery Act; the Comprehensive Environmental Response, Compensation, and Liability Act; and other EPA regulatory programs. "Oil and grease" is a conventional pollutant under the Clean Water Act and codified at 40 CFR 401.16. The term "n-hexane extractable material" reflects that this method can be used to determine materials other than oils and greases. Similarly, the term "silica gel treated n-hexane extractable material" reflects that this method can be used to determine material that is not adsorbed by silica gel (non-polar material).

1.3 If only SGT-HEM is required, the HEM determination can be skipped only if the HEM concentration is known. If the HEM concentration is not known, then the HEM determination must be performed prior to the SGT-HEM determination.

1.4 This method is applicable to surface waters, sewages, and aqueous wastes containing approximately 5 mg/L to approximately 1000 mg/L using a 1L sample. The reporting limit for both HEM and SGT-HEM is 5 mg/L. These limits may be extended to higher levels by analysis of a smaller sample volume collected separately. Dilutions are not appropriate for this procedure, except as specifically described for silica gel treatment in section 10.23.2.

1.5 On occasion clients may request slight modifications to this SOP. Any modifications to this procedure must be approved by the QA department or the Laboratory or Technical Director and documented fully in an NCM that accompanies the analytical batch.

2.0 Summary of Method

2.1 Using a solid phase extraction manifold, a 1.0 L aqueous sample (acidified to pH <2) is filtered through an extraction disk. The extractable materials are rinsed from this disk with n-hexane, collected into a 40 ml VOA vial. The extract is filtered through phase separation filter paper into a pre-weighed pan, for evaporation on the Speedvap 9000 II. This step concentrates the HEM portion of the sample. The pans are allowed to cool in a desiccator, and then re-weighed to determine the HEM concentration.

2.2 In order to determine the SGT-HEM (TPH), the residue from the sample is then reconstituted with hexane and treated with silica gel. The silica gel treated extract is filtered into a pre-weighed pan, for evaporation on the Speedvap 9001. This step concentrates the SGT-HEM portion of the sample. The pans are allowed to cool in a desiccator, and then re-weighed to determine the SGT-HEM concentration.

3.0 Definitions

3.1 N-hexane extractable material (HEM): Material that is extracted from a sample with hexane and determined by this method (oil and grease). Examples of this material include relatively non-volatile hydrocarbons, vegetable oils, animal fats, waxes, soaps, greases, and other materials with similar characteristics.

3.2 Silica gel treated n-hexane extractable material (SGT-HEM): Material that is extracted from a sample with hexane and determined by this method that is not adsorbed by silica gel (TPH).

3.3 TALS: TestAmerica Laboratory Information Management System

3.4 NCM: Non-Conformance Memo, a communication system for the laboratory to document anomalies or non-conformances encountered with samples and their analysis.

3.5 See TestAmerica Pittsburgh Laboratory's Quality Assurance Manual (PT-QA-M-001) for glossary of common terms.

4.0 Interferences

4.1 This method is not designed to quantify substances which volatilize at temperatures below approximately 85°C. Petroleum products, ranging from gasoline to #2 fuel oils, may be partially lost during the extraction and concentration process. Certain crude or heavy oils contain portions of material not soluble in n-hexane, often resulting in low recoveries.

4.2 All glassware and other apparatus in contact with the sample must be thoroughly washed and solvent rinsed to eliminate contamination from artifacts. In addition, reagent grade or better solvents must be used to ensure that the glassware, etc. is free of artifacts. The hexane, silica gel, phase separation paper, and any drying agents used, must be shown to be free from residue down to the MDL level.

4.3 For samples consisting of complex matrices (such as particulates or detergents) that may interfere with the extraction process, a smaller sample size may be used.

4.4 Care must be taken to avoid contamination of the final extract with sodium sulfate, silica gel and/or water. This can be avoided by filtering the HEM or SGT-HEM very slowly. Should contamination occur, apparent by visible amounts of crystalline material in the residue, the residue must be re-suspended in n-hexane.

The re-suspended material can be filtered with additional solvent washings of the flask through phase separator filter paper. The method then resumes with the evaporation process.

4.5 Samples must be brought to room temperature prior to analysis in order to reduce the amount of solid material adhering to the walls of the collection containers. Erroneous low results may be obtained from samples analyzed at refrigeration temperatures.

5.0 Safety

5.1 Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual (CW-E-M-001), the Pittsburgh Facility Addendum EH&S Manual (PT-HS-001) and this document. This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

5.2 The following is a list of the materials used in this method, which have a serious or significant hazard rating. This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the SDS for each of the materials listed in the table. A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the SDS for each material before using it for the first time or when there are major changes to the SDS.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Acetone	Flammable	1000 ppm-TWA	Inhalation of vapors irritates the respiratory tract. May cause coughing, dizziness, dullness, and headache.
Hexane	Flammable Irritant	500 ppm-TWA	Inhalation of vapors irritates the respiratory tract. Overexposure may cause lightheadedness, nausea, headache, and blurred vision. Vapors may cause irritation to the skin and eyes.
Hydrochloric Acid	Corrosive Poison	5 ppm-Ceiling	Inhalation of vapors can cause coughing, choking, inflammation of the nose, throat, and upper respiratory tract, and in severe cases, pulmonary edema, circulatory failure, and death. Can cause redness, pain, and severe skin burns. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
Methanol	Flammable Poison Irritant	200 ppm-TWA	A slight irritant to the mucous membranes. Toxic effects exerted upon nervous system, particularly the optic nerve. Symptoms of overexposure may include headache, drowsiness and dizziness. Methyl alcohol is a defatting agent and may cause skin to become dry and cracked. Skin absorption can occur; symptoms may parallel inhalation exposure. Irritant to the eyes.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

5.3 Eye protection that protects against splash, Laboratory coat, and appropriate gloves must be worn while samples, standards, solvents, and reagents are being handled. . Cut resistant gloves must be worn doing any other task that presents a strong possibility of getting cut. Disposable gloves that have been contaminated will be removed and discarded; other gloves will be cleaned immediately.

5.4 Exposure to chemicals must be maintained as low as reasonably achievable, therefore, unless they are known to be non-hazardous, all samples must be opened, transferred and prepared in a fume hood, or under other means of mechanical ventilation. Solvent and waste containers will be kept closed unless transfers are being made.

5.5 The preparation of standards and reagents will be conducted in a fume hood with the sash closed as far as the operations will permit, or under other adequate ventilation.

5.6 All work must be stopped in the event of a known or potential compromise to the health and safety of an Eurofins TestAmerica Pittsburgh Associate. The situation must be reported immediately to a supervisor or the EH&S coordinator.

6.0 Equipment and Supplies

The following items are recommended for performing this procedure. Equivalent items should only be used when they result in an improvement in quality, efficiency, productivity, or cost. An item can be considered equivalent if with its use, the analytical and QA/QC requirements in this SOP can be met.

6.1 Analytical balance capable of weighing to 0.1 mg

6.2 Vacuum source and tubing to connect apparatus

6.3 Waste receptacle and tubing to connect manifold

6.4 Horizon SpeedVap 9000II

6.5 UCT ENVIRO-CLEAN® Oil and Grease extraction apparatus equipped for each unit with the following:

- 6.5.1 1.0 L reservoir funnel
- 6.5.2 Rinsing flask (one per manifold set-up)
- 6.5.3 UCT ENVIRO-CLEAN® Oil and Grease cartridges – 37mm diameter polypropylene cartridge design
- 6.5.4 Oil-less vacuum pump for the UCT cartridge system (Rocker Model 400)

6.6 Desiccator

6.7 70 & 150 ml Disposable Evaporation Pans

6.8 Glass, class A, graduated cylinders, 1 L and 100 mL

6.9 pH indicator strips

6.10 40 mL VOA vials or other suitable glass container used as Collection flasks

6.11 Phase separation filter paper

6.12 Glass filter funnels

6.13 Glass Stir Rods, 4 x 125 mm or similar

7.0 Reagents and Standards

The following items are recommended for performing this procedure. Equivalent items should only be used when they result in an improvement in quality, efficiency, productivity, or cost. An item can be considered equivalent if with its use, the analytical and QA/QC requirements in this SOP can be met. Please refer to the SDS prior to the use of any reagent or standard. Quantities different from what is specified can be prepared, provided that the ratios remain the same.

All chemicals must be ACS reagent grade or better. Solvents must be of a grade shown to produce residue <1.0mg/L when evaporated.

7.1 Reagent water- Super Q or DI water

7.2 Hydrochloric acid or sulfuric acid, 6N: Mix equal volumes of concentrated HCl and reagent water or 1 part H_2SO_4 and 5 parts reagent water to produce an approximately 6N solution.

7.3 Acetone

7.4 n-Hexane, 85% minimum purity, 99.0% minimum saturated C6 isomers

7.5 Methanol

7.6 Silica Gel: Anhydrous, 75-150µm, 30 Å pore size (Dry at 200-250^{o}C for a minimum 24 hours and store in a desiccator or tightly sealed containers)

7.6.1 Verify that the n-hexane soluble material content of the silica gel is <5mg per 30g. Extract 30g of silica gel with 40-50mL of n-hexane by shaking in a closed container, using a wrist action or shaker table, for 15 minutes. Filter the extract into a tared evaporating dish with phase separation filter paper and evaporate the solvent. The final residue must be <0.0050 g (<0.17mg residue / g silica gel).

7.7 Hexadecane/stearic acid Spiking Standard (Used for LCS and MS/MSD)

7.7.1 Purchased Speedisk Oil and Grease standards kit of 2 mg/mL stearic acid and 2 mg/mL hexadecane in acetone. Spike 10.0 mL of the solution into a 1 L aliquot of acidified reagent water (acidified with 5 mL of 1:1 HCl)for the LCS, or separate sample bottles for MS/MSD. The resulting concentrations in the LCS are 40 mg/L HEM and 20 mg/L SGT-HEM (stearic acid is removed by the silica gel treatment. Refer to the manufacturer's expiration date.

7.8 Laboratory Prepared Spiking Solution (optional)

7.8.1 The spiking solution may be prepared by weighing 200 ± 2 mg hexadecane and 200 ± 2 mg stearic acid into a 100 mL volumetric flask. Dilute to

volume with acetone. Store in the dark at room temperature. To minimize evaporation of acetone, wrap the stopper and neck area of the flask with parafilm. Sonicate and warm to provide adequate dissolution of the solution. Prior to use, verify that the solution level is at the 100mL mark. In the event that some acetone has evaporated, bring the solution to volume again with acetone. Standard expiration is 6 months from the date prepared.

Note: Stearic acid tends to stick to everything. It is very important that all surfaces of the sample bottle, and anything else the sample comes in contact with, are rinsed well with hexane to ensure complete recovery.

8.0 Sample Collection, Preservation, Shipment and Storage

Listed below are the sampling requirements and holding time for this method.

Matrix	Sample Container	Min. Sample Size	Preservation	Holding Time	Reference
Water	1 L amber *	1 L amber *	>0.0℃ but ≤6.0℃, pH to <2 with HCl or H_2S0_4	28 days	40 CFR Part 136

* Smaller sample containers may be used, but RLs will be affected.

8.1 Sample bottles are pre-preserved using 5 mL of 1:1 HCl or 5 mL of 1:1 H_2SO_4 for a 1 L sample. This should be sufficient to adjust the sample pH to <2. The sample pH must be confirmed prior to extraction.

8.2 Collect approximately one liter of representative sample in a glass bottle following conventional sampling practices, except that the bottle must not be pre-rinsed with sample before collection. To allow for potential QC failures, it is recommended that additional sample aliquots be collected. To allow for MS and/or MSD analysis, collect a 2nd and/or 3rd aliquot for at least one sample. When collecting additional aliquots, either collect simultaneously in parallel, if possible, or collect as grab samples in rapid succession.

8.3 The high probability that extractable matter may adhere to sampling equipment and result in measurements that are biased low precludes the collection of composite samples for determination of oil and grease. Therefore, samples must be collected as grab samples. If a composite measurement is required, individual grab samples collected at prescribed time intervals must be analyzed separately and the concentrations averaged. Alternatively, samples can be collected in the field and composited in the laboratory. For example, collect four individual 250-mL samples over the course of a day. In the laboratory, pour each 250-mL sample into a 1 Liter pre-preserved Glass Bottle, rinse each of the four bottles (and caps)

sequentially with 30 mL of n-hexane, and use the 30 mL of n-hexane for the extraction.

8.4 If a sample is known or suspected to contain greater than 500mg/L of extractable material, collect a smaller volume of sample (the volume required will depend upon the estimated amount of extractable material) in another glass bottle. Add a proportionately smaller amount of HCl or H_2S0_4 solution to the smaller sample for preservation.

9.0 Quality Control

9.1 The following quality control samples are prepared with each batch of samples**.**

Quality Controls	Frequency	Control Limit[1]	
		HEM	SGT-HEM
Laboratory Control Sample (LCS)	1 per batch of 20 samples or less	78-114% recovery	64-132% recovery
Method Blank (MB)	1 per batch of 20 samples or less	< RL	< RL
Matrix Spike (MS)	1 per batch of 20 samples or less	78-114% recovery	64-132% recovery
Matrix Spike Duplicate (MSD)	1 per batch of 20 samples or less	78-114% recovery, RPD <18%	64-132% recovery, RPD <34%

[1]Control Limits are method specified and are maintained in LIMS

[2]The MS/MSD parent sample is randomly selected, unless specifically requested by a client.

9.2 Balance check: criteria for 2mg weight check is ±10%. Criteria for 1g weight check is ±0.5%.

9.2.1 If the balance check is outside of acceptance criteria, do not begin weighing pans. Clean the balance pan, recalibrate the balance and check the required weights again.

9.3 The Method Blank is 1 L of laboratory reagent water added to a pre-preserved bottle and taken through the same extraction and analysis process as the samples in the batch. One MB is prepared along with each batch of 20 or fewer samples. MB must have a residue < 5 mg/L, and should have residue below the current MDL. Sample results will be qualified on the final report for MB's with results above the MDL.

9.3.1 It is not permissible to use blank correction on sample results.

9.3.2 Samples associated with method blanks, which fail the acceptance criteria, must be re-prepared and reanalyzed with an acceptable blank, unless:

- Sample results are non-detect – in which case results may be reported using the "Method Blank – Report, ND" NCM.

- Sample results are greater than 10 times the concentration seen in the blank – in which case results may be reported using the "Method Blank – Report, 10X" NCM.

9.3.3 If there is insufficient sample to reanalyze, inform the project manager of the blank contamination and report results using the "Method Blank – Insufficient Sample" NCM to document the failure.

9.4 The LCS is prepared along with each batch of 20 or fewer samples in the same manner as the Method Blank, with 10mL of spike solution added as in section 7.7. Results must the control limits given in the QC table above.

9.4.1 Samples associated with a LCS, which fails the acceptance criteria, must be prepared and analyzed again with an acceptable LCS unless the LCS recovery is above the control limit, and the associated sample is non-detect. Non-detect results may be reported along with the "LCS/LCSD - %R High" NCM.

9.4.2 If there is insufficient sample volume to repeat the analysis, inform the project manager of the blank contamination and report results using the "LCS/LCSD – Insufficient Sample" NCM to document the failure.

9.5 A minimum of a Matrix Spike (MS) analysis must be performed with each analytical batch of 20 or fewer samples. If there is sufficient volume provided, a Matrix Spike Duplicate (MSD) shall also be included. If insufficient sample is available to prepare a MS, this must be documented with the "MS/MSD – Insufficient Volume" NCM.

9.5.1 If the MS (or MSD) does not meet the acceptance criteria given in the QC table above, evaluate the batch QC and parent sample result.

- If the batch LCS has acceptable recovery, results are reported using the "MS/MSD - %R, LCS Pass" NCM.
- If the parent sample concentration is greater than 4x the spiked amount, the MS recovery cannot be accurately calculated, and the "MS/MSD – High Targets" NCM should be used to document this.

9.6 If sample reanalysis due to QC failure will be past the holding time, inform the project manager of the QC failure and obtain client instructions on how to proceed. The decision on whether to report the original result or perform reanalysis past the holding time must be documented in the case narrative.

9.7 The cleanliness of the silica gel (see section 7.6) must be documented for each lot prior to use.

9.8 Each time a modification is made to this written procedure, including types of equipment used, the laboratory must repeat the initial demonstration of capability and MDL study. (See requirements in section 12.)

10.0 Procedure

10.1 Calibrate the analytical balance at 2 mg and 1000 mg using class "S" weights before and after weighing each batch of samples. (See section 9.2.)

10.2 Allow the samples to warm to room temperature before beginning. (However, do not leave samples on the bench for more than 4 hours.)

10.3 Verify that the pH of the sample is less than 2 using the following procedure:

10.3.1 Dip a glass stir rod into the well-mixed sample.

10.3.2 Withdraw the glass stir rod and allow a drop of sample to fall on a pH strip. Record the sample pH on the TALS worksheet.

10.3.3 As some oil and grease may adhere to the stir rod, rinse the stir rod with hexane and collect into the funnel for filtering the extract into the final collection vessel. Phase separation filter paper must be used so that any water rinsed off by the hexane does not end up in the evaporating dish.

10.3.4 Use a new stir rod for each sample to avoid cross-contamination.

10.3.5 If the sample is at neutral or greater pH, add 5-6 mL of 6N HCl or H_2SO_4 solution or other appropriate amount to adjust the pH of the 1 L sample to < 2. Document the final pH on the TALS worksheet if the sample pH requires adjustment.

10.4 Weigh Disposable Evaporation Pans using an analytical balance and record the weights on the TALS worksheet. Always handle pans with tongs or gloves. Do not touch the pans with bare fingers, or oils from you skin may add weight to the pan.

10.5 Assemble a UCT ENVIRO-CLEAN® Oil and Grease Cartridge Extraction Station and mount a collection chamber on each active port.

10.6 Mount the cartridge and wash the sides with approximately 10 mL of hexane. Allow the cartridge to soak in the hexane for 1 minute. Draw the hexane through the cartridge using the vacuum pump and discard the hexane as waste. Repeat two more times. Draw full vacuum through the cartridge for 2 minutes to remove all the hexane.

10.7 Add 10 mL of methanol to the cartridge and slowly draw the methanol through the cartridge leaving a layer on the surface of the cartridge. **Do not let the sorbent dry out.** Soak for one minute then add 30 mL of DI water to the cartridge. Draw the water through the cartridge to waste.

10.7.1 **Do not allow the sorbent to completely dry before adding the sample**, otherwise repeat steps 10.6 and 10.7.

10.8 Mark the meniscus of the water on the sample bottle. For the LCS and MS/MSD aliquots, spike each in the bottle with the Spiking Standard (Refer to Section 7.7). Mix well.

10.9 Add the 1 liter water sample directly to the cartridge and draw through under full vacuum. This may take several minutes for complete flow through the cartridge depending upon the level of solids in the sample. Use a bottle holder on top of the cartridge if necessary to hold the upside-down bottle securely.

10.9.1 If very high solids are present, add glass wool to the cartridge prior to adding the sample to prevent clogging and achieve good flow. The glass wool must be thoroughly rinsed with hexane before use.

10.10 After the entire sample has filtered, allow the cartridge to dry under full vacuum for 10 minutes to remove excess water. Remove any water remaining in the bottom support of the cartridge with a paper towel. Do not touch the inside of the cartridge.

10.11 Rinse the sample bottle with 10 mL of hexane. Add the hexane to the cartridge, rinsing the sides of the cartridge and the bottle holder, if used. Soak for 3 minutes. Slowly draw the hexane through the cartridge and into the collection vial. Do not allow the vacuum to continually blow on the extract.

10.12 Immediately repeat step 10.11 two more times using 10 mL of hexane.

10.13 Fill the rinsed sample bottle to the meniscus marking, and measure the volume contained using a 1 Liter, class A, graduated cylinder. Record the sample volume on the batch worksheet.

10.14 Add another 10 mL of hexane to the cartridge, rinsing the bottle holder. Allow the cartridge to soak for 3 minutes. Draw the hexane through the cartridge. The total volume of the extract will be about 35 mL.

10.15 Remove the collection vial from the manifold and cover with a screw cap.

10.16 Place a piece of phase separation filter paper into a filter funnel. Rinse three times with hexane and dispose of the rinsate.

10.17 Filter the extract through phase separator filter paper into a pre-weighed 70 mL evaporation pan. Rinse the collection vial and filter paper twice with approximately 5 mL portions of hexane.

10.18 Set the Speedvap to 50^{o}C. Place the evaporating pans into the Speedvap and evaporate to near dryness, visibly. (Evaporating to total dryness may also remove some of the lighter hydrocarbons from the residue.)

10.19 Remove the pans from the Speedvap, and place them in a desiccator for at least one hour.

10.20 Weigh the pans on an analytical balance and record the weights to the nearest 0.1 mg on the TALS worksheet.

10.21 Repeat the cycle of desiccating for 1 hour and weighing until the sample weight loss is < 4% for the previous weight or less than 0.5 mg, whichever is less. If pan weight is not within 0.5 mg, put the sample back in the desiccator for 1 hour and perform a third weighing.

10.22 If only HEM (Oil & Grease) is required to be reported, proceed to result calculation in section 11.2. If SGT-HEM (TPH) is also required, continue with section 10.23.

10.23 SGT-HEM Determination:

If the HEM result is less than the SGT-HEM reporting limit (or MDL), the analyst may report SGT-HEM as less than the reporting limit and stop at this point. SGT-HEM should always be less than or equal to HEM.

10.23.1 Rinse the residue from the HEM determination into a suitable glass container with approximately 40 mL of hexane. This re-dissolved HEM is referred to as “the extract”.

10.23.2 Add 3.0 g of silica gel for every 100 mg of HEM in the extract up to a maximum of 30 grams of silica gel (maximum of 1000 mg of HEM).

- If the amount of silica gel needed exceeds 30 grams, then only process a portion of the extract. For example- if the amount of silica gel needed is calculated to be 60 grams, then use ½ of the extract. Account for this “dilution” in the final calculation for SGT-HEM.

For example: 6.4294 g Residue x 1000 = 6429.4 mg Residue

Based on the maximum concentration of 30 g of silica gel per 1000 mg of HEM a 10X dilution is done on this sample to bring the sample within range, resulting in 642.94 mg of Residue.

Then use 3 g of silica gel per 100 mg of HEM as follows:

642.94 mg/100 mg = 6.4294 mg HEM

6.4294 mg x 3 g silica gel = 19.2882 or 19.3 g of silica gel

So a 10X dilution would need approximately 19 g of silica gel

- The HEM in the extract should be rounded up to the next 100 mg. For example, if the HEM is 735 mg, the amount of silica gel added should be based on 800 mg or 24 grams of silica gel (3 g. x 8).

10.23.3 Shake or stir the sample flask for 15 minutes.

10.23.4 Filter with phase separator paper into a previously weighed 70 mL evaporation pan. Rinse the flask, silica gel and filter paper with several small amounts of hexane to complete the transfer.

10.23.5 Repeat steps 10.18 through 10.21.

10.23.6 Determine the SGT-HEM concentration using the equation in section 11.2.

11.0 Calculations / Data Reduction

11.1 **LCS % Recovery** = $\frac{\text{observed concentration}}{\text{known concentration}} \times 100$

Note: the spike concentration for SGT-HEM is half of the HEM concentration due to the removal of stearic acid during the silica gel treatment.

11.2 **HEM or SGT-HEM (mg/L)** = $\frac{[\ A - B\ ] \times 1{,}000{,}000 \times DF}{\text{mL of sample}}$

Where:
A = final pan weight in grams.
B = initial pan weight in grams.
DF = Dilution factor (= 1 if no dilution performed)

11.3 MS/MSD $\%\ Spike\ Recovery\ (\%R) = \frac{SSR - SR}{SA} \times 100$

where:
SSR=Spiked Sample Result
SR=Sample Result
SA=Spike Added

11.4 Relative Percent Difference (RPD) between the MS and MSD (or LCS/LCSD):

$$RPD = \frac{|Conc.1 - Conc.2|}{(Conc.1 + Conc.2)/2} \times 100$$

Where: Conc. 1 and Conc. 2 are the concentrations (mg/L HEM or SGT-HEM) in the MS and MSD samples, respectively.

Note: the spike concentration for SGT-HEM is half of the HEM concentration due to the removal of stearic acid during the silica gel treatment.

12.0 Method Performance

12.1 The supervisor has the responsibility to ensure that an analyst who has been properly trained in its use and has the required experience performs this procedure. The training documentation will be forwarded to QA for inclusion in the training files. DOCs will be documented for each analyst in the training file.

12.2 **Demonstration of Capabilities(DOC)**

12.2.1 This requires the analysis of four LCS samples. The LCS sample is a well-characterized, laboratory-generated sample used to monitor method performance, which should contain the analyte(s) of interest. The results of the initial demonstration study must be acceptable before analysis of samples under this SOP may begin. Four aliquots of the LCS are prepared and analyzed using the procedures detailed in this SOP.

12.2.2 Recovery of each LCS standard analyzed as part of the IDOC must meet recovery limits of 83-101% for HEM and 83-116% for SGT-HEM. Precision between the four standards must be ≤ 11%RSD for HEM and ≤ 28%RSD for SGT-HEM.

12.2.3 Annually, a Continuing Demonstration of Capability must be documented for each analyst performing this method, using 4 consecutive batch LCS's. The LCS data may come from a single batch or multiple batches. LCS recovery limits listed in Table 9.1 must be met for all points. The %RSD of the set must meet precision criteria of the IDOC given in section 12.2.2.

12.3 **Method Detection Limit Study (MDL)**

12.3.1 A Method Detection Limit (MDL) study, as described in the Detection Limits SOP PT-QA-007, must be performed initially and any time a significant change is made to the analytical system. The MDL must be evaluated at least every 12 months.

12.3.2 The calculated MDL for HEM and SGT-HEM from the MDL study must be ≤ 1.4 mg/L, or ≤ ⅓ the regulatory compliance limit, whichever is greater. Because compliance limits can vary, the laboratory will use 1.4 mg/L as the upper limit for MDL.

13.0 Pollution Control

13.1 It is Eurofins TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage and reagent stability). Employees must abide by the policies in Section 13 of the "Eurofins TestAmerica Environmental Health and Safety Manual

(CW-E-M-001) for "Waste Management and Pollution Prevention" and the Pittsburgh Facility Addendum EH&S Manual (PT-HS-001).

14.0 Waste Management

14.1 Waste management practices are conducted consistent with all applicable rules and regulations. Excess reagents, samples and method process wastes are disposed of in an accepted manner. Waste description rules and land disposal restrictions are followed. Waste disposal procedures are incorporated by reference to PT-HS-001. The following waste streams are produced when this method is carried out.

14.2 The following waste streams are produced when this method is carried out.

14.3 Solvent or oily waste. This waste is collected in a waste container identified as "Mixed Flammable Solvent Waste", Waste #3.

14.4 Contaminated filter Paper with Sodium Sulfate, Speed Disc, and aluminum pan. This waste is collected in containers identified as "Lab Trash", Waste #12.

15.0 References

15.1 Method 1664, Revision A: N-Hexane Extractable Material (HEM; Oil and Grease) and Silica Gel Treated N-Hexane Extractable Material (SGT-HEM; Non-polar Material) by Extraction and Gravimetry, EPA-821-R-98-002, February 1999

15.2 Method 1664, Revision B: N-Hexane Extractable Material (HEM; Oil and Grease) and Silica Gel Treated N-Hexane Extractable Material (SGT-HEM; Non-polar Material) by Extraction and Gravimetry, EPA-821-R-10-001, February 2010

15.3 EPA 2000. Analytical Method Guidance for EPA Method 1664A Implementation and Use (40 CFR part 136), EPA/821-R-00-003, January 2000

15.4 Bakerbond application note. Extraction of Oil & Grease from water, EPA Method 1664

15.5 SW-846 Method 9070A: n-Hexane Extractable Material for Aqueous Samples

15.6 PT-QA-M-001, Pittsburgh Laboratory Quality Assurance Manual

15.7 PT-QA-003, Glassware Clean-up for Organic/Inorganic Procedures

15.8 PT-QA-006, Procurement of Standards and Materials; Labeling and Traceability

15.9 PT-QA-007, Detection Limits

15.10 PT-QA-009, Rounding and Significant Figures

15.11 PT-QA-016, Nonconformance & Corrective Action System

15.12 PT-QA-018, Technical Data Review Requirements

15.13 PT-QA-021, Pittsburgh Quality Control Program

15.14 PT-QA-031, Internal Chain of Custody

15.15 PT-HS-001, Pittsburgh Facility Addendum EH&S Manual

16.0 Method Modifications

16.1 Because a solid-phase extraction is performed; an allowable modification per Method 1664, Revision B, Section 1.7.1.1; the final extract will not have the water content potential from the separatory funnel method. For this reason, sodium sulfate is not added to the funnel for filtration of the extract prior to evaporation. Sodium sulfate was found to produce residues around or above the MDL in sample extracts filtered through it. The extracts are filtered through phase separation paper only.

17.0 Attachments

17.1 Figure 1 - Example HEM and SGT-HEM TALS worksheet

18.0 Revision History

18.1 Revision 6, 11/12/2008

18.2 Revision 7, 6/11/2009

18.3 Revision 8, 9/21/200

18.4 Revision 9, 7/1/2010

18.5 Revision 10, 11/4/2010

18.6 Revision 11, 6/8/2012

18.7 Revision 12, 6/3/2013

18.8 Revision 13, 6/16/2015

18.9 Revision 14, 7/20/2016

18.10 Revision 15, 11/24/2017

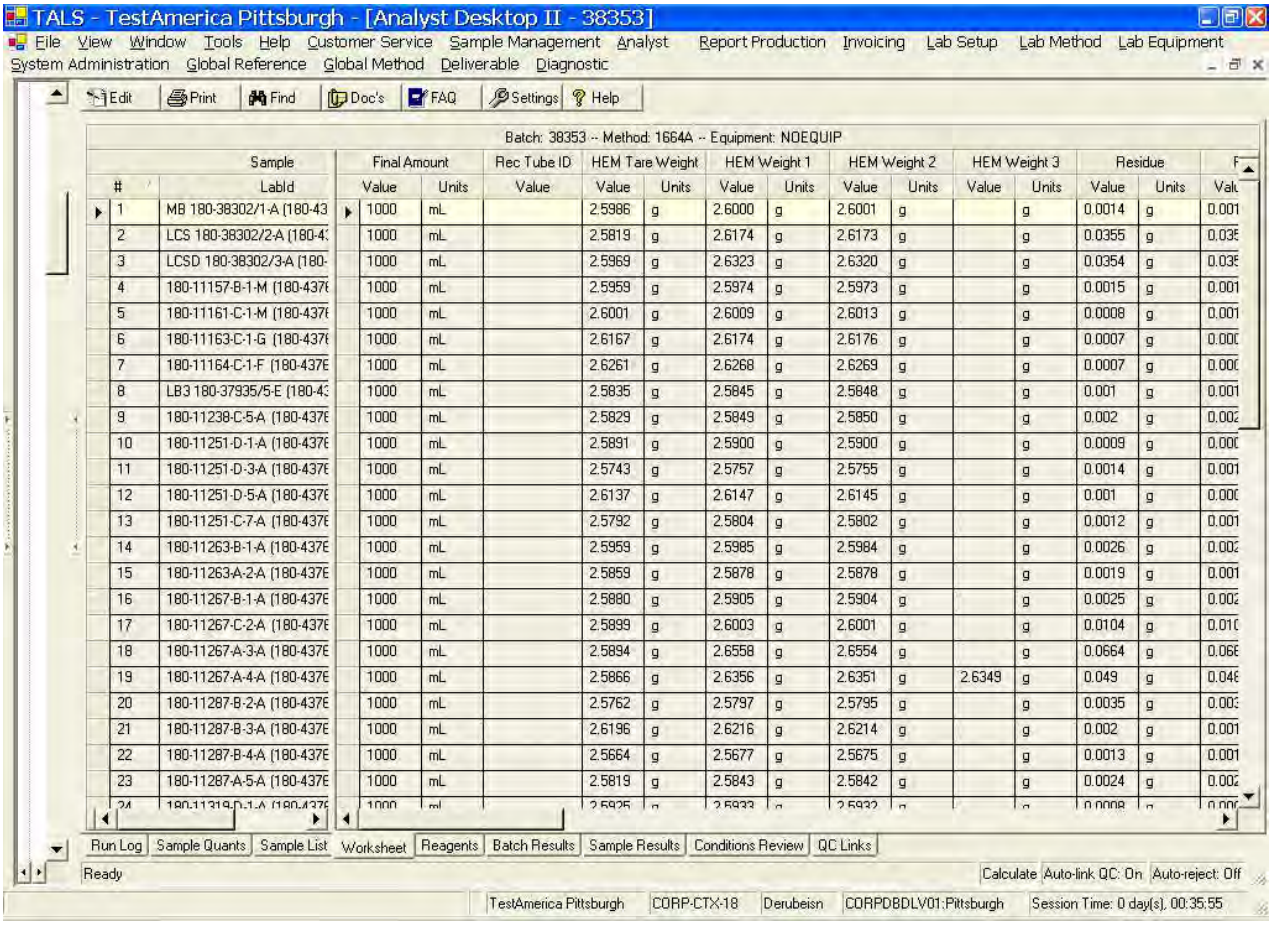
18.11 Revision 15.1, 5/14/2019

18.12 Changes to current revision

SOP section	Change from	Change to	Reason
1.5	Remove old text on method modifications	Added SOP Checklist text on method modifications	SOP Checklist Format
7.7.1		Added as the last sentence “Refer to the manufacturer’s expiration date.”	Clarification
7.8.1		Added as the last sentence “Standard expiration is 6 months from date prepared.”	Clarification

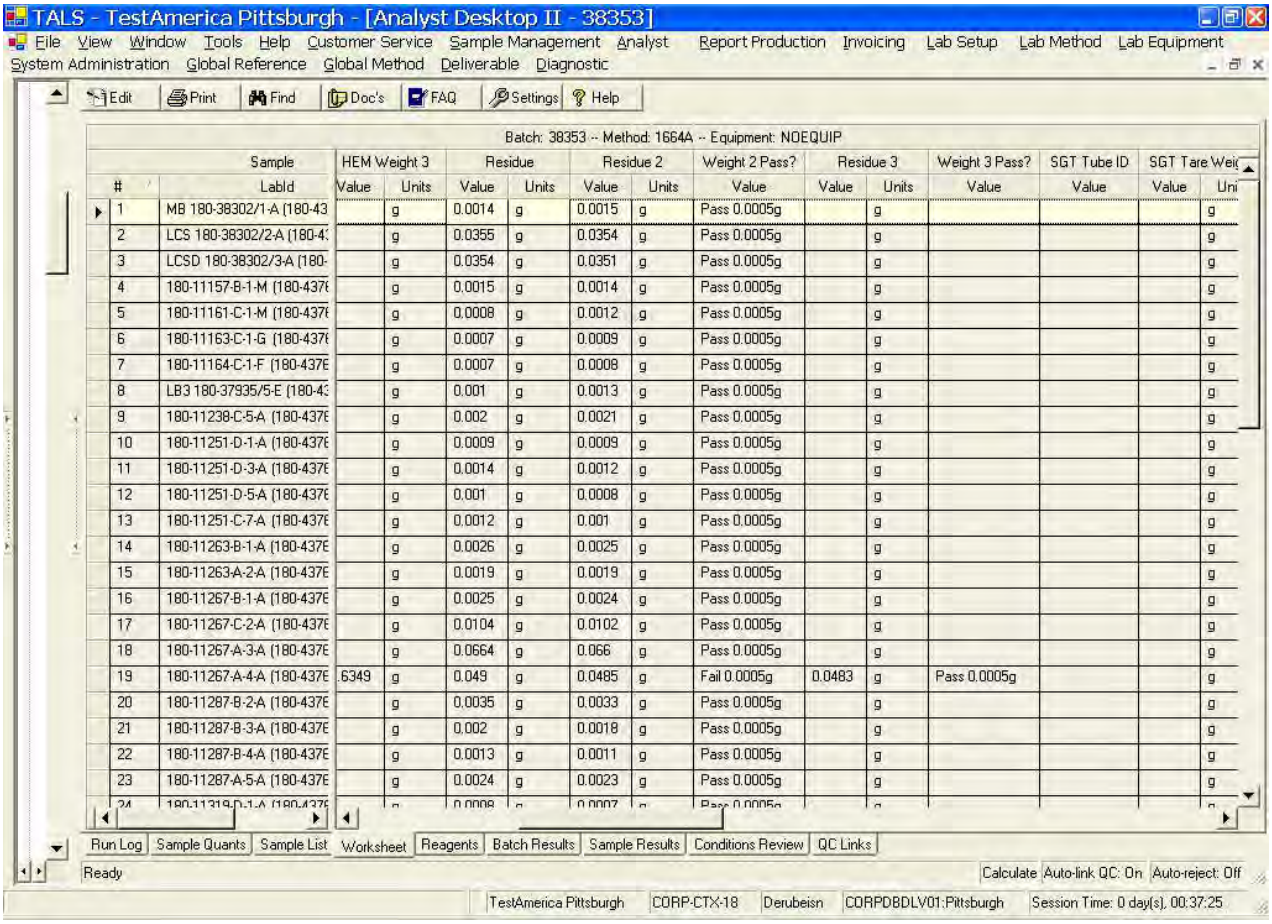
10.6		Added two additional rinsings to the cartridge cleaning	Correction

FIGURE 1 – Example TALS Worksheet for HEM



#	Sample LabId	Final Amount Value	Final Amount Units	Rec Tube ID Value	HEM Tare Weight Value	HEM Tare Weight Units	HEM Weight 1 Value	HEM Weight 1 Units	HEM Weight 2 Value	HEM Weight 2 Units	HEM Weight 3 Value	HEM Weight 3 Units	Residue Value	Residue Units
1	MB 180-38302/1-A (180-43	1000	mL		2.5986	g	2.6000	g	2.6001	g		g	0.0014	g
2	LCS 180-38302/2-A (180-4	1000	mL		2.5819	g	2.6174	g	2.6173	g		g	0.0355	g
3	LCSD 180-38302/3-A (180-	1000	mL		2.5969	g	2.6323	g	2.6320	g		g	0.0354	g
4	180-11157-B-1-M (180-437	1000	mL		2.5959	g	2.5974	g	2.5973	g		g	0.0015	g
5	180-11161-C-1-M (180-437	1000	mL		2.6001	g	2.6009	g	2.6013	g		g	0.0008	g
6	180-11163-C-1-G (180-437	1000	mL		2.6167	g	2.6174	g	2.6176	g		g	0.0007	g
7	180-11164-C-1-F (180-437	1000	mL		2.6261	g	2.6268	g	2.6269	g		g	0.0007	g
8	LB3 180-37935/5-E (180-4	1000	mL		2.5835	g	2.5845	g	2.5848	g		g	0.001	g
9	180-11238-C-5-A (180-437	1000	mL		2.5829	g	2.5849	g	2.5850	g		g	0.002	g
10	180-11251-D-1-A (180-437	1000	mL		2.5891	g	2.5900	g	2.5900	g		g	0.0009	g
11	180-11251-D-3-A (180-437	1000	mL		2.5743	g	2.5757	g	2.5755	g		g	0.0014	g
12	180-11251-D-5-A (180-437	1000	mL		2.6137	g	2.6147	g	2.6145	g		g	0.001	g
13	180-11251-C-7-A (180-437	1000	mL		2.5792	g	2.5804	g	2.5802	g		g	0.0012	g
14	180-11263-B-1-A (180-437	1000	mL		2.5959	g	2.5985	g	2.5984	g		g	0.0026	g
15	180-11263-A-2-A (180-437	1000	mL		2.5859	g	2.5878	g	2.5878	g		g	0.0019	g
16	180-11267-B-1-A (180-437	1000	mL		2.5880	g	2.5905	g	2.5904	g		g	0.0025	g
17	180-11267-C-2-A (180-437	1000	mL		2.5899	g	2.6003	g	2.6001	g		g	0.0104	g
18	180-11267-A-3-A (180-437	1000	mL		2.5894	g	2.6558	g	2.6554	g		g	0.0664	g
19	180-11267-A-4-A (180-437	1000	mL		2.5866	g	2.6356	g	2.6351	g	2.6349	g	0.049	g
20	180-11287-B-2-A (180-437	1000	mL		2.5762	g	2.5797	g	2.5795	g		g	0.0035	g
21	180-11287-B-3-A (180-437	1000	mL		2.6196	g	2.6216	g	2.6214	g		g	0.002	g
22	180-11287-B-4-A (180-437	1000	mL		2.5664	g	2.5677	g	2.5675	g		g	0.0013	g
23	180-11287-A-5-A (180-437	1000	mL		2.5819	g	2.5843	g	2.5842	g		g	0.0024	g

FIGURE 1 – Example TALS Worksheet for HEM cont.



#	Sample LabId	HEM Weight 3 Value	HEM Weight 3 Units	Residue Value	Residue Units	Residue 2 Value	Residue 2 Units	Weight 2 Pass? Value	Residue 3 Value	Residue 3 Units	Weight 3 Pass? Value	SGT Tube ID Value	SGT Tare Wei Value	SGT Tare Wei Uni
1	MB 180-38302/1-A (180-43		g	0.0014	g	0.0015	g	Pass 0.0005g		g				g
2	LCS 180-38302/2-A (180-4		g	0.0355	g	0.0354	g	Pass 0.0005g		g				g
3	LCSD 180-38302/3-A (180-		g	0.0354	g	0.0351	g	Pass 0.0005g		g				g
4	180-11157-B-1-M (180-437		g	0.0015	g	0.0014	g	Pass 0.0005g		g				g
5	180-11161-C-1-M (180-437		g	0.0008	g	0.0012	g	Pass 0.0005g		g				g
6	180-11163-C-1-G (180-437		g	0.0007	g	0.0009	g	Pass 0.0005g		g				g
7	180-11164-C-1-F (180-437		g	0.0007	g	0.0008	g	Pass 0.0005g		g				g
8	LB3 180-37935/5-E (180-4		g	0.001	g	0.0013	g	Pass 0.0005g		g				g
9	180-11238-C-5-A (180-437		g	0.002	g	0.0021	g	Pass 0.0005g		g				g
10	180-11251-D-1-A (180-437		g	0.0009	g	0.0009	g	Pass 0.0005g		g				g
11	180-11251-D-3-A (180-437		g	0.0014	g	0.0012	g	Pass 0.0005g		g				g
12	180-11251-D-5-A (180-437		g	0.001	g	0.0008	g	Pass 0.0005g		g				g
13	180-11251-C-7-A (180-437		g	0.0012	g	0.001	g	Pass 0.0005g		g				g
14	180-11263-B-1-A (180-437		g	0.0026	g	0.0025	g	Pass 0.0005g		g				g
15	180-11263-A-2-A (180-437		g	0.0019	g	0.0019	g	Pass 0.0005g		g				g
16	180-11267-B-1-A (180-437		g	0.0025	g	0.0024	g	Pass 0.0005g		g				g
17	180-11267-C-2-A (180-437		g	0.0104	g	0.0102	g	Pass 0.0005g		g				g
18	180-11267-A-3-A (180-437		g	0.0664	g	0.066	g	Pass 0.0005g		g				g
19	180-11267-A-4-A (180-437	.6349	g	0.049	g	0.0485	g	Fail 0.0005g	0.0483	g	Pass 0.0005g			g
20	180-11287-B-2-A (180-437		g	0.0035	g	0.0033	g	Pass 0.0005g		g				g
21	180-11287-B-3-A (180-437		g	0.002	g	0.0018	g	Pass 0.0005g		g				g
22	180-11287-B-4-A (180-437		g	0.0013	g	0.0011	g	Pass 0.0005g		g				g
23	180-11287-A-5-A (180-437		g	0.0024	g	0.0023	g	Pass 0.0005g		g				g

TITLE: Paint Filter – Free Liquids Test

Methods: SW-846 Method 9095B

Approvals (Signature/Date):

Roseann Ruyechan Inorganics Department Manager	11/9/2020 Date
Steve Jackson Regional Safety Coordinator	11/9/2020 Date
Virginia Zusman Quality Assurance Manager	11/9/2020 Date
Deborah L. Lowe Laboratory Director	11/23/2020 Date

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1. SCOPE AND APPLICATION

1.1 This method determines the presence of free liquids in a representative sample of waste.

1.2 On occasion clients may request slight modifications to this SOP. Any modifications to this procedure must be approved by the QA department of the Laboratory or Technical Director, and must be documented fully in an NCM that accompanies the analytical batch.

2. SUMMARY OF METHOD

2.1 A predetermined amount of sample is placed in a paint filter. If any liquid portion of the sample passes through and drops from the filter within the 5-minute test period, the sample is deemed to contain free liquids.

3. DEFINITIONS

3.1 TALS – Eurofins TestAmerica Laboratory Information Management System

3.2 NCM - Non-Conformance Memo - a system within TALS for the lab to communicate to project management and others when there is an anomaly seen with the samples or batch, or a QC failure.

3.3 Refer to the glossary in the Laboratory Quality Assurance Manual (PT-QA-M-001), current version, for more definitions related to analytical procedures.

4. INTERFERENCES

4.1 Filter media were observed to separate from the filter cone on exposure to alkaline materials. This development causes no problem if the sample is not disturbed.

4.2 Temperature can affect the test results if the test is performed below freezing point of any liquid in the sample. Tests must be performed above the freezing point and can, but are not required to, exceed room temperature of 25°C.

5. SAFETY

5.1 Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual (CW-E-M-001), the Pittsburgh Facility Addendum EH&S Manual (PT-HS-001), and this document. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the

method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

5.2 There are no materials used in this method that have a significant or serious hazard rating. A complete list of materials used in the method can be found in the reagents and standards section.

5.3 Eye protection that protects against splash, laboratory coat, and appropriate gloves must be worn while samples are being handled. Cut resistant gloves must be worn doing any other task that presents a strong possibility of getting cut. Disposable gloves that have been contaminated will be removed and discarded; other gloves will be cleaned immediately.

5.4 Exposure to chemicals must be maintained as low as reasonably achievable; therefore, unless they are known to be non-hazardous, all samples should be opened, transferred, and prepared in a fume hood, or under other means of mechanical ventilation. Waste containers will be kept closed unless transfers are being made.

5.5 All work must be stopped in the event of a known or potential compromise to the health and safety of a TestAmerica associate. The situation must be reported immediately to a laboratory supervisor or EH&S coordinator.

6. EQUIPMENT AND SUPPLIES

The following items are recommended for performing this procedure. Equivalent items should only be used when they result in an improvement in quality, efficiency, productivity, or cost. An item can be considered equivalent if with its use, the analytical and QA/QC requirements in this SOP can be met.

6.1 Conical Paint Filter: Mesh number 60 ± 5% (fine meshed size), available at local paint or hardware stores or ordered through Amazon.

6.2 Glass Funnel: If the paint filter, with the waste, cannot sustain its weight on the beaker or in the ring, then a fluted glass funnel or glass funnel with a mouth large enough to allow at least 1 inch of the filter mesh to protrude shall be used to support the filter. The funnel is to be fluted or have a large open mouth in order to support the paint filter yet not interfere with the movement to the beaker of the liquids that passes through the filter mesh.

6.3 Ring stand and ring or tripod

6.4 Glass beaker or disposable plastic cup, 100mL size or larger

6.5 Graduated cylinder, class A, various sizes

6.6 Top-loading Balance, capable of weighing to the nearest 0.01 grams

7. REAGENTS AND STANDARDS

7.1 Not Applicable

8. SAMPLE COLLECTION, PRESERVATION, SHIPMENT AND STORAGE

8.1 Sample Container: Plastic or Glass

8.2 No chemical preservation is required. Samples must be kept at >0°C but ≤6°C during transport and storage prior to analysis.

8.3 No holding time is specified in the method. A six-month holding time is assigned to this test by the lab.

9. QUALITY CONTROL

9.1 A Sample Duplicate is analyzed with every set of 20 or fewer samples.

9.2 Because this procedure does not report numeric results, no %RPD can be applied to the duplicate. The sample and duplicate must agree on the presence or absence of free liquid. If liquid is collected from one and not the other, 2 more portions of the sample must be analyzed.

9.3 No other QC type is applicable to this test.

10. PROCEDURE

10.1 Calibration and Standardization

10.1.1 The balance calibration must be verified daily before using to weigh the sample to be analyzed with weights that bracket the range of use. See SOP PT-QA-012, current version, for balance calibration requirements and procedure.

10.2 A 100 mL or 100g representative sample is required for the test. If it is not possible to obtain a sample of 100 mL or 100g that is sufficiently representative of the waste, the analyst may use larger size samples in multiples of 100 mL or 100g, i.e., 200, 300, 400 mL or g. When larger sample sizes are used, however, analysts shall divide the sample into 100 mL or 100g portions and test each portion separately. If any portion contains

free liquids, the entire sample is considered to have free liquids. If the sample is measured volumetrically, then it should lack major air spaces or voids.

10.3 Assemble test apparatus by placing the paint filter in a ring or top of a beaker that allows sufficient space beneath the filter tip for liquid to be collected without touching the filter. The filter must be supported enough to not fall through the ring, or collapse when filled with the sample.

10.4 Place the appropriate sized, representative sample in the filter. A glass funnel may be used to provide support for the paint filter if necessary. If the sample is of such bulk density that it overflows the filter, then the sides of the filter can be extended upward by taping filter paper to the inside of the filter and above the mesh. Settling the sample into the paint filter may be facilitated by lightly tapping the side of the filter as it is being filled.

10.5 Allow sample to drain for 5 minutes into the beaker. If any liquid portion collects in the beaker during the 5 minute time period, the material is deemed to contain free liquids.

11. CALCULATION / DATA REDUCTION

11.1 No calculations are used for this method

11.2 If no liquid is collected, record the sample weight(s) used in the “Final Amount Value” column on the TALS batch Worksheet. Enter the sample result as “CNF” for Collected No Free liquid (see Figure 1).

11.3 If liquid is collected, record the sample weight(s) used in the “Final Amount Value” column on the TALS batch Worksheet. Enter the sample result as “CFL” for Collected Free Liquid (see Figure 2).

12. METHOD PERFORMANCE

12.1 The supervisor has responsibility to ensure that an analyst who performs this procedure is properly trained in its use and has the required experience. Performance is monitored by direct observation of the testing.

12.2 Demonstration of Capabilities – Prior to the analysis of samples, a Demonstration of Capabilities (DOC) as described in the QA Manual, must be performed initially, annually and any time a significant change is made to the analytical system.

12.3 MDLs are not performed for this procedure.

13. POLLUTION CONTROL

13.1 This method does not contain any specific modifications that serve to minimize or prevent pollution.

13.2 It is Eurofins TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage and reagent stability). Employees must abide by the policies in Section 13 of the Corporate Environmental Health and Safety Manual (CW-E-M-001) for "Waste Management and Pollution Prevention."

14. WASTE MANAGEMENT

14.1 Waste management practices are conducted consistent with all applicable rules and regulations. Excess reagents, samples and method process wastes are disposed of in an accepted manner. Waste description rules and land disposal restrictions are followed. Waste disposal procedures are incorporated by reference to PT-HS-001 (Waste Collection, Accumulation and Storage). The following waste streams are produced when this method is carried out:

14.1.1 Solid materials (gloves, paper products) go into waste stream #12 Lab Trash.

15. REFERENCES

15.1 Method 9095B, SW-846, Test Methods for Evaluating Solid Waste, Third Edition, Final Update IV, Revision 2, November 2004

15.2 PT-HS-001, Pittsburgh Facility Addendum Environmental Health & Safety Manual

15.3 PT-QA-M-001, Pittsburgh Laboratory Quality Assurance Manual

15.4 CW-E-M-001, Corporate Health and Safety Manual for "Waste Management and Pollution Prevention"

15.5 SOP PT-QA-021, Quality Control Program

15.6 SOP PT-QA-012, Selection and Calibration of Balances and Weights

15.7 SOP PT-QA-031, Internal Chain of Custody

16. METHOD MODIFICATIONS:

16.1 None

17. ATTACHMENTS

17.1 Figure 1 – TALS Batch Results for no liquid collected
17.2 Figure 2 – TALS Batch Results for liquid collected

18. REVISION HISTORY

18.1 Revision 1, 2/5/2008
18.2 Revision 2, 9/14/2010
18.3 Revision 3, 11/24/2014
18.4 Revision 4, 12/9/2016
18.5 Revision 4.1, 5/14/2019
18.6 Changes to current revision

SOP Section	Change from	Change to	Reason
1.2		Added text for handling modifications	Correction
3.2		Added NCM definition	Correction
6.1		Added Amazon as an alternate filter source	Correction

Figure 1 – TALS Batch Results for no liquid collected

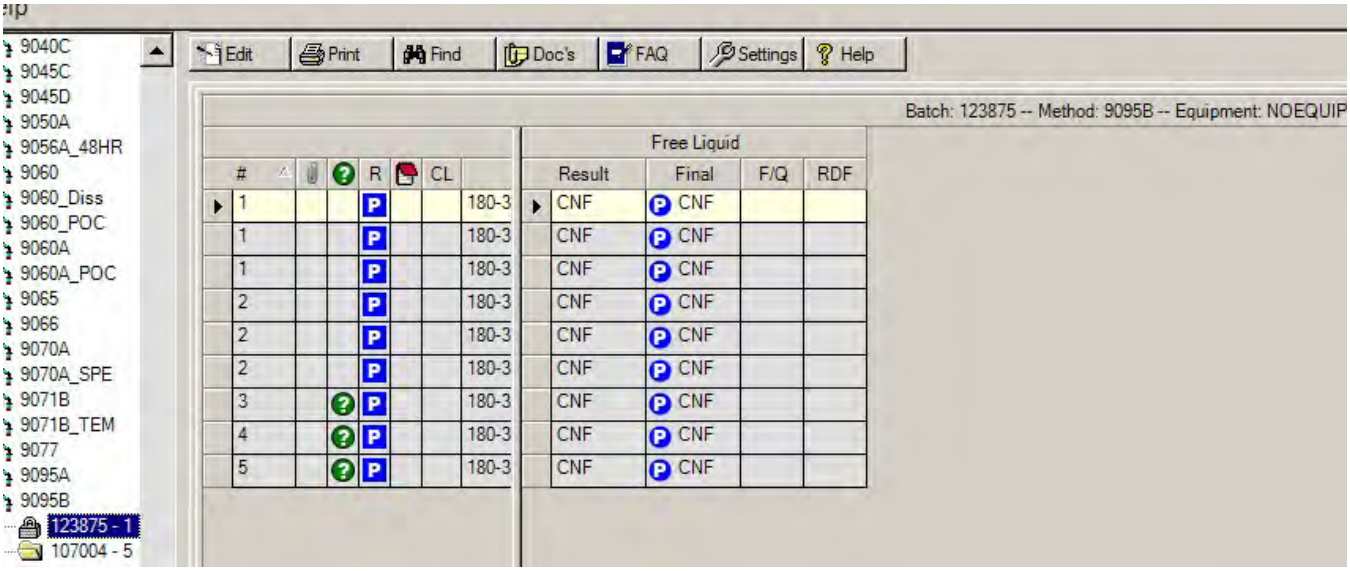
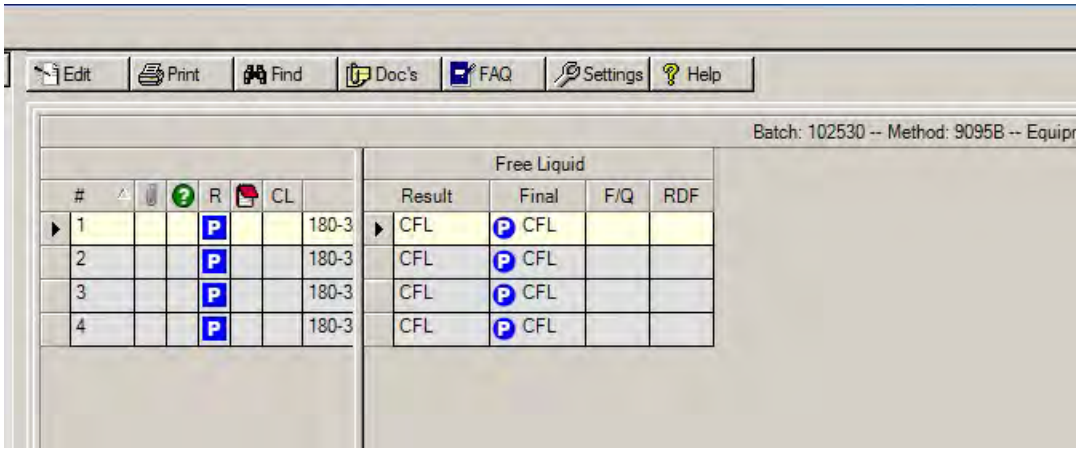


Figure 2 – TALS Batch Results for liquid collected



SOP # with Revision: PT-WC-036, Rev. 7
SOP Title: Flash Point by Setaflash (Small Scale) Closed-Cup Apparatus
Method: EPA 1020B, 1020C and ASTM Standard D 3278-96
Amendment Number: 1

Effective Date of Amendment:
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1. Briefly explain the reason for the update: Add information on second order curve which is allowed per method. Update of reference year.

2. Category for SOP Change:

☒ Typographical Corrections (Non-Technical) – Retraining Not Required.

☐ Typographical Corrections (Technical- define below) – Retraining is required.

☐ Procedural Changes (Define Below) – Retraining Required.

3. Summary of Procedure Change (circle to indicate if there are attachments to this form: No / Yes: # pages attached = ___)

Flash Point by Setaflash (Small Scale) Closed-Cup Apparatus
Method: EPA 1020B, 1020C and ASTM Standard D 3278-96

Does this change result in a Method Modification? Yes (No)
If yes, please explain below.

Any method modification will be incorporated into section 16 of the SOP at the next revision.

10.6.21
Approval Signature/Date: Technical

Amanda L Dull 10/06/21
Approval Signature/Date: QA Manager or Designee

Title: Flash Point by Setaflash (Small Scale) Closed-Cup Apparatus

Method: EPA 1020B and ASTM Standard D 3278-96

Approvals (Signature/Date):

1/13/2021	1/12/2021
Roseann Ruyechan Date Inorganics Department Manager	Steve Jackson Date Regional Safety Coordinator
12/31/2020	1/5/2021
Virginia Zusman Date Quality Assurance Manager	Deborah L. Lowe Date Laboratory Director

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1.0 Scope and Application

1.1 This standard operating procedure (SOP) describes the determination of whether a material will or will not flash at a specified temperature. This procedure is used in the characterization of the flash point of liquids, solids, wastes and products.

1.2 On occasion clients may request slight modifications to this SOP. Any modifications to this procedure must be approved by the QA department of the Laboratory or Technical Director, and documented fully in an NCM that accompanies the analytical batch.

2.0 Summary of Method

2.1 For liquid and wastes, 2 mls of sample, using a plastic transfer pipette, is introduced through a leak proof entry port into the tightly closed small scale tester or directly into the cup, which has been brought to within 3 ^{0}C (5 ^{0}F) below the expected flash point. For solid samples 1 to 5 grams of sample is transferred to the bottom of the cup with minimal exposure to air and tested.

2.2 As a flash / no- flash test, the expected flash point temperature may be a specification (e.g.140 ^{0}F). For specification testing, the temperature of the apparatus is raised to precise temperature of the specification flash point by slight adjustment by Temperature dial. After one minute, a test flame is applied inside the cup and note is taken as to whether the test sample flashes or not. The laboratory uses a set temperature of 140 ^{0}F, unless the project specifies a different temperature.

2.3 The flash point temperature is one measure of the tendency of the test specimen to form a flammable mixture with air under controlled laboratory conditions. It is only one of a number of properties which must be considered in assessing the overall flammability hazard of a material. Flash point is used in shipping and safety regulations to define *flammable* and *combustible* materials. One should consult the particular regulation involved for precise definitions of these classifications.

3.0 Definitions

3.1 Flash point – the lowest temperature at which application of an ignition source causes the vapors of a specimen of the sample to ignite under specified conditions of the test. The test specimen is deemed to have flashed when a flame appears and instantaneously propagates itself over the entire surface of the test specimen.

3.2 TALS – TestAmerica Laboratory Information Management System

3.3 NCM- Non-Conformance Memo - a system within TALS for the lab to communicate to project management and others when there is an anomaly seen with the samples or batch, or a QC failure.

3.4 Refer to the glossary in the Laboratory Quality Assurance Manual (PT-QA-M-001), current version, for general laboratory definitions.

4.0 Interferences

4.1 In order to eliminate contaminates from the test cup, it must be cleaned prior to being utilized. Clean cup with DI water after soil, solid or inorganic liquid analysis. Clean with acetone then DI water after organic sample analysis.

4.2 In certain mixtures, a non-flammable component of the sample may make inert the vapor space above the liquid, thus preventing a flash. Under this condition, the flash point is masked (no distinct flash as defined in section 3.1 is observed) resulting in the reporting of incorrectly high flash point or no flash point. **Continued heating and testing of such a mixture may represent a potential fire hazard, if not recognized.** Analysts should not perform this test unsupervised until they are experienced with sample reactions.

5.0 Safety

Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual (CW-E-M-001), the Pittsburgh Facility Addendum EH&S Manual (PT-HS-001) and this document. This procedure may involve hazardous material, operations and equipment. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

5.1 Specific Safety Concerns or Requirements
In the event a sample ignites in the test apparatus do not attempt to remove the sample. Turn off the apparatus and flame. The flame should go out when the cup is closed. If this does not happen the flame may be extinguished by covering the sample with a non-flammable material. After the apparatus has cooled the sample may be removed.

5.2 Consult the SDS for the liquefied propane gas used prior to beginning work with this SOP.

Material	Hazards	Exposure Limit (1)	Signs and Symptoms of Exposure
Liquefied Propane gas	Extremely flammable	TWA: 1800 mg/m^3 8 hours, 1000 ppm 8 hours	No known significant effects or critical hazards
(1) Exposure limit refers to the OSHA PEL, 2/2013			

6.0 Equipment and Supplies

The following items are recommended for performing this procedure. Equivalent items should only be used when they result in an improvement in quality, efficiency, productivity, or cost. An item can be considered equivalent if with its use, the analytical and QA/QC requirements in this SOP can be met.

6.1 Setaflash closed tester

6.2 Thermometer, 20-230°F with 1° graduations.

6.3 Lighter

6.4 Plastic transfer pipettes

6.5 Timer

6.6 Wooden spatulas for solids

7.0 Reagents and Standards

7.1 Liquefied Propane gas

8.0 Sample Collection, Preservation, Shipment and Storage

Matrix	Sample Container	Min. Sample Size	Preservation	Holding Time
Liquids	Glass jar / PTFE seal	250 mL	Cool, >0.0°C but ≤6.0°C	28 Days*
Solids	Glass jar/ PTFE seal	250 g	Cool, >0.0°C but ≤6.0°C	28 Days*

*No Holding Time is specified by the referenced methods or 40 CFR Part 136.3

8.1 Sample containers should be between at least 60% full and tightly sealed to preserve sample integrity.

8.2 No chemical preservation is required or appropriate.

9.0 Quality Control

9.1 The following quality control samples are prepared with each batch of samples:

Quality Controls	Frequency	Control Limit
Sample Duplicate	1 in 20 or fewer samples	Flash / No-Flash agreement

9.2 The thermometer has its calibration verified against a NIST thermometer annually. Calibration is documented in the thermometer calibration logbook. The calibration correction factor is noted on the thermometer and must be applied to all temperature measurements. Refer to SOP PT-QA-008 for details of thermometer calibration verification and correction factor requirements.

9.3 Anomalous situations occurring during sample analysis must be documented on the bench sheet and non-conformance memo must be issued if necessary.

10.0 Procedure – Method A – Flash/No Flash

10.1 Ensure that test cup is clean, dry and at room temperature before proceeding.

10.2 Switch the heater on, if not already at standby. To rapidly approach the specification flash temperature of the material under test, turn the heater dial fully clockwise causing red light to glow. The temperatures can be preset to 140 °F (or project specified temperature). When the thermometer indicates a temperature of about 3°C (5°F) below the specification or target flash point temperature, reduce the heat input to the test cup by slowly turning the heater control dial counter clockwise until signal light goes out. Make sure to apply the thermometer's correction factor to all readings.

10.3 After the test cup temperature has stabilized at the specification or target flash point, using transfer pipette, place the sample being tested into the test cup.

10.4 Set the timer for 1 min.

10.5 Open the gas control valve and light the pilot and test flames. Adjust the test flame size to 4mm, with pinch valve. A larger flame may obscure a sample flash. See Attachment 2 for flame size reference.

10.6 After one minute, a test flame is applied inside the cup and note is taken as to whether the test sample flashes or not.

10.7 Electronically record whether, it flashes or not and report either < 140°F or > 140°F in TALS (see Attachment 1 for TALS Screenshots).

10.7.1 If the project specifies a temperature other than 140°F, samples are analyzed at that temperature and results shall be reported as < or > the specified temperature, and the temperature difference detailed in an observation NCM.

10.8 If the sample flashes, repeat the test once, using a fresh portion of sample, to verify the result.

10.8.1 If the second portion of sample does not flash, repeat the analysis a third time to confirm flash or no flash for the sample, and narrate that a third portion was required.

10.9 Any anomalies seen during the analysis shall be documented using the Anomaly NCM for inclusion in the case narrative.

11.0 Calculations / Data Reduction

11.1 No calculations are performed for this test.

12.0 Method Performance

12.1 The supervisor has responsibility to ensure that an analyst who performs this procedure is properly trained in its use and has the required experience. On-going performance is monitored through internal QC and outside performance evaluation samples.

12.2 Demonstration of Capabilities can only be performed through observation of the test by another trained analyst or supervisor.

12.3 MDLs are not performed for this procedure.

13.0 Pollution Control

13.1 It is Eurofins TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage and reagent stability). Employees must abide by the policies in Section 13 of the Corporate Environmental Health and Safety Manual (CW-E-M-001) for "Waste Management and Pollution Prevention" and the Pittsburgh Facility Addendum EH&S Manual (PT-HS-001).

14.0 Waste Management

14.1 Waste management practices are conducted consistent with all applicable rules and regulations. Excess reagents, samples and method process wastes are disposed of in an accepted manner. Waste description rules and land disposal restrictions are followed. Waste disposal procedures are incorporated by reference to PT-HS-001.

14.2 Waste from samples that flash are placed into Mixed Solvent Waste, Waste #3.

15.0 References

15.1 Test Methods for Evaluating Solid Waste (SW-846), Method 1020 B, small scale Closed-Cup Method for Determining Ignitability, Rev. 2, August 2002

15.2 ATSM D3278 – 96, Flash point of liquids by small scale closed- cup apparatus, Test Method A

15.3 Laboratory Quality Assurance Manual, PT-QA-M-001

15.4 Pittsburgh Facility Addendum EH&S Manual, PT-HS-001

15.5 Thermometer Calibration and Temperature Monitoring. PT-QA-008

15.6 Equipment Maintenance, PT-QA-022

15.7 Subsampling, PT-QA-024

15.8 Internal Chain of Custody, PT-QA-031

16.0 Method Modifications

16.1 Flashpoint is tested at 140°F rather than the ASTM method recommended 230°F.

16.2 Viscosity of the sample is not tested as per section 10.4 of ASTM method.

17.0 Attachments

17.1 Attachment 1 - TALS Screenshots for Ignitability

17.2 Attachment 2 – Example Flame Size

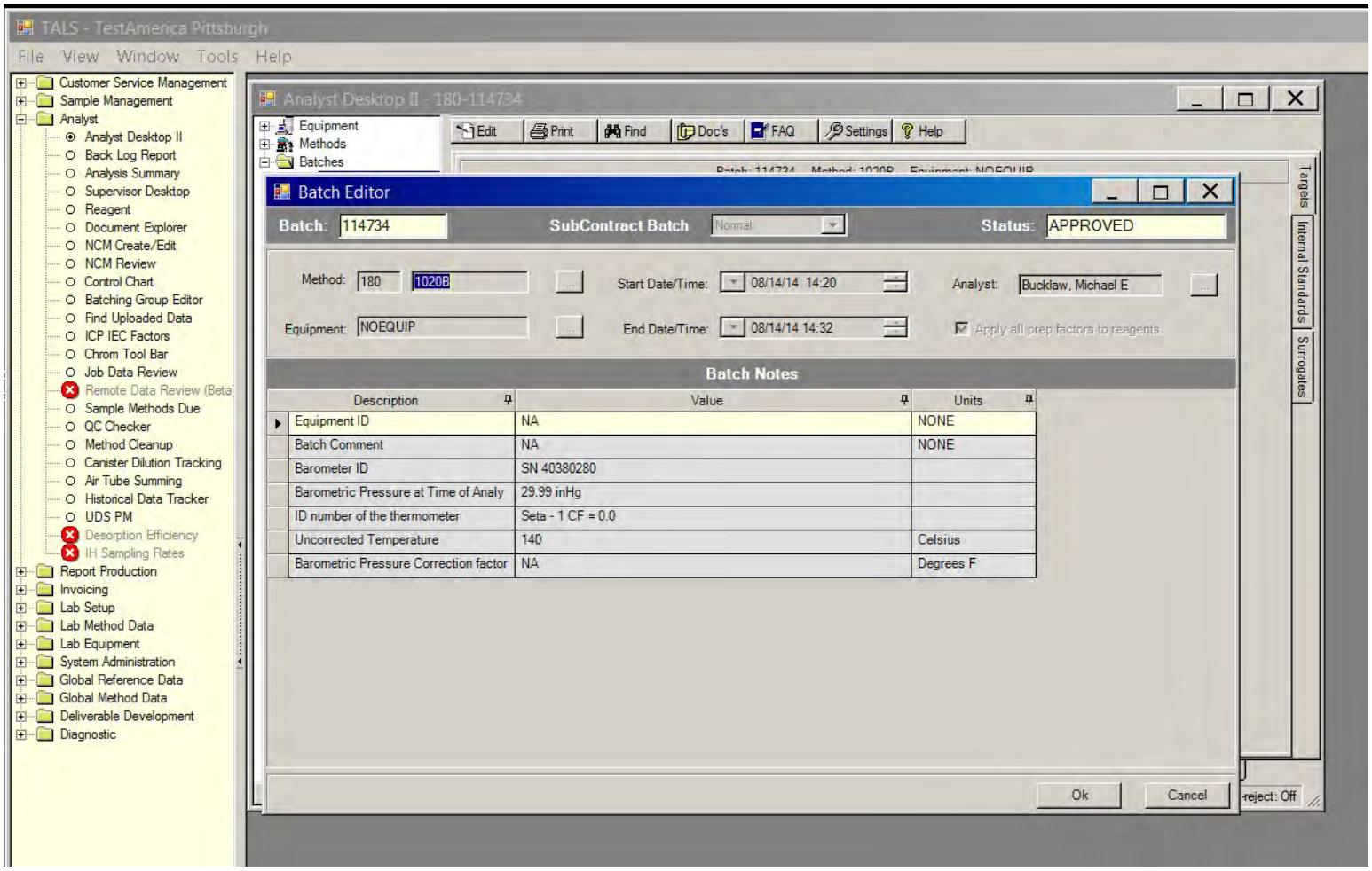
17.3 Attachment 3 – Examples of a Flash

18.0 0Revision History

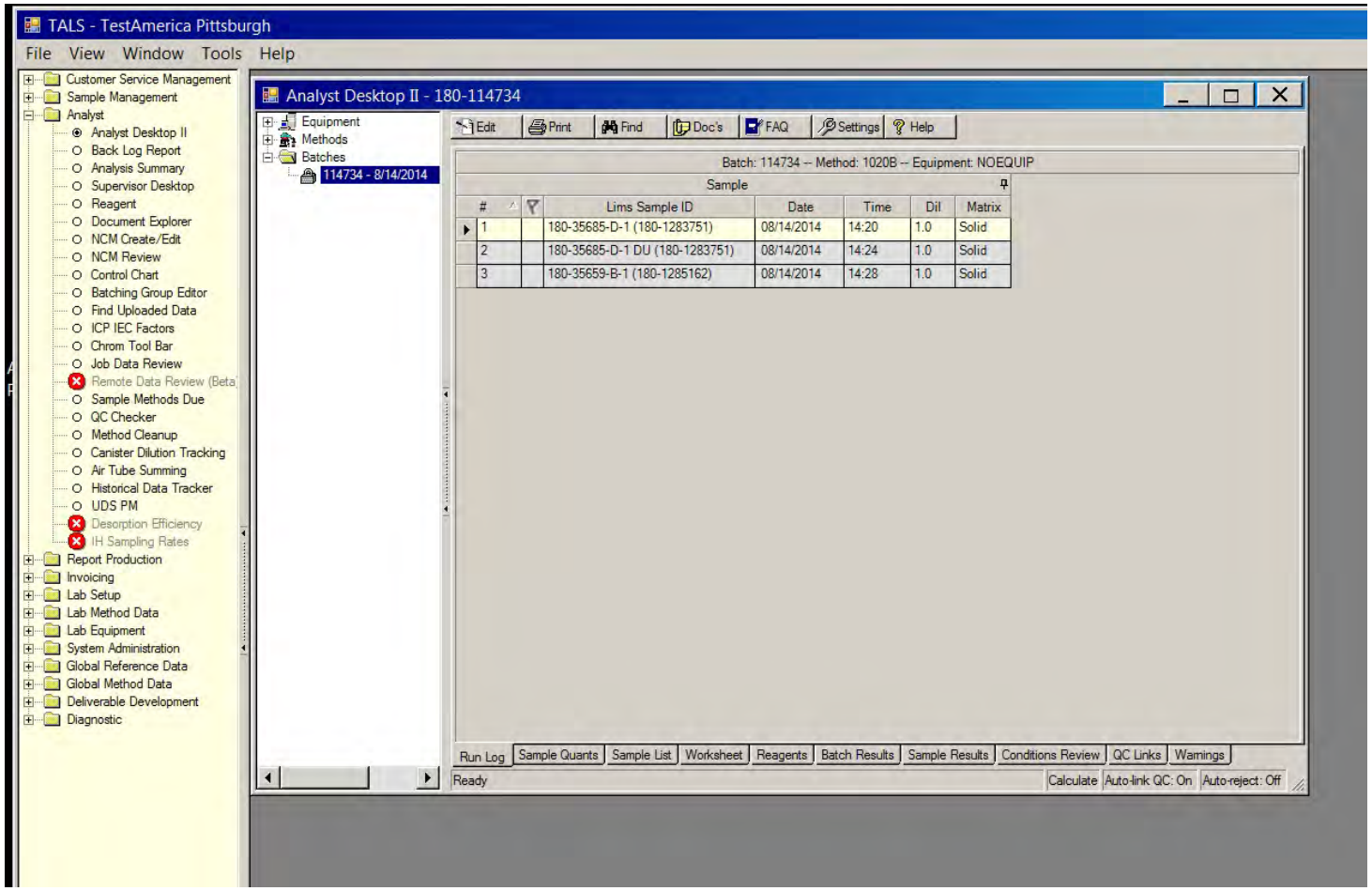
18.1 Revision 1, 10/14/2009
18.2 Revision 2, 3/2/2011
18.3 Revision 3, 5/24/2012
18.4 Revision 4, 9/11/2014
18.5 Revision 5, 1/6/2017
18.6 Revision 6, 2/1/2019
18.7 Revision 6.1, 5/14/2019
18.8 Changes to current revision

SOP section	Change from	Change to	Reason
1.2	Modifications per QAM	Modifications must be approved and documented	Clarification
10.5		Added larger flames could obscure flash & a reference to example flame in Attachment 2	Clarification
17.2 & Attachment 2		Added picture example for flame size	Clarification
17.3 & Attachment 3		Added picture examples of a flash	Clarification

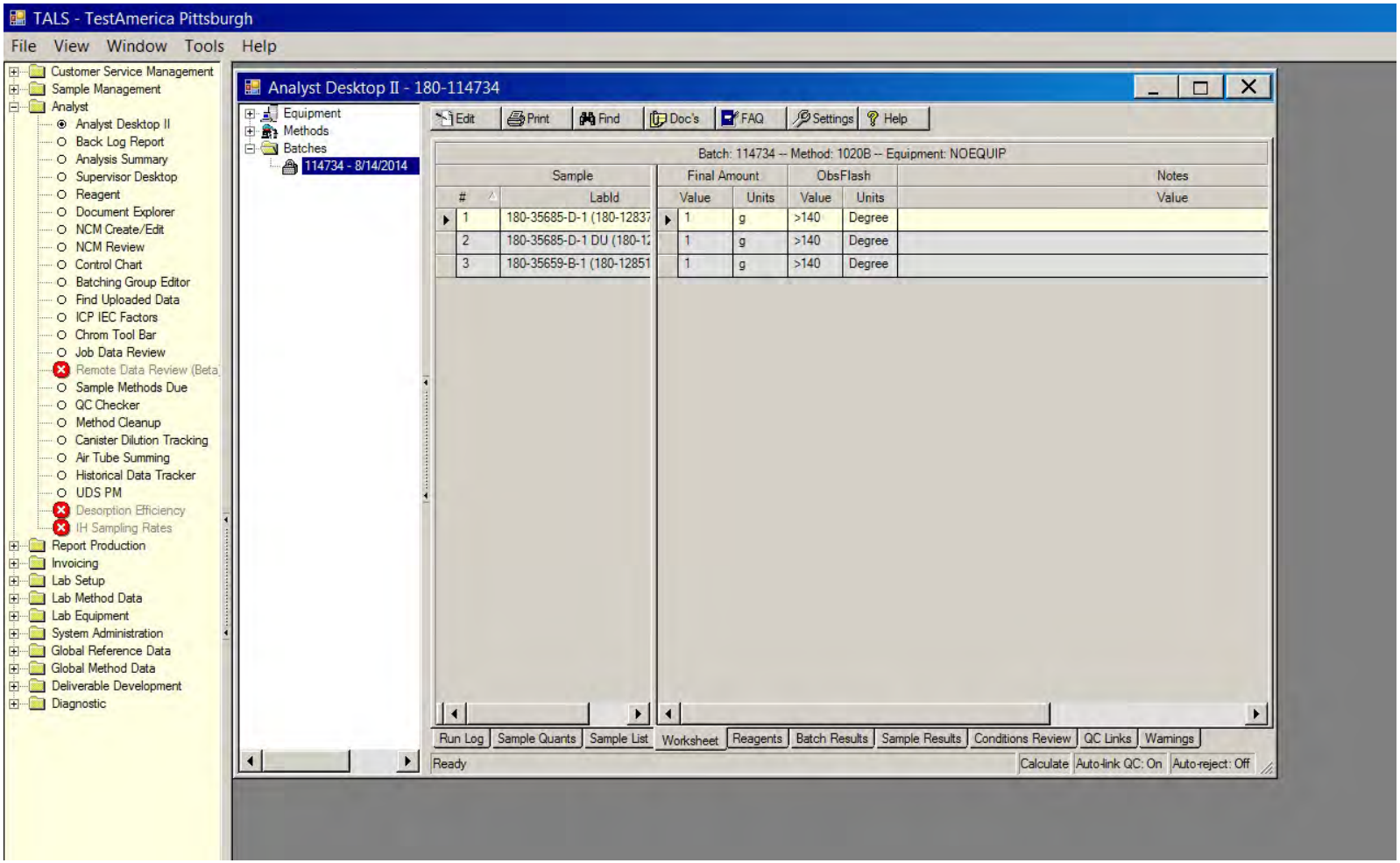
Attachment 1 – TALS Screenshots for Ignitability



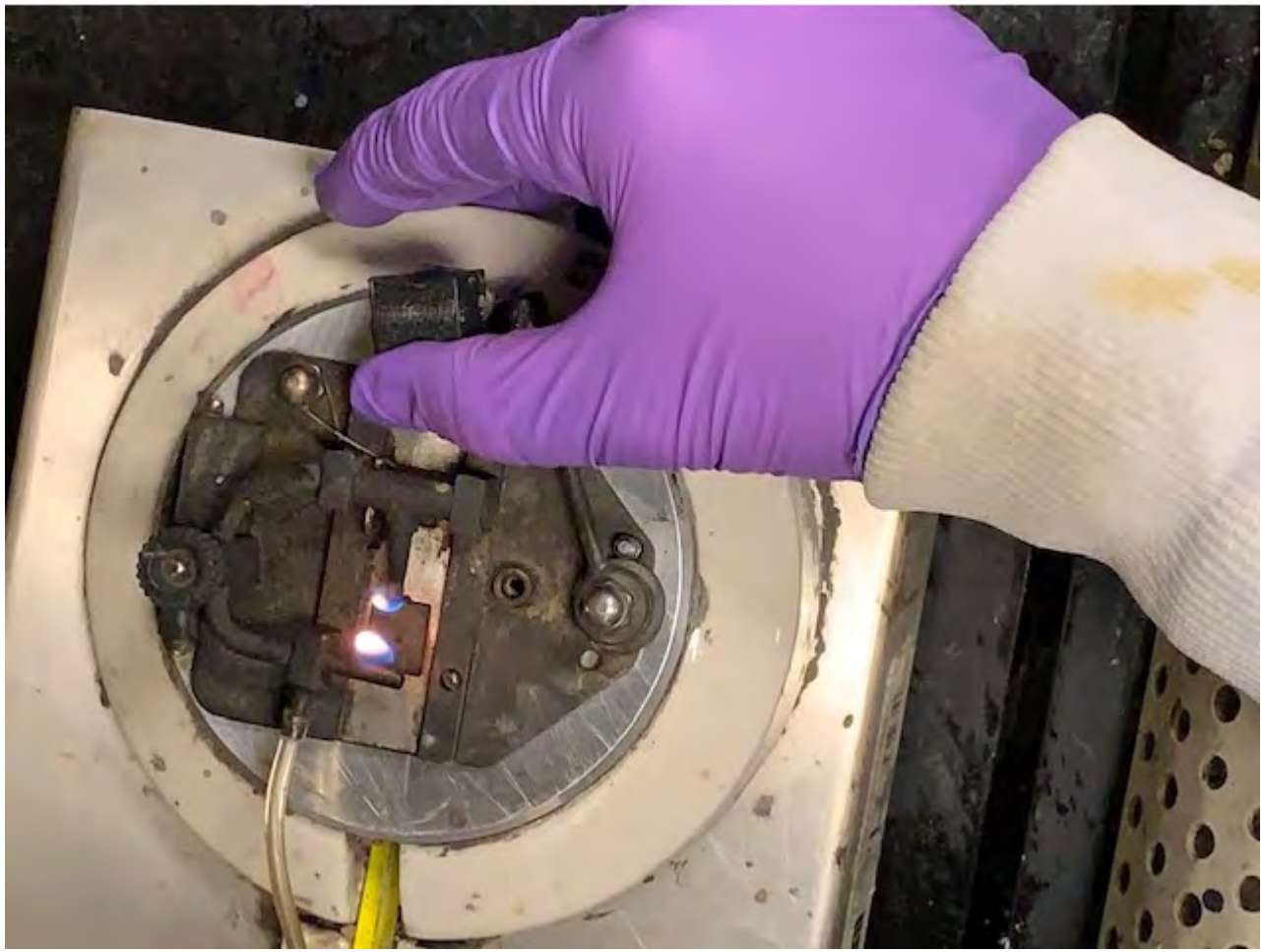
Attachment 1 – TALS Screenshots for Ignitability (cont.)



Attachment 1 – TALS Screenshots for Ignitability (cont.)



Attachment 2 – Flame Size Example



Attachment 3 – Examples of Flash





SOP # with Revision: PT-WC-043 Rev. 4
SOP Title: Determination of Total and Volatile Suspended Solids in Waters and Wastes
Amendment Number: 1

Effective Date of Amendment:
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1. Briefly explain the reason for the update: 40 CFR Part 136 Update of Reference Year

2. Category for SOP Change:

☒ Typographical Corrections (Non-Technical) – Retraining Not Required.

☐ Typographical Corrections (Technical- define below) – Retraining is required.

☐ Procedural Changes (Define Below) – Retraining Required.

3. Summary of Procedure Change (circle to indicate if there are attachments to this form: No / Yes: # pages attached = ___)

15.1. Standard Methods for the Examination of Water and Waste Water, 2011, 2015; Methods 2540D and 2540E, on-line editions

Does this change result in a Method Modification? Yes (No)
If yes, please explain below.

Any method modification will be incorporated into section 16 of the SOP at the next revision.

8.23.21
5.16.21

Approval Signature/Date: Technical

Amanda L Gulli 08/12/21

Approval Signature/Date: QA Manager or Designee

Title: Determination of Total and Volatile Suspended Solids in Waters and Wastes

Method(s): EPA 160.4 and Standard Methods 2540D & 2540E

Approvals (Signature/Date):

3/9/2020	3/11/2020
Roseann Ruyechan Date	Steve Jackson Date
Inorganics Department Manager	Regional Safety Coordinator
3/6/2020	3/12/2020
Virginia Zusman Date	Deborah L. Lowe Date
Quality Assurance Manager	Laboratory Director

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1. SCOPE AND APPLICATION

1.1. This SOP is applicable to the determination of total suspended solids and volatile suspended solids using gravimetric techniques. This SOP is based on the residue methods contained in EPA MCAWW and Standard Methods for the Examination of Water and Wastes (SM).

1.2. This SOP is applicable to drinking, surface, and saline waters and domestic and industrial wastes.

1.3. The Total Suspended Solids (TSS), or Non Filterable Residue, analysis protocol is based on SM 2540D.

1.4. The Volatile Suspended Solids (VSS) analysis protocol is based on EPA Method 160.4 and SM 2540E.

1.5. The methods cover a practical range of 2 mg/L to 20,000 mg/L.

1.6. The reporting limit is 2 mg/L for TSS and VSS, for a 1000mL sample. Analyzing smaller sample volumes will result in proportionally higher reporting limits.

1.7. On occasion clients may request slight modifications to this SOP. Any modifications must be approved by the QA department of the Laboratory of Technical Director, and documented fully in an NCM that accompanies the analytical batch.

2. SUMMARY OF METHOD

2.1. Total Suspended Solids (TSS): A well-mixed sample is filtered through a pre-weighed glass fiber filter with a pore size of 2.0µm (or smaller). The residue on the filter is dried to constant weight at 103 -105°C. The increase in weight over that of the pre-weighed filter represents the TSS content. The filtrate from this procedure may be used for TDS determination. The filter from this procedure may also be used for VSS analysis.

2.2. Volatile Suspended Solids (VSS): The residue obtained from the determination of total suspended solids is ignited at 550°C. VSS is determined from the weight loss after ignition.

2.3. Total Dissolved Solids may be performed at the same time, using the filtrate from the TSS filtration. Refer to SOP PT-WC-045 for details of this analysis.

3. DEFINITIONS

3.1. TALS – TestAmerica Laboratory Information Management System

3.2. NCM – Non-Conformance Memo - a system within TALS for the lab to communicate to project management and others when there is an anomaly seen with the samples or batch, or a QC failure.

3.3. Refer to the glossary in the Laboratory Quality Assurance Manual (PT-QA-M-001), current version.

4. INTERFERENCES

4.1. Method interferences may be caused by contaminants, reagents, glassware, and other sample processing hardware. All these materials must be routinely demonstrated to be free from interferences under the conditions of analysis by running method blanks.

4.2. Non-homogeneous samples may give erroneous results. Samples should be mixed as thoroughly as possible before removing an aliquot for analysis.

4.3. Non-representative particulates such as leaves, sticks, and other detritus should be excluded from the sample if it is determined that their inclusion is not desired in the final result. The presence/removal of these artifacts should be noted on the benchsheet.

4.4. Samples containing large amounts of solids may filter slowly. Prolonged filtration times resulting from filter clogging may produce high TSS results due to increased colloidal materials captured on the clogged filter. Analysis volumes should be reduced for such samples.

4.5. Oil and grease in the samples will cause unreliable results due to difficulty in drying to constant weight. Floating oil and grease, if present, should be included in the sample and dispersed by a blender device before aliquoting.

4.6. Filtration apparatus, filter material, pre-washing, post-washing, and drying temperatures are specified because these variables have been shown to affect the results.

4.7. The temperature at which the residue is dried has an important bearing on the results because weight losses due to volatilization of organic matter, mechanically occluded water, water of crystallization, and gases from heat-induced chemical decomposition, as well as weight gains due to oxidation, depend on temperature and time of heating.

4.8. Each sample requires close attention to desiccation after drying. Minimize opening the desiccator because moist air enters. Some samples may be more hygroscopic than those used as the desiccant and may take absorb additional water from the air as they cool.

4.9. Too much residue on the filter will crust over, entrapping water that will not be driven off during drying. Total residue must be limited to 200 mg (0.200g).

4.10. For samples high in dissolved solids, thoroughly wash the filter to ensure removal of dissolved material prior to TSS determination.

4.11. The volatile solids tests are subject to many errors due to the loss of water of crystallization, loss of volatile organic matter prior to combustion, incomplete oxidation of certain complex organics and decomposition of mineral salts during combustion. The results should not be considered an accurate measure of organic carbon in the sample.

4.12. Oils from hands can be transferred to the filters, causing a positive interference with these analyses. Handle filters only with forceps.

5. SAFETY

5.1. Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual (CW-E-M-001), the Pittsburgh Facility Addendum EH&S manual (PT-HS-001), and this document. This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

5.2. Oily samples or those that contain volatile chemicals may ignite during this procedure. In the case of a fire, the muffle should be turned off and allowed to cool before the sample can be removed and put under a hood.

5.3. Always use tongs, forceps or heat resistant gloves when placing filter pans in or removing them from ovens. Extra care should be used when using a muffle furnace, as temperatures are extremely high. Gloves designed for dealing with extreme temperatures must be used when handling any sample container that has been in a muffle furnace.

5.4. There are no materials used in this method that have a significant or serious hazard rating. A complete list of materials used in the method can be found in the reagents and

materials section. Employees must review the information in the SDS for each material before using it for the first time or when there are major changes to the SDS.

5.5. Eye protection that protects against splash, laboratory coat, and appropriate gloves must be worn while samples, standards, solvents, and reagents are being handled. Cut resistant gloves must be worn doing any other task that presents a strong possibility of getting cut. Disposable gloves that have been contaminated will be removed and discarded; other gloves will be cleaned immediately.

5.6. Exposure to chemicals must be maintained as low as reasonably achievable; therefore, unless they are known to be non-hazardous, all samples should be opened, transferred, and prepared in a fume hood, or under other means of mechanical ventilation. Solvent and waste containers will be kept closed unless transfers are being made.

5.7. All work must be stopped in the event of a known or potential compromise to the health and safety of a TestAmerica associate. The situation must be reported immediately to a laboratory supervisor or EH&S coordinator.

6. EQUIPMENT AND SUPPLIES

The following items are recommended for performing this procedure. Equivalent items should only be used when they result in an improvement in quality, efficiency, productivity, or cost. An item can be considered equivalent if with its use, the analytical and QA/QC requirements in this SOP can be met.

6.1. Analytical balance capable of weighing to 0.0001 g

6.2. Vacuum filtration apparatus

6.3. Vacuum pump equipped with moisture trap

6.4. Glass fiber filter disks, 47 mm, pore size $\leq$ 2µm, without organic binder - Whatman 934-AH, pre-cleaned and pre-weighed, or ProWeigh filters from Environmental Express.

6.4.1. If VSS analysis is to be performed, Environmental Express Proweigh Filters (part # F93447VOL) are used. Glass fiber filter disks, 47 mm, pore size $\leq$ 2µm, without organic binder, pre-cleaned and dried at 550^{o}C and pre-weighed

6.4.2. If pre-cleaned filters are not used, the preparation steps in section 10.3.1.2 must be used prior to filtering samples.

6.5. Desiccators providing sufficient space for storage of samples in process separate from filters and evaporating dishes

6.6. Desiccant containing a color indicator of moisture concentration or an instrumental indicator

6.7. Drying oven set at 103 -105°C

6.8. Muffle furnace set at 550°C ± 50°C

6.9. Thermometers, NIST traceable

6.10. Graduated cylinders, to deliver (TD), assorted sizes, Class A

6.11. Aluminum weighing dishes large enough to hold a 47 mm filter - (Pre-weighed filters are purchased already in Aluminum dishes.)

6.12. Forceps

6.13. Zetex gloves or other gloves capable of providing protection at 550°C

6.14. Wide-bore serological volumetric pipettes

7. REAGENTS AND STANDARDS

The following items are recommended for performing this procedure. Equivalent items should only be used when they result in an improvement in quality, efficiency, productivity or cost. An item can be considered equivalent if with its use, the analytical and QA/QC requirements in this SOP can be met. Please refer to the SDS prior to the use of any reagent or standard.

7.1. Reagent water - Deionized water, with conductance <1 umhos/cm – to be used for blanks and rinses

7.2. The LCS standard solution is commercially purchased. The true value varies from lot to lot when received from the manufacturer and the calculation is adjusted accordingly by the laboratory. The standard is received with a certificate of analysis with the true value listed on it and is retained by the laboratory.

7.2.1. If the solution needs to be made by the laboratory the procedure is as follows: LCS solution (500 mg/L TSS and TDS, 1000 mg/L TS): Place 500.0 mg of diatomaceous earth (infusorial earth or celite 545) and 500 mg sodium chloride into a 1000 mL volumetric flask and dilute to volume with deionized water. Mix well. Prepare fresh every three months.

8. SAMPLE COLLECTION, PRESERVATION, SHIPMENT AND STORAGE

8.1. Samples should be collected in either plastic or glass bottles. No chemical preservation is appropriate.

8.2. Samples must be stored at >0.0℃ but ≤6.0℃ to minimize microbiological decomposition of solids. Bring samples to room temperature before analysis.

8.3. To meet method SM2540D requirements, sufficient sample volume, **up to 1 L**, must be filtered to produce at least 2.5mg (0.0025g) of final residue on the filter.

8.3.1. Smaller samples sizes may be used for samples that are high in solids. Historical data or visual inspection may be used to choose a different sample size for initial analysis, as long as the minimum (2.5mg or 0.0025g) and maximum (200mg or 0.200g) limits are met.

8.4. Holding time for TSS and VSS determination is 7 days from the date of sampling to begin the analysis.

9. QUALITY CONTROL

The TestAmerica QC Program document (PT-QA-021) provides further details of the QC and corrective action guidelines presented in this SOP. Refer to this document if additional guidance is required.

9.1. Method Blank (MB): One method blank must be processed with each batch of 20 or fewer samples. The method blank consists of 1L of reagent water that is carried through the entire analytical procedure, including filtration as applicable. The method blank is used to identify any system and process interferences or contamination of the analytical system that may lead to the reporting of elevated analyte concentrations or false positive data. Blank results must be less than the RL. If the MB result is above the RL, all associated samples must be reanalyzed unless one of the following conditions exists.

- If a Blank result is above the RL, but solids are not detected above the RL in the associated samples, the data may be reported with qualifiers and using the “Method Blank – Report, ND” NCM.

- If the sample concentrations are greater than 10x the concentration seen in the MB, results may be reported with qualifiers and using the "Method Blank – Report, 10X" NCM

9.1.1. High blank results indicate contamination. Make sure the filtering apparatus is clean before repeating the analysis.

9.1.2. If MB results are negative (filter weighs less after analysis), this indicates that the balance was not operating correctly or the filter/weighing dish was not completely dry when the initial weights were measured. If MB results are below the negative RL (a change of -0.0025g for example), all samples with concentrations <10X the RL should be reanalyzed if possible. If reanalysis is not possible, the negative blanks and possible negative bias for the sample results must be narrated using the "Other – Deficiency" NCM.

9.2. Laboratory Control Sample (LCS): One LCS must be processed with each batch of 20 or fewer samples. The LCS must be carried through the entire analytical procedure. The LCS is used to monitor the accuracy of the analytical process. On-going monitoring of the LCS results provides evidence that the laboratory is performing the method within acceptable accuracy and precision guidelines.

9.2.1. No LCS is available for volatile residue procedures.

9.2.2. Refer to Section 7.2.1 for instructions on how to prepare the LCS solution, unless using a purchased standard. The LCS analysis volume must be calculated to produce a final residue between 2.5 and 200mg (0.0025 and 0.20g).

9.2.3. The LCS percent recovery must be within 80 -120% of the true or certified value.

9.2.4. If LCS recovery is outside the control limits, all associated samples must be reanalyzed with an acceptable LCS, unless the original LCS recovery is high and samples results are <RL.

9.2.4.1. If reanalysis is not possible due to limited sample quantity or holding time considerations, the project manager must be notified so that the client can be informed. Results reported associated with a failed LCS must be reported with the appropriate result qualifier and the failure narrated through the appropriate NCM:

- For high LCS recovery and associated samples <RL, use "LCS/LCSD - %R High".
- If samples are reanalyzed due to failed LCS, and the reanalysis occurs past the holding time, use "LCS/LCSD - %R, RX/RA Batch, Outside".
- If there is insufficient sample volume to perform a reanalysis, use "LCS/LCSD – Insufficient Sample".

9.2.5. Poor LCS results are usually the result of contamination or poor mixing. Make sure to shake the LCS and all samples very well before pouring out the portion to be filtered or analyzed. Do not allow the sample to settle in the graduated cylinder.

9.3. Duplicate (D): A duplicate sample analysis is required with every 10 samples or less for all methods.

9.3.1. Sample and Duplicate results must agree within 10%RPD The failure of the sample duplicates to meet precision criteria must be addressed in the case narrative.

9.3.2. Make sure to shake all samples very well before pouring out each portion to be filtered or analyzed to improve homogenization and precision. Do not allow the sample to settle in the graduated cylinder before analyzing.

9.3.3. On-going method precision is documented using LCS control charting.

10. PROCEDURE

10.1. Calibration and Standardization

10.1.1. Proper balance operation will be verified daily or prior to sample analysis by following the balance calibration SOP, PT-QA-12. Analytical balance calibration must be verified at least every 24 hours, and prior to using the balance each day. Weights used for calibration verification, prior to initial and read-back weighings, are documented in the TALS batch information.

10.1.1.1. For TSS and VSS, balances are verified using 2mg, 0.300g, 10g and 120g weights.

10.1.1.2. Balance reading for the 2mg weight must be 0.0018g - 0.0022g; for the 0.300 g weight must be 0.2995g – 0.3005 g; for the 10g weight must be 9.9900g – 10.0100g and for the 120g weight must be 119.88000g – 120.1200g. If any reading falls outside these limits, the balance must be recalibrated and the verification performed again with all weights in the group.

10.1.2. Oven temperature must be checked daily and recorded in the oven temperature logbook. Oven temperature must be kept between 103-105^{o}C. Volatile Suspended Solids are ignited in the muffle furnace at 550 $\pm$ 50^{o}C. The temperature of the muffle furnace used for volatile residues analyses is documented at the time of use.

Note: If oven temperatures are not within the required range, the temperature setting must be adjusted to bring it within range. The drying/burn time for the samples does not begin until oven or furnace is within the required temperature range.

10.2. Procedure

10.2.1. Any variation in procedure shall be completely documented using a Nonconformance Memo and must be approved by a Supervisor or Manager. If contractually required, the client shall be notified.

10.2.2. All samples are to be electronically checked out of sample control with the TALS internal chain of custody (ICOC) program.

10.2.3. Proper sample identification is extremely important in any analytical procedure. Labeling of filters holders must be done in a manner to ensure connection with the proper sample. Labeling must be done in a way that will not be compromised by the heat used. For VSS, the sample ID is pressed into the aluminum pan.

10.2.4. Non-representative particulates such as leaves, sticks, and other detritus should be excluded from the sample if it is determined that their inclusion is not desired in the final result. The presence/removal of these artifacts should be noted on the TALS Worksheet Notes or Batch Information Comments.

10.2.5. If samples are visibly oily, this should be noted on the TALS Worksheet Notes or Batch Information Comments.

10.2.6. If there is limited sample volume or high solids content, smaller amounts of sample may need to be processed than detailed in the following sections. This occurrence must be noted on the TALS Worksheet. Reporting limits are adjusted according to the actual sample volume used.

10.2.7. All weights are recorded to the nearest 0.1mg directly from the balance into the TALS Worksheet using balance upload. If the balance upload is not working, the weights are recorded in the Wet Chemistry Raw Data Logsheet for transfer to the TALS Worksheet. If the manual Logsheet is used, it must be scanned to a pdf and attached to the TALS batch as raw data.

10.2.8. Prepare TALS analytical batches prior to beginning analysis, as the batch must be available to upload initial weights and record sample volumes used.

10.2.9. Scan the barcode on the filter's pan for pre-weighed filters. This will upload both the pan ID and the filter weight into the "Dish" column on the TALS Worksheet. Manually enter the filter weight into the "Tare Weight Value" column. (See Figure 4.)

10.3. Total Suspended Solids – SM 2540D

10.3.1. Preparation of Glass Fiber Filter Disc:

10.3.1.1. Prepared and pre-weighed filters are purchased for use under this method. The certificate of analysis is retained for the filters. The filter lot number is documented in the TALS Batch Information. Scan the barcode on the filter's pan to enter the tare weight of the filter into the TALS Batch Worksheet.

- When a new lot/box of pre-weighed filters is opened for use, 5% of the filters are chosen at random, and weighed to confirm that the weights provided are correct, within 0.0004g. If the filter weight does not agree, that filter is not used. If more than 5 filters checked per box do not agree, the box is not used for analysis.
- For TSS only analysis, use Whatman 934-AH filters or ProWeigh filters from Environmental Express. If VSS analysis is to be performed, use only Environmental Express ProWeight filters.

10.3.1.2. If prepared/pre-weighed filters are not used:

- Place the glass fiber filter discs, one at a time, on the membrane filter apparatus with wrinkled surface side up.
- While vacuum is applied, wash the disc with three successive 20 mL volumes of reagent water.
- Remove all traces of water by continuing to apply vacuum after water has passed through and discard washings.
- Remove filter from membrane filter apparatus and place in a labeled, aluminum weighing dish. Dry the prepared filters in an oven at 103 -105°C for at least one hour.
- If volatile suspended solids (VSS) are to be determined as well as total suspended solids, the filter must be heated at 550 $\pm$ 50^{o}C for 15 minutes.
- Using forceps, remove the pans from the oven and place in a desiccator and cool to room temperature.
- Weigh the cooled filter using an analytical balance to the nearest 0.1 mg. Handle the filters or pan/filter only with forceps. This weight is automatically uploaded into the TALS Worksheet and associated with the TALS sample batch using the Balance Link program (see Figure 4).
- Repeat the drying, cooling, and weighing cycle until a constant weight is obtained or weight loss is less than 0.5 mg.

10.3.2. Selection of Sample Volume

10.3.2.1. For the Method Blank and clean samples, filter 1000mL.

10.3.2.2. If during filtration of this initial volume, the filtration rate drops rapidly or if filtration time exceeds 5 - 10 minutes, a smaller volume of sample should be processed. Sample cannot be poured out of the filtration apparatus once added. If a smaller sample size is needed, the analysis must be started over.

10.3.2.3. If the sample appears high in TSS, start with a sample volume < 100mL. If the sample filters rapidly and no significant residue remains on the filter, more volume can be added. If volumes less than 10 mL are required, measure the sample volume using a wide-bore serological pipette, so as not to exclude larger particles from the sample aliquot.

10.3.2.4. For TSS, a residue of at least 2.5mg must be obtained, or a liter of water must be filtered.

10.3.3. Sample Analysis:

10.3.3.1. Assemble the filtering apparatus, and rinse with DI water.

10.3.3.2. Weigh filter, or scan pre-weighed filter pan, and enter the filter weight into the TALS worksheet. Place the filter in the apparatus, wrinkled surface side up. Pre-wet the filter using a few mL of reagent water and begin suction.

10.3.3.3. Shake the sample vigorously and quantitatively transfer the appropriate volume of the sample to the filter using a graduated cylinder or wide-bore pipette. For samples which are visibly high in TSS or are otherwise slow to filter, filter 25mL at a time until filtration slows. Record the volume of sample filtered (to the nearest mL) on the TALS Worksheet.

Note: If Total Dissolved Solids (TDS) is also required, the filtrate from the TSS analysis may be used. Refer to SOP PT-WC-045 for details of TDS analysis.

10.3.3.4. Remove all traces of water by continuing to apply vacuum after the sample has passed through.

10.3.3.5. With suction on, rinse the graduated cylinder, filter, suspended solids residue, and filter funnel wall with three approximately 10 mL portions of reagent water allowing complete drainage between washings.

10.3.3.6. Remove all traces of water by continuing to apply vacuum for about three minutes after the sample has passed through.

10.3.3.7. Carefully remove the filter from the filter support and transfer back to its aluminum weighing dish.

10.3.3.8. Dry the filter at least one hour at 103 -105°C.

10.3.3.9. Record the date, time, and oven temperature on the TALS Batch Information when the samples are initially placed in the oven and again when they are removed from the oven (see Figure 3).

10.3.3.10. Using forceps, remove the weighing dish from the oven and place in a desiccator.

10.3.3.11. Cool the samples to room temperature in the desiccator. Weigh the filter and upload the weight to the TALS Worksheet (see Figure 4).

10.3.3.12. If the residue is >0.2g (200mg), start the analysis over again with a smaller sample volume. If insufficient volume is available for reanalysis, proceed with the steps below, and narrate that the results were determined from a residue >0.2g. Longer drying times may be required to reach constant weight.

10.3.3.13. Return the samples to the oven for at least another hour, cool in a desiccator, and reweigh. Repeat the drying, cooling, desiccating, and weighing cycle until a constant weight is obtained. Constant weight is considered a weight change < 0.5 mg or < 4% of the previous weight, whichever is less. If constant weight has not been achieved after three cycles of heating, cooling and weighing; document the 3 weights and do not perform any further cycles. Document that only 3 heating cycles were performed using an NCM. The lowest weight of the 3 is used for calculating the final result.

NOTE: All dates, times, temperatures and weights are recorded in the TALS Batch.

10.3.3.14. If volatile suspended solids are to be determined, treat the residue according to Section 10.4.

10.3.3.15. TALS will automatically calculate the sample result from the data entered into the Worksheet. Check the calculations using the formula given in Section 11.1. Use the lowest final weight achieved for calculating TSS.

10.4. Volatile Suspended Solids – 160.4 and SM 2540E

10.4.1. Heat the muffle furnace up to temperature (550 ± 50 °C).

10.4.2. Place the filter from the TSS analysis (Section 10.3.3.) into an aluminum pan that does not have a paper label. Press the sample ID into the pan. Place the pans into the muffle furnace to ignite the residue.

10.4.3. Record the date, time, and muffle furnace temperature in the TALS Batch Information when the samples are initially placed in the oven and again when they are removed from the oven (see Figure 5 or 7).

10.4.4. Heat the samples for at least 20 minutes. Typically, 15 - 20 minutes ignition is sufficient for 200 mg of residue. Because TSS residue is restricted to <200mg, only a single ignition is required for VSS.

10.4.5. Using tongs and heat resistant gloves, remove the weighing dish from the muffle furnace and place in a desiccator and cool to room temperature.

10.4.6. Weigh the cooled pan, and upload the weight into the TALS Batch Worksheet (see Figure 6 or 8).

10.4.7. Return filter to furnace for another 15 minutes. Cool in desiccator. Weigh and upload weight into TALS.

10.4.8 TALS will calculate the final results. Check calculations using the formula given in Section 11.2. Use the lowest final weight achieved for calculating VSS.

11. CALCULATIONS / DATA REDUCTION

11.1. Calculate Total Suspended Solids as follows:

$$\text{Total Suspended Solids, mg / L} = \frac{(A - B) \times 1000}{C}$$

Where: A = weight of filter + residue (mg)
B = weight of filter (mg)
C = volume of sample filtered (mL)

11.2. Calculate Volatile Suspended Solids as follows:

$$\text{Volatile Suspended Solids, mg / L} = \frac{(A - B) \times 1000}{C}$$

Where: A = weight of residue + filter before ignition (mg)
B = weight of residue + filter after ignition (mg)
C = volume of sample (mL)

11.3. The relative percent difference (RPD) of duplicates are calculated according to the following equation:

$$RPD = 100\left[\frac{|DU1 - DU2|}{\left(\frac{DU1 + DU2}{2}\right)}\right]$$

Where: DU1 = Sample result
DU2 = Sample duplicate result

11.4. LCS Percent Recovery

$$\text{LCS Percent Recovery} = \left(\frac{\text{Observed Conc. in LCS}}{\text{True LCS Conc.}}\right) \text{x } 100 \text{ Percent}$$

11.5. If multiple weighing cycles are required, the lowest final sample weight is used for calculating solids content.

11.6. If smaller or larger sample volumes are processed than are specified in the method, the reporting limit must be adjusted accordingly.

11.7. Sample results should be reported according to the following significant figure rules:

Significant Figures	Sample Result
2	< 10
3	≥ 10

12. METHOD PERFORMANCE

12.1. The supervisor has responsibility to ensure that an analyst who performs this procedure is properly trained in its use and has the required experience. Performance is monitored through internal QC and outside performance evaluation samples. Please refer to the QA Manual for additional information concerning Precision and Accuracy.

12.2. Demonstration of Capabilities – Prior to the analysis of samples, a Demonstration of Capabilities (DOC) as described in the QA Manual, must be performed initially, annually and any time a significant change is made to the analytical system.

12.3. MDLs are calculated annually from Method Blank data according to SOP PT-QA-007, and are compared to the Reporting Limit. Results are not reported below the RL.

13. POLLUTION CONTROL

13.1. It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage and reagent stability). Employees must abide by the policies in Section 13 of the Corporate Environmental Health and Safety Manual (CW-E-M-001) for "Waste Management and Pollution Prevention" and the Pittsburgh Facility Addendum EH&S Manual (PT-HS-001).

13.2. This method does not contain any specific modifications that serve to minimize or prevent pollution

14. WASTE MANAGEMENT

14.1. Waste management practices are conducted consistent with all applicable rules and regulations. Excess reagents, samples and method process wastes are disposed of in an accepted manner. Waste description rules and land disposal restrictions are followed. Waste disposal procedures are incorporated by reference to PT-HS-001.

14.2. The following waste streams are produced when this method is carried out.

14.2.1. Unpreserved waste samples. This waste is disposed down a lab sink.

14.2.2. Aluminum weighing tins, contaminated filter and filter residue generated by the analysis. This waste is disposed of in trashcans with the building trash.

15. REFERENCES

15.1. Methods for the Chemical Analysis of Water and Wastes, EPA-600/4-79-020, March 1979: Method 160.4.

15.2. Standard Methods for the Examination of Water and Waste Water, 2011; Methods 2540D and 2540E, on-line editions

15.3. SOP PT-QA-007, Detection Limits

15.4. SOP PT-QA-021, TestAmerica Pittsburgh QC Program

15.5. SOP PT-QA-012, Selection and Calibration of Balances and Weights

15.6. TestAmerica Pittsburgh Laboratory Quality Assurance Manual, PT-QA-M-001

15.7. SOP PT-QA-006, Procurement of Standards and Materials; Labeling and Traceability

15.8. SOP PT-QA-008, Thermometer Calibration and Temperature Monitoring

15.9. SOP PT-QA-016, Nonconformance & Corrective Action System

15.10. SOP PT-QA-031, Internal Chain of Custody

15.11. SOP PT-HS-001, Pittsburgh Facility Addendum EH&S Manual

16. METHOD MODIFICATIONS

16.1. Samples are not taken through the drying, cooling, and weighing cycle more than three times. If the sample fails to reach constant weight within three cycles, this is documented in a Nonconformance Memo and the lowest final weight is used for the calculation.

17. ATTACHMENTS

17.1. Figure 1: Total Suspended Solids (TSS) Flow Chart
17.2. Figure 2: Volatile Suspended Solids (VSS) Flow Chart
17.3. Figure 3: TALS Batch Information – Method 2540D
17.4. Figure 4: TALS Batch Worksheet – Method 2540D
17.5. Figure 5: TALS Batch Information – Method 2540E
17.6. Figure 6: TALS Batch Worksheet – Method 2540E
17.7. Figure 7: TALS Batch Information – Method 160.4
17.8. Figure 8: TALS Batch Worksheet – Method 160.4

18. REVISION HISTORY

18.1. Revision 0, 11/24/2014
18.2. Revision 1, 1/6/2017
18.3. Revision 2, 1/31/19
18.4. Revision 3, 4/26/2019
18.5. Changes to current revision

SOP section	Change from	Change to	Reason
9.3.1	Removed 5x RL statement		Correction
10.1.2		Added note that is oven is outside temperature range the setting must be adjusted to bring it within range.	Clarification
12.3 & 15.3	CA-Q-A-006	PT-QA-007	Document change

Figure 1 - Total Suspended Solids (TSS)

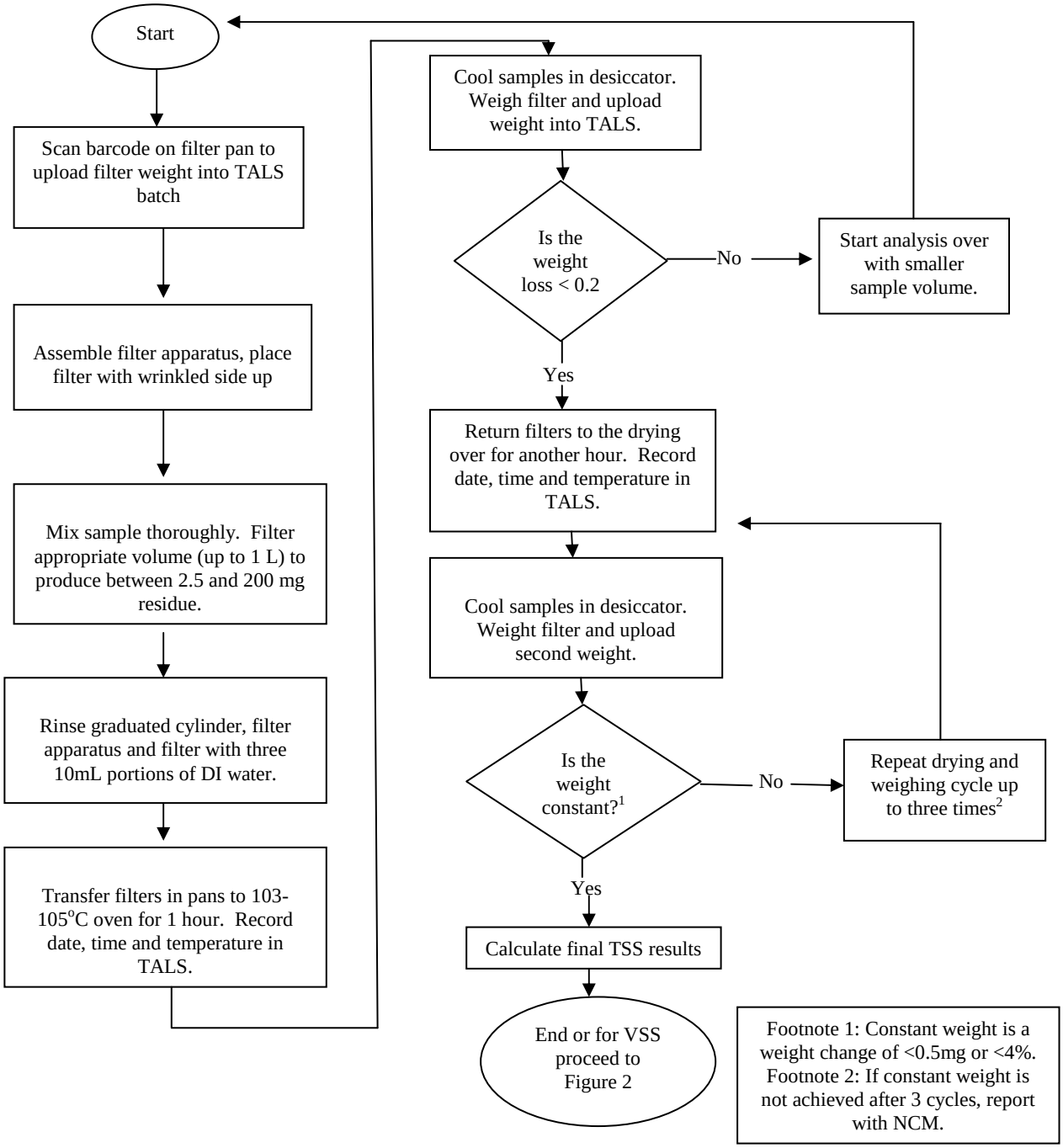
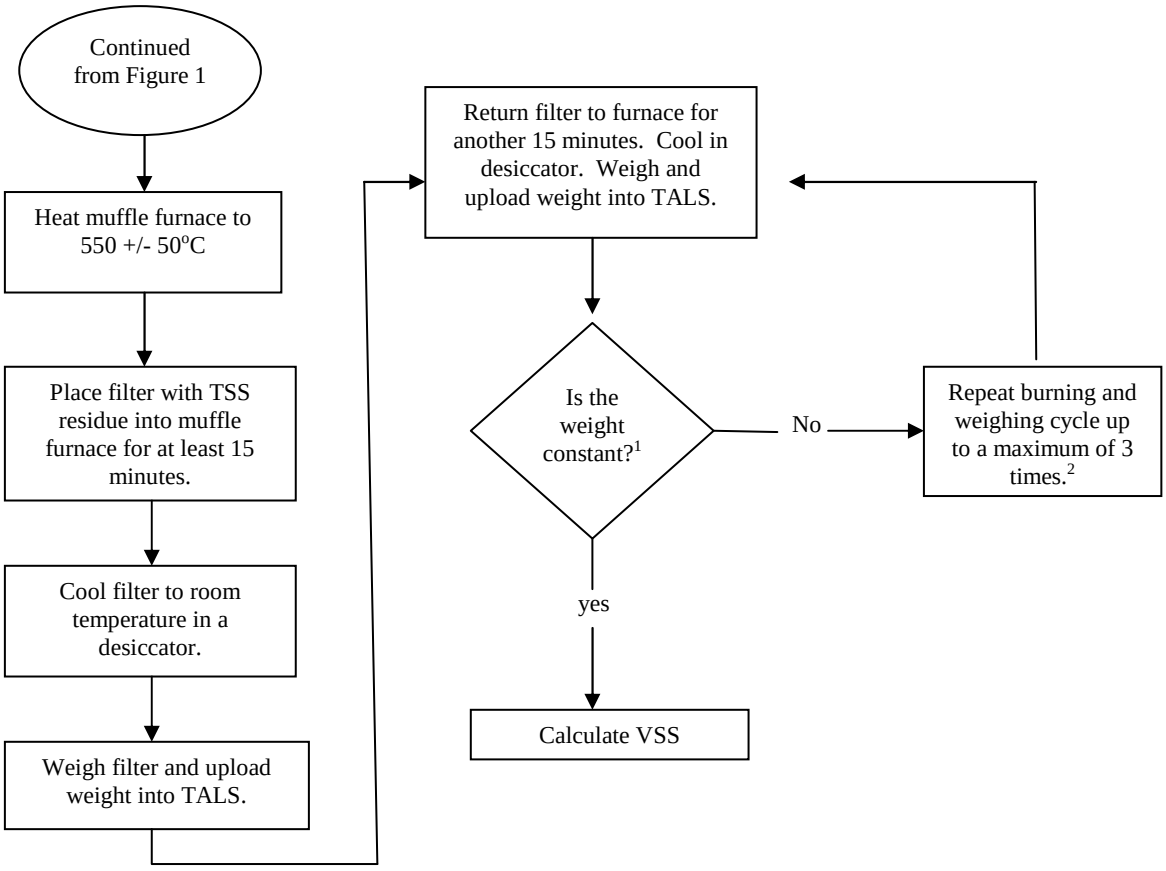
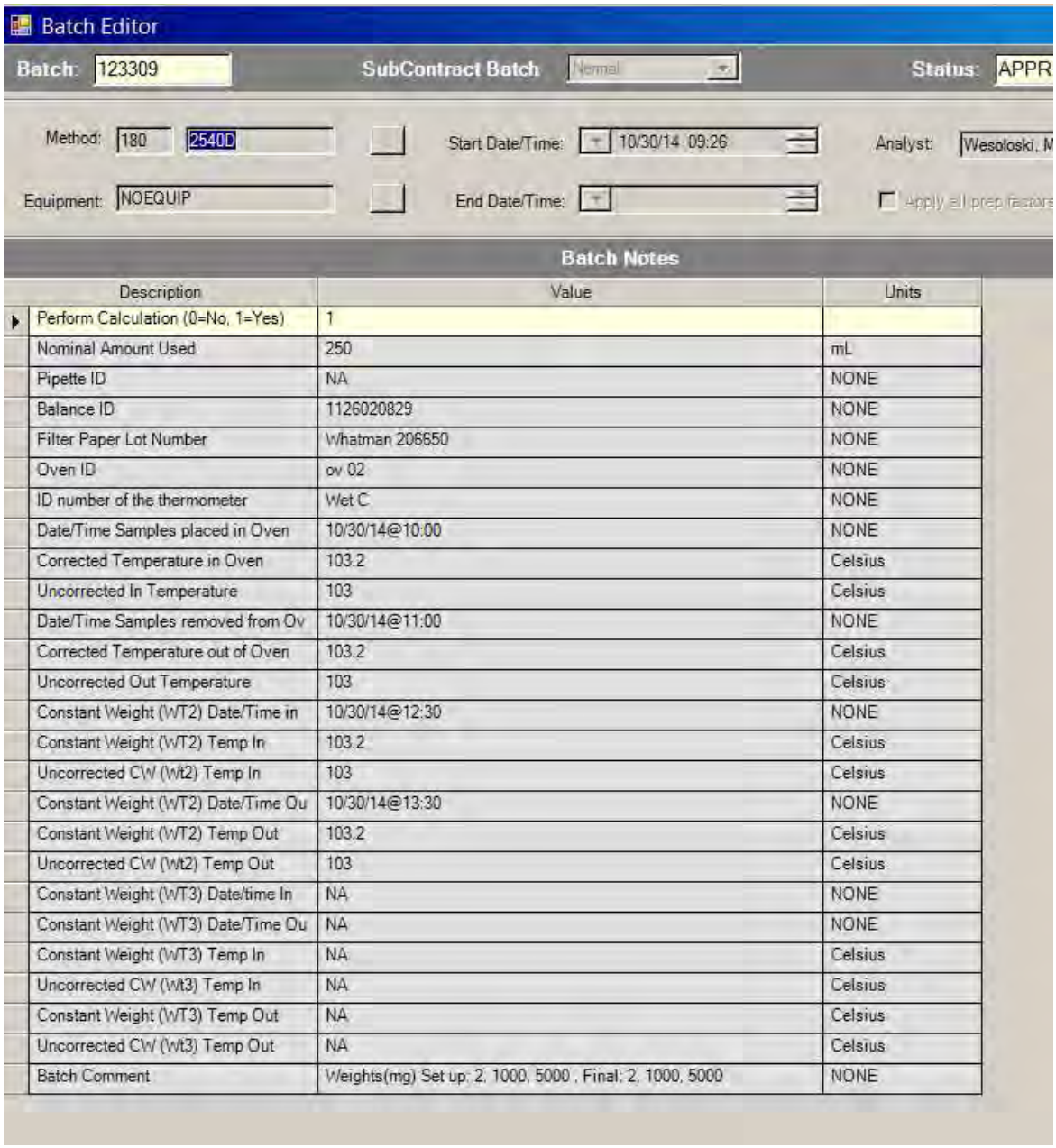


Figure 2 - Volatile Suspended Solids (VSS)



Footnote 1: Constant weight is a weight change of <0.5mg or <4%.
Footnote 2: If constant weight is not achieved after 3 cycles, report with NCM.

Figure 3 - TALS Batch Information – Method 2540D



Batch Editor

Batch: 123309 SubContract Batch Normal Status: APPR

Method: 180 2540D Start Date/Time: 10/30/14 09:26 Analyst: Wesoloski, M

Equipment: NOEQUIP End Date/Time: Apply all prep factors

Batch Notes

Description	Value	Units
Perform Calculation (0=No, 1=Yes)	1	
Nominal Amount Used	250	mL
Pipette ID	NA	NONE
Balance ID	1126020829	NONE
Filter Paper Lot Number	Whatman 206650	NONE
Oven ID	ov 02	NONE
ID number of the thermometer	Wet C	NONE
Date/Time Samples placed in Oven	10/30/14@10:00	NONE
Corrected Temperature in Oven	103.2	Celsius
Uncorrected In Temperature	103	Celsius
Date/Time Samples removed from Ov	10/30/14@11:00	NONE
Corrected Temperature out of Oven	103.2	Celsius
Uncorrected Out Temperature	103	Celsius
Constant Weight (WT2) Date/Time in	10/30/14@12:30	NONE
Constant Weight (WT2) Temp In	103.2	Celsius
Uncorrected CW (Wt2) Temp In	103	Celsius
Constant Weight (WT2) Date/Time Ou	10/30/14@13:30	NONE
Constant Weight (WT2) Temp Out	103.2	Celsius
Uncorrected CW (Wt2) Temp Out	103	Celsius
Constant Weight (WT3) Date/time In	NA	NONE
Constant Weight (WT3) Date/Time Ou	NA	NONE
Constant Weight (WT3) Temp In	NA	Celsius
Uncorrected CW (Wt3) Temp In	NA	Celsius
Constant Weight (WT3) Temp Out	NA	Celsius
Uncorrected CW (Wt3) Temp Out	NA	Celsius
Batch Comment	Weights(mg) Set up: 2, 1000, 5000 ; Final: 2, 1000, 5000	NONE

Figure 4 - TALS Batch Worksheet – Method 2540D

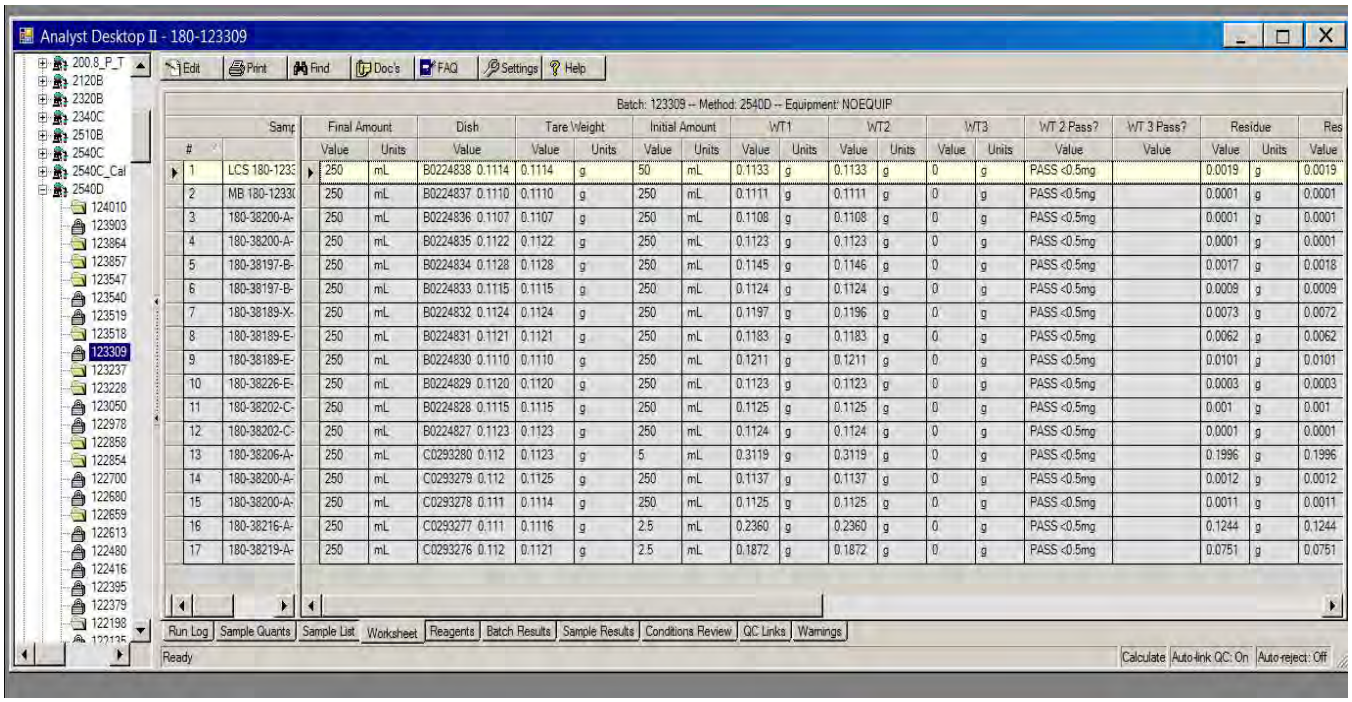
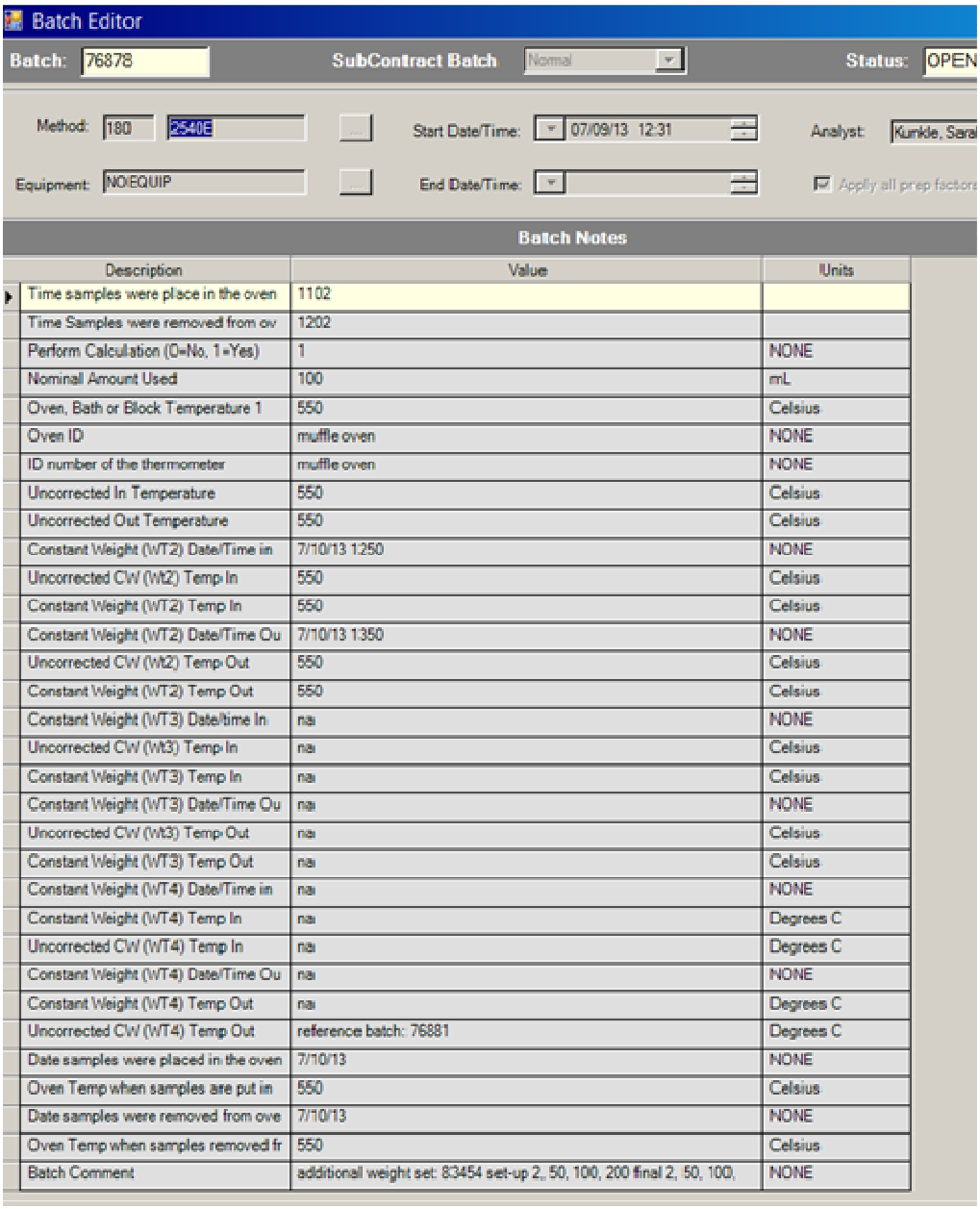


Figure 5 - TALS Batch Information – Method 2540E



Batch Editor

Batch: 76878 SubContract Batch: Normal Status: OPEN

Method: 180 2540E Start Date/Time: 07/09/13 12:31 Analyst: Kunkle, Sara

Equipment: NOEQUIP End Date/Time: Apply all prep factors

Batch Notes

Description	Value	Units
Time samples were place in the oven	1102	
Time Samples were removed from ov	1202	
Perform Calculation (0=No, 1=Yes)	1	NONE
Nominall Amount Used	100	mL
Oven, Bath or Block Temperature 1	550	Celsius
Oven ID	muffle oven	NONE
ID number of the thermometer	muffle oven	NONE
Uncorrected In Temperature	550	Celsius
Uncorrected Out Temperature	550	Celsius
Constant Weight (WT2) Date/Time in	7/10/13 1250	NONE
Uncorrected CW (Wt2) Temp In	550	Celsius
Constant Weight (WT2) Temp In	550	Celsius
Constant Weight (WT2) Date/Time Ou	7/10/13 1350	NONE
Uncorrected CW (Wt2) Temp Out	550	Celsius
Constant Weight (WT2) Temp Out	550	Celsius
Constant Weight (WT3) Date/time In	na	NONE
Uncorrected CW (Wt3) Temp In	na	Celsius
Constant Weight (WT3) Temp In	na	Celsius
Constant Weight (WT3) Date/Time Ou	na	NONE
Uncorrected CW (Wt3) Temp Out	na	Celsius
Constant Weight (WT3) Temp Out	na	Celsius
Constant Weight (WT4) Date/Time in	na	NONE
Constant Weight (WT4) Temp In	na	Degrees C
Uncorrected CW (WT4) Temp In	na	Degrees C
Constant Weight (WT4) Date/Time Ou	na	NONE
Constant Weight (WT4) Temp Out	na	Degrees C
Uncorrected CW (WT4) Temp Out	reference batch: 76881	Degrees C
Date samples were placed in the oven	7/10/13	NONE
Oven Temp when samples are put in	550	Celsius
Date samples were removed from ove	7/10/13	NONE
Oven Temp when samples removed fr	550	Celsius
Batch Comment	additional weight set: 83454 set-up 2, 50, 100, 200 final 2, 50, 100,	NONE

Figure 6 - TALS Batch Worksheet – Method 2540E

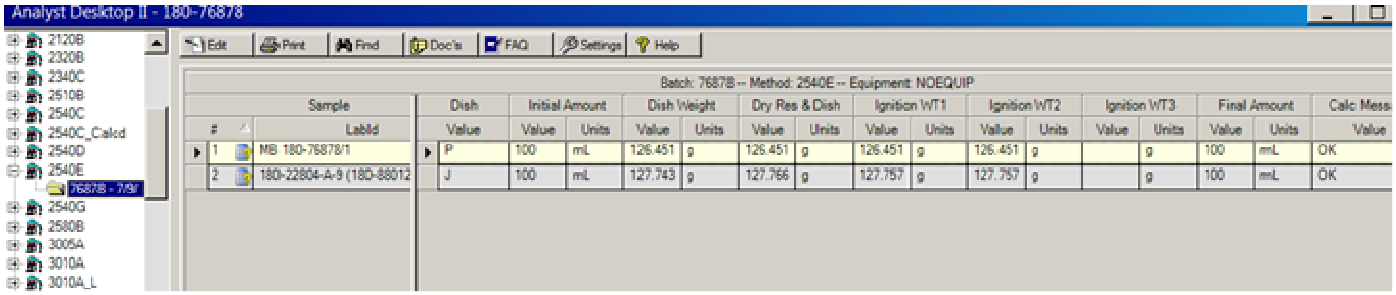
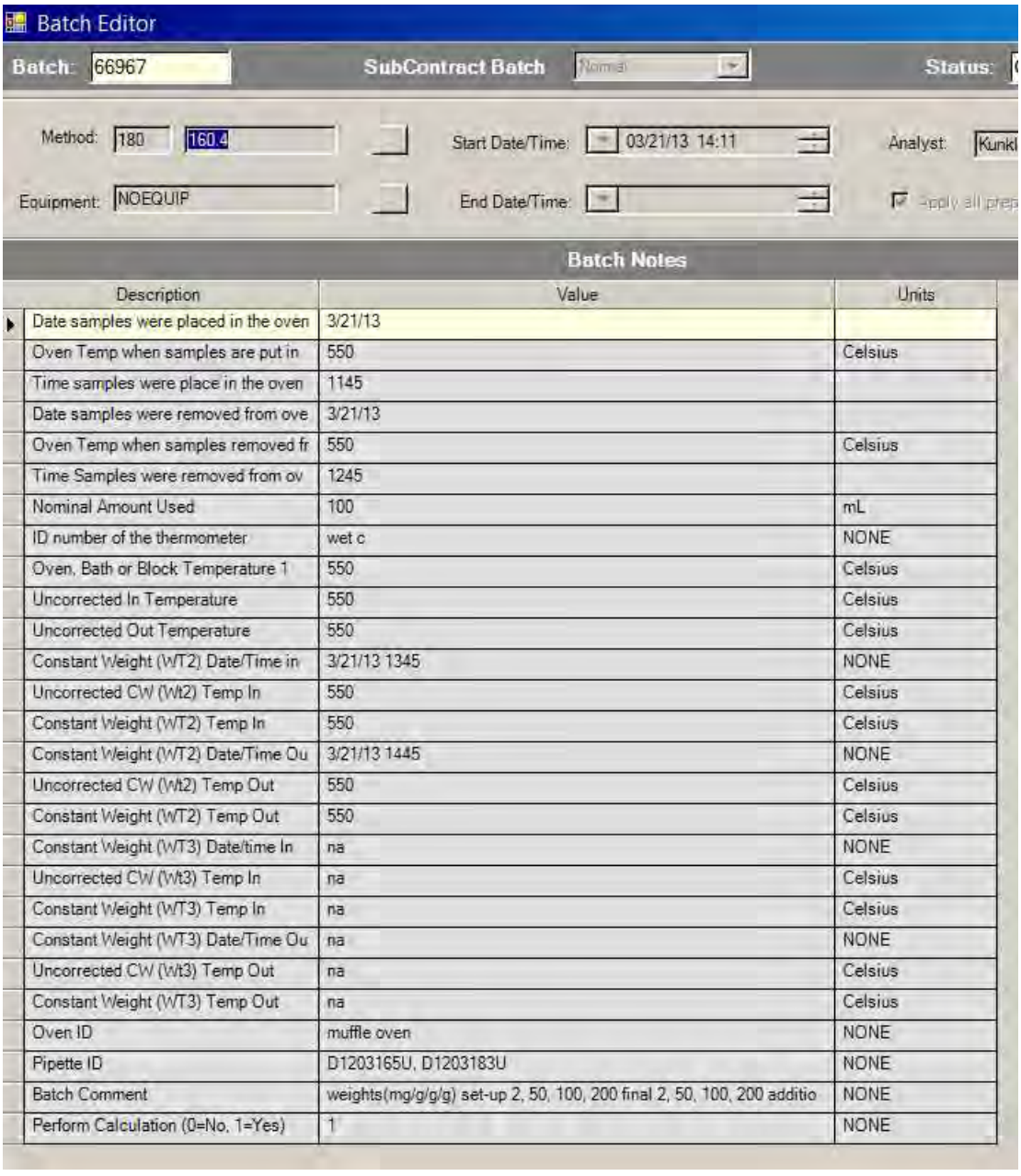


Figure 7 - TALS Batch Information – Method 160.4



Batch Editor

Batch: 66967 SubContract Batch Status:

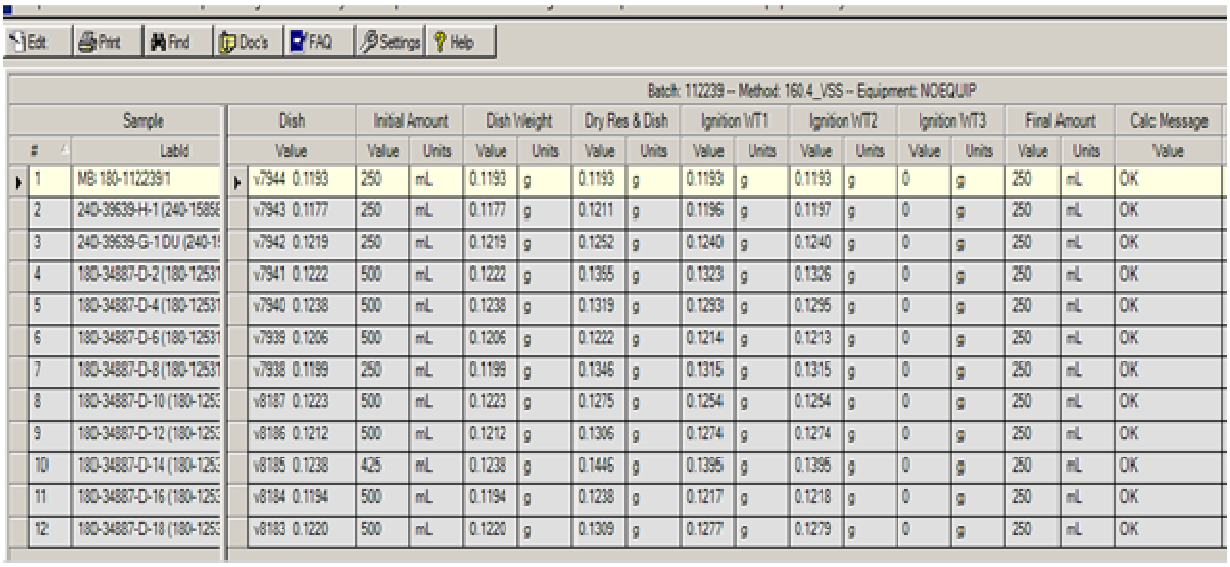
Method: 180 160.4 Start Date/Time: 03/21/13 14:11 Analyst: Kunkl

Equipment: NOEQUIP End Date/Time:

Batch Notes

Description	Value	Units
Date samples were placed in the oven	3/21/13	
Oven Temp when samples are put in	550	Celsius
Time samples were place in the oven	1145	
Date samples were removed from ove	3/21/13	
Oven Temp when samples removed fr	550	Celsius
Time Samples were removed from ov	1245	
Nominal Amount Used	100	mL
ID number of the thermometer	wet c	NONE
Oven, Bath or Block Temperature 1	550	Celsius
Uncorrected In Temperature	550	Celsius
Uncorrected Out Temperature	550	Celsius
Constant Weight (WT2) Date/Time in	3/21/13 1345	NONE
Uncorrected CW (Wt2) Temp In	550	Celsius
Constant Weight (WT2) Temp In	550	Celsius
Constant Weight (WT2) Date/Time Ou	3/21/13 1445	NONE
Uncorrected CW (Wt2) Temp Out	550	Celsius
Constant Weight (WT2) Temp Out	550	Celsius
Constant Weight (WT3) Date/time In	na	NONE
Uncorrected CW (Wt3) Temp In	na	Celsius
Constant Weight (WT3) Temp In	na	Celsius
Constant Weight (WT3) Date/Time Ou	na	NONE
Uncorrected CW (Wt3) Temp Out	na	Celsius
Constant Weight (WT3) Temp Out	na	Celsius
Oven ID	muffle oven	NONE
Pipette ID	D1203165U, D1203183U	NONE
Batch Comment	weights(mg/g/g/g) set-up 2, 50, 100, 200 final 2, 50, 100, 200 additio	NONE
Perform Calculation (0=No, 1=Yes)	1	NONE

Figure 8 - TALS Batch Worksheet – Method 160.4



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Batch: 112239 -- Method: 160.4_VSS -- Equipment: NOEQUIP

Sample		Dish	Initial Amount		Dish Weight		Dry Res & Dish		Ignition WT1		Ignition WT2		Ignition WT3		Final Amount		Calc Message
#	LabId	Value	Value	Units	Value	Units	Value	Units	Value	Units	Value	Units	Value	Units	Value	Units	Value
1	MB-180-112239/1	v7944 0.1193	250	mL	0.1193	g	0.1193	g	0.1193	g	0.1193	g	0	g	250	mL	OK
2	240-39639-H-1 (240-15855	v7943 0.1177	250	mL	0.1177	g	0.1211	g	0.1196	g	0.1197	g	0	g	250	mL	OK
3	240-39639-G-1 DU (240-15	v7942 0.1219	250	mL	0.1219	g	0.1252	g	0.1240	g	0.1240	g	0	g	250	mL	OK
4	180-34887-D-2 (180-12531	v7941 0.1222	500	mL	0.1222	g	0.1355	g	0.1323	g	0.1326	g	0	g	250	mL	OK
5	180-34887-D-4 (180-12531	v7940 0.1238	500	mL	0.1238	g	0.1319	g	0.1293	g	0.1295	g	0	g	250	mL	OK
6	180-34887-D-6 (180-12531	v7939 0.1206	500	mL	0.1206	g	0.1222	g	0.1214	g	0.1213	g	0	g	250	mL	OK
7	180-34887-D-8 (180-12531	v7938 0.1199	250	mL	0.1199	g	0.1346	g	0.1315	g	0.1315	g	0	g	250	mL	OK
8	180-34887-D-10 (180-1253	v8187 0.1223	500	mL	0.1223	g	0.1275	g	0.1254	g	0.1254	g	0	g	250	mL	OK
9	180-34887-D-12 (180-1253	v8186 0.1212	500	mL	0.1212	g	0.1306	g	0.1274	g	0.1274	g	0	g	250	mL	OK
10	180-34887-D-14 (180-1253	v8185 0.1238	425	mL	0.1238	g	0.1445	g	0.1395	g	0.1395	g	0	g	250	mL	OK
11	180-34887-D-16 (180-1253	v8184 0.1194	500	mL	0.1194	g	0.1238	g	0.1217	g	0.1218	g	0	g	250	mL	OK
12	180-34887-D-18 (180-1253	v8183 0.1220	500	mL	0.1220	g	0.1309	g	0.1277	g	0.1279	g	0	g	250	mL	OK

SOP # with Revision: PT-WC-045 Rev. 4
SOP Title: Determination of Total and Volatile Suspended Solids in Waters and Wastes
Amendment Number: 1

Effective Date of Amendment:
Append this form to the front of the SOP copy.

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The following SOP change is in effect as of the stated date. *This form will remain attached to the referenced SOP until such a time that the SOP is updated, approved, and redistributed, at which time it will become part of the historical SOP record.*

1. Briefly explain the reason for the update: 40 CFR Part 136 Update of Reference Year

2. Category for SOP Change:

☒ Typographical Corrections (Non-Technical) – Retraining Not Required.

☐ Typographical Corrections (Technical- define below) – Retraining is required.

☐ Procedural Changes (Define Below) – Retraining Required.

3. Summary of Procedure Change (circle to indicate if there are attachments to this form: No / Yes: # pages attached = ___)

15.1. Standard Methods for the Examination of Water and Waste Water, 2011, 2015; Methods 2540D and 2540E, on-line editions

Does this change result in a Method Modification? Yes (No)
If yes, please explain below.

Any method modification will be incorporated into section 16 of the SOP at the next revision.

8-23-21
8-16-21

Approval Signature/Date: Technical

Amanda L Gallo 08/12/21

Approval Signature/Date: QA Manager or Designee

Title: Determination of Total Dissolved and Volatile Dissolved Solids in Waters and Wastes

Method(s): Standard Methods 2540C & 2540E

Approvals (Signature/Date):

4/7/2020	4/7/2020
Roseann Ruyechan Date	Steve Jackson Date
Inorganics Department Manager	Regional Safety Coordinator
4/3/2020	4/15/2020
Virginia Zusman Date	Deborah L. Lowe Date
Quality Assurance Manager	Laboratory Director

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1. SCOPE AND APPLICATION

1.1. This SOP is applicable to the determination of total dissolved solids and volatile dissolved solids using a gravimetric technique. This SOP is based on the residue methods contained in Standard Methods for the Examination of Water and Wastes (SM), 2011.

1.2. This SOP is applicable to drinking, surface, and saline waters and domestic and industrial wastes.

1.3. The Total Dissolved Solids (TDS), or Filterable Residue, analysis protocol is based on SM 2540C.

1.4. The Volatile Dissolved Solids (VDS) analysis protocol is based on SM 2540E.

1.5. The methods cover a practical range of 10 mg/L to 20,000 mg/L

1.6. The reporting limit is 10 mg/L for TDS and VDS.

1.7. On occasion clients may request slight modifications to this SOP. Any modifications to this procedure must be approved by the QA department or the Laboratory or Technical Director, and documented fully in an NCM that accompanies the analytical batch.

2. SUMMARY OF METHOD

2.1. Total Dissolved Solids (TDS): A well-mixed sample is filtered through a glass fiber filter with a pore size of 2.0µm (or smaller). The filtrate is quantitatively transferred into a preweighed evaporating dish and is evaporated to dryness and then dried to constant weight at 180°C. The increase in weight over that of the empty dish represents the total dissolved solids. The filter from this procedure may also be used for TSS/VSS determination.

2.2. Volatile Dissolved Solids (VDS): The residue obtained from the determination of total dissolved solids is ignited at 550°C in a muffle furnace. The loss of weight on ignition is reported as mg/L volatile dissolved solids.

2.3. Total Suspended Solids may be performed at the same time, using the filter from the TDS filtration. Refer to SOP PT-WC-043 for details of this analysis.

3. DEFINITIONS

3.1. TALS – TestAmerica Laboratory Information Management System

3.2. NCM - Non-Conformance Memo - a system within TALS for the lab to communicate to project management and others when there is an anomaly seen with the samples or batch, or a QC failure.

3.3. Refer to the glossary in the Laboratory Quality Assurance Manual (PT-QA-M-001) for additional definitions.

4. INTERFERENCES

4.1. Method interferences may be caused by contaminants, reagents, glassware, and other sample processing hardware. All these materials must be routinely demonstrated to be free from interferences under the conditions of analysis by running method blanks.

4.2. Non-homogeneous samples may give erroneous results. Samples should be mixed as thoroughly as possible immediately before removing an aliquot for analysis.

4.3. Samples containing large amounts of suspended solids may filter slowly. Prolonged filtration time and pressure may cause breakthrough of some suspended material into the filtrate. Analysis volumes should be reduced for such samples.

4.4. Oil and grease in samples which permeates the filter will cause unreliable results due to difficulty in drying to constant weight. If present, oil & grease should be included in the sample and dispersed by a blender device before aliquoting.

4.5. Filtration apparatus, filter material, pre-washing, post-washing, and drying temperatures are specified because these variables have been shown to affect the results.

4.6. The temperature at which the residue is dried has an important bearing on the results. Weight losses due to volatilization of organic matter, mechanically occluded water, water of crystallization, and gases from heat-induced chemical decomposition, as well as weight gains due to oxidation, depend on temperature and time of heating.

4.7. Each sample requires close attention to desiccation after drying. Minimize opening the desiccator because moist air enters. Some samples may be more hygroscopic than those used as the desiccant and may take absorb additional water from the air as they cool.

4.8. Highly mineralized waters containing significant concentrations of calcium, magnesium, chloride, and/or sulfate may be hygroscopic and will require prolonged drying, desiccation and rapid weighing.

4.9. Samples containing high concentrations of bicarbonate may require careful and possibly prolonged drying to ensure that all the bicarbonate is converted to carbonate.

4.10. Too much residue in the drying vessel will crust over, entrapping water that will not be driven off during drying. Total residue must be limited to 200 mg (0.200g).

4.11. Some samples may have fine suspended solids that will pass through the glass fiber filter causing high TDS results. If solids are visible in the filtered sample, this must be documented in the batch comments.

4.12. Aluminum pans must not be used for TDS/VDS analyses. Components in some samples may react to form aluminum compounds, causing unreliable results.

4.13. For samples high in dissolved solids, thoroughly wash the filter after the sample has passed through to ensure removal of dissolved material prior to TSS determination, if included.

4.14. The volatile solids tests are subject to many errors due to the loss of water of crystallization, loss of volatile organic matter prior to combustion, incomplete oxidation of certain complex organics and decomposition of mineral salts during combustion. The results should not be considered an accurate measure of organic carbon in the sample.

4.15. Oils from hands can be transferred to the drying vessels or filters, causing a positive interference with these analyses. Handle crucibles, beakers and filters only with tongs or gloves.

5. SAFETY

5.1. Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual (CW-E-M-001), the Pittsburgh Facility Addendum EH&S manual (PT-HS-001), and this document. This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

5.2. Oily samples or those that contain volatile chemicals may ignite during this procedure. In the case of a fire, the muffle should be turned off and allowed to cool before the sample can be removed and put under a hood.

5.3. Always use tongs, forceps or heat resistant gloves when placing sample vessels in or removing them from ovens. Extra care should be used when using a muffle furnace, as temperatures are extremely high. Gloves designed for dealing with extreme temperatures must be used when handling any samples or glassware that has been in a muffle furnace.

5.4. There are no materials used in this method that have a significant or serious hazard rating. A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the SDS for each material before using it for the first time or when there are major changes to the SDS.

5.5. Eye protection that protects against splash, laboratory coat, and appropriate gloves must be worn while samples, standards, solvents, and reagents are being handled. Cut resistant gloves must be worn doing any other task that presents a strong possibility of getting cut. Disposable gloves that have been contaminated will be removed and discarded; other gloves will be cleaned immediately.

5.6. Exposure to chemicals must be maintained as low as reasonably achievable; therefore, unless they are known to be non-hazardous, all samples should be opened, transferred, and prepared in a fume hood, or under other means of mechanical ventilation. Solvent and waste containers will be kept closed unless transfers are being made.

5.7. All work must be stopped in the event of a known or potential compromise to the health and safety of a TestAmerica associate. The situation must be reported immediately to a laboratory supervisor or EH&S coordinator.

5.8. This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

6. EQUIPMENT AND SUPPLIES

The following items are recommended for performing this procedure. Equivalent items should only be used when they result in an improvement in quality, efficiency, productivity, or cost. An

item can be considered equivalent if with its use, the analytical and QA/QC requirements in this SOP can be met.

6.1. Analytical balance capable of weighing to 0.0001 g

6.1.1. Environmental Express StableWeigh system for use with TDS bags – fitted to balance to remove static from bags before weighing.

6.2. Vacuum filtration apparatus

6.3. Vacuum pump equipped with moisture trap

6.4. Glass fiber filter disks, 47 mm, pore size $\leq$ 2µm, without organic binder - Whatman 934-AH, pre-cleaned and pre-weighed (If pre-cleaned filters are not used, the preparation step in section 10.2 must be used prior to filtering samples.)

6.5. Evaporating dishes, 120 mL, made of porcelain. These must be thoroughly cleaned, rinsed with deionized water, and baked at 180 ± 2°C for at least one hour before use for TDS and baked at 550$\pm$50^{o}C for at least 1 hour for use for VDS. Store prepared dishes in a desiccator.

Note: 150mL glass beakers may also be used for TDS determinations. Glass beakers may not be used for procedures requiring use of a muffle furnace (VDS).

Note: Environmental Express StableWeigh pre-dried and pre-weighed TDS bags (part # TDS100) may also be used for TDS determinations. Bags may not be used for procedures requiring use of a muffle furnace (VDS). At least 2 bags per lot must be checked before use to verify the weights are accurate.

6.6. Desiccators providing sufficient space for storage of samples in process separate from filters and evaporating dishes

6.7. Desiccant containing a color indicator of moisture concentration or an instrumental indicator

6.8. Drying ovens set at 103 -105 °C and 180 ± 2 °C. Separate ovens should be maintained at appropriate temperatures.

Note: Environmental Express StableWeigh TDS HotBlocks (Model # TDS024) are used for the initial drying at 103-105°C of the TDS bags. Bags are then transferred to a 180 ± 2°C oven to complete drying.

6.9. Muffle furnace (550 °C ± 50 °C)

6.10. Thermometers, NIST traceable

6.11. Conductivity meter and associated apparatus

6.12. Graduated cylinders, to deliver (TD), assorted sizes, Class A

6.13. Crucible tongs

6.14. Zetex gloves or other gloves capable of providing protection at 550 °C.

7. REAGENTS AND STANDARDS

The following items are recommended for performing this procedure. Equivalent items should only be used when they result in an improvement in quality, efficiency, productivity or cost. An item can be considered equivalent if with its use, the analytical and QA/QC requirements in this SOP can be met. Please refer to the SDS prior to the use of any reagent or standard.

7.1. Reagent water - Deionized water, with conductance <1 umhos/cm – to be used for blanks and rinsing

7.2. The LCS standard solution is commercially purchased. The true value varies from lot to lot when received from the manufacturer and the calculation is adjusted accordingly by the laboratory. The standard is received with a certificate of analysis with the true value listed on it and is retained by the laboratory. Follow manufacturer's instructions for storage and expiration date.

7.2.1. If the solution needs to be made by the laboratory the procedure is as follows: LCS solution (500 mg/L TSS and TDS, 1000 mg/L TS): Place 500.0 mg of diatomaceous earth (infusorial earth or celite 545) and 500 mg sodium chloride into a 1000 mL volumetric flask and dilute to volume with deionized water. Mix well. Store at room temperature. Prepare fresh every three months.

8. SAMPLE COLLECTION, PRESERVATION, SHIPMENT AND STORAGE

8.1. Samples should be collected in either plastic or glass bottles.

8.2. Samples must be stored at >0.0℃ but ≤6.0℃ to minimize microbiological decomposition of solids. Bring samples to room temperature before analysis.

8.3. To achieve the reporting limits listed in Section 1, the recommended minimum volume for samples is 100mL. If less than minimum volumes are used because of limited sample or solids loading this should be documented using an NCM to be included in the report case narrative.

8.3.1. Smaller samples sizes may be used for samples that are high in solids. Historical data, visual inspection and conductivity readings may be used to choose a different sample size for initial analysis.

8.4. Holding times for TDS and VDS determination is 7 days from the date of sampling to begin the analysis.

9. QUALITY CONTROL

9.1. Method Blank (MB): One method blank must be processed with each batch of 20 or fewer samples. The method blank consists of reagent water that is carried through the entire analytical procedure, including filtration as applicable. The method blank is used to identify any system and process interferences or contamination of the analytical system that may lead to the reporting of elevated analyte concentrations or false positive data.

9.1.1. Use 100mL of reagent water for MB analysis.

9.1.2. Corrective Action for Blank Contamination:

If the analyte level in the method blank is $\geq$ the RL for the analyte of interest in the sample, all associated samples with reportable levels of analyte must be reanalyzed, unless the sample concentrations are >10x the blank concentration. If reanalysis is not possible due to limited sample quantity or holding time considerations, the project manager must be notified so that the client can be informed. Results reported associated with a contaminated method blank (unless sample >10X blank) must be reported with a B qualifier and the failure narrated through the appropriate NCM:

- For samples that have results <RL, use “Method Blank – Report, ND”
- For samples that have results >RL and <10x MB, use “Method Blank - >1/2RL” and correct the associated narrative to state “greater than the reporting limit”.
- For samples that have results >10x MB concentration, use “Method Blank – Report, 10X”

9.1.3. High blank results indicate contamination. Make sure the glassware and filtering apparatus is clean before repeating the analysis.

9.1.4. If MB results are negative (dish weighs less after analysis), this indicates that the balance was not operating correctly or the filter/weighing dish was not completely dry when the initial weights were measured. If MB results are below the negative RL (a change of -0.0010g for example), all samples with concentrations <10X the RL should be reanalyzed if possible. If reanalysis is not possible, the negative blanks and possible negative bias for the sample results must be narrated using the "Other – Deficiency" NCM.

9.2. Laboratory Control Sample (LCS): One LCS must be processed with each batch of 20 or fewer samples. The LCS must be carried through the entire analytical procedure. The LCS is used to monitor the accuracy of the analytical process. On-going monitoring of the LCS results provides evidence that the laboratory is performing the method within acceptable accuracy and precision guidelines.

9.2.1. No LCS is available for the volatile residue procedure.

9.2.2. Refer to Section 7.2 for instructions on how to prepare the LCS solution, unless using a purchased standard. The LCS analysis volume must be calculated to produce a final residue between 2.5 and 200mg.

9.2.3. The LCS percent recovery must be within 80 -120% of the true or certified value.

9.2.4. If LCS recovery is outside the control limits, all associated samples must be reanalyzed with an acceptable LCS, unless the original LCS recovery is high and samples results are <RL.

9.2.4.1. If reanalysis is not possible due to limited sample quantity or holding time considerations, the project manager must be notified so that the client can be informed. Results reported associated with a failed LCS must be reported with the appropriate result qualifier and the failure narrated through the appropriate NCM:

- For high LCS recovery and associated samples <RL, use "LCS/LCSD - %R High".
- If samples are reanalyzed due to failed LCS, and the reanalysis occurs past the holding time, use "LCS/LCSD - %R, RX/RA Batch, Outside".
- If there is insufficient sample volume to perform a reanalysis, use "LCS/LCSD – Insufficient Sample".

9.2.5. Poor LCS results are usually the result of contamination or poor mixing. Make sure to shake the LCS and all samples very well before pouring out the portion to be filtered or analyzed. Do not allow the sample to settle in the graduated cylinder.

9.3. Duplicate (D): A duplicate sample analysis is required with every 10 samples or less for all methods.

9.3.1. Sample and Duplicate results must agree within 10% RPD.

9.3.2. The failure of the sample duplicates to meet precision criteria must be addressed in the case narrative.

9.3.3. Make sure to shake all samples very well before pouring out the portion to be filtered or analyzed to improve homogenization and precision. Do not allow the sample to settle in the graduated cylinder before analyzing.

9.3.4. On-going method precision is documented using LCS control charting.

10. PROCEDURE

10.1. Calibration and Standardization

10.1.1. Proper balance operation will be verified daily or prior to sample analysis by following the balance calibration SOP, PT-QA-12. Analytical balance calibration must be verified at least every 24 hours, and prior to using the balance each day. Weights used for calibration verification, prior to initial and read-back weighings, are documented in the TALS batch information.

10.1.1.1. For TDS and VDS, balances are verified using 2mg, 0.300g, 10g and 120g weights.

10.1.1.2. Balance reading for the 2mg weight must be 0.0018g - 0.0022g; for the 0.300 g weight must be 0.2995g – 0.3005 g; for the 10g weight must be 9.9900g – 10.0100g and for the 120g weight must be 119.88000g – 120.1200g. If any reading falls outside these limits, the balance must be recalibrated and the verification performed again with all weights in the group..

10.1.2. Oven temperature must be checked daily and recorded in the oven temperature logbook. Oven temperatures must be kept between 103-105^{o}C and 178-182^{o}C. The temperature of the Muffle furnace used for volatile residues analyses is documented at the time of use.

10.1.3. Conductivity of the water must be monitored and recorded in the Conductivity Logbook daily. The maximum permissible conductivity is 1.0 umhos/cm (at 25 °C). If the conductivity reading on the water system exceeds this level, do not use the water for these procedures and notify the supervisor immediately.

10.2. Procedure

10.2.1. All samples are to be electronically checked out of sample control with the TALS internal chain of custody (ICOC) program.

10.2.2. Proper sample identification is extremely important in any analytical procedure. Labeling of evaporating dishes and filters holders must be done in a manner to ensure connection with the proper sample. Labeling must be done in a way that will not be compromised by the heat used.

10.2.3. If samples are visibly oily, this should be noted on the TALS Worksheet Notes or Batch Information Comments.

10.2.4. If there is limited sample volume or high solids content, smaller amounts of sample may need to be processed than detailed in the following sections. This occurrence must be noted on the TALS Worksheet. Reporting limits are adjusted according to the actual sample volume used.

10.2.5. All weights are recorded to the nearest 0.1mg directly from the balance into the TALS Worksheet using balance upload. If the balance upload is not working, the weights are recorded in the Wet Chemistry Raw Data Logsheet for transfer to the TALS Worksheet. If the manual Logsheet is used, it must be scanned to a pdf and attached to the TALS batch as raw data.

10.2.6. Prepare TALS analytical batches prior to beginning analysis, as the batch must be available to upload initial weights and record sample volumes used.

10.2.7. The conductance of each sample is checked prior to sample filtration, to be used to determine the appropriate sample volume to process.

10.2.7.1. Allow the sample to warm to near room temperature before checking conductivity. Shake the sample well, rinse the conductivity cell with two portions of the sample, and then allow the conductivity reading of a third portion to settle. Record the conductivity on the TALS Worksheet (see Figure 2). Rinse the conductivity cell with laboratory reagent water between samples.

10.2.7.2. TDS is typically 55 - 90% of the conductance result. The exact relationship depends on the compounds present in the samples and may not hold for very high concentrations or samples containing non-ionic species or samples with conductance greater than 10,000 µmho/cm or less than 50 µmho/cm.

- If the sample has a conductance less than 2500 µmhos/cm, 100 mL should be used.

- If the sample has a conductance over 3,000 µmhos/cm, to achieve a residue of <200mg, the following formula should be used to approximate the required sample volume:

  (2000 x 100) / Conductivity = mL sample to use

  Note: To avoid odd reporting limits, and make measuring easier, round the determined sample volume to one of the following volume increments: 5, 10, 20, 25, 50, or 100 mL.

10.3. Total Dissolved Solids – SM 2540C

10.3.1. Preparation of Glass Fiber Filter Disc, if not purchased prepared for use:

- Place the glass fiber filter discs, one at a time, on the membrane filter apparatus with wrinkled surface side up.
- While vacuum is applied, wash the disc with three successive 20 mL volumes of reagent water.
- Remove all traces of water by continuing to apply vacuum after water has passed through and discard washings.
- If TSS analysis is to be performed along with the TDS, follow the additional filter drying steps in SOP PT-WC-043.

10.3.2. Preparation of Evaporating Dishes: (Evaporating dishes must be used if VDS is to be determined.)

10.3.2.1. If only total dissolved solids are to be measured, heat a clean evaporating dish or glass beaker to 180 ± 2 °C for one hour. If volatile dissolved solids are to be measured in addition to TDS, ignite the clean evaporating dish at 550 ± 50 °C for one hour in a muffle furnace.

10.3.2.2. Heat resistant gloves and tongs must be used when removing items from the muffle furnace.

10.3.2.3. Store and cool dish in desiccator until dish reaches room temperature or until needed.

10.3.2.4. Weigh immediately before use to the nearest 0.1 mg. Upload weight into the TALS Worksheet for the associated sample (see Figure 2).

10.3.3. If Environmental Express StableWeigh bags are used, the bags require no pre-drying or weighing. Scan the barcode on the bag to enter the bag weight into the TALS worksheet.

10.3.4. Sample Analysis:

10.3.4.1. Thoroughly rinse the entire filtration apparatus with reagent water before filtering each sample. Remove as much of the rinse water as possible from the filtration flask.

10.3.4.2. Assemble the filtering apparatus. Place a glass fiber filter on the funnel base, wrinkled surface side up. Attach the funnel cup. Pre-wet the filter using a few mL of reagent water and begin suction.

Note: If the sample also requires TSS, follow the analytical steps in SOP PT-WC-043 for recording the initial filter weights.

10.3.4.3. Shake the sample vigorously and rapidly transfer 100 mL, or a smaller portion which will yield between 10 and 200 mg dried residue, to the funnel by means of a graduated cylinder. (Pre-determine the volume to use following step 10.2.8 above.)

- If total dissolved solids content is low, <2.5mg of residue is obtained from the initial analysis, successive sample volumes may be filtered and added to the original dish.

- If more than 10 minutes are required to complete filtration, decrease sample size.

10.3.4.4. Record the volume of sample used (to the nearest mL) on the benchsheet.

10.3.4.5. For the method blank, process 100 mL of reagent water as the sample.

10.3.4.6. For the LCS, process 50 mL of the LCS Solution and dilute to a final volume of 100 mL with Reagent water. Refer to Section 7.2 for instructions on how to prepare the LCS if not using a purchased standard.

10.3.4.7. Filter the sample through the glass fiber filter.

10.3.4.8. Rinse the graduated cylinder, funnel walls, and filter with three successive 10 mL portions of reagent water and allow for complete drainage between washings. Continue to apply vacuum for about three minutes after filtration is complete to remove as much water as possible.

10.3.4.9. Transfer the filtrate (including the washings) to a pre-weighed evaporating dish. Rinse the receiving flask with 10 - 25 mL of reagent water and transfer washings into the dish to ensure complete transfer of the sample.

Note: If TSS analysis is being performed along with the TDS, refer to SOP PT-WC-043 for steps to take with the filter for this analysis.

Note: If StableWeigh bags are used, the sample has been filtered directly into the drying vessel (bag). (See Figure 5 at the end of this document.) Remove the bag from the filtering apparatus and transfer it to the rack.

10.3.4.10. Place the dishes into the 103-105°C oven overnight to evaporate the water.

Note: If StableWeigh bags are being used, transfer the racks to the StableWeigh HotBlocks. HotBlocks are maintained at 103-105°C. Bags are heated to dryness in the block, or may be left in the block overnight.

10.3.4.11. Transfer the evaporated sample containers (dishes or bags) into the 180 ± 2°C oven, and bake for at least one hour after the temperature returns to 180 ± 2°C.

10.3.4.12. Record the dates, times, and oven temperatures for in and out of the ovens or block on the TALS Batch Information (see Figure 9).

10.3.4.13. Using tongs, remove the weighing vessel from the oven and place in a desiccator and cool to room temperature.

10.3.4.14. Weigh the vessel to the nearest 0.1 mg. This weight is automatically uploaded into the TALS Worksheet using the Balance Link program (see Figure 2).

10.3.4.15. If the residue is >0.2g, start the analysis over again with a smaller sample volume. If insufficient volume is available for reanalysis, proceed with the steps below, and narrate that the results were determined from a residue >200mg.

10.3.4.16. Return the samples to the oven for at least another hour, cool in a desiccator, and reweigh. Repeat the drying, cooling, desiccating, and weighing cycle until a constant weight is obtained. Constant weight is considered a weight change < 0.5 mg or < 4% of the previous weight, whichever is less. If more than three cycles are required, use the third cycle weights and initiate a Nonconformance Memo.

Note: When weighing dried sample, be alert to change in weight due to air exposure and/or sample degradation. Note any change in sample residue appearance in the TALS Worksheet Notes.

10.3.4.17. TALS will automatically calculate the sample result from the data entered into the Worksheet. Check the calculations according to the equation in Section 11.1. Use the lowest final weight achieved for calculating TDS.

10.4. Volatile Dissolved Solids – SM2540E

10.4.1. Heat muffle furnace up to temperature (550 ± 50 °C).

10.4.2. Place evaporating dish containing residue generated by Total Dissolved Solids protocol (Section 10.3.3.17) in muffle furnace to ignite the residue.

10.4.3. Record the date, time, and muffle furnace temperature in the TALS Batch Information when the samples are initially placed in the oven and again when they are removed from the oven (see Figure 3).

10.4.4. Heat samples for at least 15 minutes. Typically 15-20 minutes ignition is sufficient for 200 mg of residue. However, more than one sample and/or heavier residues may necessitate longer ignition times.

10.4.5. Using tongs and heat resistant gloves, remove the weighing dish from the muffle furnace and place in a desiccator and cool to room temperature. Weigh dish when it has cooled to room temperature, and upload the weight into the TALS Worksheet (see Figure 4).

10.4.6. Return the samples to the furnace for another 15 minutes, cool in a desiccator and reweigh. Repeat the drying, cooling, desiccating and weighing cycle until a constant weight is obtained. Constant weight is considered a weight change of < 0.5mg or < 4% of the previous weight, whichever is less. If constant weight has not been achieved after 3 cycles of heating, cooling and weighing; document the 3 weights and do not perform any further cycles. Document that only 3 heating cycles were performed, and the lowest weight of the 3 is used for reporting in an NCM.

10.4.7. TALS will calculate the final results. Check calculations using the formula given in Section 11.2. Use the lowest final weight achieved for calculating VDS.

11. CALCULATIONS / DATA REDUCTION

11.1. Calculate Total Dissolved Solids as follows:

$$\text{Total Dissolved Solids, mg / L} = \frac{(A - B) \times 1000}{C}$$

Where: A = weight of dried residue + dish (mg)
B = weight of dish (mg)
C = volume of sample (mL)

11.2. Calculate Volatile Dissolved Solids as follows:

$$\text{Volatile Dissolved Solids, mg / L} = \frac{(A - B) \times 1000}{C}$$

Where: A = weight of residue + dish before ignition (mg)
B = weight of residue + dish after ignition (mg)
C = volume of sample (mL)

11.3. The relative percent difference (RPD) of duplicates are calculated according to the following equation:

$$RPD = 100\left[\frac{|DU1 - DU2|}{\left(\frac{DU1 + DU2}{2}\right)}\right]$$

Where: DU1 = Sample result
DU2 = Sample duplicate result

11.4. LCS Percent Recovery

$$\text{LCS Percent Recovery} = \left(\frac{\text{Observed Conc. in LCS}}{\text{True LCS Conc.}}\right) \text{x } 100 \text{ Percent}$$

11.5. If multiple weighing cycles are required, the lowest final sample weight is used for calculating solids content.

11.6. If smaller or larger sample volumes are processed than are specified in the method, the reporting limit must be adjusted accordingly.

11.7. Sample results should be reported according to the following significant figure rules:

Significant Figures	Sample Result
2	< 10
3	≥ 10

12. METHOD PERFORMANCE

12.1. The supervisor has responsibility to ensure that an analyst who performs this procedure is properly trained in its use and has the required experience. Performance is monitored through internal QC and outside performance evaluation samples. Please refer to the QA Manual for additional information concerning Precision and Accuracy.

12.2. Demonstration of Capabilities – Prior to the analysis of samples, a Demonstration of Capabilities (DOC) as described in the QA Manual, must be performed initially, annually and any time a significant change is made to the analytical system.

12.3. An MDL standard study is not performed for this procedure. The reporting limit is verified annually by Method Blank MDL calculation. Results are not reported below the RL.

13. POLLUTION CONTROL

13.1. It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage and reagent stability). Employees must abide by the policies in Section 13 of the Corporate Environmental Health and Safety Manual (CW-E-M-001) for "Waste Management and Pollution Prevention" and the Pittsburgh Facility Addendum EH&S Manual (PT-HS-001).

13.2. This method does not contain any specific modifications that serve to minimize or prevent pollution

14. WASTE MANAGEMENT

14.1. Waste management practices are conducted consistent with all applicable rules and regulations. Excess reagents, samples and method process wastes are disposed of in an accepted manner. Waste description rules and land disposal restrictions are followed. Waste disposal procedures are incorporated by reference to PT-HS-001.

14.2. The following waste streams are produced when this method is carried out.

14.2.1. Unpreserved, non-hazardous samples are disposed down a lab sink.

14.2.2. Oily waste samples are disposed of into waste stream #10 – flammable sample waste.

15. REFERENCES

15.1. Standard Methods for the Examination of Water and Waste Water, 2011 Method 2540C & 2540E
15.2. SOP PT-QA-021, TestAmerica Pittsburgh QC Program
15.3. SOP PT-QA-012, Selection and Calibration of Balances and Weights.
15.4. TestAmerica Pittsburgh Laboratory Quality Assurance Manual, PT-QA-M-001
15.5. SOP PT-QA-006, Procurement of Standards and Materials; Labeling and Traceability
15.6. SOP PT-QA-008, Thermometer Calibration and Temperature Monitoring
15.7. SOP PT-QA-016, Nonconformance & Corrective Action System
15.8. SOP PT-QA-031, Internal Chain of Custody
15.9. SOP PT-HS-001, Pittsburgh Facility Addendum EH&S Manual

16. METHOD MODIFICATIONS

16.1. Samples are not taken through the drying, cooling, and weighing cycle more than three times. If the sample fails to reach constant weight within three cycles, this is documented in a Nonconformance Memo and the lowest final weight is used for the calculation.

17. ATTACHMENTS

17.1. Figure 1: TALS Batch Information – Method 2540C
17.2. Figure 2: TALS Batch Worksheet – Method 2540C
17.3. Figure 3: TALS Batch Information – Method 2540E
17.4. Figure 4: TALS Batch Worksheet – Method 2540E
17.5. Figure 5: Stable Weigh TDS set up

18. REVISION HISTORY

18.1. Revision 0, 12/3/2014
18.2. Revision 1, 1/4/2017
18.3. Revision 2, 7/28/2017
18.4. Revision 3, 5/10/19
18.5. Changes to current revision

SOP section	Change from	Change to	Reason
1.7	Modifications per QAM	Modifications must be approved and documented	Clarification
6.1.1, 6.5, 6.8		Added Environmental Express StableWeigh information	Correction
9.3.1	Remove caveat for concentrations < 5x RL		Correction
10.2.1	Removed modification statement		Redundant with 1.7
10.2.7.2	3000	2500	Correction
10.3.3, 10.3.4.9, 10.3.4.10, 10.3.4.12		Added instructions for StableWeigh bag use.	Correction
17.5		Added Figure 5	Clarification

Figure 1 - TALS Batch Information – Method 2540C - 1

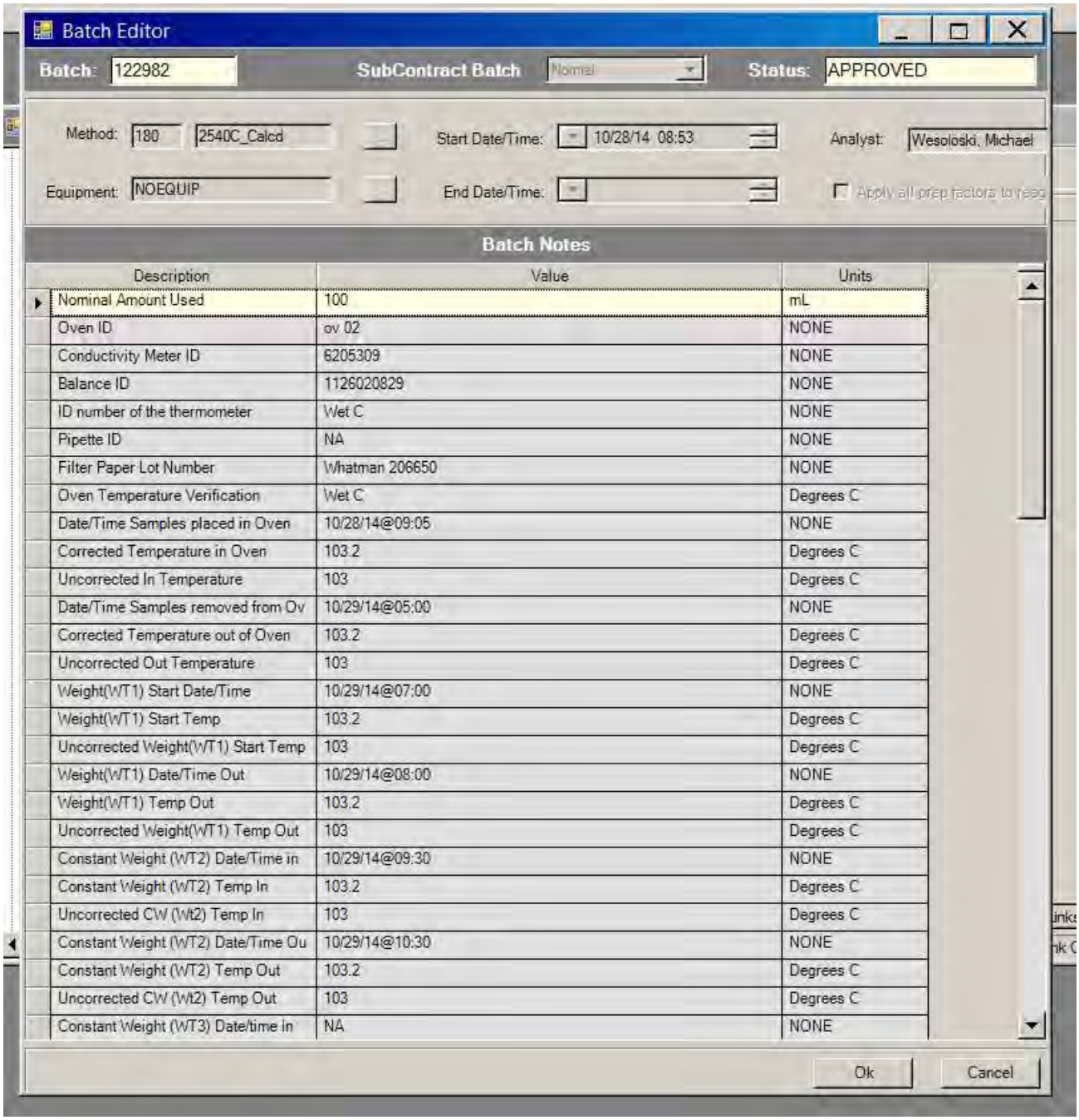
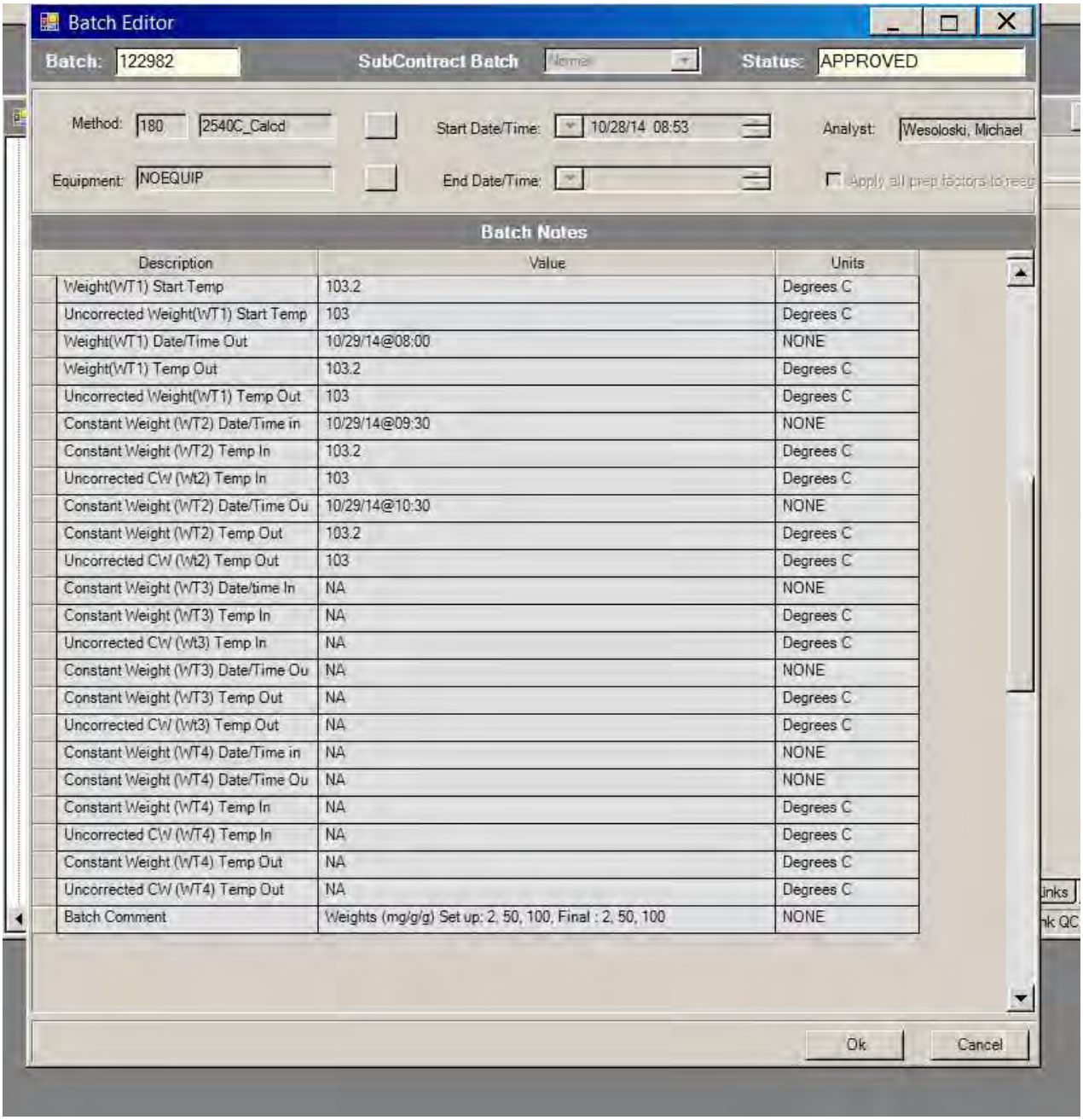


Figure 1 - TALS Batch Information – Method 2540C – 2



Description	Value	Units
Weight(WT1) Start Temp	103.2	Degrees C
Uncorrected Weight(WT1) Start Temp	103	Degrees C
Weight(WT1) Date/Time Out	10/29/14@08:00	NONE
Weight(WT1) Temp Out	103.2	Degrees C
Uncorrected Weight(WT1) Temp Out	103	Degrees C
Constant Weight (WT2) Date/Time in	10/29/14@09:30	NONE
Constant Weight (WT2) Temp In	103.2	Degrees C
Uncorrected CW (Wt2) Temp In	103	Degrees C
Constant Weight (WT2) Date/Time Ou	10/29/14@10:30	NONE
Constant Weight (WT2) Temp Out	103.2	Degrees C
Uncorrected CW (Wt2) Temp Out	103	Degrees C
Constant Weight (WT3) Date/time In	NA	NONE
Constant Weight (WT3) Temp In	NA	Degrees C
Uncorrected CW (Wt3) Temp In	NA	Degrees C
Constant Weight (WT3) Date/Time Ou	NA	NONE
Constant Weight (WT3) Temp Out	NA	Degrees C
Uncorrected CW (Wt3) Temp Out	NA	Degrees C
Constant Weight (WT4) Date/Time in	NA	NONE
Constant Weight (WT4) Date/Time Ou	NA	NONE
Constant Weight (WT4) Temp In	NA	Degrees C
Uncorrected CW (WT4) Temp In	NA	Degrees C
Constant Weight (WT4) Temp Out	NA	Degrees C
Uncorrected CW (WT4) Temp Out	NA	Degrees C
Batch Comment	Weights (mg/g/g) Set up: 2, 50, 100, Final : 2, 50, 100	NONE

Figure 2 - TALS Batch Worksheet – SM2540C

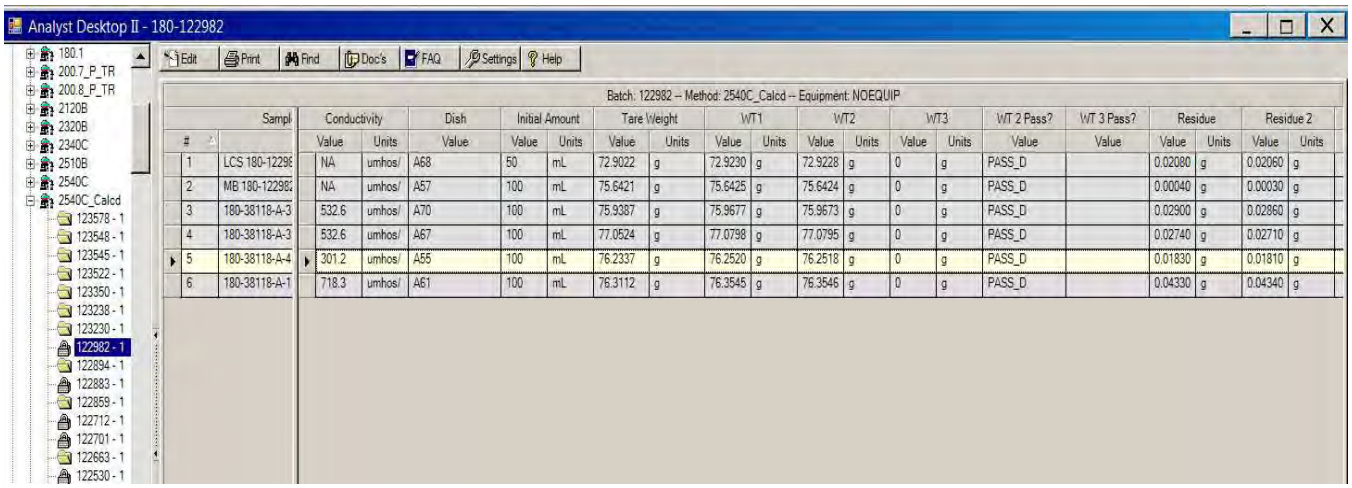
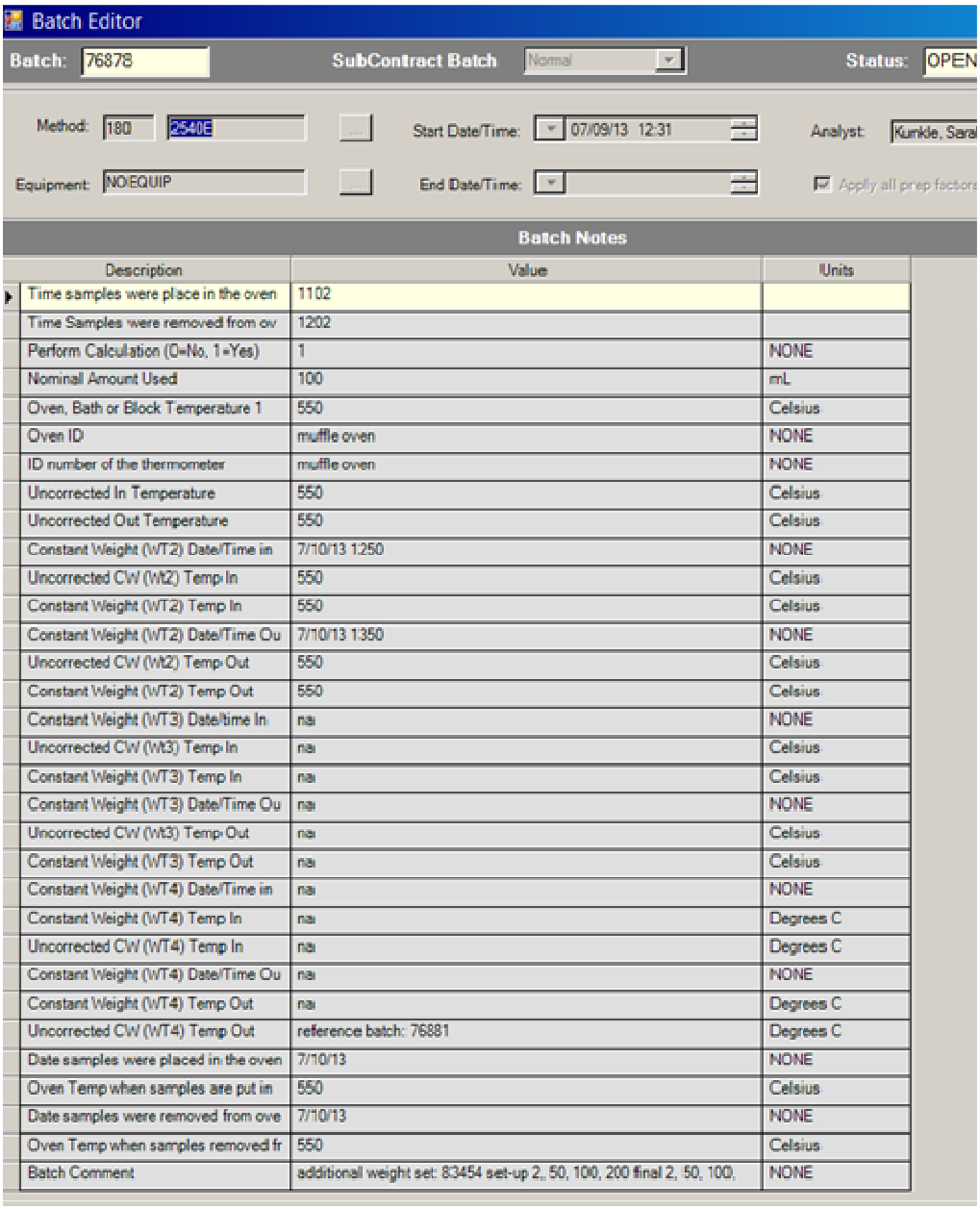


Figure 3 - TALS Batch Information – Method 2540E



Batch Editor

Batch: 76878 SubContract Batch: Normal Status: OPEN

Method: 180 2540E Start Date/Time: 07/09/13 12:31 Analyst: Kunkle, Sara

Equipment: NOEQUIP End Date/Time: Apply all prep factors

Batch Notes

Description	Value	Units
Time samples were place in the oven	1102	
Time Samples were removed from ov	1202	
Perform Calculation (0=No, 1=Yes)	1	NONE
Nominall Amount Used	100	mL
Oven, Bath or Block Temperature 1	550	Celsius
Oven ID	muffle oven	NONE
ID number of the thermometer	muffle oven	NONE
Uncorrected In Temperature	550	Celsius
Uncorrected Out Temperature	550	Celsius
Constant Weight (WT2) Date/Time in	7/10/13 1250	NONE
Uncorrected CW (Wt2) Temp In	550	Celsius
Constant Weight (WT2) Temp In	550	Celsius
Constant Weight (WT2) Date/Time Ou	7/10/13 1350	NONE
Uncorrected CW (Wt2) Temp Out	550	Celsius
Constant Weight (WT2) Temp Out	550	Celsius
Constant Weight (WT3) Date/time In	na	NONE
Uncorrected CW (Wt3) Temp In	na	Celsius
Constant Weight (WT3) Temp In	na	Celsius
Constant Weight (WT3) Date/Time Ou	na	NONE
Uncorrected CW (Wt3) Temp Out	na	Celsius
Constant Weight (WT3) Temp Out	na	Celsius
Constant Weight (WT4) Date/Time in	na	NONE
Constant Weight (WT4) Temp In	na	Degrees C
Uncorrected CW (WT4) Temp In	na	Degrees C
Constant Weight (WT4) Date/Time Ou	na	NONE
Constant Weight (WT4) Temp Out	na	Degrees C
Uncorrected CW (WT4) Temp Out	reference batch: 76881	Degrees C
Date samples were placed in the oven	7/10/13	NONE
Oven Temp when samples are put in	550	Celsius
Date samples were removed from ove	7/10/13	NONE
Oven Temp when samples removed fr	550	Celsius
Batch Comment	additional weight set: 83454 set-up 2, 50, 100, 200 final 2, 50, 100,	NONE

Figure 4 - TALS Batch Worksheet – Method 2540E

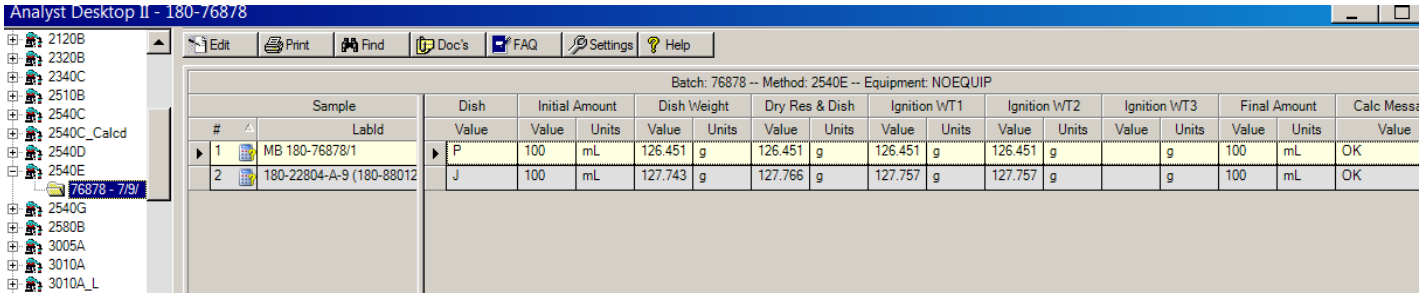


Figure 5 – Stable Weigh TDS Bag Filtration Setup



TITLE: TOXICITY CHARACTERISTIC LEACHING PROCEDURE (TCLP) AND SYNTHETIC PRECIPITATION LEACHING PROCEDURE (SPLP)

METHODS: SW-846 1311 AND 1312

Approvals (Signature/Date):

3/20/2020	3/23/2020
Sharon Bacha, Technical Manager — Date	Steve Jackson, Regional Safety Coordinator — Date
3/20/2020	4/2/2020
Virginia Zusman, Quality Assurance Manager — Date	Deborah L. Lowe, Laboratory Director — Date

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1. SCOPE AND APPLICATION

1.1 This SOP describes the application of the Toxicity Characteristic Leaching Procedure (TCLP), SW846 Method 1311. The Toxicity Characteristic (TC) of a waste material is established by determining the levels of 8 metals and 31 organic chemicals in the aqueous leachate of a waste. The TC is one of four criteria in 40 CFR Part 261 to determine whether a solid waste is classified as a hazardous waste. The TC Rule utilizes the TCLP method to generate the leachate under controlled conditions that are designed to simulate leaching through a landfill. EPA's "worst case" waste disposal model assumes mismanaged wastes will be exposed to leaching by the acidic fluids generated in municipal landfills. The EPA's model also assumes the acid/base characteristics of the waste will be dominated by the landfill fluids. The TCLP procedure directs the testing laboratory to use a more acidic leaching fluid if the sample is an alkaline waste, again in keeping with the model's assumption that the acid fluids will dominate leaching chemistry over time.

1.2 A specific list of TC analytes and regulatory limits may be found in Appendix A, Table 3.

1.2.1 **Note:** The list in Appendix A does not include the December 1994 EPA rule for Universal Treatment Standards for Land Disposal Restrictions. Those requirements include 216 specific metallic and organic compounds and, in some cases, lower detection limit requirements (see 40 CFR 268.40). TCLP leachates are part of the new Universal Treatment Standards, but the conventional analytical methods will not necessarily meet the new regulatory limits. Consult with the client and with TestAmerica Technical Specialists before establishing the instrumental methods for these regulations.

1.3 This SOP also describes the application of the Synthetic Precipitation Leaching Procedure (SPLP) that was designed to simulate the leaching that would occur if a waste was disposed in a landfill and exposed only to percolating rain water. The procedure is based on SW846 Method 1312. The list of analytes for SPLP may extend beyond the toxicity characteristic compounds shown in Appendix A. With the exception of the use of a modified extraction fluid, the SPLP and TCLP protocols are essentially equivalent. Where slight differences may exist between the SPLP and TCLP they are distinguished within this SOP.

1.4 The procedure is applicable to liquid, solid, wastes and multiphasic wastes.

1.5 The results obtained are highly dependent on the pH of the extracting solution, the length of time that the sample is exposed to the extracting solution, the temperature during extraction, and the particle size/surface area of the sample. These parameters must be carefully controlled. Any deviations from the method affecting these parameters must be documented as a nonconformance, with a cause and corrective action described.

1.6 The reporting limits are based on the individual samples as well as the individual analysis techniques. However, the sample is determined to be hazardous if it contains any analyte at levels greater than or equal to the regulatory limits.

1.7 If a total analysis of the waste demonstrates that individual analytes are not present in the waste, or that they are present but at such low concentrations that the appropriate regulatory levels could not possibly be exceeded, the TCLP procedure need not be run. If the total analysis results indicate that TCLP is not required, the decision to cease TCLP analysis should be remanded to the client.

1.8 If an analysis of any one of the liquid fractions of the procedure leachate indicates that a regulated compound is present at such a high concentration that, even after accounting for dilution from the other fractions of the leachate, the concentration would be equal to or above the regulatory level for that compound, then the waste is hazardous and it may not be necessary to analyze the remaining fractions of the leachate. However, the remaining analyses should not be terminated without the approval of the client.

1.9 Volatile organic analysis of the leachate obtained using a bottle extraction, normally used for extractable organics and metals, can be used to demonstrate that a waste is hazardous, but only the ZHE option can be used to demonstrate that the concentration of volatile organic compounds is below regulatory limits.

1.10 On occasion clients may request modifications to this SOP. These modifications are handled as indicated in PT-QA-M-001, Quality Assurance Manual.

1.11 One-time procedural variations are allowed only if deemed necessary in the professional judgment of management to accommodate variation in sample matrix, radioactivity, chemistry, sample size, or other parameters. Any variation in procedure shall be completely documented on a Nonconformance Memo kept in the project file and described in the final report. The variation must be approved by the Project Manager and Technical Director or QA Manager. Any unauthorized deviations from this procedure must also be documented as a nonconformance, with a cause and corrective action described.
NOTE: TCLP is a “method defined parameter” as such any changes from the method may invalidate the analytical results. Careful consideration by the technical staff and discussion with the QA department is required before any procedural changes occur.

1.12 For subsampling procedures refer to SOP PT-QA-024.

2. SUMMARY OF METHOD

2.1 For liquid wastes that contain less than 0.5% dry solid material, the waste, after filtration through 0.6 to 0.8 µm glass fiber filter, is defined as the TCLP or SPLP leachate.

2.2 For wastes containing greater than or equal to 0.5% solids, the liquid, if any, is separated from the solids and stored for later analysis. The particle size of the remaining solid phase is reduced, if necessary. The solid phase is extracted with an

amount of extraction fluid equal to 20 times the weight of the solid phase. For TCLP, the extraction fluid employed for extraction of non-volatile analytes is a function of the alkalinity of the solid phase of the waste. For SPLP, the extraction fluid employed is a function of the region of the country where the sample site is located if the sample is a soil. Two leachates may be generated: a) one for analysis of non-volatile constituents (semi-volatile organics, pesticides, herbicides and metals and/or b) one from a Zero Headspace Extractor (ZHE) for analysis of volatile organic constituents. Following extraction, the liquid leachate is separated from the solid phase by filtration through a 0.6 to 0.8 µm fiber filter.

2.3 If compatible (i.e., multiple phases will not form on combination), the initial liquid phase of the waste is added to the liquid leachate and these are prepared and analyzed together. If incompatible, the liquids are analyzed separately and the results are mathematically combined to yield a volume-weighted average concentration.

3. DEFINITIONS

3.1 Leachate - the TCLP or SPLP extract (aqueous solution) generated from this procedure.

3.2 Percent Wet Solids - that fraction of a waste sample (as a percentage of the total sample) from which no liquid may be forced out by an applied pressure.

3.3 TALS – TestAmerica Laboratory Information Management System

3.4 NCM - Non-Conformance Memo – a system within TALS for the lab to communicate to project management and others when there is an anomaly seen with the samples or batch, or a QC failure.

3.5 Refer to the glossary in the Quality Assurance Manual PT-QA-M-001 for additional definitions.

4 INTERFERENCES

4.5 Oily wastes may present unusual filtration and drying problems. As recommended by EPA (see Figure 3), oily wastes will be evaluated on an individual basis. This applies specifically to samples containing viscous non-aqueous liquids that would be difficult to filter.

4.6 Wastes containing free organic liquids (i.e., those with separable non-aqueous liquid phases) will be assumed to be 100% liquid and totals analysis will be performed to determine if the oil exceeds TCLP limits.

4.7 Solvents, reagents, glassware and other sample processing hardware may yield artifacts and/or interferences to sample analysis. All these materials must be

demonstrated to be free from interferences under the conditions of the analysis by analyzing method blanks as described in the Section 9.0 and the individual determinative SOPs.

4.8 Glassware and equipment contamination may result in analyte degradation. Soap residue on glassware and equipment may contribute to this. All glassware and equipment should be rinsed very carefully to avoid contamination.

4.9 Phthalates may be eliminated by proper glassware cleanup and by avoiding plastics. Only glass, Teflon, or Type 316 stainless steel tumblers may be used for leachates to be analyzed for organics. Plastic tumblers may be used for leachates to be analyzed for metals only.

4.10 Overexposure of the sample to the environment will result in the loss of volatile components.

4.11 Potential interferences that may be encountered during analysis are discussed in the individual analytical methods.

5 SAFETY

5.1 Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual (CW-E-M-001), the Pittsburgh Facility Addendum EH&S Manual (PT-HS-001) and this document. This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

5.2 The following is a list of the materials used in this method, which have a serious or significant hazard rating. This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the SDS for each of the materials listed in the table. A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the SDS for each material before using it for the first time or when there are major changes to the SDS.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Acetic Acid	Corrosive Poison Flammable	10 ppm-TWA	Contact with concentrated solution may cause serious damage to the skin and eyes. Inhalation of concentrated vapors may cause serious damage to the lining of the nose, throat, and lungs. Breathing difficulties may occur.
Hydrochloric Acid	Corrosive Poison	5 ppm-Ceiling	Inhalation of vapors can cause coughing, choking, inflammation of the nose, throat, and upper respiratory tract, and in severe cases, pulmonary edema, circulatory failure, and death. Can cause redness, pain, and severe skin burns. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
Nitric Acid	Corrosive Oxidizer Poison	2 ppm-TWA 4 ppm-STEL	Nitric acid is extremely hazardous; it is corrosive, reactive, an oxidizer, and a poison. Inhalation of vapors can cause breathing difficulties and lead to pneumonia and pulmonary edema, which may be fatal. Other symptoms may include coughing, choking, and irritation of the nose, throat, and respiratory tract. Can cause redness, pain, and severe skin burns. Concentrated solutions cause deep ulcers and stain skin a yellow or yellow-brown color. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
Sodium Hydroxide	Corrosive	2 Mg/M3-Ceiling	Severe irritant. Effects from inhalation of dust or mist vary from mild irritation to serious damage of the upper respiratory tract, depending on severity of exposure. Symptoms may include sneezing, sore throat or runny nose. Contact with skin can cause irritation or severe burns and scarring with greater exposures. Causes irritation of eyes, and with greater exposures it can cause burns that may result in permanent impairment of vision, even blindness.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

5.3 Eye protection that protects against splash, laboratory coat, and appropriate gloves must be worn while samples, standards, solvents, and reagents are being handled. Cut resistant gloves must be worn doing any other task that presents a strong possibility of getting cut. Disposable gloves that have become contaminated will be removed and discarded, other gloves will be cleaned immediately.

5.4 Gas pressurized equipment is employed in this procedure. Be sure all valves and gauges are operating properly and that none of the equipment, especially tubing, is over-pressurized. **CAUTION:** Do not open equipment that has been pressurized until it has returned to ambient pressure.

5.5 A rotary agitation apparatus is used in this procedure. Certain samples may break the glass jars used in the procedure. For these samples, extra caution, including plastic or polyethylene overwraps of the glass jar, may be necessary.

5.6 Secure the tumbler and extraction apparatus before starting rotary agitation apparatus. Once the tumbler apparatus is started the area around the apparatus is roped off to prevent injury.

5.7 During sample rotation, pressure may build up inside the bottle. Periodic venting of the bottle will relieve pressure.

5.8 Exposure to chemicals must be maintained as low as reasonably achievable, therefore, unless they are known to be non-hazardous, all samples must be opened, transferred and prepared in a fume hood, or under other means of mechanical ventilation. Solvent and waste containers will be kept closed unless transfers are being made.

5.9 The preparation of standards and reagents and glassware cleaning procedures that involve solvents such as methylene chloride will be conducted in a fume hood, outside of the TCLP room, with the sash closed as far as the operation will permit.

5.10 All work must be stopped in the event of a known or potential compromise to the health and safety of a TestAmerica associate. The situation must be reported **immediately** to a laboratory supervisor or EH&S coordinator.

5.11 Due to the potential for ignition and/or flammability, do not attempt to dry non-aqueous liquid samples in an oven.

6 EQUIPMENT AND SUPPLIES

The following items are recommended for performing this procedure. Equivalent items should only be used when they result in an improvement in quality, efficiency, productivity, or cost. An item can be considered equivalent if with its use, the analytical and QA/QC requirements in this SOP can be met.

6.1 Extraction vessels

6.1.1 For volatile analytes - zero-headspace extraction (ZHE) vessel, gas-pressure actuated, Millipore YT3009OHW or equivalent (see Figure 2 in Appendix B).

6.1.1 For metals - either borosilicate glass jars (2.5 L plastic-coated, with Teflon lid inserts) or 2 L HDPE (Nalgene or equivalent) bottles may be used.

6.1.2 For non-volatile organics - only borosilicate glass may be used.

6.2 Vacuum filtration apparatus, capable of 0 - 50 psi and stainless steel pressure filtration apparatus (142 mm diameter), capable of 0 - 50 psi.

6.3 Borosilicate glass fiber filters, 0.6 - 0.8 µm (Whatman GF/F 14.2 cm, 0.7 µm or equivalent). When analyzing for metals, wash the filters with 1 N nitric acid and de-ionized water prior to use. As an alternative, certified pre-washed filters may be used. Glass fiber filters are fragile and should be handled with care.

6.4 Rotary agitation apparatus, multiple-vessel - Associated Design and Manufacturing Company 3740-6 or equivalent (see Figure 1). The apparatus must be capable of rotating the extraction vessel in an end-over-end fashion at 30 ± 2 rpm.

6.5 ZHE Extract Collection Devices are used to collect the initial liquid phase and the final extract of the waste from the ZHE device, either of the following may be used:

6.5.1 Gas-tight syringes, 100 mL capacity, Hamilton 0158330 or equivalent,

6.5.2 or dispense leachate directly into a VOA vial.

6.6 Top loading balance, capable of 0 - 4000 ± 0.01g (all measurements are to be within ± 0.1 grams).

6.7 pH meter and probe capable of reading to the nearest 0.01 unit, and with automatic temperature compensation. (Accumet XL150 pH meter or equivalent)

6.8 Magnetic stirrer/hotplate and stirring bars

6.9 VOA vials, 40 mL, with caps and septa

6.10 Glass jars, 1/2 - 1 gallon, with Teflon lid-liners

6.11 Nalgene plastic bottles, 1 liter

6.12 Peristaltic pump, Cole Parmer or equivalent

6.13 Miscellaneous laboratory glassware and equipment

7 REAGENTS AND STANDARDS

The following items are recommended for performing this procedure. Equivalent items should only be used when they result in an improvement in quality, efficiency, productivity, or cost. An item can be considered equivalent if with its use, the analytical and QA/QC requirement in this SOP can be met. Please refer to the SDS prior to the use of any reagent or standard.

Reagent grade, or better, chemicals must be used unless otherwise indicated. Follow manufacturer's instructions for storage and expiration.

7.1 Reagent water for non-volatile constituents must be produced by a Millipore DI system or equivalent. For volatile constituents, water must be passed through an activated carbon filter bed (Milli-Q or tap water passed through activated carbon). Reagent water must be free of the analytes of interest as demonstrated through the analysis of method blanks.

7.2 Hydrochloric acid, 1 N: Carefully add 83.3 mL concentrated HCl to 800 mL reagent water, cool and dilute to 1 liter with reagent water. Cap and shake to mix well. Store at room temperature. This solution expires 1 year after preparation.

CAUTION: Always add acid to water to avoid splattering.

7.3 Nitric acid, concentrated, (HNO_3)

7.4 Sodium hydroxide, 1 N, purchased or prepared: Carefully add 40 g reagent grade NaOH pellets to 800 mL reagent water, stir until the pellets are completely dissolved, cool and dilute to 1 liter with reagent water. Store at room temperature. If prepared in the laboratory, this solution expires 1 year after preparation.

CAUTION: Significant heat is generated in the preparation of this reagent.

7.5 Acetic acid, glacial: concentrated, reagent grade liquid (HOAc).

7.6 pH calibration solutions: pH Buffers of 2, 4, 7, 10 and 13 - Purchased with certificate of analysis. A fresh portion of buffer solution must be used each day of analysis.

7.7 **TCLP Leaching Fluids**

7.7.1 The pH of both solutions listed below must be checked and recorded daily with use. The pH meter/probe must be calibrated prior to daily use.

7.7.2 TCLP Leaching Fluid 1 is purchased from Ricca Chemical Company, TCLP Leaching Fluid 2 is purchased from Lab Chem. Fluids may be prepared following the procedure outlined in sections 7.7.5 and 7.7.6. If prepared in the

laboratory, the solution expires 1 year after preparation, or if the pH falls outside the required range.

7.7.3 The leaching fluids MUST be prepared correctly. If the desired pH range is not achieved and maintained, the TCLP may yield erroneous results due to improper leaching. If the pH is not within the specifications, the fluid must be discarded and fresh extraction fluid prepared. The pH of purchased and prepared TCLP fluids must be checked prior to each day's use, and the actual pH is recorded in the TALS batch.

7.7.4 Additional volumes of extraction fluids listed above may be prepared by multiplying the amounts of acetic acid and NaOH by the number of liters of extraction fluid required.

7.7.5 TCLP Fluid #1: Carefully add 5.7 mL glacial acetic acid and 64.3 mL of 1 N NaOH to 500 mL reagent water in a 1 liter volumetric flask. Dilute to a final volume of 1 L with reagent water, cap and shake to mix well. When correctly prepared, the pH of this solution is 4.93 ± 0.05.

7.7.6 TCLP Fluid #2: Carefully add 5.7 mL glacial acetic acid to 500 mL reagent water in a 1 liter volumetric flask. Dilute to a final volume of 1 L with reagent water, cap and shake to mix well. When correctly prepared, the pH of this solution is 2.88 ± 0.05.

7.8 Nitric acid, 20% solution: Slowly and carefully add 200 mL concentrated HNO_3 to 800 mL reagent water. Cap and shake to mix well. Store at room temperature. This solution expires 1 year after preparation.

7.9 Sulfuric acid / nitric acid - 60/40 weight percent mixture. Cautiously mix 60 g of concentrated sulfuric acid with 40 g of concentrated nitric acid. Store at room temperature. This solution expires 1 year after preparation.

7.10 SPLP Leaching fluids

7.10.1 SPLP Leaching Fluids are purchased from Lab Chem Inc. or prepared following the procedures outlined below. The pH of SPLP fluids must be checked prior to each day's use. If not within specifications, the fluid is discarded and fresh fluid prepared.

7.10.2 SPLP fluid #1: Add 60/40 weight percent mixture of sulfuric and nitric acids (7.9) to reagent water until the pH is 4.20 ± 0.05. This fluid is used for soils from a site that is east of the Mississippi River and for wastes and wastewaters.

7.10.3 SPLP fluid #2: Fluid #1 is diluted with laboratory reagent water until the pH is 5.00 ± 0.05. This fluid is used for soils from a site that is west of the Mississippi River.

7.10.4 DI Water for SPLP Volatiles leaching - This is laboratory reagent water. SPLP leaching for Volatiles and always performed with DI water only. Additionally, any cyanide-containing waste or soil is leached with DI water because leaching of cyanide containing samples under acidic conditions may result in the formation of hydrogen cyanide gas.

7.11 Methanol - used to aid in cleaning oil-contaminated equipment

7.12 Metals Spiking Standards – See Table 4 at the end of this document for details

7.13 Nitric Acid, concentrated – used for preserving leachates' MS/MSD volume for metals analysis. Sample aliquots for metals are filtered directly into pre-preserved bottles containing nitric acid.

8 SAMPLE COLLECTION, PRESERVATION, SHIPMENT AND STORAGE

8.1 Samples being analyzed for non-volatile organic compounds should be collected and stored in glass containers with Teflon lid liners.

8.2 Samples being analyzed for metals only can be collected in either glass or polyethylene containers.

8.3 When the waste is to be evaluated for volatile analytes, care should be taken to minimize the loss of volatiles. Samples shall be collected and stored in a manner intended to prevent the loss of volatile analytes (e.g., samples should be collected in Teflon lined septum capped vials with minimal headspace and stored at >0.0 °C but ≤6.0°C).

8.3.1 Samples should not be cooled for shipment or storage <u>if doing so will change the composition of the sample</u>.

8.3.2 Samples should be opened only immediately prior to extraction.

8.4 Samples must not have any chemical preservation prior to leaching. Chemical preservatives are never added to leachates generated for organic analysis. Chemical preservation is added only to inorganic parameters after leaching and filtration.

8.5 Samples should be refrigerated to >0.0 °C but ≤6.0°C, unless refrigeration results in irreversible physical changes to the waste. If precipitation occurs, the entire sample (including precipitate) should be extracted.

8.6 The minimum TCLP or SPLP sample collection size is determined by the physical state or states of the waste and the analytes of concern. The amount of waste required varies with the percent solids. The lower the percent solids, the more waste will be required for preliminary and final testing. For aqueous samples containing between 0.5 and 10% solids, several kilograms of sample are required to complete the analyses. The general minimal requirements when the samples are 100% solids include: 1 - 32 oz jar for semi-volatile organic analysis and metals, and 1 - 4 oz jar for volatile organic analysis. Low-density sample materials, such as rags or vegetation, will require larger volumes of sample. For liquid samples (less than 50% solids), minimum requirements are 2 - 32 oz jars for semi-volatile organic analysis and metals, and 2 - 8 oz jars for volatile organic analysis. If volatile organic analysis is the only requested parameter, 2 separate jars are required. If matrix spike or duplicate control samples are requested, additional sample volume is required. If sufficient sample volumes were not received, analyses cannot be started and the client should be notified as soon as possible. Reduced volume will only be done if there is insufficient sample received from the client.

8.7 TCLP or SPLP leachates should be prepared for analysis and analyzed as soon as possible following extraction. Leachates or portions of leachates for metallic analyte determinations must be acidified with nitric acid to a pH less than 2, unless precipitation occurs. If precipitation occurs upon addition of nitric acid to a small aliquot of the leachate, then the remaining portion of the leachate shall not be acidified and the leachate shall be analyzed as soon as possible. All other leachates should be stored under refrigeration (>0.0 °C but ≤6.0°C) until analyzed. ZHE leachates must be stored in VOA vials filled to eliminate all headspace.

8.8 Samples are subject to appropriate treatment within the following time periods:

Table 1 – Holding Times

Parameter	Collection to Start of TCLP/SPLP Leach	End of TCLP/SPLP Tumble to Semi-Volatile Extraction	End of TCLP/SPLP Leach or Semi-volatile Extraction to Analysis
Volatiles	14	N/A	14
Semivolatiles	14	7	40
Mercury	28	N/A	28
Other Metals	180	N/A	180

NOTE: The initial holding time is measured from date of collection to date TCLP/SPLP extraction started. (This should be the TCLP/SPLP extraction date in TALS LIMS.) Semi-volatile method prep holding time is measured from the day tumbling is complete to the start of method extraction. Subsequent analysis holding times are measured from the date extraction (TCLP/SPLP or method prep) starts**.** If sample holding times are exceeded, the values obtained will be considered minimal concentrations. Exceeding holding times is not acceptable in establishing that a waste does not exceed the regulatory level. Exceeding the holding time will not invalidate characterization if the waste exceeds the regulatory limit. The Total Elapsed Time is to be used as guidance. If preps are initiated at the last possible moment of a holding time, the elapsed times may be exceeded.

9. QUALITY CONTROL

9.1 A Quality Control Batch (QC Batch) is a set of up to 20 field samples of similar matrix that behave similarly and are processed using the same procedures, reagents and standards within the same time period. The same lot of reagents must be used within a batch. A minimum of one TCLP/SPLP extraction blank (Method Blank), and enough leachate to prepare one Matrix Spike (MS), and one Matrix Spike Duplicate (MSD) are required with each TCLP/SPLP leachate batch.

9.2 TCLP/SPLP Extraction Blanks - A minimum of one blank (using the same extraction fluid as used for the samples) must be prepared and analyzed for every batch of

samples extracted in a particular vessel type. The blanks are generated in the same way as the samples (i.e., blanks will be tumbled and filtered with the samples). Consult the individual analytical Method SOPs for blank acceptance criteria.

Note: ZHE vessels are uniquely numbered. Each time a new batch is set up the blank should be rotated sequentially to the next vessel to ensure all vessels are periodically checked. A blank is required to be performed at least every 20th time a vessel is used (see Appendix C for Example Blank Vessel Rotation Forms).

9.3 Matrix Spike (MS/MSD) - Matrix spikes are used to monitor the performance of the analytical methods on the matrix and to assess the presence of interferences. MS/MSD generation will be done when client requested with each batch of 20 samples.

9.3.1 Matrix spikes are added after filtration of the TCLP/SPLP leachate. Spikes are not added prior to the TCLP/SPLP leaching. For metals, matrix spikes are to be added before preservation with nitric acid.

- MS/MSD for Mercury are spiked with 0.25 mL of the 1000 ug/L TCLP Hg spiking solution to 50 mL sample.

- MS/MSD for Metals are spike with 0.50 mL of the solutions listed in Table 4 at the end of this document, according to the elements that are requested, to 50 mL of sample.

9.3.2 The use of internal calibration or alternate methods may be needed when the recovery of the matrix spike is below the expected performance (see Section 9.4.2).

9.3.3 Consult the individual analysis SOPs for additional guidance on spike compounds and levels.

9.4 Corrective Actions

9.4.1 Consult the Laboratory QC Program SOP (PT-QA-021) and individual analysis SOPs for corrective action for QC failures.

9.4.2 Method of Standard Additions (MSA) shall be used for metals if all of the following conditions are met:

- Recovery of the analyte in matrix spike is not at least 50%,

- The concentration of the analyte does not exceed the regulatory level, and

- The concentration of the analyte measured in the sample is within 20% of the appropriate regulatory level.

If the matrix spike recovery is 5% or less due to dilution or matrix interference, contact the project manager and client for guidance. The client should also be contacted prior to initiation of any MSA steps. Refer to the individual analysis SOPs for details on how to perform MSA analysis.

9.4.3 If the temperature during the tumbling procedure is not maintained at 23 ± 2℃ then the samples are required to be re-tumbled. If insufficient sample remains to repeat the tumbling process, the Project Manager will be notified and will call the client for direction on how to proceed.

10. PROCEDURE

10.1 PRELIMINARY SAMPLE EVALUATIONS (Refer to Flow Chart #1, Appendix D)

10.1.1 Preliminary TCLP evaluations (percent solids, particle size, selection of extraction fluid, and fluid/leachate compatibility) are required to be done using a minimum of a 100 gram aliquot of waste. This aliquot may also undergo the actual TCLP or SPLP extraction for Non-volatiles ONLY IF it has NOT been oven dried. If the solid portion is oven dried, a separate aliquot must be used for the actual leaching procedure.

10.1.2 Consult the holding times for the appropriate tests (Section 8.8) and prioritize extractions such that holding times are not exceeded.

10.1.3 All non-aqueous samples must be leached with a minimum100g to 2 L for TCLP and SPLP. The following sample volumes are given to the laboratory for further preparation and analysis after leaching and/or filtration.

Table 2 - Minimum Required Leachate Volume

Analysis	Required Volume (mL)
Volatiles	2 x 40
Semi-volatiles	200
Pesticides	100
Herbicides	100
Metals	150

10.1.3.1 For TCLP and SPLP samples designated or chosen for matrix spike and matrix spike duplicate analysis, three times the listed volumes are required. Spiking is performed after leaching and/or filtration.

10.1.4 SAMPLE DESCRIPTION

10.1.4.1 Observe the number of phases present in the sample according to apparent density. (Note: It may be impossible to distinguish apparent density if only one liquid phase is observed and there is no indication on the COC form. If this is the case, record it as aqueous material and let the subsequent analytical record show if the liquid is organic.) It is common that when more than one container of multi-phasic materials is received from the field, each container will show different amounts of each phase.

10.1.4.2 If the sample has multiple phases and is received in more than 1 bottle then the contents of each bottle should be combined in a single larger container prior to processing the sample further. If this is not possible, then the alternate procedure described in the following section should be used. This procedure is not appropriate if testing will be done for volatile organic compounds.

10.1.4.3 Properly record the relative amounts of each phase by measuring the depth of the layers in each container after the contents have been allowed to settle. Determine the combined volume of each phase for all containers. These values are needed to determine the correct volume/mass adjustments on the final result.

10.1.5 PERCENT SOLID PHASE

10.1.5.1 Percent Solids and ZHE Extractions - The ZHE filtration apparatus cannot accurately determine percent solids less than 5%. If an extraction is to be performed solely for volatile organic compounds and the percent solids concentration is apparently greater than 5%, proceed to Section 10.3 (Procedure: ZHE Extraction Procedure, Volatile Constituents). Otherwise, continue with the steps in this section. The aliquot of sample used here cannot be used again for the ZHE extraction.

10.1.5.2 Determine Type of Filtration Apparatus Needed

- If the waste will obviously yield no free liquid when subjected to pressure filtration (i.e., it is 100% solid), then proceed to Section 10.1.6 (Particle-size Reduction).

- If the sample is mostly a non-viscous liquid (water or non-viscous organic liquid) of low solids content (<10%) or a highly granular, liquid containing waste vacuum filtration may be used.

- If the sample is viscous (sludge or has high solids content), use pressure filtration.

10.1.5.3 Weight of filter - Measure and record this value before loading the filter into the filter holder.

10.1.5.4 Weight of subsample and filtrate for percent solids measurement.

- Assemble the filtration apparatus (use blunt forceps to handle the 0.6 to 0.8 μm filter membrane).

- Homogenize the waste in the sample container. Measure and record the gross weight.

- Measure and record the tare weight of the filtration vessel, screen and filter paper.

- Transfer the sample to the filtration device attempting to spread the waste sample evenly over the surface of the filter. Measure and record the tare weight of the empty sample container and any residual sample.

- Calculate and record the net weight of sample used for testing.

10.1.5.5 Filtration for percent solids

- Slowly apply gentle pressure or vacuum of 10 psi to the filtration apparatus. Allow the sample to filter until no SIGNIFICANT additional liquid has passed through the filter during a 2 minute period.

- Repeat previous step by increasing the pressure in 10 psi increments until a maximum of 50 psi is reached. Stop the filtration when no additional filtrate is generated within a 2 minute period.

Note: Some samples will contain liquid material that does not filter (e.g., oil). Do not attempt to filter the sample again by exchanging filters. Viscous oils or any wastes which do not pass through the filter are classified as a solid.

- Remove the filtrate collection vessel, weigh and record the gross weight.
- Calculate and record the net weight of filtrate. This result will be used in the percent solids calculation.
- Pour the filtrate into a graduated cylinder. Measure and record the volume of the aqueous phase. Measure and record the volume of any organic phase. If more than one organic phase is observed, provide a description. These results will be used in the final sample calculations.
- Retain the filtrate for use in Section 10.1.8 (Determination of Filtrate/Extraction Fluid Compatibility), and for possible recombination with the filtrate obtained in Section 10.2.13.

10.1.5.6 Percent of Wet Solids

- Subtract the net weight of the filtrate `from the net weight of the subsample to calculate the total weight of wet solids.
- Calculate the weight percent of wet solids using the Equation in Section 11.1.1.
- If the percent wet solids result is ≥ 0.5% and < 5.0%, and it is noticed that a small amount of the aqueous filtrate is entrained in the wetting of the filter, proceed to Section 10.1.5.7 to complete the percent solids measurement on a dry-weight basis. **Note:** If obviously oily (non-aqueous) material is entrained on the filter, do not dry the filter; proceed to Section 10.1.7 (Determination of Appropriate Extraction Fluid).
- If the percent wet solids result is greater than 5.0%, proceed to Section 10.1.6 (Particle-Size Reduction).
- If the percent wet solids result is less than 0.5%, the sample is treated as a TCLP/SPLP liquid. Discard the solid phase. No leaching will be necessary; the filtrate is the final TCLP/SPLP leachate.

10.1.5.7 Weight percent of dry solids (skip this step and refer to Appendix B for oily samples). Note: These steps are required only if it is noticed that a small amount of the filtrate is entrained in wetting of the filter and the percent wet solids content is ≥ 0.5% and < 5.0 %

- Remove the filter with the wet solids from the filtration apparatus.
- Dry the filter and solid phase at 100 ± 20 ° C.
- Remove the filter from the oven and allow it to cool in a desiccator.
- Weigh and record the gross dry weight.
- Repeat the drying step. Weigh and record the second gross dry weight. If the two weighings do not agree within 1%, perform additional drying and weighing until successive weighings agree within 1%.
- Calculate the weight percent of dry solids.
- If the dry solids result is ≥ 0.5% and the sample will be extracted for non-volatile constituents, proceed to Section 10.1.6 (Particle Size Reduction) using a fresh wet portion of waste.
- If the percent solids result is less than 0.5%, discard the solid phase. No leaching will be necessary; the filtrate is the TCLP leachate. Proceed to Section 10.1.8 (Determination of Filtrate/Leachate Compatibility) to determine whether or not the material is a non-aqueous, immiscible liquid.

10.1.6 PARTICLE-SIZE REDUCTION FOR FLUID SELECTION

10.1.6.1 This procedure is not applicable to oily or organic liquid samples that are to be treated as a TCLP/SPLP solid.

10.1.6.2 The subsample used for fluid selection must consist of particles less than 1 mm in diameter (versus the less than 1 cm requirement for the material used for the actual extraction). Inappropriate use of coarser materials could result in the selection of the wrong fluid type.

10.1.6.3 Surface area exclusion - size reduction is not required if the sample surface area is greater than or equal to 3.1 cm^2 per gram.

10.1.6.4 If the sample contains particles greater than 1 mm in diameter, crush, cut, or grind the solids to the required size.

10.1.6.5 Consult your supervisor or manager when dealing with unusual sample matrices (e.g., wood, cloth, metal, brick).

10.1.6.6 Equipment blank will be generated when samples undergo particle size reduction. This blank is used to evaluate sieve cleanliness. The blank is generated after the sample goes through the sieve and the sieve is washed and methanol rinsed.

10.1.7 DETERMINATION OF APPROPRIATE EXTRACTION FLUID

10.1.7.1 If the solid content is greater than or equal to 0.5%, and if the sample is being analyzed for metals or nonvolatile organic compounds, the type of leaching solution must be determined.

10.1.7.2 Follow times, temperature, and particle size specified in this section as closely as possible. If reaction time between the acid solution and solid waste is too short or too long, the procedure may produce false pH readings.

10.1.7.3 For SPLP, refer to Section 7.10 for fluid selection. Matrix type must be specified by the client. Check special instructions or see the project manager, then record the fluid type selected on the SPLP worksheet.

10.1.7.4 The TCLP leaching fluid for all volatiles is Fluid #1.

10.1.7.5 For TCLP leach fluid determination for non-volatile analytes, continue with the following steps.

10.1.7.6 Calibrate the pH meter with fresh buffer solutions, using buffers that bracket the anticipated range of samples. (Instructions are for the Accumet XL150 pH meter. If another meter type is used, follow the manufacturer's instructions for calibration.) The meter is calibrated daily with use.

a. Touch **Standardize** on the pH measure screen to access the standardization screen. Touch "**Clear**" to clear the last calibration.

b. Rinse the electrode with reagent water and place in the pH 2.0 buffer. Enter the value (2.0) of the standard into the pH meter using the touch screen, and touch **Enter**. When the reading stabilizes, **STABLE** will appear on the screen. Touch **Confirm** to accept that buffer standardization.

c. Rinse the probe and place it into the 4.0 buffer. Touch **Standardize** to access the standardization screen again, and repeat the steps in b.

d. Repeat b. and c. for 7.0, 10.0 and 13.0 buffers.

e. The Slope of the calibration curve is displayed on the pH Measure screen. The slope must meet the of ≥95% (or other manufacturer's recommended limit) for the calibration to be acceptable.

f. If the slope requirement is not met, clean the probe and repeat calibration with fresh buffers. If the second calibration does not meet the acceptance limit, change the KCl solution in the electrode, and repeat calibration.

g. Each buffer reading must be within 0.05 pH units of the true buffer value. If readings do not meet this requirement, repeat with a second fresh portion of buffer, or repeat the calibration.

h. After the calibration the ICV/LCS, a second source pH 7 buffer, is analyzed before sample analysis. The pH readings must be within 0.05 pH units.

i. Document the calibration and verification in the meter calibration log daily.

10.1.7.7 Weigh out a 5.0 ± 0.1 g subsample (less than 1 mm particle size) of the solid phase into a 250-mL beaker and enter the actual weight on the TCLP worksheet.

10.1.7.8 Add 96.5 ± 1.0 mL of reagent water, cover with a watchglass, and stir for 5 minutes on a stirrer and enter the actual volume on the TCLP worksheet.

10.1.7.9 Measure and record the sample pH. **Note**: To avoid damaging the pH probe when organic liquid (i.e., Oily samples) is present, use narrow range pH indicator paper to determine the pH of this type of sample.

10.1.7.10 If the pH is less than or equal to 5.00, use Fluid #1 and proceed to Section 10.1.8 (Fluid Compatibility).

10.1.7.11 If the fluid pH is greater than 5.0, add 3.5 mL 1 N HCl, cover with a watchglass. Slurry the sample briefly then heat at 50 ^{o}C ± 5°C for 10 minutes. Record the temperature of the hotplate on the TCLP worksheet. **Note**: The heating cycle is a critical step. If the solid waste does not remain in contact with the acidic solution under specified time and temperature conditions, an erroneous pH may be measured.

10.1.7.12 Cool to room temperature.

10.1.7.13 Measure and record the pH immediately after the sample has reached room temperature.

- If the pH is less than or equal to 5.00, use Fluid #1.
- If the pH is greater than 5.00, use Fluid #2.

10.1.8 DETERMINATION OF FILTRATE/EXTRACTION FLUID COMPATIBILITY (skip this step for SPLP extractions)

10.1.8.1 Place 5 mL of the appropriate leaching fluid (determined in the previous step) into a 20-25 mL vial. **Note**: Use fluid type # 1 if simply testing the filtrate for a sample with less than 0.5% solids.

10.1.8.2 Add 5 mL of the initial filtrate, cap and shake.

10.1.8.3 If the phases are miscible, the initial filtrate and solid phase leachate will be physically recombined upon completion of the leachate generation.

10.1.8.4 If the phases are NOT miscible, the initial filtrate and the solid phase leachate will be prepared and analyzed separately and the results mathematically combined (see Section 11.1.4).

10.1.9 For samples requiring analysis for semi-volatile organics, pesticides, herbicides or metals proceed to Section 10.2.

10.1.10 For samples requiring analysis for volatile organics (ZHE), proceed to Section 10.3.

10.2 BOTTLE EXTRACTION PROCEDURE: NON-VOLATILE CONSTITUENTS: SEMI-VOLATILES, PESTICIDES, HERBICIDES, METALS (Refer to Flow Chart #2, Appendix D)

10.2.1 Record all masses to the nearest 0.1 g.

10.2.2 The aliquot used in the Preliminary Evaluation may be used for this procedure only if it was not oven dried. If the sample is 100% solid or if the preliminary aliquot was not oven dried proceed directly to Section 10.2.7 (Particle Size Reduction). If the Preliminary Evaluation aliquot was oven dried then, using a fresh aliquot of sample, continue as described in Sections 10.2.3 through 10.2.6.

10.2.3 Examine the sample and determine the type of filtration to employ per Section 10.1.5.2.

10.2.4 Determine and record the volume (mass) of the initial filtrate. Cover with aluminum foil and retain for use as defined in Section 10.2.13.

10.2.5 Determine and record the "solid" phase mass by subtracting the mass of the liquid filtrate from the mass of the subsample.

10.2.6 Evaluate the solid portion of the waste for particle size. If it contains particles greater than 1 cm in size, prepare the solid portion of the waste for leaching by crushing, cutting, or grinding such that all particles are less than 1 cm in size (i.e., capable of passing through a 9.5 mm, 0.375 inch, standard sieve). Size reduction is not required if the sample surface area is greater than or equal to 3.1 cm^2 per gram.

10.2.6.1 Consult your supervisor or manager when dealing with unusual sample matrices (e.g., wood, cloth, metal, brick). Scissors or shears may be used to cut cloth, plastic or sheet metal. Saws may be used for wood or solid metal. Bricks, rocks, or other solids amenable to grinding should be subcontracted out for particle size reduction. (Contact the assigned Project Manager.) Note that size reduction to fine powder is not appropriate, and could invalidate results. If necessary, consult client for guidance.

10.2.7 Weigh out 100g of solid sample, or the weight required to provide 100g of solid if adjusted for the percent moisture present in the sample.

10.2.8 NOTE: If the solid sample contains 10% moisture that is not filterable liquid, the weight of sample required for 100g of solids is 110g. The corresponding leaching fluid to be added is then reduced for the 10% moisture content of the sample, or 10mL, to 1990mL.

10.2.9 Weigh the required mass of solid phase into an appropriate bottle (plastic for metals only, glass for all others) and slowly add 2000mL, or the volume adjusted for the percent moisture present in the solid sample, of appropriate leaching fluid as determined under Section 10.1.7 (e.g., 100 g of sample would require 2000 g of leaching fluid). Record the weight of the sample aliquoted for the extraction on and the amount of extraction fluid added on the TCLP worksheet.

10.2.9.1 Record the vessel number used for ZHE leaching for each sample on the TCLP worksheet. Note that the same vessel should not be used repeatedly for the method blank, if practical.

10.2.9.2 Vessels are labeled and tracked in a logbook. The date that the vessel is first used and the date it is disposed of are included in this logbook.

10.2.10 Ensure any effervescence has stopped before capping the bottle tightly. Secure in a rotary agitator and rotate end-over-end at 28-32 rpm for 16-20 hours. The temperature of the room must be maintained at 23 ± 2℃ throughout the tumbling period. Start and end times for tumbling are recorded in the TALS batch. The room temperature is monitored using the min/max thermometer that has been set at the beginning of the tumbling period. Both the minimum and maximum temperature of the room during the tumbling period are recorded on the TALS worksheet. The actual tumbler speed (RPM's) will be monitored at the start of each batch and is recorded in the TALS batch information. As agitation continues, pressure may build up within the bottle for some types of wastes. To relieve excessive pressure, the bottle may be removed and opened periodically in a properly vented hood to relieve any built-up pressure.

10.2.11 Remove the bottle and filter the sample using vacuum or pressure filtration by filtering through a new glass fiber filter as discussed in Sections 10.1.5.2. For final filtration of the TCLP/SPLP leachate, the glass fiber filter may be changed, if necessary, to facilitate filtration. Filters must be acid washed if metals are to be determined (see Section 6.3). The entire sample need not be filtered; however, sufficient volume should be generated to support the required analyses. Record the date and time the filtration is started on the TCLP/SPLP worksheet.

10.2.12 If the waste contained no initial filtrate, this solution from 10.2.11 is defined as the TCLP/SPLP leachate.

10.2.13 If the waste did yield an initial filtrate, consult the worksheet for initial filtrate/leachate compatibility. If they are compatible, they are to be combined in the correct proportions (see Section 11.1.4) and mixed well. This combined solution is defined as the TCLP/SPLP leachate.

10.2.14 If the individual phases are NOT compatible, they are to be prepared and analyzed separately and the results combined mathematically. (See Section 11.1.5.)

10.2.15 Measure the pH of the TCLP leachate after filtration and record on the TCLP/SPLP worksheet in TALS. (Do not attempt to measure the pH of oily samples as the probe may be rendered inoperable. Measure the pH of oily samples with pH strips.)

10.2.16 Prepare subsamples for metals for MS/MSD quality control testing using the appropriate TCLP/SPLP spiking solution (do not spike for organics). Record the lot number of the spiking solution on the TCLP/SPLP worksheet. Refer to the

appropriate determinative SOPs for further guidance on the spike components, levels and action criteria.

10.2.17 Immediately preserve the leachate as follows:

- Metals: pH < 2 w/50% HNO_3 for non-oils (do not acidify oils or non-aqueous miscible liquids. Do an NCM along with the batch to let the lab and PM know the sample was not preserved.)
  - o All metals aliquots other then the MS and MSD volumes shall be filtered into a pre-preserved 125mL bottle containing nitric acid.
  - o The preservation pH shall be checked by dripping a few drops of the preserved sample onto acid-range pH paper. The pH of the preserved leachate must be ≤2.
  - o MS and MSD volumes are spiked according to section 9.3, and then preserved with nitric acid to pH ≤2. Start by adding 0.5mL concentrated nitric acid to the spiked sample, add an additional 0.5mL if the pH is not ≤2.
  - o Document the preserved pH in the comment column of the TALS batch.

- All other parameters: Refrigerate to >0.0˚C but ≤6.0˚C

Note: Refer to Section 8.7 if precipitation occurs upon preservation.

10.2.18 Label each sample with the appropriate information and submit to the appropriate analytical groups for leachate extraction/digestion and analysis with copies of the TCLP preparation worksheets.

10.3 ZHE EXTRACTION PROCEDURE: VOLATILE CONSTITUENTS (Refer to Flow Chart #3, Appendix D)

10.3.1 Use the ZHE device to obtain a TCLP/SPLP leachate for analysis of volatile compounds only. Leachate resulting from the use of the ZHE shall NOT be used to evaluate the mobility of non-volatile analytes (e.g., metals, pesticides, etc.).

10.3.2 Due to some shortcomings of the method, losses of volatile compounds may occur. Extra care should be observed during the ZHE procedure to ensure that such losses are minimized. Charge the ZHE with sample only once and do not open the device until the final extract has been collected. Do not allow the waste, the initial liquid phase or the extract to be exposed to the atmosphere any longer than necessary.

10.3.3 If the TCLP/SPLP extraction is for volatile components only, refer to Section 10.1.5.1 before proceeding.

10.3.4 All masses should be recorded to the nearest 0.1 g.

10.3.5 Assemble the ZHE apparatus. Test for leakage by closing all valves except the gas inlet/outlet valve and pressurizing to 50 psi. Allow to stand for 15 minutes and check the pressure on the built-in gauge to make sure it is not leaking. If the pressure is NOT 50 psi, consult your supervisor.

10.3.6 Adjust the ZHE piston in the ZHE body to the appropriate height (slightly moisten the O-rings with leaching fluid if necessary).

10.3.7 Consult the backlog for matrix designation and examine the sample. If the sample appears to be different from the preliminary information found on the backlog, consult your supervisor.

10.3.8 If the preliminary evaluations indicated the need for particle size reduction, homogenize the waste, weigh out a sufficient size subsample and prepare for leaching by crushing, cutting, or grinding such that all particles are less than 1 cm in size as measured with a ruler (Do NOT sieve the sample). Size reduction is not required if the sample surface area is greater than or equal to 3.1 cm^2 per gram. **Note:** To minimize loss of volatiles, samples for volatiles that require particle size reduction should be kept in sample storage (>0.0 ºC but ≤6.0°C) until immediately before size reduction. Aggressive reduction, which would generate heat, should be avoided and exposure of the waste to the atmosphere should be avoided to the extent possible. Size reduction to a fine powder is not appropriate.

10.3.8.1 Consult your supervisor or manager when dealing with unusual sample matrices (e.g., wood, cloth, metal, brick). Scissors or shears may be used to cut cloth, plastic or sheet metal. Saws may be used for wood or solid metal. Bricks, rocks, or other solids amenable to grinding should be subcontracted out for particle size reduction.

10.3.9 Place the ZHE apparatus on the balance and tare the balance.

10.3.10 Determine the appropriate size subsample to weigh using the percent solids information from Section 10.1.5, based on 25 g of solid.

10.3.10.1 For wastes that are 100% solids, a 25 g sample is used.

10.3.10.2 For wastes containing < 0.5% solids the liquid portion of the waste, after filtration, is defined as the TCLP/SPLP leachate. Filter enough of the sample to support all of the volatile analyses required.

10.3.10.3 For wastes containing ≥ 0.5% and < 5.0% solids, a 500 g subsample of waste is recommended.

10.3.10.4 If the sample has ≥ 5.0% solids, the appropriate sample size should be determined using the equation in Section 11.2. **Note:** For wastes containing greater than 0.5% wet or dry solids (Section 10.1.5), the "solids" value from the ZHE filtration process may be used to determine the volume of fluid to load into the ZHE. This approach is recommended since the solids value from Section 10.1.5 may differ from the filtration solids due to sample variability or differences in the filtration apparatus.

10.3.11 Homogenize and transfer an appropriate size subsample of the waste into the ZHE and record the mass.

10.3.12 Carefully place the glass fiber filter between the support screens and secure to the ZHE. Tighten all the fittings.

10.3.13 Place the ZHE in a vertical position; open both the gas AND liquid inlet/outlet valves. Attach a gas line to the gas inlet/outlet valve.

10.3.14 If the waste is 100% solid, slowly increase the pressure to a maximum of 50 psi to force out as much headspace as possible and proceed to Section 10.3.19.

10.3.15 If the waste is < 100% solids, carefully apply gentle pressure of 10 PSI (or more, if necessary) to force all headspace slowly out of the ZHE. At the FIRST appearance of liquid from the liquid inlet/outlet valve, quickly close the valve and discontinue gas pressure.

10.3.16 Assemble a syringe and place the plunger in all the way. Adjust the tension on the plunger to provide slight drag. Attach the pre-weighed syringe or Tedlar bag to the liquid inlet/outlet valve and open the valve. Record the tare weight of the collection device.

10.3.17 Carefully apply gas pressure of no more than 10 PSI to force out the liquid phase. Allow the sample to filter until no SIGNIFICANT additional filtrate has passed in a 2 minute period. **Note**: If the capacity of the syringe is reached, close the liquid inlet/outlet valve, discontinue gas pressure, remove the syringe and return to Section 10.3.15.

10.3.18 Repeat previous step increasing the pressure in 10 PSI increments until 50 PSI is reached and no significant liquid has passed in a 2 minute period. Remove the collection device and record the total weight of the collection device with filtrate. Close the valve and discontinue gas pressure. Transfer the filtrate to VOA vials and label appropriately. Calculate the weight of filtrate collected. **Notes:** If the original waste contained less than 0.5% solids (Section 11.2.5), this filtrate is defined as the TCLP/SPLP leachate and you may proceed to

Section 10.3.28. Otherwise, save the vials by storing at 4 C under minimal headspace conditions, for recombination as in Section 10.4.27. The material remaining in the ZHE is defined to be the "solid" phase. Calculate the weight of the solid phase.

10.3.19 Based on the information from Sections 10.1.5 and 10.3.10 and using the formula in 11.1.3, determine the weight of fluid to load into the ZHE on the "solid" phase. The ZHE device has approximately a 500-mL capacity. Based on the need to add an amount of extraction fluid equal to 20 times the mass of the "solid" phase, the ZHE can therefore accommodate a maximum of 25 grams of "solid". **Note:** The TCLP ZHE prep uses only TCLP fluid #1; the SPLP ZHE prep uses only DI Water.

10.3.20 Load the fluid transfer reservoir with an excess of Fluid #1 for TCLP or DI Water for SPLP and preflush the transfer line to eliminate air pockets. Be sure the required volume remains.

10.3.21 Using a stainless steel syringe transfer the required volume into the ZHE and close the valve.

10.3.22 Check the ZHE to make sure all the valves are closed and manually rotate the ZHE (end-over-end) 2 or 3 times. Reposition the ZHE in the vertical position.

10.3.23 Pressurize the ZHE to 5-10 PSI. Allow to stand for 10 minutes, and then recheck the pressure. If the ZHE appears to be leaking, follow the corrective action protocols recommended by the manufacturer and repeat the test.

10.3.24 Slowly open the liquid inlet/outlet valve to bleed out any headspace that may have been introduced during the introduction of the Fluid. Upon the first sign of liquid from the valve, close the valve.

10.3.25 Repressurize the ZHE to 5-10 PSI and place in the rotary agitator. Rotate at 28-32 rpm for 16-20 hours. Room temperature should be 23 ± 2 ℃. The room temperature and time should be checked at both the start and end of the extraction and recorded on the ZHE worksheet.

10.3.26 Confirm that the pressure of 5-10 PSI was maintained throughout the leaching. If it was not maintained, return to Section 10.3.1 and repeat the leachate with a new aliquot of sample. Contact the PM if there is insufficient sample to repeat the leaching procedure.

10.3.27 Attach a syringe or Tedlar bag and open the liquid inlet/outlet valve to collect the aqueous leachate and proceed as outlined in 10.3.19 - 10.3.20. Record the volume/mass of the leachate and any oil phase. Record the date and time the

filtration is completed on the ZHE worksheet. **Note:** If the waste contained an initial liquid phase, the liquid may be filtered directly into the same collection device holding the initial liquid phase of the waste. A separate filtrate collection container must be used if combination would create multiple phases or there is not enough volume left within the filtrate collection container.

10.3.28 If the waste contained an initial filtrate (Section 10.3.18) that is miscible with the solid phase leachate (as determined in Section 10.1.8), the solid phase leachate and the initial filtrate are directly recombined in the correct proportions (see Section 11.1.4). If the individual phases are NOT compatible, they are to be collected, prepped and analyzed separately. **Note:** Chill the filtrate and receiving vessels before recombining.

10.3.29 Following collection, store the TCLP/SPLP leachate in 2 40-mL VOA vials with minimal headspace at 4 ± 2 °C and prepare for analysis as soon as possible using the appropriate organic extraction procedure. Due to space limitations, the rest of the leachate is disposed of.

10.3.30 If the individual phases are analyzed separately, combine the results mathematically by using the recombination calculation in Section 11.1.5.

11. CALCULATIONS/DATA REDUCTION

11.1 Calculation of Percent Wet Solids:

$$Percent\ \ Wet\ \ Solids = 100\left(\frac{Mass,"solid"\ phase}{Mass,initial\ subsample}\right)$$

11.2 Calculation of weight of waste to charge to ZHE:

$$Weight\ of\ \ waste\ to\ charge\ to\ ZHE = 100\left(\frac{25}{\%\ wet\ solids}\right)$$

11.3 Calculation of weight of extraction fluid to use:

$$Weight\ of\ \ Extraction\ \ fluid = \frac{20\ x\ \%\ wet\ solids\ x\ weight\ of\ \ waste\ to\ be\ extracted}{100}$$

11.4 Calculation of volume of initial filtrate phase to recombine with solid phase leachate:

$$\text{Volume of filtrate for recombination} = \left(\frac{Weight\ of\ solids\ leached}{Total\ weight\ of\ solids}\right)\left(\frac{Leachate\ recovered}{Fluid\ added}\right)\left(Volume\ of\ initial\ aqueous\ filtrate\right)$$

11.5 Mathematical recombination of analytical results:

$$Final\ Analyte\ Concentration = \frac{(V_1 \times C_1) + (V_2 \times C_2)}{V_1 + V_2}$$

V_1 = total volume of the initial filtrate phase (L).
C_1 = analyte concentration in initial filtrate phase (mg/L).
V_2 = volume of the theoretical solid phase leachate (L).
C_2 = analyte concentration in solid phase leachate (mg/L).,

11.6 REPORTING REQUIREMENTS

11.6.1 Samples generated with method deviations, such as temperature exceedances, insufficient sample for TCLP/SPLP complaint tumble, etc…, must be reported as **NOT** TCLP/SPLP compliant through an NCM and narrated in the Case Narrative of the final report.

11.6.2 Follow these reporting conventions for multi-phase samples:

11.6.2.1 If both phases have positive results, use the values from each phase to calculate the recombined result. Use the reporting limit for each phase to calculate the recombined reporting limit.

11.6.2.2 If both phases are “ND,” not detected, the recombined result is “ND,” and the reporting limit is calculated from the reporting limit for each phase.

11.6.2.3 If one phase is “ND” and the other phase has a positive result, use the reporting limit for the “ND” phase and the positive value for the other phase to calculate the combined result. The combined reporting limit is based on the reporting limit for both phases. If the combined result is less than the combined reporting limit, then supply a footnote to indicate that “a positive result was detected below the calculated detection limit.”

11.6.3 Units - regardless of the nature of the sample, all TCLP and SPLP results are reported in units of mg/L.

11.6.4 For limits and significant figures, consult the appropriate analytical methods.

11.6.5 Anomalies - all anomalies observed during the leach procedure must be noted on the worksheet or an anomaly form. Some examples of such anomalies are:

11.6.5.1 Sample was monolithic - subsample was obtained by crushing, cutting, grinding, sawing, etc.

11.6.5.2 Insufficient sample - less than the required 100 g minimum was available.

11.6.5.3 If less than the required minimum sample is available, the client will be contacted to determine whether or not to proceed with the extraction. If the client chooses to proceed, an explanation will be included in the Case Narrative.

11.6.5.4 Multiple phases - "X" phases were present.

11.6.5.5 Sample was oil - single phase.

11.6.5.6 Sample contained liquid that did not filter under test conditions.

11.7 REVIEW REQUIREMENTS

11.7.1 Review all applicable holding times. If a holding time was exceeded, confirm that a holding time violation form was properly documented and routed.

11.7.2 If Total analysis results are available, those results may be compared with the TCLP/SPLP analysis results according to the following:

$$Total \geq 20 \times TCLP/SPLP$$

NOTE: Assumes the sample is 100% Solids.

11.7.3 Total constituent analysis results can be used to demonstrate the TCLP/SPLP protocol is unnecessary. In performing a TCLP/SPLP analysis, there is a 20x dilution of the original sample with the leaching solution. Thus, if the "total constituent" result is less than 20 times the TCLP/SPLP action level, it is impossible for the leachate to "fail" and the TCLP/SPLP does not need to be performed. For example, the TC level for lead is 5.0 mg/L (ppm). Therefore, if a sample of lead-contaminated soil contains less than 100 ppm total lead, a TCLP/SPLP test need not be run for lead.

12. METHOD PERFORMANCE

12.1 The supervisor has responsibility to ensure that an analyst who performs this procedure is properly trained in its use and has the required experience. Performance is monitored through internal QC and outside performance evaluation samples. Please refer to the QA Manual (PT-QA-M-001) for additional information concerning Precision and Accuracy.

12.2 Demonstration of Capability – Prior to the independent analysis of client samples, A Demonstration of Capability (DOC), as described in the QA Manual, must be performed. DOCs are repeated annually and any time a significant change is made to the analytical system.

12.3 TCLP/SPLP leaching is not included in MDL studies.

13. POLLUTION PREVENTION

13.1 It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage and reagent stability). Employees must abide by the policies in Section 13 of the Corporate Environmental Health and Safety Manual (CW-E-M-001) for "Waste Management and Pollution Prevention" and the Pittsburgh Facility Addendum EH&S Manual (PT-HS-001).

13.2 This method does not contain any specific modifications that serve to minimize or prevent pollution.

14. WASTE MANAGEMENT

14.1 Waste management practices are conducted consistent with all applicable rules and regulations. Excess reagents, samples and method process wastes are disposed of in an accepted manner. Waste description rules and land disposal restrictions are followed. Waste disposal procedures are incorporated by reference to PT-HS-001. The following waste streams are produced when this method is carried out.

14.1.1 Remaining TCLP/SPLP extracts: Fluid is collected in a Hazardous Water Drum, Waste #24.

14.1.2 Solids: This waste includes soils, filter paper, paper towels, etc. This waste is collected in containers identified as "Lab Trash", Waste #12.

15. REFERENCES

15.1 Method 1311, Toxicity Characteristic Leaching Procedure, Revision 0, July 1992, SW-846 Final Update IV

15.2 Method 1312, Synthetic Precipitation Leaching Procedure, Revision 0, September 1994, SW-846 Final Update IV

15.3 Toxicity Characteristic: Corrections to Final Rule. Method 1311, Federal Register, Vol. 55, No. 126, Friday, June 29, 1990

15.4 Toxicity Characteristic: Final Rule. Method 1311, Federal Register, Vol. 55, No. 61, Thursday, March 29, 1990

15.5 Technical Background Document and Response To Comments, Method 1311, Toxicity Characteristic Leaching Procedure, USEPA/OSW, April, 1989

15.6 PT-QA-021, TestAmerica Pittsburgh QC Program

15.7 PT-QA-024, Subsampling

15.8 PT-QA-031, Internal Chain of Custody

15.9 PT-QA-M-001, Quality Assurance Manual

15.10 PT-HS-001, Pittsburgh Facility Addendum EH&S Manual

16. METHOD MODIFICATIONS

16.1 Section 10.1.1: Preliminary Evaluations. Section 7.1 of source method SW846 1311 states that the sample aliquot used for the preliminary evaluation "...may not actually undergo TCLP extraction." Section 7.1.5 of the source method indicates that the portion used for the preliminary evaluation may be used for either the ZHE or non-volatile extraction if the sample was 100% solid. Section 7.1.5 further indicates that if the sample was subjected to filtration (i.e., < 100% solid) that this aliquot may be used for the non-volatile extraction procedure only as long as sufficient sample is available (minimum 100 g). Samples which have been subjected to the ovendrying step may not be used for TCLP extraction because solid phase degradation may result upon heating.

16.2 Section 10.1.5.6: Percent Solids Determination. Section 7.1.2 of source method SW846 1311 indicates that "if the percent wet solids is ≥ 0.5% and it is noticed that a small amount of the filtrate is entrained in wetting of the filter" that the filter should be oven dried to determine percent dry solids ". Drying of oil or organic matrices can both

be hazardous and inappropriate. Additionally, it may be impossible to achieve a constant weight when performing this step. Due to safety concerns, if obviously oily or heavy organic matrices are entrained on the filter, the filter is not oven dried.

16.3 Section 10.1.8: Preliminary Determination of Filtrate/Extraction Fluid Compatibility. Section 7.2.13 of the source method SW846 1311 provides no guidance as to how to make this determination. As a result, the procedure herein was developed and incorporated into the Preliminary Determinations section.

16.4 Section 9.2: TCLP/SPLP Extraction Blanks. Section 8.1 of the source methods state that a minimum of one blank for every 20 extractions "...that have been conducted in an extraction vessel." TestAmerica has interpreted this to mean one blank per twenty samples leached per TYPE of leaching vessel (i.e., Bottle or ZHE) per leach fluid used. Only ZHEs are tracked, as bottles used for other tumbling are not re-used.

16.5 Section 10.1.7.10: Determination of Appropriate Extraction Fluid. SW846 Method 1311 does not address the appropriate approach to take if the pH equals 5.0. This SOP requires that Fluid #1 must be used if the pH is less than or equal to 5.0.

16.6 Section 9.3: QA/QC - Matrix Spikes. Section 8.2 of the source method SW846 1311 states "A matrix spike shall be performed for each waste type..." and "A minimum of one matrix spike must be analyzed for each analytical batch." Further, Section 8.2.3 of the source method also states "The purpose of the matrix spike is to monitor the performance of the analytical methods used, and to determine whether matrix interferences exist." The standard TestAmerica QAPP is designed to address the performance monitoring of analytical methodology through the LCS program. A minimum of one MS and MSD will be prepared for each TCLP/SPLP leachate batch. The MS/MSD results are used to determine the effect of a matrix on the precision and accuracy of the analytical process. Due to the potential variability of the matrix of each sample, the MS/MSD results have immediate bearing only on the specific sample spiked and not all samples in the batch.

16.6.1 Section 8.2.2 of the source method states that "In most cases, matrix spikes should be added at a concentration equivalent to the corresponding regulatory level." The method also states "If the analyte concentration is less than one half the regulatory level, the spike concentration may be as low as one half of the analyte concentration but may not be less than five times the method detection limit". For several analytes, spiking at the regulatory level is inappropriate to the range of analysis afforded by the determinative methods. Due to the wide range in these levels, TestAmerica spikes at the levels specified in the determinative SOPs.

17. ATTACHMENTS

17.1 APPENDIX A – Tables

17.2 APPENDIX B – Figures

17.3 APPENDIX C – Example Worksheets/Logbooks

17.4 APPENDIX D - Flowcharts

18. REVISION HISTORY

18.1 Revision 4, 1/31/2009

18.2 Revision 5, 5/8/2012

18.3 Revision 6, 6/09/2013

18.4 Revision 7, 6/30/2015

18.5 Revision 8, 9/27/2017

18.6 Revision 9, 10/29/2018

18.7 Revision 9.1, 4/29/2019

18.8 Changes to current revision

SOP section	Change from	Change to	Reason
7.10.4, 10.3.19 and 10.3.20	SPLP Fluid #3	DI Water	Clarificationt
7.12		Added metals spiking solutions	Clarification
7.13		Added conc. Nitric acid for preservation	Clarification
9.3.1		Added details for spiking metals MS/MSD	Clarification
10.2.12		Added details for metals preservation	Clarification
Table 4		Added for metals spike concentrations	Clarification

APPENDIX A

TABLES

Table 3 - Toxicity Characteristic Analytes and Regulatory Levels (Final Rule)

Contaminant	mg/L
Arsenic	5.0
Barium	100.0
Benzene	0.5
Cadmium	1.0
Carbon tetrachloride	0.5
Chlordane	0.03
Chlorobenzene	100.0
Chloroform	6.0
Chromium	5.0
o-Cresols	200.0
m-Cresols	200.0
p-Cresols	200.0
Total Cresols (used if isomers not resolved)	200.0
2,4-D	10.0
1,4-Dichlorobenzene	7.5
1,2-Dichloroethane	0.5
2,4-Dinitrotoluene	0.13
1,1-Dichloroethylene	0.7
Endrin	0.02
Heptachlor (& epoxide)	0.008
Hexachlorobenzene	0.13
Hexachlorobutadiene	0.5
Hexachloroethane	3.0
Lead	5.0
Lindane	0.4
Mercury	0.2
Methoxychlor	10.0
Methyl ethyl ketone	200.0
Nitrobenzene	2.0
Pentachlorophenol	100.0
Pyridine	5.0
Selenium	1.0
Silver	5.0
Tetrachloroethylene	0.7
Toxaphene	0.5
Trichloroethylene	0.5
2,4,5-Trichlorophenol	400.0
2,4,6-Trichlorophenol	2.0
2,4,5-TP (Silvex)	1.0
Vinyl chloride	0.2

Table 4 – Metals Spikes concentrations (mg/L)

Element	LPTCLP Spike	TA-Spike 1	TA-Spike 2	TA-Spike 3 INT
Al	--	--	1000	--
Ag	100	--	--	50
As	500	200	--	--
B	--	--	--	250
Ba	500	200	--	--
Be	--	100	--	--
Ca	--	--	5000	--
Cd	100	100	--	--
Co	--	100	--	--
Cr	500	100	--	--
Cu	--	100	--	--
Fe	--	--	1000	--
K	--	--	5000	--
Li	--	100	--	--
Mg	--	--	5000	--
Mn	--	100	--	--
Mo	--	100	--	--
Na	--	--	5000	--
Ni	--	100	--	--
Pb	500	100	--	--
Sb	--	--	--	50
Se	100	200	--	--
Si	--	200	--	--
Sn	--	200	--	--
Sr	--	100	--	--
Ti	--	100	--	--
Tl	--	200	--	--
V	--	100	--	--
Zn	--	--	--	50

APPENDIX B

FIGURES

Figures 1 & 2 - Rotary Agitation Apparatus and Zero Headspace Extraction Vessel (ZHE)

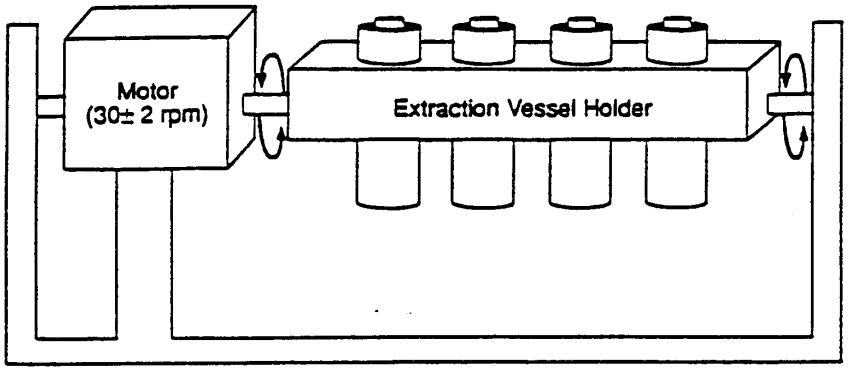


Figure 1. Rotary Agitation Apparatus

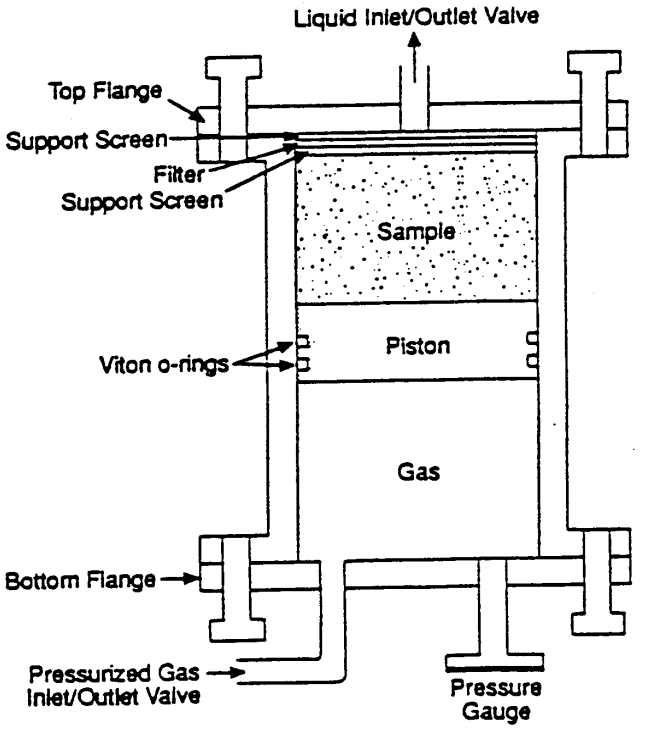


Figure 2. ZHE Vessel

Figure 3 - US Environmental Protection Agency Memorandum #35, Page 1



UNITED STATES ENVIRONMENTAL PROTECTION AGENCY
WASHINGTON, D.C. 20460

OFFICE OF
SOLID WASTE AND EMERGENCY RESPONSE

MEMORANDUM # 35

DATE: June 12, 1992

SUBJECT: Notes on RCRA Methods and QA Activities

From: Gail Hansen, Chief
Methods Section (OS-331)

This memo addresses the following topics:

- o 1992 Symposium on Waste Testing and Quality Assurance
- o SW-846 Update
  - Final Rule for January 23, 1989 Proposed Rule
  - Notice, Proposed Rulemaking for the Second Update to the Third Edition
- o Chlorofluorocarbon 113 (CFC-113) Solvent Replacement Update
- o Environmental Monitoring Methods Index (EMMI)
- o Sampling Work Group Formation
- o MICE Update
- o Oily Waste Analysis
- o Electronic SW-846 Availability.

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Figure 3 - US Environmental Protection Agency Memorandum #35, Page 10

Oily Waste Analysis

One of the most frequently asked questions on the MICE Service concerns the application of the TCLP, Method 1311, to oily wastes. Many callers request technical guidance on the extraction of oily wastes due to the difficulty in the filtration on these types of waste. In many cases, an oily waste does not filter completely due to premature clogging of the glass fiber filter. This can result in the retention of standing liquid on the glass fiber filter. Material that do not pass through the glass fiber filter at the conclusion of the filtration step is defined by the method as the solid phase of the waste. The solid phase is then subjected to the leaching procedure of the TCLP. For oily wastes, clogging of the glass fiber filter can result in an overestimation of the amount of solid material available for leaching.

To solve this problem, the Agency recommends a conservative approach, one that probably will overestimate the amount of leaching. Rather than performing the TCLP extraction on the unfiltered portion of the oily waste, assume the waste is 100% liquid (e.g., will pass through the glass fiber filter) and perform a totals analysis on the oily waste to determine if the oil exceeds the appropriate regulatory level.

Filterable waste oil generated during the TCLP must be analyzed for a variety of organic and inorganic analytes. The OSW recognizes the difficulty in achieving acceptable performance for the analysis of waste oil using methods currently provided in SW-846. As a result, the Agency will provide several new methods for the preparation and analysis of oil samples to the Organic Methods Workgroup in July. In addition, a microwave assisted digestion procedure should improve the analysis of metals and will be proposed as part of the Second Update of the Third Edition of SW-846. Brief descriptions of these techniques are provided below, for additional information on the organic procedures contact Barry Lesnik at (202) 260-7459. For additional information on microwave digestion contact Ollie Fordham (202) 260-4778.

The use of purge-and-trap (Method 5030) for volatiles in oil generally results in severe contamination of analytical instrumentation. Traps, transfer lines and chromatography columns may become contaminated with oil. This leads to elevated baselines, hydrocarbon background in subsequent analyses, and cross-contamination. Headspace (Method 3810) is currently allowed only as a screening procedure in SW-846. The Agency is evaluating the use of headspace in conjunction with isotope dilution mass spectrometry for the quantitative analysis of volatiles in oil. Headspace reduces interference problems encountered with purge-and-trap. However, headspace quantitation can be questionable because the distribution of analytes is not

10

APPENDIX C

EXAMPLE WORKSHEETS/LOGBOOKS

Example – ZHE Blank Vessel Tracking Logbook

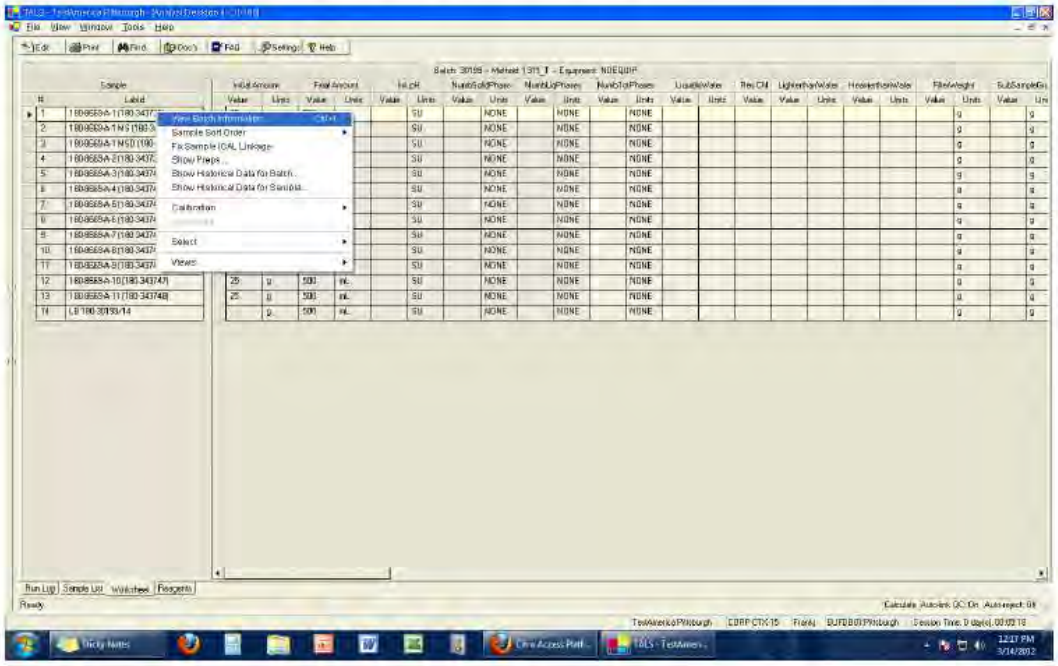
TestAmerica Pittsburgh Year: _____
Work Instruction #: PT-LP-W-002 R1
TCLP: Zero Headspace (ZHE) Extraction Blank Vessel Tracking Logbook
Circled extraction date number indicates that the vessel was used for a blank

ZHE No.	No. of Extractions																			
	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17	18	19	20
Prep Analyst																				
A2																				
A7																				
B2																				
B7																				
B10																				
B101																				
BC																				
BE																				
BI																				
BV																				
C2																				
CF																				
D34																				
DG																				

Page 1 of 2 PT-LP-W-002 R1

TALS LIMS Screenshots - TCLP

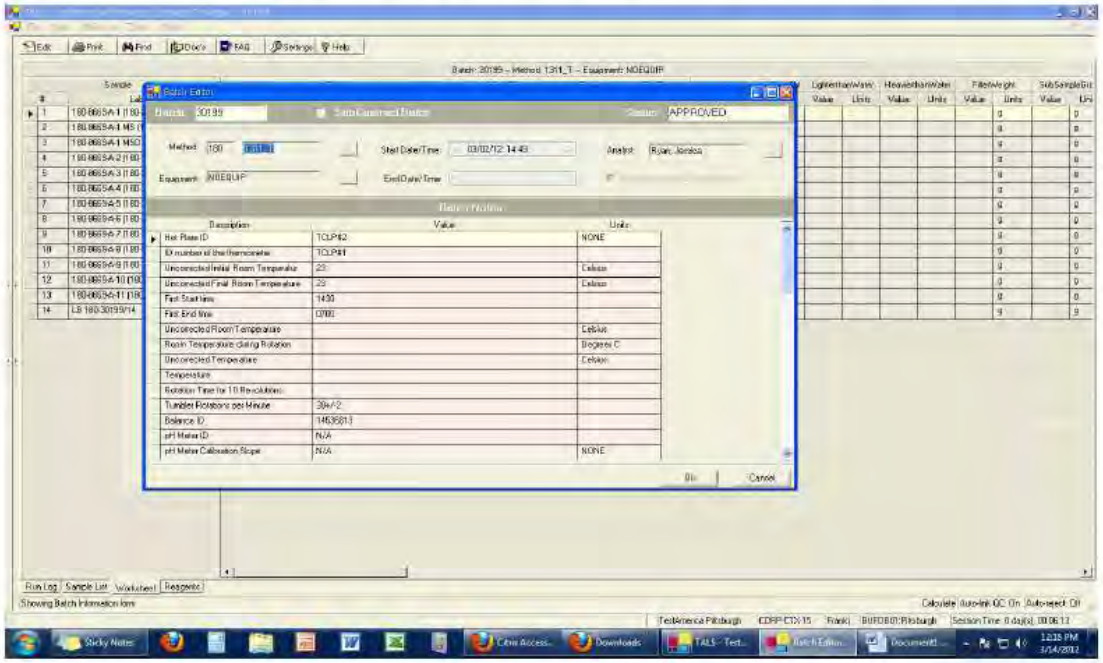
TCLP Method 1311: Batch Info and Worksheet



To View/Edit Batch info once in a batch, right-click and select View Batch Info.

TALS LIMS Screenshots – TCLP (cont.)

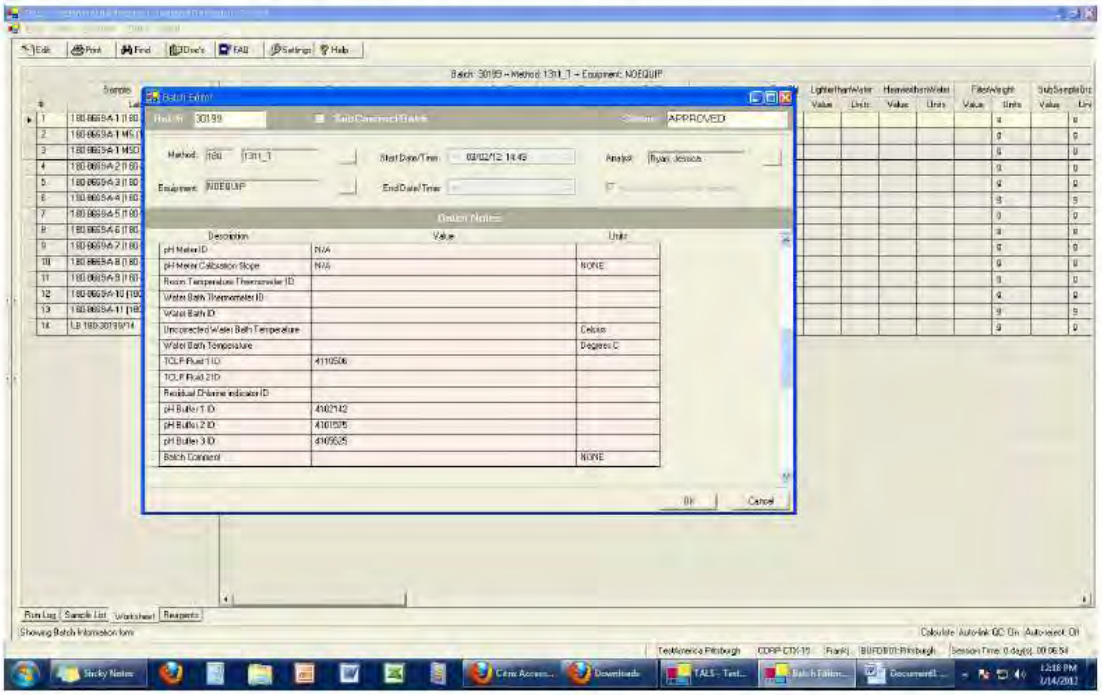
TCLP Method 1311: Batch Info and Worksheet



This is the Batch Info window. (Only ½ of the Batch Info in this screenshot)

TALS LIMS Screenshots – TCLP (cont.)

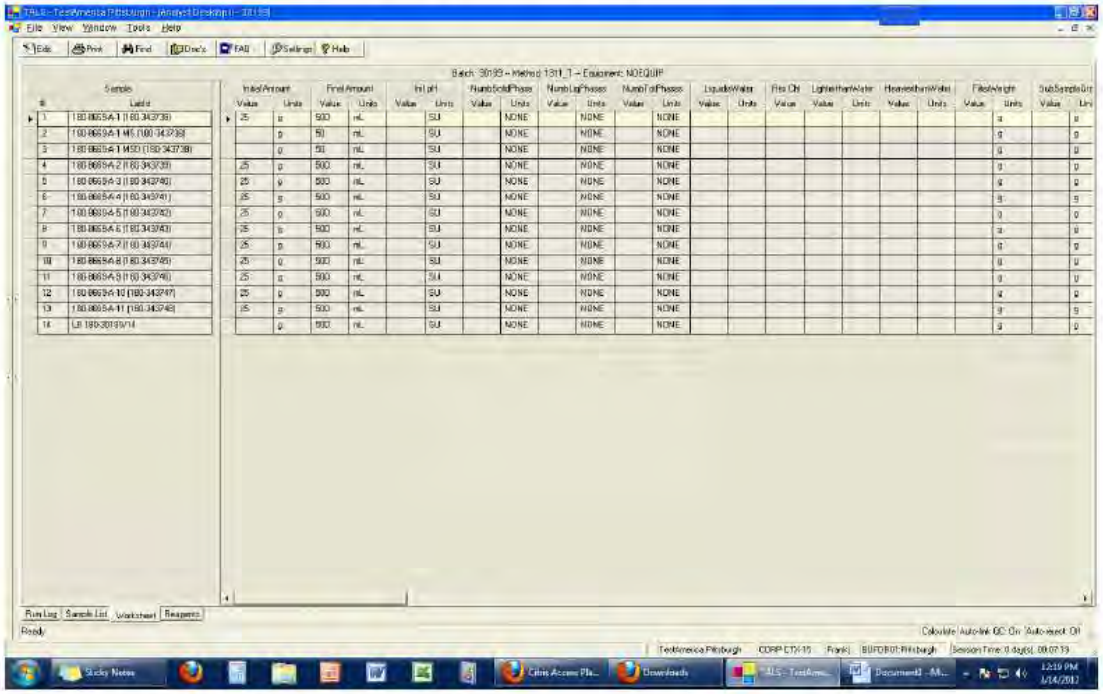
TCLP Method 1311: Batch Info and Worksheet



This is the rest of the Batch Info screen

TALS LIMS Screenshots – TCLP (cont.)

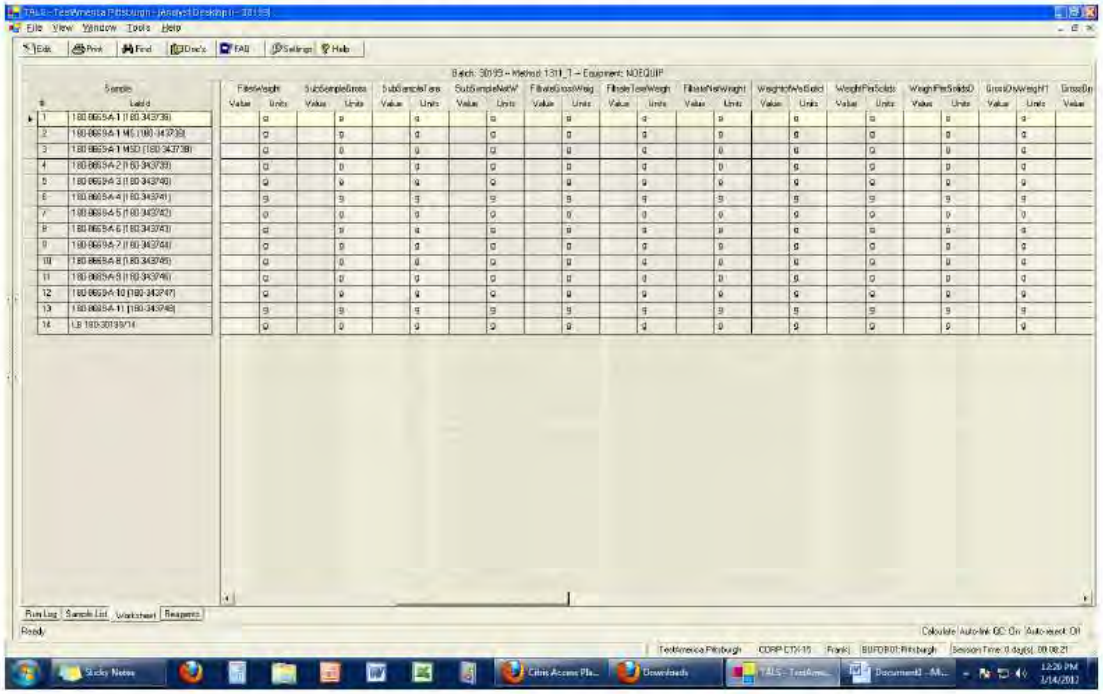
TCLP Method 1311: Batch Info and Worksheet



This is screenshot 1 of 5 of the Worksheet for Method 1311.

TALS LIMS Screenshots – TCLP (cont.)

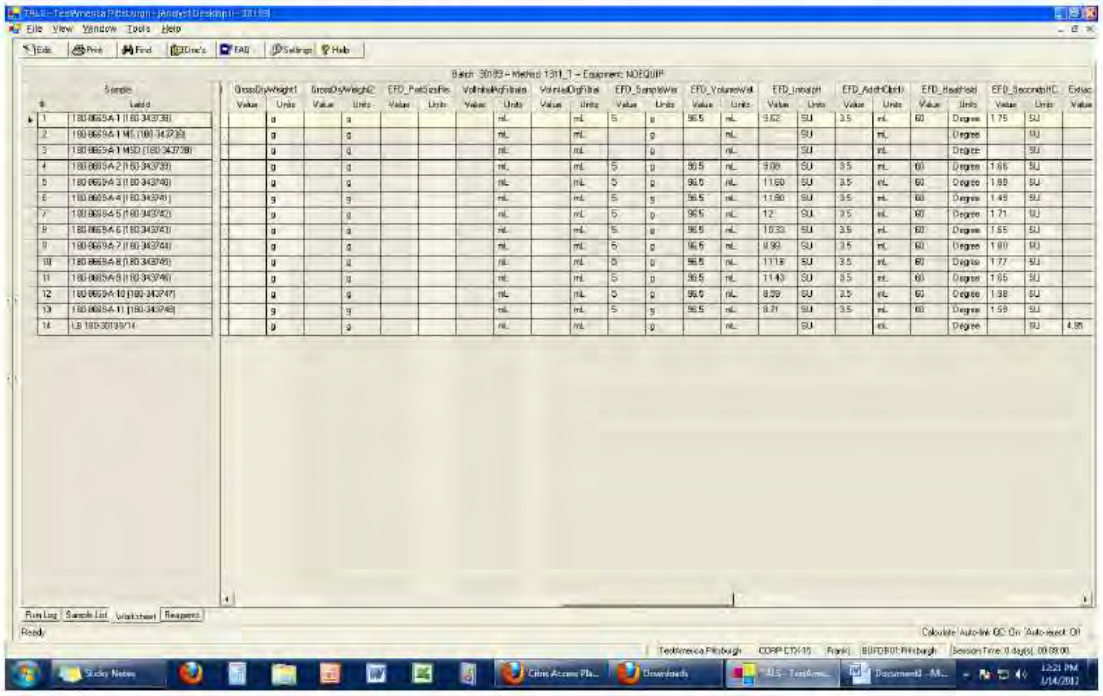
TCLP Method 1311: Batch Info and Worksheet



Screenshot 2 of 5

TALS LIMS Screenshots – TCLP (cont.)

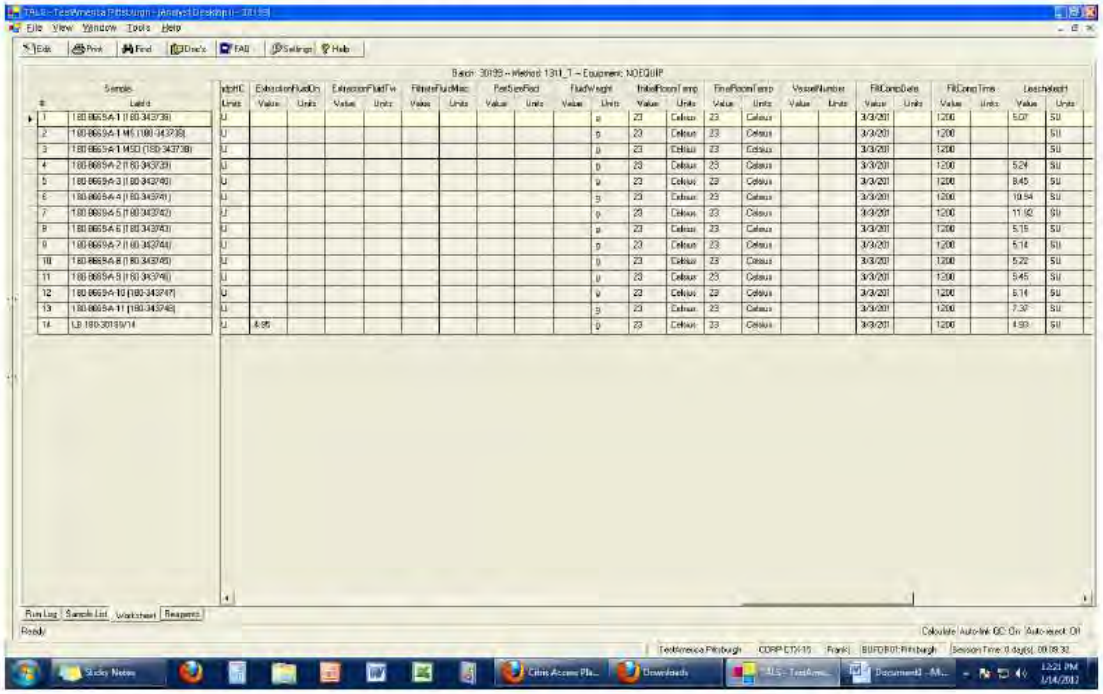
TCLP Method 1311: Batch Info and Worksheet



Screenshot 3 of 5

TALS LIMS Screenshots – TCLP (cont.)

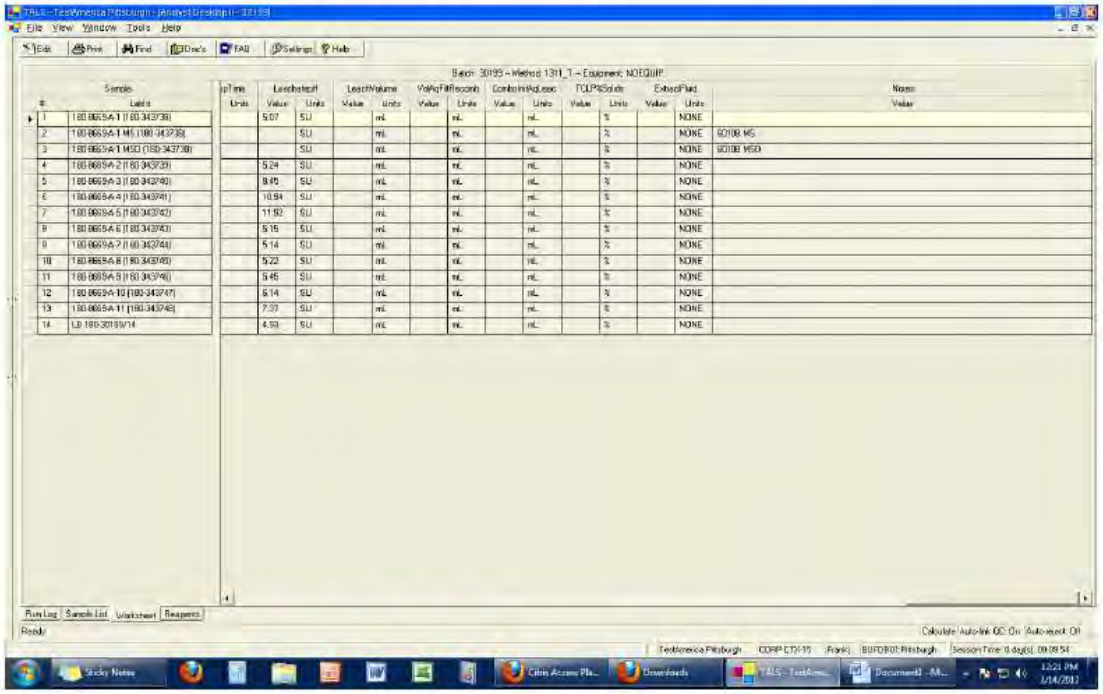
TCLP Method 1311: Batch Info and Worksheet



Screenshot 4 of 5

TALS LIMS Screenshots – TCLP (cont.)

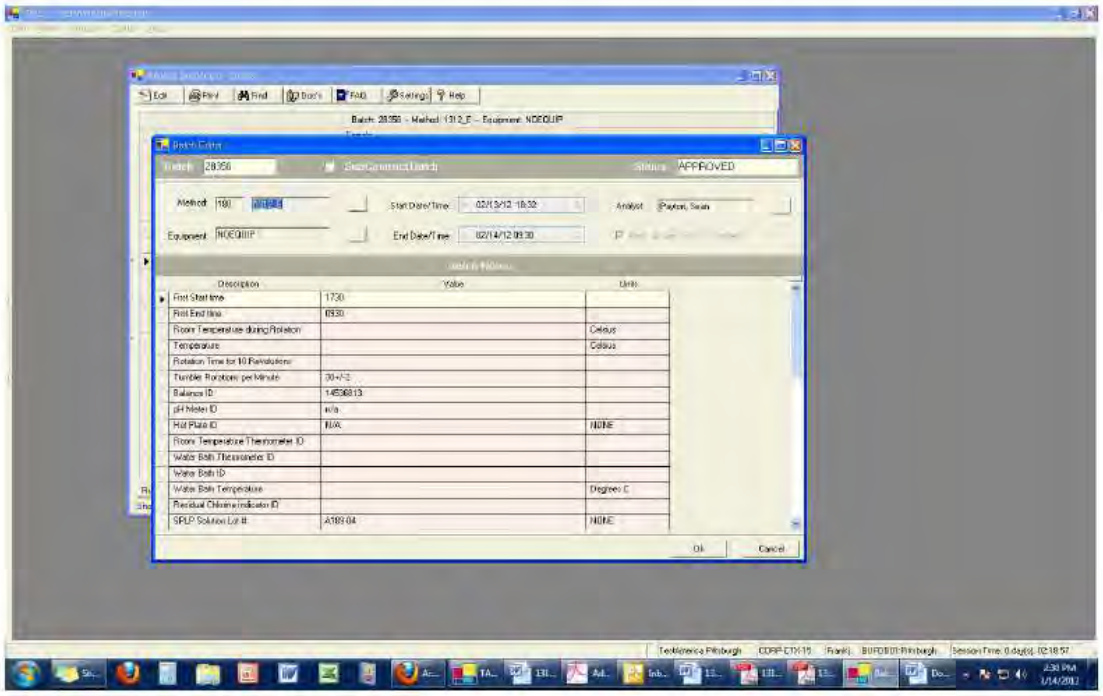
TCLP Method 1311: Batch Info and Worksheet



Screenshot 5 of 5

TALS LIMS Screenshots – SPLP

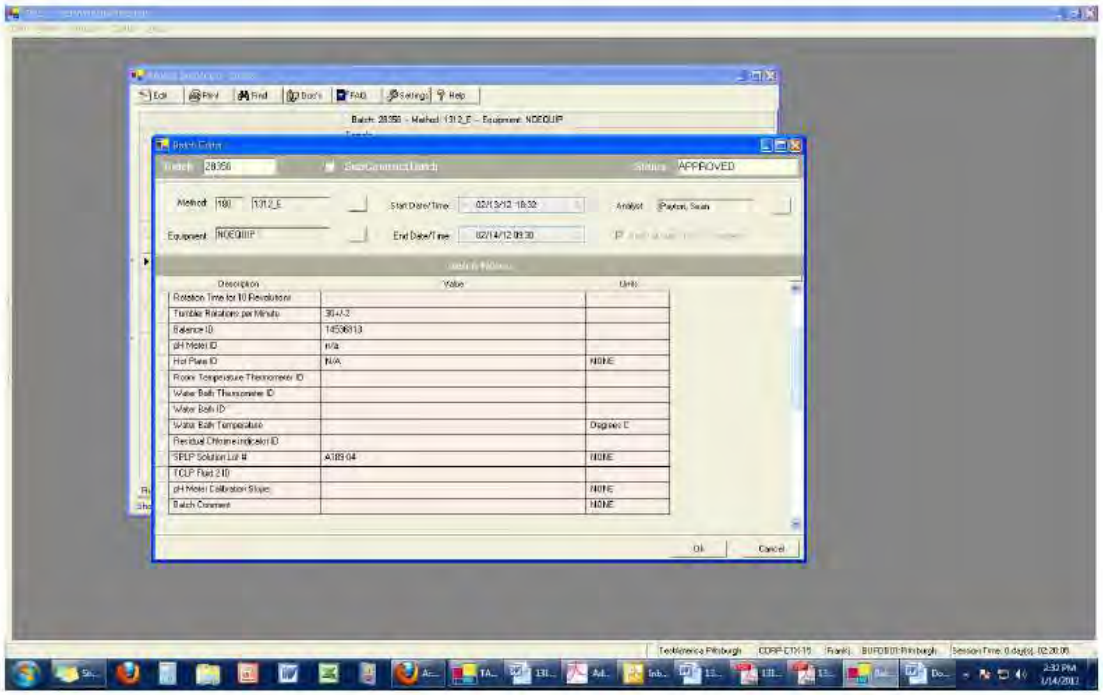
Method 1312 SPLP: Batch Info and Worksheet



1312 Method: SPLP Batch Info (Screenshot 1 of 2)

TALS LIMS Screenshots – SPLP (cont.)

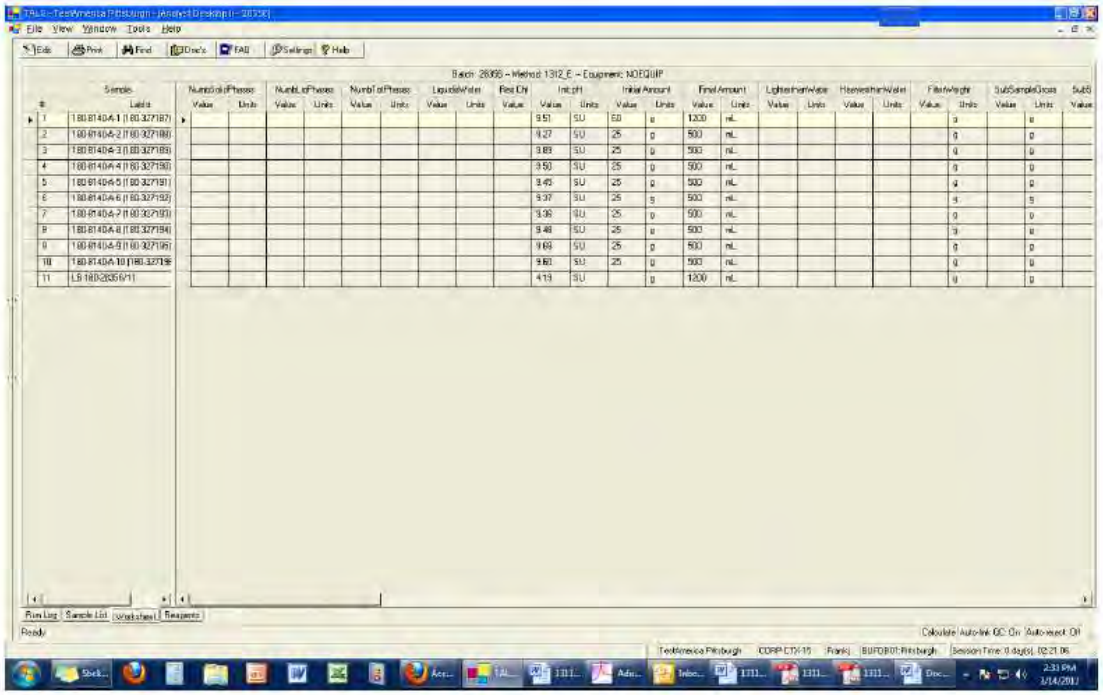
Method 1312 SPLP: Batch Info and Worksheet



1312 Method: SPLP Batch Info (Screenshot 2 of 2)

TALS LIMS Screenshots – SPLP (cont.)

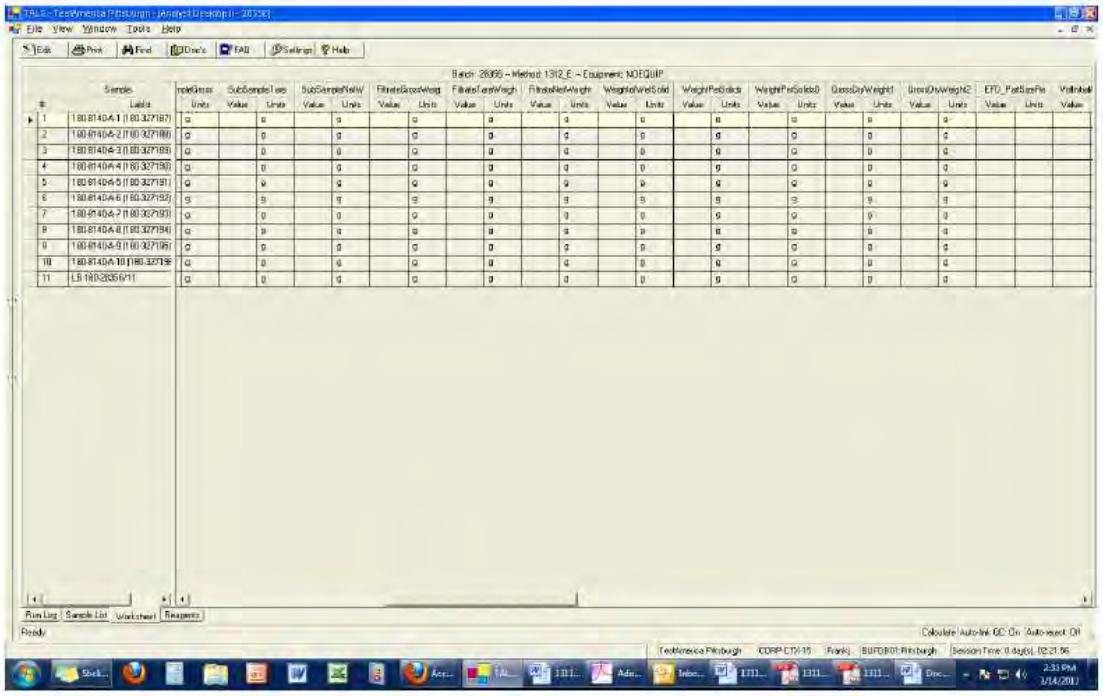
Method 1312 SPLP: Batch Info and Worksheet



SPLP Worksheet Screenshot 1 of 5

TALS LIMS Screenshots – SPLP (cont.)

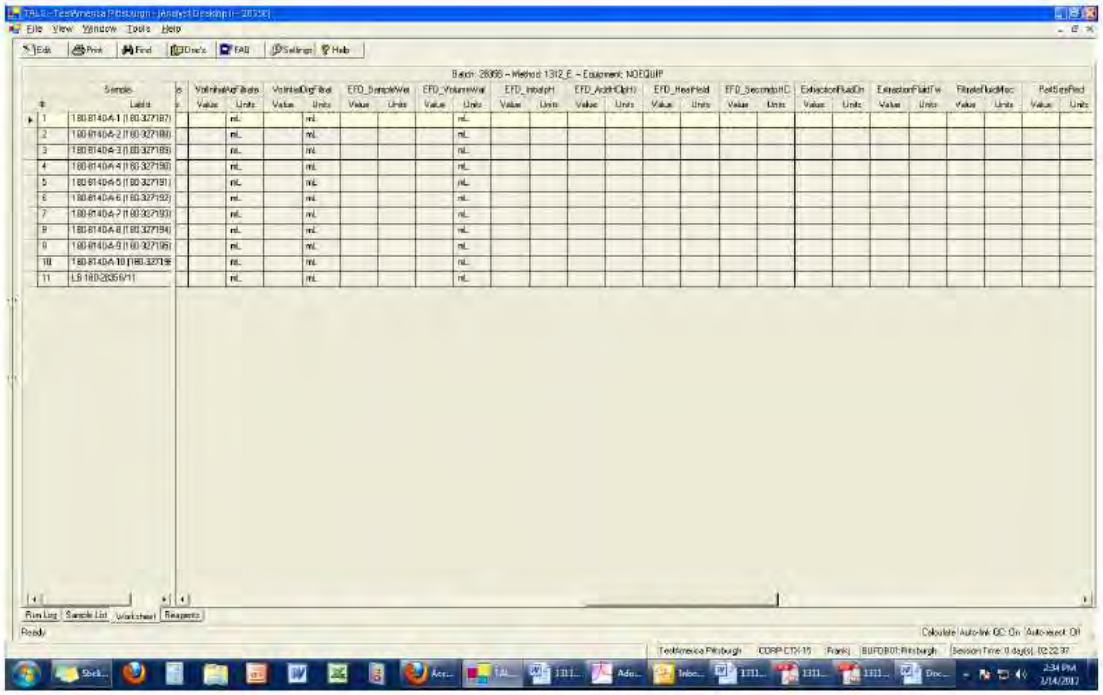
Method 1312 SPLP: Batch Info and Worksheet



SPLP Worksheet Screenshot 2 of 5

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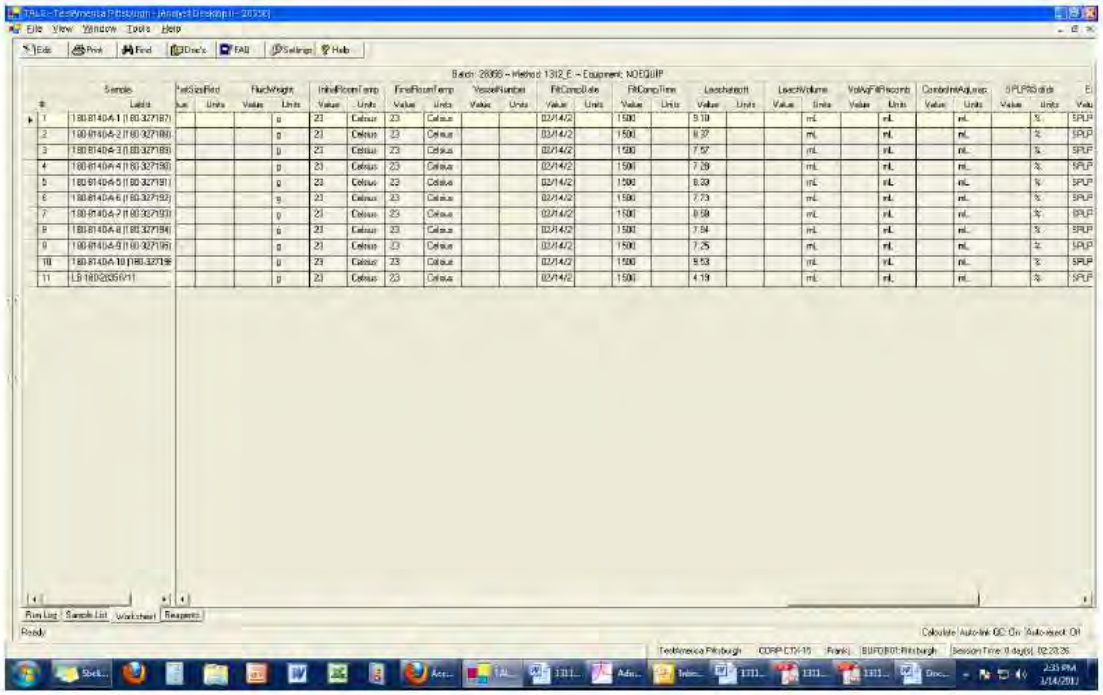
Method 1312 SPLP: Batch Info and Worksheet



SPLP Worksheet Screenshot 3 of 5

TALS LIMS Screenshots – SPLP (cont.)

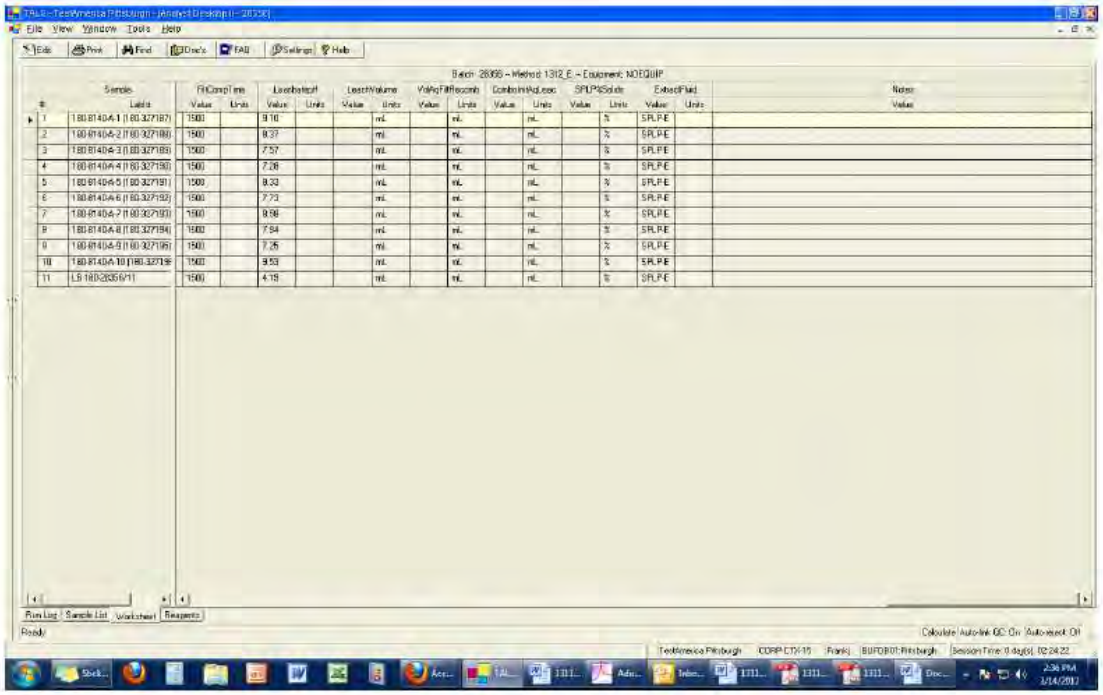
Method 1312 SPLP: Batch Info and Worksheet



SPLP Worksheet Screenshot 4 of 5

TALS LIMS Screenshots – SPLP (cont.)

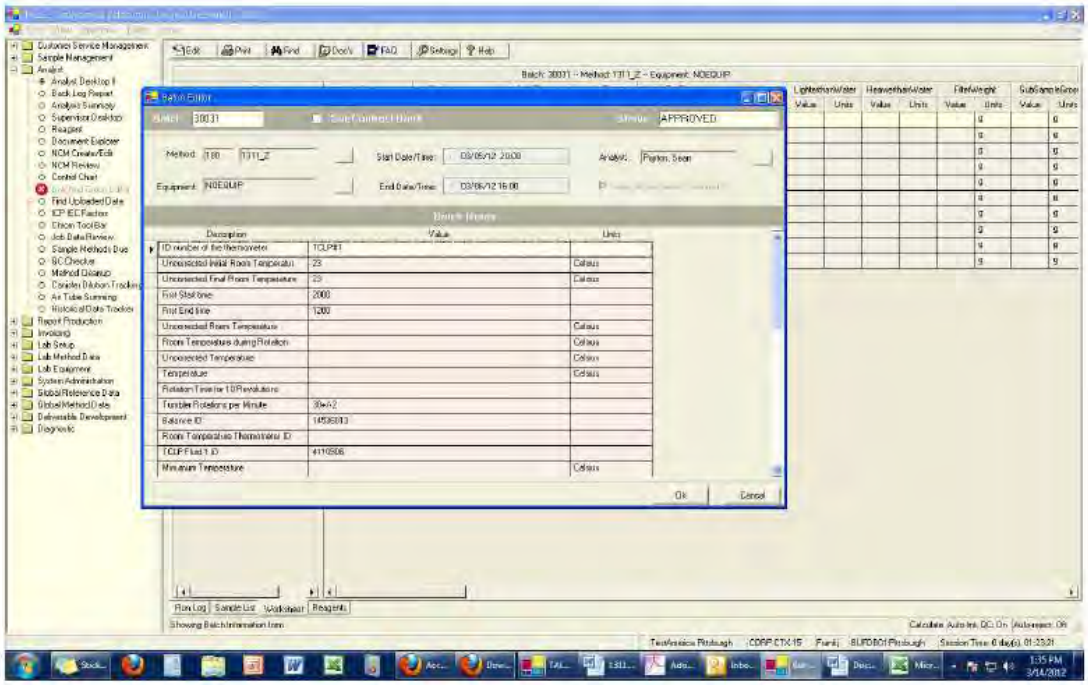
Method 1312 SPLP: Batch Info and Worksheet



SPLP Worksheet Screenshot 5 of 5

TALS LIMS Screenshots – ZHE

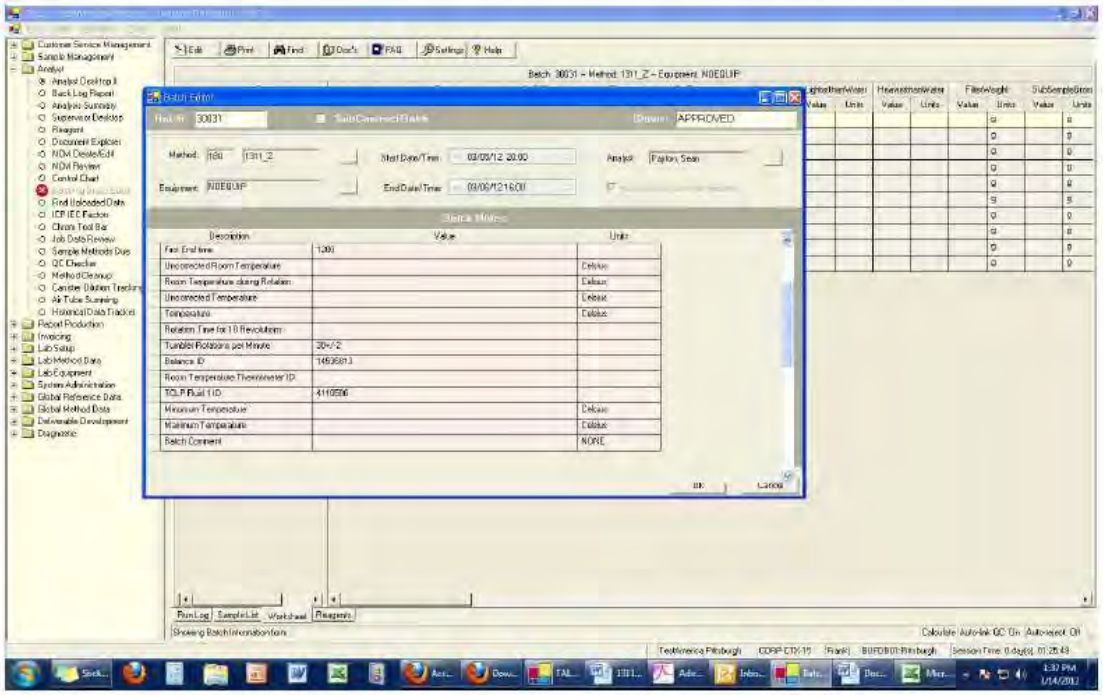
TCLP Method 1311 ZHE: Batch Info and Worksheet



1311 Method: ZHE Batch Info (Screenshot 1 of 2)

TALS LIMS Screenshots – ZHE

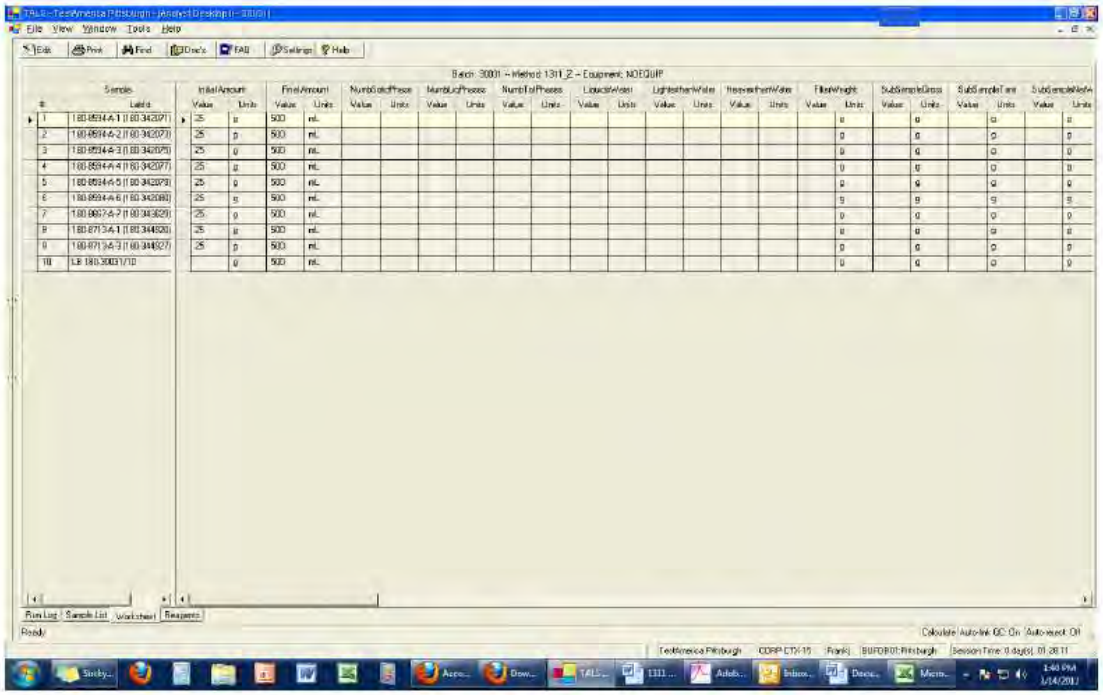
TCLP Method 1311 ZHE: Batch Info and Worksheet



1311 Method: ZHE Batch Info (Screenshot 2 of 2)

TALS LIMS Screenshots – ZHE

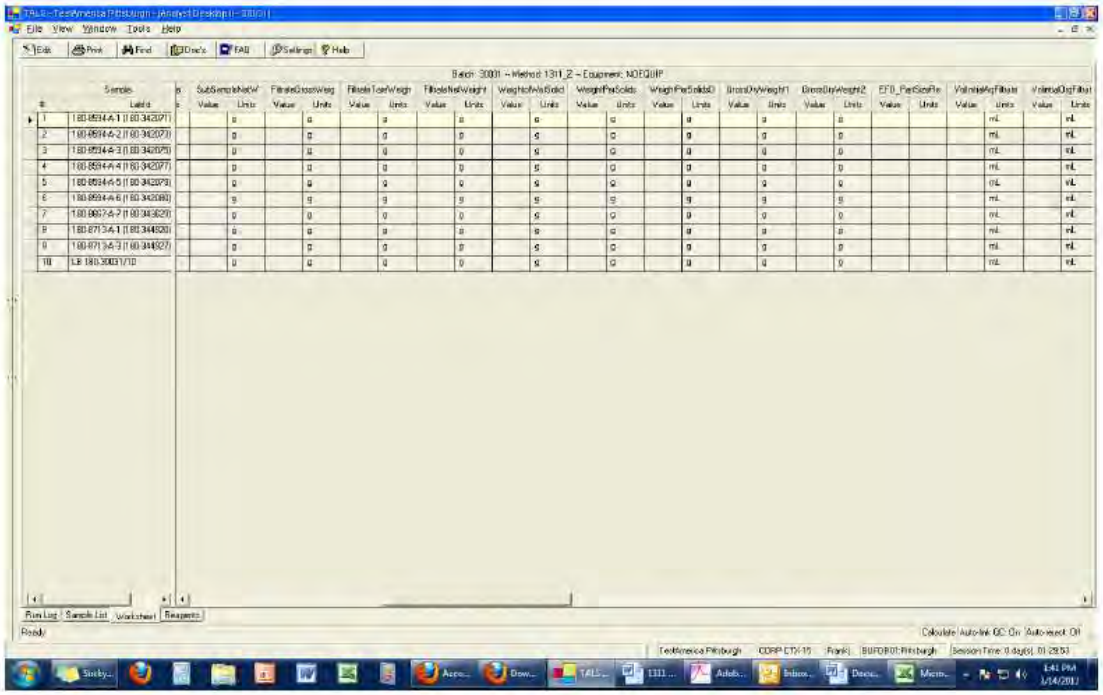
TCLP Method 1311 ZHE: Batch Info and Worksheet



ZHE Worksheet Screenshot 1 of 5

TALS LIMS Screenshots – ZHE

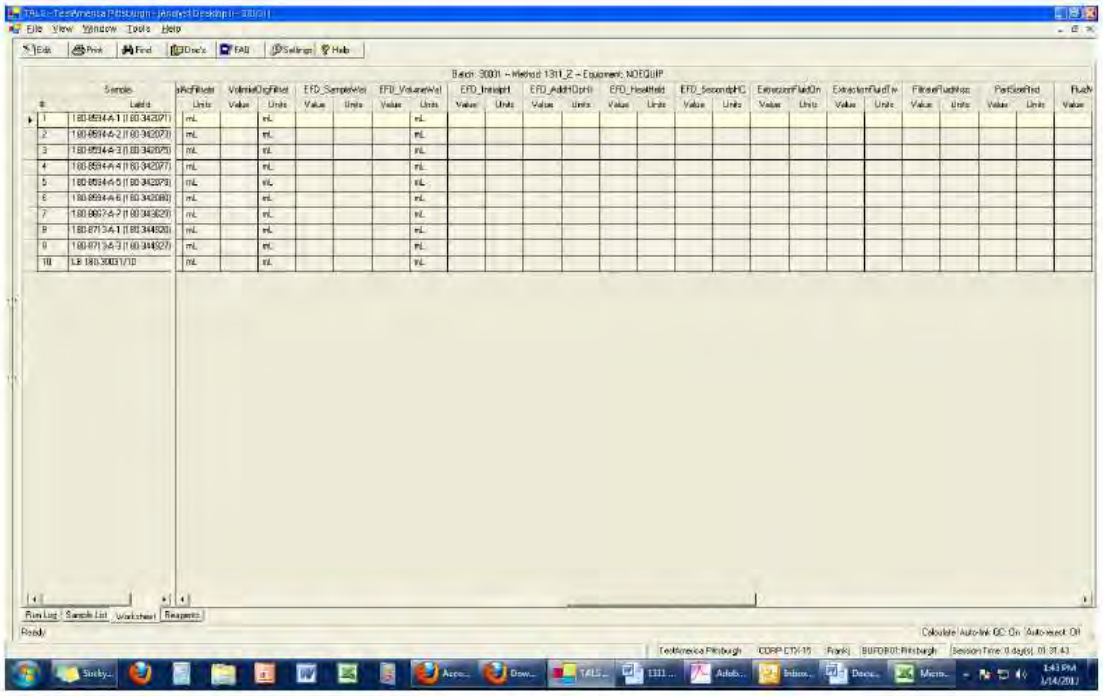
TCLP Method 1311 ZHE: Batch Info and Worksheet



ZHE Worksheet Screenshot 2 of 5

TALS LIMS Screenshots – ZHE

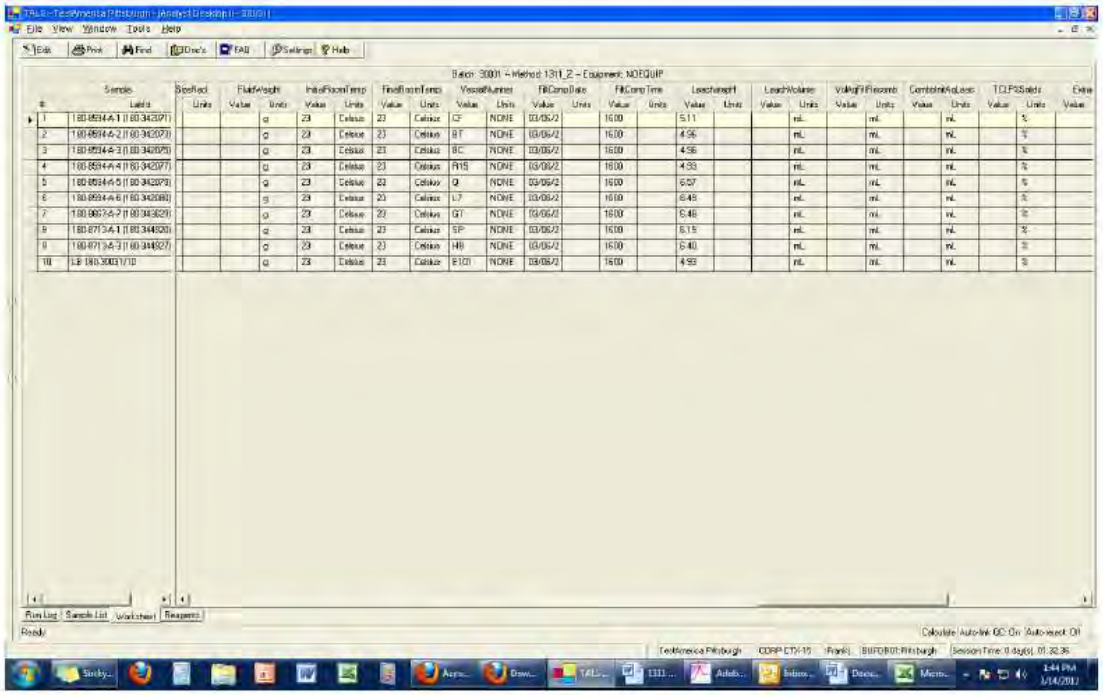
TCLP Method 1311 ZHE: Batch Info and Worksheet



ZHE Worksheet Screenshot 3 of 5

TALS LIMS Screenshots – ZHE

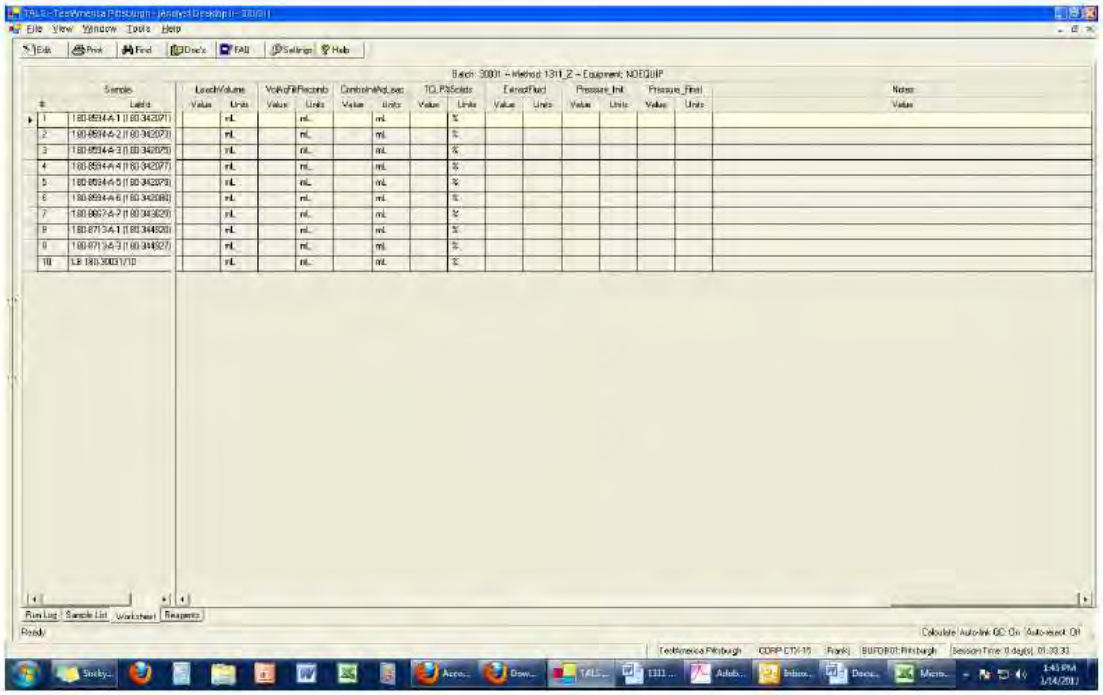
TCLP Method 1311 ZHE: Batch Info and Worksheet



ZHE Worksheet Screenshot 4 of 5

TALS LIMS Screenshots – ZHE

TCLP Method 1311 ZHE: Batch Info and Worksheet



ZHE Worksheet Screenshot 5 of 5

TestAmerica Pittsburgh ***EXTRACTIONS*** LOGBOOK ID: OP2914

TCLP VESSEL MAINTENANCE LOG

Date	Analyst	Vessel #	Maintenance Performed

INITIAL and DATE every entry

printed on: 23-Sep-08 9:09:26 AM

TestAmerica Pittsburgh ***EXTRACTIONS*** LOGBOOK ID: OP2916

ZHE MAINTENANCE LOG

Date	Analyst	Vessel #	Maintenance Performed

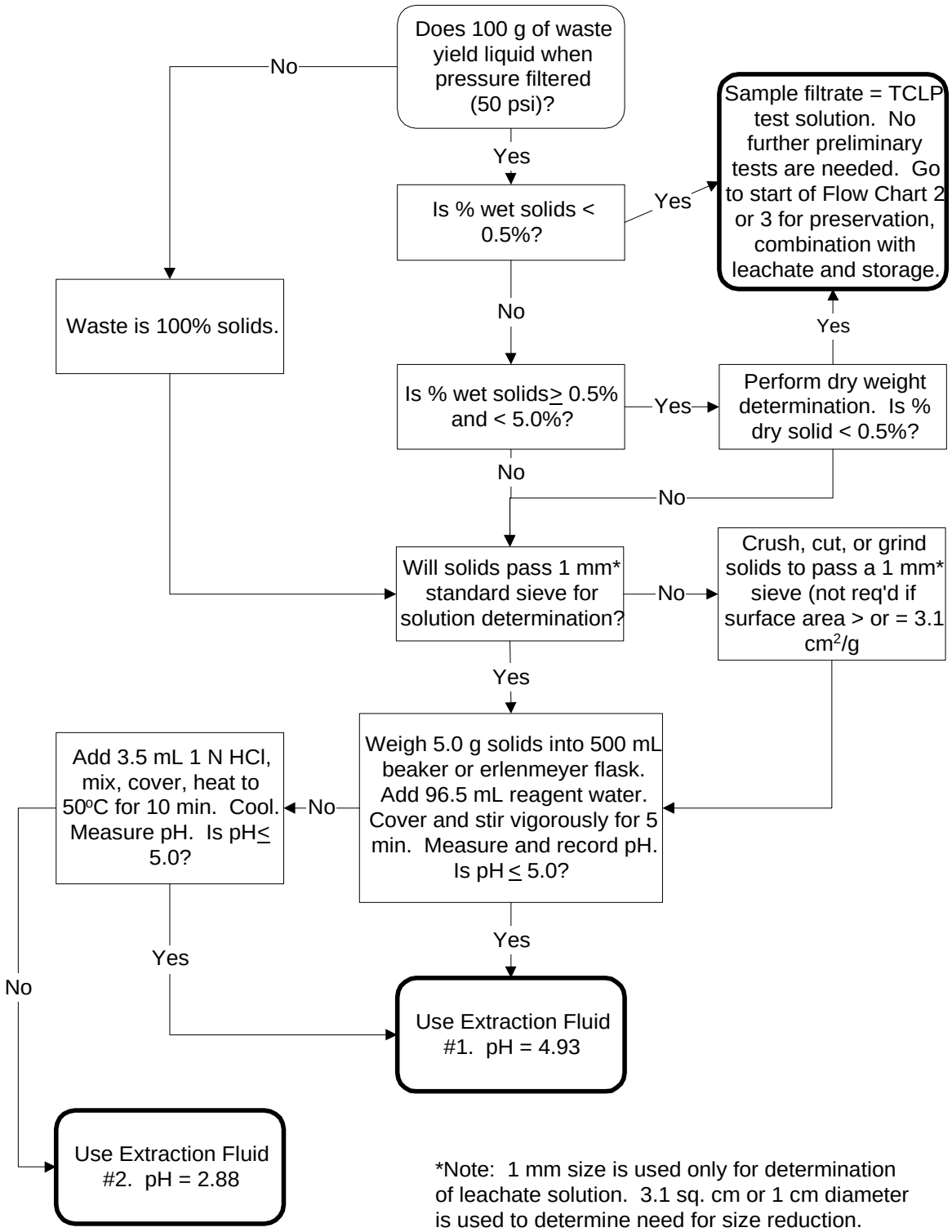
INITIAL and DATE every entry

printed on: 23-Sep-08 9:11:00 AM Page 1 of 1

APPENDIX D

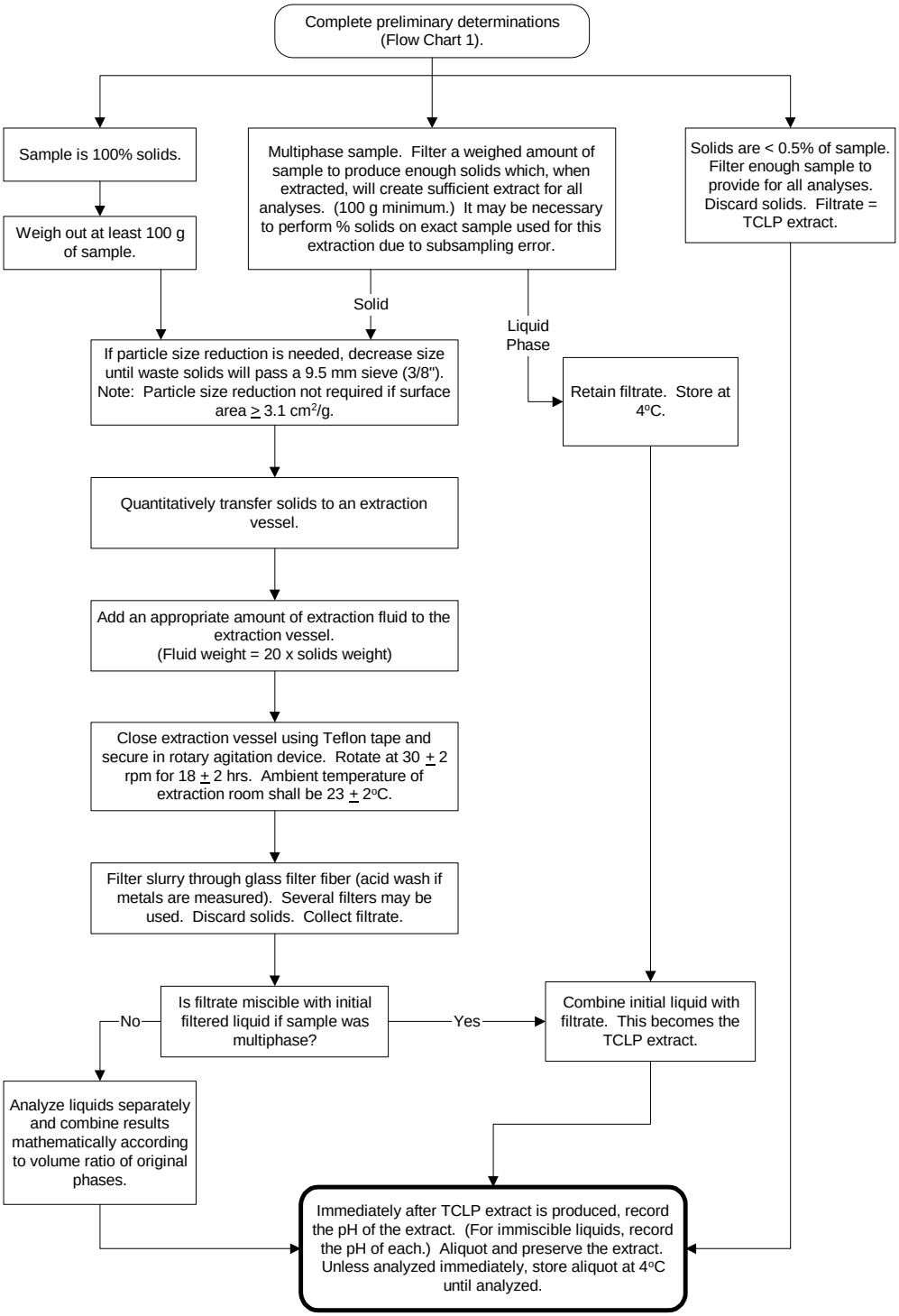
FLOW CHARTS

Flow Chart 1 - Preliminary Sample Evaluation (Section 10.2)

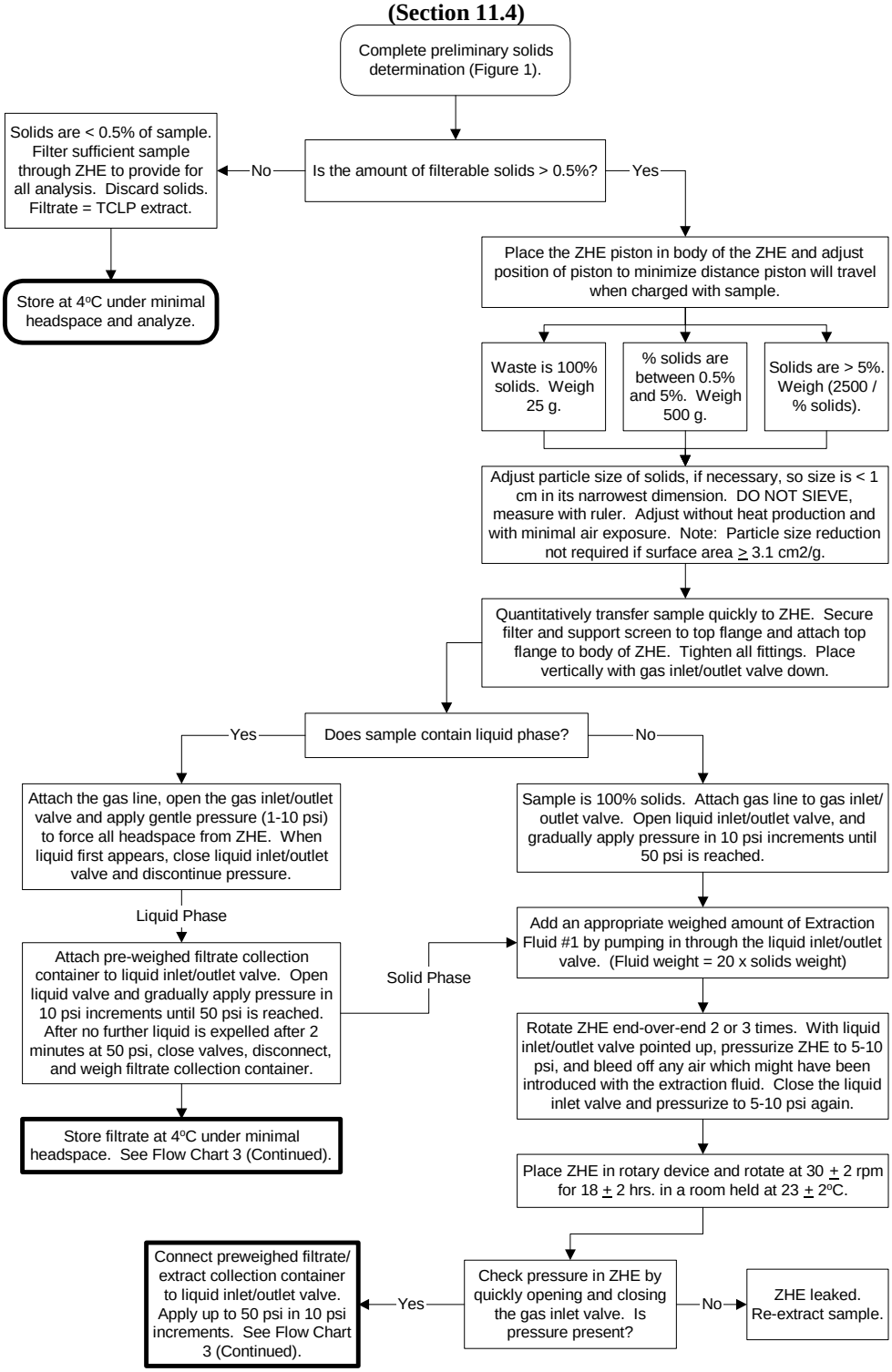


*Note: 1 mm size is used only for determination of leachate solution. 3.1 sq. cm or 1 cm diameter is used to determine need for size reduction.

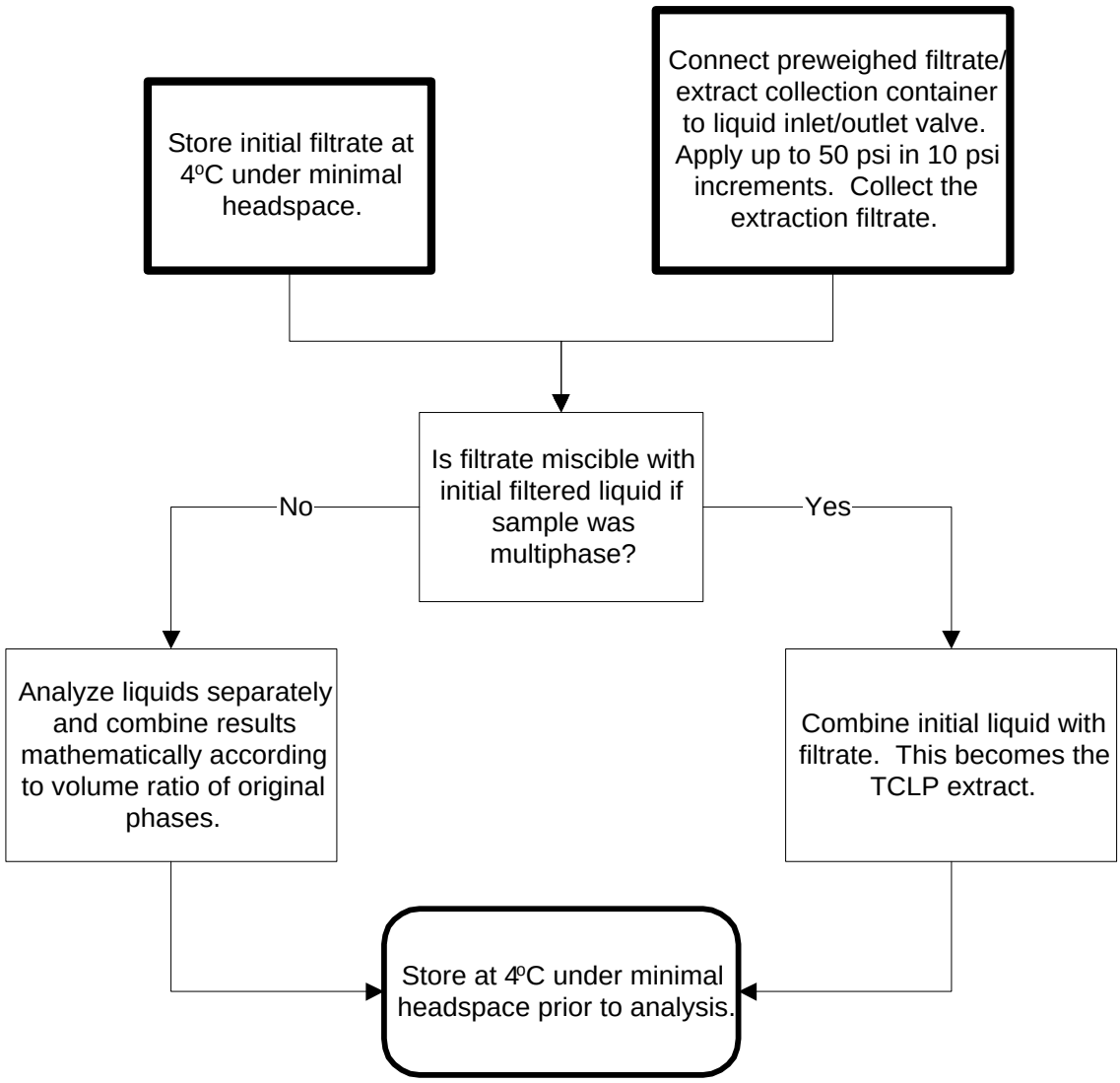
Flow Chart 2. Bottle Extraction, Non-Volatile Constituents (Section 11.3)



Flow Chart 3. ZHE Extraction, Volatile Constituents (Section 11.4)



Flow Chart 3. ZHE Extraction
(Continued)



MEASUREMENT OF ANALYTES USING THE KONELAB ANALYZER

(Methods: Various)

Approvals (Signature/Date):

Kimberly Chamberlain 12/29/2021

Kimberly Chamberlain Date
Quality Assurance Manager / Environmental Health and Safety Coordinator

12/29/2021

Adriana Geiger Date
Department Manager

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1.0 Scope and Application

This SOP gives the procedures for the determination of various analytes in water and soil samples using the Konelab Autoanalyzer. The following analytes may be determined:

Analyte	Reference Method	SOP Summary
Ammonia	EPA 350.1 SM4500-NH3 B (AQ prep) COE 3-154 (SO Prep) SM4500-NH3 G (analysis)	Attachment 1a
Chloride	EPA 325.2 EPA 9251 SM4500-Cl^- E	Attachment 1b
Total Chlorine	EPA 5050 / EPA 9251	Attachment 1b
Ferrous iron	SM3500-Fe D (SM 21st Edition) SM3500-Fe B (SM On-Line Edition)	Attachment 1c
Ferric Iron	SM3500-Fe B (Calculation)	Attachment 1c
Hexavalent Chromium	EPA 7196A SM3500-Cr D (SM 21st Edition) SM3500-Cr B (SM On-Line Edition)	Attachment 1d
Trivalent Chromium	EPA 7196A SM3500-Cr B (Calculation)	Attachment 1d
Ortho-Phosphate	EPA 365.1 SM4500-P F	Attachment 1e
Sulfate	EPA 375.4 EPA 9038	Attachment 1f
Total Sulfur	EPA 5050 / EPA 9038	Attachment 1f
BTU	ASTM D240-87	Attachment 1f

The reporting limits (RL), the method detection limits (MDL), and the accuracy and precision criteria associated with each method are provided in the TALS Method Limit Groups (MLGs).

This SOP was written by and for Eurofins Savannah laboratory.

2.0 Summary of Method

Samples and reagents are dispensed into disposable, acrylic multi-cell cuvettes that hold 12 separate analyses. During the incubation period(s), the sample cuvettes are maintained at 37°C. The measurement system is a single channel interference filter photometer with beam splitting reference. The color wheel can be configured with up to 15 different filters. Fiber optic cabling transfers the light signal from the source to the detector. The lamp is a halogen lamp.

The SOP Attachments contain the method parameters required to perform each analysis, a summary of the "chemistries" (reagents, standards, procedures, wavelengths, etc.) used to measure each target analyte, sample preparation information, as well as any method-specific information, requirements, and/or criteria. Method-specific requirements contained in the Attachments supersede the requirements outlined within the body of this SOP.

This SOP is based on the methods listed in Section 1.0.

3.0 Definitions

Refer to the Glossary Section of the *Quality Assurance Manual* (QAM) for a complete listing of applicable definitions and acronyms.

4.0 Interferences

4.1 Procedural Interferences

4.1.1 Interferences may be caused by contaminants in solvents, reagents, glassware, and other sample processing apparatus and can make identification and/or quantification of the target analytes difficult.

4.1.2 All sample collection containers are single-use disposable containers which limits the potential for contamination. All non-disposable labware must be scrupulously cleaned in accordance with the posted Labware Cleaning Instructions to ensure it is free from contaminants and does not contribute artifacts.

4.1.3 High purity reagents and solvents are used to help minimize interference problems. Acetone, hydrochloric acid, methanol, nitric acid, and sulfuric acid must be verified prior to use in accordance with the Eurofins Environment Testing Solvent Lot Testing Program.

4.1.4 Instrument and/or method blanks are routinely used to demonstrate all reagents and apparatus are free from interferences under the conditions of the analysis.

4.1.5 Refer to the appropriate SOP Attachment, as listed in Section 1, for any method-specific procedural interference information.

4.2 Matrix Interferences

4.2.1 Matrix interferences may be caused by contaminants that are co-extracted from the sample matrix. The sample may require cleanup or dilution prior to analysis to reduce or eliminate the interferences. For this procedure, the only cleanup procedure available is filtration through a 0.45um filter. This filtration step will remove turbidity that is present that may interfere with analysis.

4.2.2 Interfering contamination may occur when a sample containing low concentrations of analytes is analyzed immediately following a sample containing relatively high concentrations of analytes. As such, samples known to be clean should be analyzed first. To prevent carryover into subsequent samples, analysis of reagent blanks may be needed after the analysis of a sample containing high concentrations of analytes.

4.2.3 Refer to the appropriate SOP Attachment, as listed in Section 1, for any method-specific matrix interference information.

5.0 Safety

Employees must abide by the policies and procedures in the Eurofins Environment Testing Environmental Health and Safety Manual (EHSM), the Eurofins Savannah Addendum to the EHSM, and this document.

This procedure may involve hazardous materials, operations, and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user to follow appropriate safety, waste disposal, and health practices under the assumption that all samples and reagents are potentially hazardous.

The analyst must protect himself/herself from exposure to the sample matrix. Many of the samples that are tested may contain hazardous chemical compounds or biological organisms. The analyst must, at a minimum, wear protective clothing (lab coat), eye protection (safety glasses or face shield), disposable nitrile gloves, or equivalent, and closed-toe, nonabsorbent shoes when handling samples.

5.1 Specific Safety Concerns or Requirements

The toxicity or carcinogenicity of chemicals used in this method has not been precisely defined. Each chemical should be treated as a potential health hazard, and exposure to these chemicals should be minimized.

Mercuric thiocyanate will give off hydrogen cyanide (HCN) gas if combined with strong acids. Inhalation of CN gas can cause irritation, dizziness, nausea, unconsciousness and potentially death.

Sulfuric acid is a strong oxidizer and is a corrosive. It will react violently when combined with organic compounds, possibly producing fire. Inhalation can cause irritation of the

nose, throat, mucus membranes, and upper respiratory tract. Contact with the eyes can cause blurred vision, redness, pain, and even blindness.

Acetic acid is a corrosive. Contact with concentrated acetic acid can cause damage to the skin and eyes. Inhalation of concentrated vapors may cause damage to the lining of the nose, throat, and lungs.

Acetone is a flammable solvent. It can cause irritation to the respiratory tract. Overexposure can cause fatigue, lightheadedness, headache, dizziness, and blurred vision.

Nitric and hydrochloric acids are extremely hazardous as oxidizers, corrosives, poisons, and are reactive. Inhalation of the vapors can cause coughing, choking, irritation of the nose, throat, and respiratory tract, breathing difficulties, and lead to pneumonia and pulmonary edema. Contact with the skin can cause severe burns, redness, and pain. Nitric acid can cause deep ulcers, and staining of the skin to a yellow or yellow-brown color. These acid vapors are irritating and can cause damage to the eyes. Contact with the eyes can cause permanent damage.

Methanol is a flammable solvent. It can cause irritation to the respiratory tract. Overexposure can cause fatigue, confusion, headache, dizziness, and drowsiness.

5.2 Primary Materials Used

The following is a list of the materials used in this procedure, which have a serious or significant hazard rating, and a summary of the primary hazards listed in their MSDS/SDS.

NOTE: This list does not include all materials used in the procedure. A complete list of materials used in this procedure can be found in the Reagents and Standards Section and the Equipment and Supplies Section of this SOP

Employees must review the information in the MSDS/SDS for each material before using it for the first time or when there are major changes to the MSDS/SDS. Electronic copies of MSDS/SDS can be found using the “MSDS” link on the Oasis homepage, on the EH&S webpage on Oasis, and via TALS File System Shares.

Material	Hazards	Exposure Limit[1]	Signs and Symptoms of Exposure
Acetic Acid	Corrosive Poison Flammable	10ppm TWA	Contact with concentrated solution may cause serious damage to the skin and eyes. Inhalation of concentrated vapors may cause serious damage to the lining of the nose, throat, and lungs. Breathing difficulties may occur.
Acetone	Flammable	1000ppm TWA	Inhalation of vapors irritates the respiratory tract. May cause coughing, dizziness, dullness, and headache.
Barium Chloride	Poison Irritant	0.5mg/m^3 TWA	May be fatal if swallowed. Harmful if inhaled. Avoid contact with eyes, skin, and clothing. Avoid breathing dust. Keep container closed and when in use adequate ventilation.
Ferric Nitrate	Oxidizer	None	Causes irritation to the respiratory tract. Causes irritation, redness, and pain to the skin and eyes.
Hydrochloric Acid[2]	Corrosive Poison	5ppm Ceiling	Inhalation of vapors can cause coughing, choking, inflammation of the nose, throat, and upper respiratory tract, and in severe cases, pulmonary edema, circulatory failure, and death. Can cause redness, pain, and severe skin burns. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
Mercuric Thiocyanate	Poison	0.1 mg/m^3 Ceiling (Mercury Compounds)	Extremely Toxic. Causes irritation to the respiratory tract. **May produce Hydrogen Cyanide gas if combined with strong acids.** Causes irritation. Symptoms include redness and pain. May cause burns. May cause sensitization. Can be absorbed through the skin with symptoms to parallel ingestion. **May affect the central nervous system.** Causes irritation and burns to eyes. Symptoms include redness, pain, and blurred vision; may cause serious and permanent eye damage.
Methanol	Flammable Poison Irritant	200ppm TWA	A slight irritant to the mucous membranes. Toxic effects exerted upon nervous system, particularly the optic nerve. Symptoms of overexposure may include headache, drowsiness and dizziness. Methyl alcohol is a defatting agent and may cause skin to become dry and cracked. Skin absorption can occur; symptoms may parallel inhalation exposure. Irritant to the eyes.

Material	Hazards	Exposure Limit[1]	Signs and Symptoms of Exposure
Nitric Acid[2]	Corrosive Oxidizer Poison	2ppm TWA 4ppm STEL	Nitric acid is extremely hazardous; it is corrosive, reactive, an oxidizer, and a poison. Inhalation of vapors can cause breathing difficulties and lead to pneumonia and pulmonary edema, which may be fatal. Other symptoms may include coughing, choking, and irritation of the nose, throat, and respiratory tract. Can cause redness, pain, and severe skin burns. Concentrated solutions cause deep ulcers and stain skin a yellow or yellow-brown color. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
Phenol	Corrosive	5ppm TWA	Breathing vapor, dust or mist results in digestive disturbances. Will irritate and possibly burn respiratory tract. Rapidly absorbed through the skin with systemic poisoning effects to follow. Discoloration and severe burns may occur, but may be disguised by a loss in pain sensation. Eye burns with redness, pain, blurred vision may occur. May cause severe damage and blindness.
Phosphoric Acid	Corrosive	1mg/m^3 TWA	Inhalation is not an expected hazard unless misted or heated to high temperatures. May cause redness, pain, and severe skin burns. May cause redness, pain, blurred vision, eye burns, and permanent eye damage.
Sodium Hydroxide	Corrosive Poison	2mg/m^3 TWA	This material will cause burns if comes into contact with the skin or eyes. Inhalation of Sodium Hydroxide dust will cause irritation of the nasal and respiratory system.
Sulfuric Acid[2]	Corrosive Oxidizer Dehydrator Poison Carcinogen	1mg/m^3 TWA	Inhalation produces damaging effects on the mucous membranes and upper respiratory tract. Symptoms may include irritation of the nose and throat, and labored breathing. Symptoms of redness, pain, and severe burn can occur. Contact can cause blurred vision, redness, pain and severe tissue burns. Can cause blindness.
[1]Exposure limit refers to the OSHA regulatory exposure limit.			
[2]Always add acid to water to prevent violent reactions.			

6.0 Equipment and Supplies

All applicable laboratory support equipment and supplies (including balances; thermometers; and volumetric containers, used to determine quantitative measurements, such as mechanical pipettes, disposable graduated pipettes, gas-tight syringes, etc.) must be verified in accordance with SOP SA-AN-100: *Laboratory Support Equipment (Verification and Use)*.

6.1 Equipment and Instrumentation

- Top-loading Balance
- Tumbler
- MicroDist Apparatus
- Konelab Model 20 Autoanalyzer, or equivalent
- Data System: The software system is based on the Windows NT operating system. The hardware interface can be either RS232 or an Ethernet connection and can be integrated to both transmit and receive information from a TALS.

6.2 Analytical Data System/ Software / Hardware

The Quality Assurance Department maintains the Master Equipment Listing for the laboratory. This is a listing of all instrumentation utilized by the laboratory and includes details about the instrumentation including the software or firmware.

6.3 Volumetric Containers and Dispensers

All volumetric labware must be verified in accordance with SOP SA-AN-100: *Laboratory Support Equipment (Verification and Use).* Refer to Attachment 6 for Labware Cleaning Procedures.

Volumetric Labware	Volume	Type (Quantitative / Qualitative)	Use	Verification Frequency	Laboratory Verification Criteria
Plastic Transfer Pipettes	Various	Qualitative	pH checks, transferring sample or reagents	None	None
Disposable Medicine Cups	Various	Qualitative	Used to contain sample aliquot for pH measurement	None	None
Disposable Konelab Sample Cups	2mL	Qualitative	Holding sample on instrument	None	None
Eppendorf-Style Mechanical Pipettes	Various	QUANTITATIVE	Preparing Dilutions, Standard and Reagent Preparation and Spiking	Initially, prior to first use, then quarterly thereafter (Daily for DOD)	Accuracy = 2% Precision = 1%
Volumetric Flasks (Class A)	Various	QUANTITATIVE	Preparing Dilutions, Standard and Reagent Preparation	None (Class A)	None (Class A)
Graduated Cylinders (Class A)	Various	QUANTITATIVE	Preparing Dilutions, Standard and Reagent Preparation	None (Class A)	None (Class A)
Disposable Round Bottom Test Tubes	8mL	Qualitative	Holding sample on instrument	None	None

6.4 Lab Supplies

- pH paper (narrow and wide range)
- Residual Chlorine Check Strips – starch iodide strips or DPD pillow pops
- Detergent – Nochromix, or equivalent, used for washing non-disposable labware.
- Hengar Granules
- Magnetic Stir Bars
- Teflon Chips – used as soil blank matrix

6.5 Sample Collection Containers

All sample collection containers are single-use disposable containers which limits the potential for contamination. The routine sample collection containers supplied by the laboratory are purchased with Certificate of Analysis attesting to purity.

Refer to Attachment 2 for a cross-reference of containers to methods.

7.0 Reagents and Standards

7.1 Expiration Dates

Expiration dates (time from initial use or receipt to final use) for standard and reagent materials must be set according to the guidance in this SOP. Note: These are maximum expiration dates and are not to be considered an absolute guarantee of standard or reagent quality. Sound judgment must be used when deciding whether to use a standard or reagent. If there is doubt about the quality of a standard or reagent material, a new material must be obtained or the standard or reagent material verified. Data quality must not be compromised to extend a standard's life.

The expiration date of any standard or reagent must not exceed the expiration date of the standard or reagent that was used to prepare it.

Unless noted elsewhere in this SOP, the following expiration date requirements and storage conditions apply:

Purchased Unopened = Manufacturer's Expiration Date
Purchased Opened = 5 years from Open Date
Prepared = 3 months from Preparation Date
Storage = Room Temperature

7.2 Reagents

Reagents must be prepared and documented in accordance with SOP SA-AN-041: *Reagent and Standard Materials Procedures.* Certificates of analysis or purity must be received with all purchased reagents, and scanned and attached to the standard in TALS. Unless otherwise stated in the method-specific attachments, reagents should be stored in a cool, dry, ventilated storage area away from incompatible materials.

Refer to the appropriate SOP Attachment, as listed in Section 1, for the method-specific reagents associated with each analysis.

Acetone, hydrochloric acid, methanol, nitric acid, and sulfuric acid must be verified prior to use in accordance with the Eurofins Environment Testing Solvent Lot Testing Program.

Blank Matrix – Ottawa Sand or Teflon Chips (see associated Attachment). Used for the preparation of soil QC samples.

Laboratory Reagent Water – ASTM Type II (also referred to as DI Water)

7.3 Standards

Standards must be prepared and documented in accordance with SOP SA-AN-041: *Reagent and Standard Materials Procedures.* Certificates of analysis or purity must be received with all purchased standards, and scanned and attached to the standard in TALS.

Refer to the appropriate SOP Attachment, as listed in Section 1, for the method-specific standards associated with each analysis. Other standard concentrations may be used provided they support the reporting limit and are fully documented in accordance with SOP SA-AN-041.

8.0 Sample Collection, Preservation, Shipment, and Storage

Refer to Attachment 2 for the method-specific sample collection, preservation, shipment, and storage information.

9.0 Quality Control

SOP SA-QA-017: *Evaluation of Batch QC Data* and the SOP Summary in Attachment 3 provide requirements for evaluating QC data. Refer to the appropriate SOP Attachment, as listed in Section 1, for any method-specific quality control information associated with each analysis.

9.1 Batch QC

A preparation/analysis batch consists of up to 20 environmental samples and the associated QC items analyzed together within a 24 hour period. Refer to the Table in Attachment 3 for a summary of the method-specific batch QC items.

If there is insufficient sample volume to perform the required matrix spike(s), an NCM must be initiated on all affected samples to denote this situation. Insufficient sample volume is defined as receiving less than a total of 20mL.

If insufficient sample volume is provided to perform the Sample duplicate (SD) or matrix spike/ matrix spike duplicate (MS/MSD), the Laboratory control sample (LCS) must be prepared in duplicate (LCS/LCSD). An NCM must be initiated on all samples within the batch to denote this situation.

Note: Unless the client has specified which sample to use as the matrix spike, the matrix spike must be selected at random from the samples in the batch. Field QC (e.g., equipment blanks, trip blanks, and field blanks) must not be used for MS/MSD unless specifically requested to do so by the client or unless there is insufficient sample available for performing MS/MSD using an actual field sample.

MRL LCS for DW

The EPA Manual for the Certification of Laboratories Analyzing Drinking Water requires a Laboratory fortified blank (LFB) at the MRL to be performed each day. Therefore, if analyzing drinking water samples, a Laboratory Control Sample (LCS) at the Reporting Limit (RL) must also be included in the required batch QC.

9.2 Instrument QC

9.2.1 Initial Calibration (ICAL)

The instrument must be calibrated in accordance with SOP SA-QA-016: *Evaluation of Calibration Curves.* This SOP provides requirements for establishing the calibration curve and gives the applicable formulas.

Instrument calibration is performed by analyzing a series of known standards. The calibration curve must consist of a minimum of 3 standards and a blank. The lowest level calibration standard must be at or below the reporting limit, and the remaining standards will define the working range of the analytical system.

The regression coefficient (r^2) of the regression curve must be greater than or equal to 0.995 for the initial calibration curve to be acceptable.

Readback Criteria : Each point on the curve must be within +/- 10% of true value.

9.2.2 Second Source Initial Calibration Verification (ICV)

The calibration curve must be verified initially – prior to any sample analyses – in accordance with SOP SA-QA-016 with a standard obtained from a second source.

The ICV must be within 10% to be acceptable.

9.2.3 Initial Calibration Blank (ICB) / Continuing Calibration Blank (CCB)/ Method Blank (MB)

The instrument must be shown to be free from contamination by the analysis of calibration blanks. Initial calibration blanks are analyzed at the beginning of each analysis run. Continuing calibration blanks are analyzed following each CCV.

SM3500-Fe B, SM3500 Fe-D SM3500-Cr B: Blanks must be < MDL
Other methods: Blanks must be < ½ RL.

9.2.4 Continuing Calibration Verification (CCV)
Unless noted elsewhere in this SOP, the initial calibration curve must be verified every 10 analyses and at the end of the analysis run with a mid-level standard.

The CCV must be within 10% to be acceptable.

9.2.5 Linear Calibration Range / Linear Dynamic Range
The linear calibration range (LCR), also known as the Linear Dynamic Range (LDR), must be determined initially for the following methods:

SM3500-Fe B
SM3500-Cr B
SM4500-NH_3 G
SM4500-P F
SM4500-Cl^- E
EPA 350.1
EPA 365.1

The initial demonstration of linearity uses as many standards as necessary to ensure the curve is linear. The standards should cover the low and high ends of the calibration range. Each standard must be within +/- 10% of the true value.

The LCR/LDR must be verified every 6 months for the following methods:
EPA 350.1
EPA 365.1

The continued demonstration of linearity consists of a minimum of 3 standards and a blank. For the linearity to be verified, the standards must be within +/-10% of the initial value to be acceptable. If this criterion is not met, the initial demonstration of linearity must be performed.

Note: This study is not meant to extend the linear range of the calibration. If an analyte is measured at a concentration greater than the highest calibration standard, the sample must be analyzed at a dilution to bring the target analyte into the range of the initial calibration.

9.3 Corrective Action for Out-of-Control Data
When the quality control parameters do not meet the criteria set forth in this SOP, corrective action must be taken in accordance with SOP SA-QA-005: *Preventive and Corrective Action Procedure* and the QC Summary Table in Attachment 3. SOP SA-QA-005 provides contingencies for out-of-control data and gives guidance for exceptionally permitting departures from approved policies and procedures. Nonconformance Memos must be initiated to document all instances where QC criteria are not met and all departures from approved policies and procedures.

10.0 Procedure

10.1 Sample Preparation

Remove the samples or extracts from the refrigerator and allow them to come to room temperature.

Soil samples must be homogenized prior to preparation in accordance with SOP SA-QA-015: *Compositing, Homogenization, and Segregation of Samples*. Note: If a sample or extract is filtered, the associated QC must also be filtered.

The sample preparation procedures are summarized below. Refer to appropriate SOP Attachment for the method-specific procedures.

Analyte	Aqueous Prep	Soil Prep
Ammonia	Distillation* (EPA 350.1 / SM4500-NH_3 B) Undistilled (No Prep)*	Soil Leachate
Chloride	No Prep	Soil Leachate
Total Chlorine	EPA 5050 (SOP SA-GE-010)	EPA 5050 (SOP SA-GE-010)
Ferrous Iron (Ferric Iron calc)	Filter	
Hexavalent Chromium (Trivalent Chromium calc)	Filter**	N/A
Ortho-Phosphate	Filter **	Soil Leachate
Sulfate	No Prep	Soil Leachate
Total Sulfur	EPA 5050 (SOP SA-GE-010)	EPA 5050 (SOP SA-GE-010)
BTU	ASTM D270-87	ASTM D240-87

*Ammonia samples are distilled upon client request.
** Samples should be field filtered.

10.2 QC Sample Preparation

Refer to appropriate SOP Attachment, as listed in Section 1, for the method-specific QC sample preparation instruction associated with each procedure.

10.3 Analysis

10.3.1 Instrument Operating Conditions

The instrument conditions listed in this SOP are provided for guidance purposes. The actual conditions used by the laboratory may be slightly different from those listed here and must be documented in the instrument maintenance log, data system, and/or run log.

Instrument maintenance must be performed in accordance with Attachment 4 of this SOP.

Data System
Programmable instrument parameters include:

Volume of sample (maximum of 125uL)
Wavelength
Type and order of reagent(s) addition
Background absorbance
Time of sample/reagent mixing
Timing of absorbance readings
Dilutions
Reagents lot numbers/expiration dates

QC checks can be programmed into the methods as needed, including initial and continuing control standards, blanks, and upper and lower limits on absorbance. If samples or standards exceed these control limits, the data are flagged.

Operation
The instrument utilizes the following process:

- Sample is dispensed into the cuvettes according to the test method (maximum sample volume is 125uL)
- The reagents are added to the sample and mixed.
- The sample is incubated.
- The sample absorbance is measured (if absorbance exceeds calibration range, sample is diluted as defined in the test method and reanalyzed. If the dilution exceeds the maximum range, the data are flagged and the analyst must prepare dilution for sample).

10.3.2 Initial and Continuing Calibration

The instrument is calibrated using the standards and criteria described in the appropriate SOP Attachment, as listed in Section 1. Once the calibration has been established and verified with an ICV in accordance with Section 9.2.2, sample analysis may proceed.

Verify the calibration curve with a continuing calibration verification using the standards and criteria described given in Section 9.2.4 and the appropriate SOP Attachment, as listed in Section 1.

10.3.3 Sample Analysis

Remove the samples or extracts from the refrigerator and allow them to come to room temperature.

Samples are loaded into racks that hold 14 samples each. The autosampler holds up to 6 racks of 14 samples. The racks can be fitted to hold sample containers from 0.5mL up to 10mL. Two separate wash stations are used to minimize sampler carryover. The probe is flow-through and externally rinsed.

The sample/extract must be analyzed using the same volume used for the calibration standards. The default procedure is to include QC items (method blank, LCS, MS/MSD, and SD) in determining the maximum number of samples in the analysis.

10.3.4 Example Analytical Sequence

Refer to the appropriate SOP Attachment, as listed in Section 1, for any changes to the sequence listed below.

Description	Comments
Blank	
Initial Calibration	
ICV	Second Source
ICB	
Samples & Batch QC Items	Up to 10 analyses. Not to exceed 2 hours.
CCV	
CCB	
Samples & Batch QC Items	Up to 10 analyses. Not to exceed 2 hours.
CCV	
CCB	

11.0 Calculations / Data Reduction

11.1 Data Reduction

Data must be evaluated in accordance with SOP SA-QA-002: *Data Review and Reporting*.

11.1.1 MS/MSD Evaluation

If the concentration of a target analyte in the un-spiked (native) sample is more than four times the theoretical concentration of the matrix spike, the recovery is not reported and the data is flagged.

11.1.2 Historical Data

Many of the laboratory's clients submit samples for repeat monitoring purposes. Prior to analysis, verify TALS Worksheet Notes and/or use the TALS Historical Data Tracker Feature to determine if historical data is available for review.

11.1.3 Chemical Relationships

When available, the following chemical relationships must be evaluated for each sample. If these relationships are not met the Department Manager must be contacted immediately.

- Total Results ≥ Dissolved Results
- TKN ≥ Ammonia
- Total Phosphorus ≥ Ortho-phosphate
- TDS ≥ Individual Anions
- Ferrous Iron ≤ Total Iron

11.1.4 Drinking Water Compliance Evaluation

Public water suppliers (PWS) are governed by EPA-specified Maximum Contaminant Levels (MCL) above which indicates noncompliance. The MCLs associated with this procedure are given in Attachment 5. Notify the PM immediately via a Nonconformance Memo if any sample contains detection above these levels.

11.2 Calculations

11.2.1 The calculations associated with batch QC determinations are given in SOP SA-QA-017. Applicable calculations include accuracy (% recovery) and precision (%RPD).

11.2.2 The calculations associated with initial and continuing calibrations and are given in SOP SA-QA-016. Applicable calculations include determination for: calibration factor, standard deviation, relative response factor, and relative standard deviation.

11.2.3 The calculation to determine final concentration is given as follows:

$$FinalConcentration = CONC_{Sample} \otimes \frac{F}{I \times dw} \otimes D$$

Where:

$CONC_{Sample}$= Concentration of the sample
F = Final volume/weight
I = Initial volume/weight
dw = % Solids decimal equivalent
D = Dilution factor

Note: All dry weight corrections are performed automatically in TALS.

12.0 Method Performance

12.1 Reporting Limit Verification (RLV)

At a minimum, RLVs must be performed initially upon method set-up in accordance with SOP SA-QA-007: *Determination and Verification of Detection and Reporting Limits*.

For analytes and methods certified by DOD ELAP, RLVs must be performed quarterly thereafter.

12.2 Method Detection Limit (MDL) Study

The method detection limits (MDL) is defined as the minimum measure concentration of a substance that can be reported with 99% confidence that the measured concentration is distinguishable from the method blank results. MDLs reflect a calculated (statistical) value determined under ideal laboratory conditions in a clean matrix and may not be achievable in all environmental matrices. The current MDLs associated with this procedure are given in the Method Limit Group (MLG) in TALS.

At a minimum, MDL Studies must be performed initially upon method set-up in accordance with SOP SA-QA-007: *Determination and Verification of Detection and Reporting Limits*.

Note: MDL Studies are not required for non-routine analytes provided results are not reported below the RL (i.e., MDL equals RL in TALS).

12.3 Method Detection Limit Verification (MDLV)

At a minimum, MDLVs must be performed initially upon method set-up in accordance with SOP SA-QA-007: *Determination and Verification of Detection and Reporting Limits*.

12.4 QC Limit Generation, Control Charting, and Trend Analysis

The control limits for the batch QC items (LCS, MS/MSD, SD) for this procedure are specified in the reference method and cannot be broadened; therefore, the laboratory defaults to the method-defined limits and does not utilize in-house or laboratory-derived limits for the evaluation of batch QC items.

Although the laboratory must default to the method-defined QC limits, control charting is a useful tool and is performed to assess analyte recoveries over time to evaluate trends. Control charting must be performed periodically (at a minimum annually) in accordance with SOP SA-QA-017: *Evaluation of Batch QC Data.*

12.5 Demonstrations of Capability

Initial and continuing demonstration of capability must be performed in accordance with SOP SA-QA-006*: Training Procedures.*

Prior to performing this procedure unsupervised, each new analyst who performs this analysis must demonstrate proficiency per method/analyte combination by successful completion of an initial demonstration of capability. The IDOC is performed by the analysis of 4 consecutive LCSs that meet the method criteria for accuracy and precision. The IDOC must be documented and routed to the QA Department for filing.

Annual continuing demonstrations of capability (CDOCs) are also required per analyst per method/analyte combination. The CDOC requirement may be met by the consecutive analysis of four LCS all in the same batch, by the analysis of four LCS analyzed in four consecutive batches (in different batches on different days), via acceptable results on a PT study, or analysis of client samples with statistically indistinguishable results when compared to another certified analyst. The CDOC must be documented and routed to the QA Department for filing.

12.4 Training Requirements

All training must be performed and documented in accordance with SOP SA-QA-006: *Training Procedures*.

Note: The SOPs listed in the Reference/Cross-Reference Section are applicable to this procedure. All employees performing this procedure must also be trained on these SOPs.

13.0 Pollution Control

It is Eurofins Savannah's policy to evaluate each method and look for opportunities to minimize waste generated (e.g., examining recycling options, ordering chemicals based on quantity needed, preparing reagents based on anticipated usage and reagent stability, etc.). Employees must abide by the policies in Section 13 of the Environmental Health and Safety Manual.

This procedure has been evaluated for opportunities to minimize the waste generated. Where reasonably feasible, pollution control procedures have been incorporated.

14.0 Waste Management

Waste management practices must be conducted consistent with all applicable federal, state, and local rules and regulations. All waste (i.e., excess reagents, samples, and method process wastes) must be disposed of in accordance with Section 9 of the Eurofins Savannah Addendum to the EHSM. Waste description rules and land disposal restrictions must be followed.

14.1 Waste Streams Produced by the Method

The following waste streams are produced when this method is carried out:

- Excess aqueous samples – Dispose according to characterization by the disposal program. Neutralize non-hazardous samples before disposal into drain/sewer. Transfer hazardous samples to the waste department for disposal.

- Excess soil and solid samples – Dispose according to characterization by the disposal program. Transfer hazardous and non-hazardous samples to the yardbox in the waste storage area for disposal via incineration. Samples classified as PCB or Foreign Soil hazardous must be transferred to the waste coordinator for drumming.

- Non-hazardous acidic and alkaline wastewater and samples must be neutralized before disposal into the sewer system.

15.0 References / Cross-References

- SOP SA-AN-100: *Laboratory Support Equipment (Verification and Use)*
- SOP SA-AN-041: *Reagent and Standard Materials Procedures*
- SOP SA-ME-021: *Digestion Procedures for Solids for Hexavalent Chromium*
- SOP SA-QA-002: *Data Review and Reporting*
- SOP SA-QA-005: *Preventive and Corrective Action Procedures*
- SOP SA-QA-006*: Training Procedures*
- SOP SA-QA-007: *Determination and Verification of Detection and Reporting Limits (RLs, MDLs, and IDLs)*
- SOP SA-QA-015: *Homogenization, Compositing, and Segregation of Samples*
- SOP SA-QA-016: *Evaluation of Calibration Curves*
- SOP SA-QA-017: *Evaluation of Batch QC Data*
- Eurofins Savannah Quality Assurance Manual
- Eurofins Environmental Health and Safety Manual (CW-E-M-001)
- Eurofins Savannah Addendum to the Environmental Health and Safety Manual
- *Standard Methods for the Examination of Water and Wastewater*; American Public Health Association: Washington, DC
  - SM1020: *Quality Assurance*; 20th Edition
  - SM3020: *Quality Assurance/Quality Control*; 2005
  - SM3500-Cr B: *Chromium, Colorimetric Method*; 2001
  - SM3500-Fe B: *Ferrous Iron, Phenanthroline Method*; 1997
  - SM4020: *Quality Assurance/Quality Control*
  - SM4500-Cl^- E: *Chloride, Automated Ferricyanide Method*; (Approved 1997; Editorial Revision 2011)
  - SM4500-NH_3 B: *Nitrogen (Ammonia), Preliminary Distillation Step*; 1997
  - SM4500-P F: *Phosphorous, Automated Ascorbic Acid Reduction Method*; 1999
  - SM4500-NH_3 G: *Nitrogen (Ammonia), Automated Phenate Method*; 1997
  - SM5020: *Quality Assurance/Quality Control*
- *Methods for Chemical Analysis of Water and Wastes*; U.S. EPA Office of Research and Development: Cincinnati, OH, March, 1983
  - EPA 325.2: *Chloride (Colorimetric, Automated Ferricyanide AAII)*; 1978

- EPA 350.1: *Determination of Ammonia Nitrogen by Semi-Automated Colorimetry*; Revision 2.0, August 1993
- EPA 365.1: *Determination of Phosphorus by Semi-Automated Colorimetry*; Revision 2.0, August 1993
- EPA 375.4: *Sulfate (Turbidimetric)*; 1978

- *Test Methods for Evaluating Solid Waste, Third Edition On-line*; U.S. EPA Office of Solid Waste and Emergency Response: Washington, DC
  - EPA 5050: *Bomb Preparation Method for Solid Waste; Revision 0, September 1994*
  - EPA 7196A: *Chromium, Hexavalent (Colorimetric)*; Revision 1, July 1992
  - EPA 9038: *Sulfate (Turbidimetric)*; Revision 0, September 1986
  - EPA 9251: *Chloride (Colorimetric, Automated Ferricyanide AAII)* ; Revision 0, September 1986
- *Annual Book of ASTM Standards Section 5, Volume 05.01;* American Society of Testing and Materials: Philadelphia, 1992.
  - D240-87: *Heat of Combustion of Liquid Hydrocarbon Fuels by Bomb Calorimeter*; May 1991

16.0 Method Modifications and Clarifications

16.1 Many of the reference methods were written specifically for drinking water and source water samples; however, the laboratory may perform other types of water samples using these procedures. These procedures may be modified to analyze other matrices (e.g., wipe samples) based on the needs of the client. This will need to be arranged by the Project Manager at the initiation of the project. Wipe, waste, and tissue matrices are non-routine, and the laboratory is not currently NELAC certified for these matrices. The laboratory uses its routine soil RLs and MDLs (converted for initial and final volumes, etc.), and soil QC limits to evaluate wipe, waste, and tissue samples. Soil DOCs can be used to satisfy analyst demonstrations of capability for these types of non-routine matrices. Ottawa sand is used as the blank matrix for tissue samples unless a “true” tissue matrix is required by the project.

16.2 The EPA Manual for the Certification of Laboratories Analyzing Drinking Water requires a LFB at the MRL to be performed each day. The laboratory meets this requirement by preparing an LCS at the RL in each batch of drinking water samples.

16.3 Refer to appropriate SOP Attachment, as listed in Section 1, for any method-specific modifications and/or clarifications associated with each procedure.

16.4 The default procedure for the ammonia analysis is to analyze samples undistilled. An equivalency study has been conducted using a variety of sample matrices and shows similar results were obtained for undistilled versus distilled samples. Distillation for ammonia samples can be performed upon client request.

17.0 Attachments

The following Tables, Diagrams, and/or Validation Data are included as Attachments:

Attachment 1a: SOP Summary – Ammonia
Attachment 1b: SOP Summary – Chloride and Total Chlorine
Attachment 1c: SOP Summary – Ferrous Iron and Ferric Iron
Attachment 1d: SOP Summary – Hexavalent Chromium and Trivalent Chromium
Attachment 1e: SOP Summary – Ortho-phosphate
Attachment 1f: SOP Summary – Sulfate, Total Sulfur, and BTU
Attachment 2: Sample Collection, Preservation, and Holding Time Table
Attachment 3a: Batch QC Frequency
Attachment 3b: QC Summary
Attachment 4: Instrument Maintenance and Troubleshooting
Attachment 5: Maximum Contaminant Levels (MCL) Table
Attachment 6: Glassware Cleaning Procedures

Attachment 1a
SOP Summary
Ammonia

1.0 Summary

Ammonia reacts with hypochlorite and alkaline phenol to form indophenol blue. The blue color produced is intensified with sodium nitroprusside and measured at 660nm.

2.0 Interferences

Extreme sample pH values may interfere. Proper sample preservation and adjustment just prior to analysis should alleviate this problem. Turbidity and color in samples must be filtered and/or diluted prior to analysis. Residual chlorine should be removed at the time of sampling by the addition of sodium thiosulfate.

3.0 Reagents and Standards

3.1 Purchased Reagents

Sodium Thiosulfate ($Na_2S_2O_3 \cdot 5H_2O$) Dechlorination Reagent – reagent grade

Sulfuric Acid (H_2SO_4) – concentrated, reagent grade
Storage: Area with acid resistant floors. Store away from direct sunlight, heat, and water.

Sodium Hydroxide (NaOH) – pellets, reagent grade
Storage: Isolate from moisture and heat sources.

Potassium Chloride (KCl) – reagent grade

Phenol – solution must be $\geq$89% pure
Storage: Isolate from heat or ignition sources.

Sodium Tetraborate ($Na_2B_4O_7$) – reagent grade

Bleach/Sodium Hypochlorite – must contain 6.15% sodium hypochlorite (NaOCl)
Storage: Protect from heat and light.
Expiration: Unopened: 1 year; Opened: 6 months

Disodium Ethylenediamine Tetraacetate (EDTA) – reagent grade

Sodium Nitroprusside ($Na_2(NO)Fe(CN)_5 \cdot 2H_2O$) – reagent grade
Storage: Away from moisture and incompatibles

3.2 Prepared Reagents

Sodium Phenate Solution
Dissolve 9.3mL liquid (≥89%) phenol in 50mL ammonia free water, slowly add 3.2g NaOH and mix thoroughly and dilute to 100mL with DI Water. **CAUTION**: Wear gloves and eye protection when preparing this solution.
Expiration: Daily

Sodium Hypochlorite Solution
Dilute 25mL of 5.25% NaOCl with 50mL DI Water. (Note: If "concentrated" bleach is used (8.25% NaOCl), measure 16mL and dilute to 50mL with DI Water.)
Expiration: Daily

EDTA Buffer
Dissolve 50g of disodium ethylenediamine tetraacetate and 5.5g of NaOH in 1000mL of DI water.
Expiration: 1 year

Sodium Nitroprusside Solution
Dissolve 0.5g of $Na_2(NO)FE(CN)_5.2H_2O$ in 1000mL of DI Water.
Expiration: 1 year

5% Sulfuric Acid Solution
Add 50mL of concentrated sulfuric acid to a 1L volumetric flask containing 500mL DI water. Bring to volume with DI water.

Sulfuric Acid Solution (0.01M) – NH3_Diluent
Add 2.08mL of concentrated sulfuric acid to 1.0 gallon of water (0.56mL of concentrated H2SO4 per liter of water). The pH of this solution is 2.0. Verify the pH before use.
Expiration: 1 year

2N Sodium Hydroxide Solution
In a 1L volumetric flask add 500mL of DI water. Add 80 grams of sodium hydroxide pellets and let dissolve. Bring to volume with DI water after the pellets have dissolved. Mix well.
Expiration: 1 year

Sodium Hydroxide Solution (15N)
Add 250mL DI Water to a 500mL volumetric flask. Add a stir bar and place on stir plate. In small increments and with constant stirring, slowly add 300g NaOH to the flask. Allow to cool and dilute to volume with DI water. Transfer to a labeled storage container.
Expiration: 1 year

Potassium Chloride (1M)
Dilute 148g of potassium chloride to with 1800mL DI Water. Add 1.12mL concentrated sulfuric acid. Dilute to 2000mL and mix thoroughly.
Expiration: 1 year

O-Cresolphthalein Indicator Solution 0.2% w/v alcoholic solution.
Expiration: Manufacturer's expiration date

MicroDist Trapping Solution
Add 3.3mL of concentrated sulfuric acid to 1.0L of water.
Expiration: 1 year

Borate Buffer (MicroDist)
Add 5.0g of sodium borate decahydrate and 22g sodium hydroxide to 600mL of DI Water in a 1L volumetric flask. Stir to dissolve then add 10mL o-cresolphthalein indicator solution. Dilute to mark with DI Water. Buffer should be a deep purple color.
Expiration: 1 year

3.3 Standards

Ammonia Stock Standard (100mg/L) Purchased
Expiration: Manufacturer's expiration date

Ammonia Calibration Standards
Prepare calibration standards in 0.01M Sulfuric Acid (NH3 Diluent) according to the following table.

Standard Level	Parent Standard	Volume Intermediate (mL)	Final Volume (mL)	Final Concentration (mg/L)
1	Ammonia Stock Standard	2.0	100	2.0
2 (LCS)	Ammonia Stock Standard	10	1000	1.0
3	Ammonia Stock Standard	0.50	100	0.50
4	Ammonia Stock Standard	0.25	100	0.25
5	Ammonia Stock Standard	0.10	100	0.10
6	Cal 2	5.0	100	0.050
7	Cal 2	2.5	100	0.025

Initial Calibration Verification (ICV) – 1.0mg/L standard from NCL Labs. Adjust to pH 2 with a weak solution of H2SO4.
Expiration: manufacturer's date (1 month if no date given)

4.0 Sample Preparation

4.1 Aqueous Samples
Aqueous samples are routinely analyzed with no sample preparation. The samples can be distilled upon request.

NOTE: All compliance samples originating from West Virginia or South Carolina must be distilled prior to analysis.

Residual Chlorine Check (for Ammonia analyses only)
For each sample,
- Place a piece of starch-iodide paper in a disposable medicine cup.
- Pour a few drops of sample into the medicine cup and note the color change of the paper.
- If the paper turns blue or black, residual chlorine is present. Initiate a Nonconformance Memo and dechlorinate the sample with sodium thiosulfate.

pH Verification (for Ammonia analyses only)
For each sample,
- Create a preservation batch in LIMS (preserv_chk)
- Place a piece of pH paper in a disposable medicine cup. Use narrow range 1.0-2.5 pH paper.
- Pour a few drops of sample into the medicine cup and note the color change of the pH paper.
- If the pH is greater than 2, initiate a Nonconformance Memo. Adjust the sample pH to 2 or less, targeting a value near pH 2, using sulfuric acid. The volume of sulfuric acid must not exceed 1% of the volume of the container or sample; e.g., no more than 2.5mL of sulfuric acid per 250mL container or sample.

Note: To avoid cross-contamination, use a separate medicine cup, pH strip, and residual chlorine strip per sample. Do not dip the strip into the sample container.

Prepare QC samples in the same manner as field samples as follows:
- Laboratory Control Sample (LCS) – Prepared in same manner as Cal Level 2.
- Low-Level Laboratory Control Sample- (LLCS) (Drinking Water only) – Performed at the RL (0.25mg/L) for routine DW samples. Performed at 0.05mg/L for FP
- Matrix Spike / Matrix Spike Duplicate (MS/MSD) – Add 0.1mL of the 100mg/L Ammonia Stock Standard to 10mL of the sample selected for MS/MSD.

  Note: SM4500-NH3 G recommends the MS/MSD spike concentration to be varied periodically.

4.1.1 Ammonia Micro-Distillation Preparation

Create a Distill_Ammonia batch in LIMS.

Set controller to 120°C and turn on the heater block to warm up (~40 min). Verify the temperature of the micro-distillation apparatus with an IR gun. The temperature must be within 115-125°C. Record the corrected and uncorrected temperature in the Batch Data Types in TALS.

Determine the number of collector tubes needed and align with “M” end in the up position by placing them into a collector tube rack.

Label and fill each collector tube with 1mL of trapping solution (0.016M Sulfuric Acid). At the end of the distillation, once diluted to 6mL final volume, the final concentration will be 0.003M Sulfuric Acid.

Place the same number of sample tubes into the sample tube rack.

Label each tube and place 6mL of sample into each sample tube.

Add 1mL of 0.55M Borate Buffer to each tube. Ensure the sample plus buffer remains purple (pH >9.8). If purple color fades when the buffer is added, add additional NaOH Solution to bring the pH up until the purple color returns.

Immediately push the “D” end of the collector tube over the open end of the sample tube to start the seal.

Place the tube assembly in the press by putting the sample tube through the hole in the base.

Before pressing, place one hand at the breakaway point located on the collector tube to stabilize the assembly.

Apply pressure to the assembly with a smooth motion until the stop ring on the sample tube hits the “D” end of the collector tube.

Once complete, put on heat resistant gloves and add the tube assemblies to the pre-heated block by placing the sample tube and “D” end of the collector tube into the block.

Set the timer for 30 minutes. Document the start time in the LIMs batch.

Once time is up remove the assemblies from the block one at a time and immediately remove the sample tube from the collector tube and invert so the D side is facing up. Note: this should be done within 4 seconds to prevent “suck-back” of the sample.

Document the end time, uncorrected temperature and corrected temperature in the batch information.

Once all of the tubes have been removed and disassembled, seal with Parafilm at the “I” end of the tube and allow the tubes to cool for 10 minutes.

Hold the tube horizontally and roll the tube with your fingers to gather the droplets that are stuck to the walls. The tube can be flicked with your finger as well.

With the “D” end still up, grasp the tube and break it at the breakaway point by pulling the “D” end towards your body and twisting the tube. Discard the “D” end of the tube.

Perform a pH check of samples to ensure they are still at a pH of 2. If not, adjust with Sulfuric Acid prior to diluting up to 6mLs. Use narrow range pH paper with a range of 1-2.5 for this check.

Dilute the distillate up to the 6 mL mark with NH3 Diluent and cover the tube with Parafilm until ready for analysis.

4.2 Soil Samples

- Weigh approximately 20g of sample into a 125mL screw-cap plastic bottle. Record the weight to the nearest 0.1g. Add 100mL of 2M potassium chloride to each sample container and place in the tumbler.
- Rotate for 30-45 minutes. Remove the containers from the extractor and allow the leachates to settle.
- Filter the extract using a syringe filter with a 0.45um pore size filter and analyze the extracts as liquid samples.

Prepare QC samples in the same manner as field samples as follows:

- Method Blank – Weigh approximately 20g of Teflon chips into a 125mL screw-cap plastic bottle. Record the weight to the nearest 0.1g.
- Laboratory Control Sample – Weigh approximately 20g of Teflon chips into a 125mL screw-cap plastic bottle. Record the weight to the nearest 0.1g. Add 1mL of 100mg/L Ammonia Intermediate Standard to the extraction bottle. Spike soil prior to adding 99ml of 2M potassium chloride.
- MS/MSD – Weigh additional 20g aliquots of one of the samples for the MS/MSD. Add 1mL of 100mg/L Ammonia Intermediate Standard Ammonia Matrix Spike Solution to the sample. Spike soil prior to adding 99ml of 2M potassium chloride.

5.0 Method-Specific QC Requirements

5.1 Linear Calibration Range
EPA 350.1 – Performed initially; verified every 6 months.
SM4500-NH_3 G – Performed initially.

5.2 Method Detection Limit Studies
EPA 350.1 – Requires MDL Studies every 6 months.
SM4500-NH_3 G – Requires MDL Study to be analyzed over 3-5 days.

6.0 Method-Specific Modifications and/or Clarifications

The ammonia distillation sample volumes have been adjusted to compensate for current lab restrictions of glassware and volume of sample submitted. All reagent volumes have been adjusted accordingly.

The reference methods state to adjust sample pH with 1N NaOH. The sample is preserved with H_2SO_4 to a pH of <2. To adjust the pH with such a weak solution would require adding >5% volume and therefore diluting the sample. As such, the laboratory uses 15N NaOH to adjust the sample pH.

The default procedure for the ammonia analysis is to analyze samples undistilled. An equivalency study has been conducted using a variety of sample matrices and shows similar results were obtained for undistilled versus distilled samples. Distillation for ammonia samples can be performed upon client request.

Attachment 1b
SOP Summary
Chloride & Total Chlorine

1.0 Summary

Chloride reacts with mercuric thiocyanate forming a mercuric chloride complex. Released thiocyanate reacts with iron (III) forming a red ferric thiocyanate complex the color produced, measured at 480nm, is proportional to the chloride concentration.

Refer to SOP SA-GE-010 for information on the preparation procedures for Total Chlorine. The attached Work Instruction provides the procedural and calculation information.

2.0 Interferences

Highly colored samples can positively interfere. Turbid samples should be filtered.

3.0 Reagents and Standards

3.1 Purchased Reagents

Mercuric Thiocyanate [$Hg(SCN)_2$] – reagent grade
Storage: Protected from light.

Methanol – HPLC or P&T grade
Storage: Protect from heat, sparks, and flame in a flammable storage area.

Ferric Nitrate [$Fe(NO_3)_3.9H_2O$] – reagent grade
Storage: Away from sources of heat and moisture. Avoid storage on wooden floors.
Expiration: 1 year

Nitric Acid, concentrated – A.C.S. grade
Storage: acid resistant floors and good drainage; away from sunlight, heat, and water.

3.2 Prepared Reagents

Ferric Nitrate Solution (20.2%) – Dissolve 202g $Fe(NO_3)_3.9H_2O$ into 500mL reagent water. Carefully add 44.4mL of concentrated nitric acid. Mix and dilute to 1L with reagent water. Store in an amber bottle away from light.
Expiration: 1 Year

Mercuric Thiocyanate Solution (Saturated) – Dissolve 4.16g $Hg(SCN)_2$ into 500mL of methanol. Dilute to 1L with methanol, mix, and filter. Dispose if reagent becomes discolored or cloudy.
Storage: Protected from light.
Expiration: 1 year

Color Reagent – Add 75mL of Mercuric Thiocyanate Solution to 75mL Ferric Nitrate Solution. Mix and dilute to 500mL with reagent water. Dispose if reagent becomes discolored or cloudy.
Storage: Tightly closed-light resistant container. Protect from light.
Expiration: 3 months

3.3 Standards

Sodium Chloride (NaCl) – reagent grade

Chloride Calibration Stock (1000mg/L) – Purchased from Inorganic Ventures.
Expiration: manufacturer's expiration date or one year, whichever is shorter.

Chloride Calibration Standards

Standard Level	TALS Reagent Name	Parent Standard	Aliquot (mL)	Final Volume (mL)	Final Concentration (mg/L)
1	Cl CAL10	Cl Cal Stk	0.1	100	1
2	Cl CAL6	Cl Cal Stk	1.0	100	10
3	Cl CAL5	Cl Cal Stk	2.0	100	20
4	Cl CAL4	Cl Cal Stk	4.0	100	40
5	Cl CAL12	Cl Cal Stk	5.0	100	50

Dilute to volume in reagent water.

Chloride Laboratory Control Sample (LCS) (25mg/L) – Add 6.25mL Chloride Calibration Stock to a 250mL volumetric. Dilute to volume with reagent water. This standard is also used as the CCV.

Chloride ICV Stock (1000mg/L) – Purchased from NSI.
Expiration: manufacturer's expiration date or one year, whichever is shorter.

Chloride ICV (25mg/L) – Add 2.5mL of Chloride ICV Stock to a 100mL volumetric flask. Dilute to volume with reagent water.

4.0 Sample Preparation

4.1 Aqueous Samples
Aqueous samples are routinely analyzed with no sample preparation.

Prepare QC samples in the same manner as field samples as follows:

- Laboratory Control Sample (LCS) – prepared as described above
- Low-Level Laboratory Control Sample (LLCS) (Drinking Water only) – Lowest level standard of the initial calibration
- MS/MSD – Add 0.25mL of Chloride Calibration Stock to 10mL of the sample selected for MS/MSD. Note: SM4500-Cl^- E recommends the MS/MSD spike concentration to be varied periodically.
- Sample Duplicate (SD) – Prepare an additional aliquot of the chosen sample.

4.2 Soil Samples

- Create a preparation batch in LIMS
- Weigh approximately 5g of sample into a 125mL screw-cap plastic bottle. Record the weight to the nearest 0.1g. Add 100mL of reagent water to each sample container and place in the tumbler.
- Rotate for 15minutes.
- Remove the containers from the extractor and allow the leachates to settle. Filter the extract using a chloride-free 25mm PTFE syringe filter with a 0.20µm pore size and analyze the extracts as liquid samples.

Prepare QC samples in the same manner as field samples as follows:

- Method Blank – Weigh approximately 5g of Teflon Chips into a 125mL screw-cap plastic bottle. Record the weight to the nearest 0.1g.
- Laboratory Control Sample – Weigh approximately 5g of Teflon Chips into a 125mL screw-cap plastic bottle. Record the weight to the nearest 0.1g. Add 2.5mL of the Chloride Cal Stock (1000mg/L) Solution and 97.5mL of DI water.
- MS/MSD – Weigh 2 additional 5g aliquots of one of the samples for the MS/MSD. Add 2.5mL of the Chloride Cal Stock (1000mg/L) Solution and 97.5mL of DI water to each bottle.

5.0 Method-Specific QC Requirements

5.1 Linear Calibration Range
SM4500-Cl^- E – Performed initially.

5.2 Method Detection Limit Studies
SM4500-Cl^- E – Requires MDL Study to be analyzed over 3-5 days.

6.0 Method-Specific Modifications and/or Clarifications

6.1 The SM4500-Cl^- E reference method specifies to use 21mL nitric acid to prepare the ferric nitrate solution. The EPA 325.2 and EPA 9251 reference methods specify 31.5mL. The laboratory utilizes 44.4mL as outlined in the Konelab application notes.

6.2 The SM4500-Cl^- E reference method includes the addition of polyoxyethylene 23 lauryl ether to the color reagent. This is not required by the other methods and has been omitted from the laboratory's procedure.

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Work Instruction
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Calculation of Total Chlorine / Total Halogen

Method: SW-846 5050 in conjunction with SW-846 9056A or 9251

Summary of Procedure:

Total chlorine / total halogens (as total chlorine) in a sample is determined by oxygen bomb combustion using SOP SA-GE-010: *Bomb Preparation*. The sample is oxidized by electrical ignition in a bomb containing oxygen under pressure. Under these conditions, chlorine is converted to inorganic chloride and recovered in the DI water bomb washings. The chloride is quantitated using either ion chromatography (SOP SA-GE-115: *Ion Chromatography*) or Konelab (SOP SA-GE-001: *Measurement of Analytes using the Konelab Analyzer*).

Two fundamental conditions must apply before a sample can be considered to have combusted:

1. Some part of the sample must be heated to its ignition temperature to start the combustion. Then in burning, the sample must liberate sufficient heat to support its own combustion.
2. The combustion must produce enough turbulence within the bomb to bring oxygen into the fuel cup for burning the sample completely.

Calculation:

The calculation of Total Chlorine is performed by the LIMS system.

$$Total\ Halogen, mg/kg\ dw = \frac{A \times B \times C}{D \times E}$$

Total Chlorine / Total Halogens (as Total Chlorine)

where

A = chloride concentration determined by IC or Konelab in mg/L
B = final volume of bomb washings in liters (0.10L)
C = 1000g/kg conversion factor.
D = sample weight in grams
E = decimal fraction of the percent solids

**NOTE-- Oil matrices will not have a percent solids, therefore the E variable is dropped from the calculation, and the total halogen is reported as mg/kg as is.

Quality Control:

This procedure is a calculation only; therefore, there are no QC items associated. Formal detection limit studies, as described in 40CFR Part 136B and SOP SA-QA-07: *Determination of Detection Limits,* are not required.

Formal demonstrations of capability (DOCs) are described in section 12.2 of SOP SA-GE-010.

Attachment 1c
SOP Summary
Ferrous Iron and Ferric Iron

1.0 Summary

Ferrous iron is determined by reaction with phenanthroline in acid solution producing an orange color with a maximum absorbance of 510nm.

Ferric iron is performed via the calculation below. The Total Iron result is obtained via ICP and/or ICP/MS analysis in accordance with SOP SA-ME-070 or SA-ME-074.

Ferric Iron = Total Iron – Ferrous Iron

2.0 Interferences

The reaction of ferrous iron with 1,10-phenanthroline is free from common interferences. Long storage times or exposure of samples to light must be avoided. Protect from light and analyze as soon as possible.

3.0 Standards and Reagents

3.1 Purchased Reagents

Hydrochloric Acid (HCl) – concentrated, A.C.S. grade
Storage: Acid resistant floors and good drainage. Store away from sunlight, heat and water.

Glacial Acetic Acid (CH_3COOH) – concentrated, A.C.S. grade
Storage: Acid resistant floors and good drainage. Protect from freezing. Store away from sunlight, heat, and ignition sources.

Ammonium Acetate ($NH_4C_2H_3O_2$) – reagent grade

1,10-phenanthroline monohydrate ($C_{12}H_8N_2 \cdot H_2O$) – reagent grade

3.2 Prepared Reagents

Hydrochloric Acid (1:1) – Slowly add 500mL concentrated HCl to about 400mL DI Water in a 1-L volumetric flask, cool and dilute to volume with DI water. This solution must be prepared in a hood.
Expiration: parent's expiration date or within 1 year of preparation, whichever comes first

Ammonium Acetate Buffer Solution – Dissolve 150g $NH_4C_2H_3O_2$ into 150mL DI Water. Add 700mL concentrated glacial acetic acid. Note: Because $NH_4C_2H_3O_2$ contains a significant amount of iron, new reference standards must be made with each buffer preparation.
Expiration: 5 years (not to exceed expiration of parent reagents).

Phenanthroline Solution (Color Reagent) – Dissolve 100mg $C_{12}H_8N_2 \cdot H_2O$ into 100mL of reagent water. Add 2 drops of concentrated HCl. Discard this solution if it darkens.
Expiration 1 year.

Diluent – 30ml of reagent water acidified to pH <2.0 with 1:1 HCl. For use in Kone instrument.

3.3 Standards

Ferrous Ammonium Sulfate [$Fe(NH_4)_2(SO_4)_2 \cdot 6H_2O$] – reagent grade; Note: The laboratory utilizes two independent lots of ferrous ammonium sulfate. One lot (Mallinckrodt) is utilized as the primary source for calibration standards, laboratory control samples, and matrix spikes. The second lot (Hach) is utilized as the secondary source for the initial calibration verification (ICV).

Ferrous Iron Solution (1000mg/L) – Dissolve 0.7022g $Fe(NH_4)_2(SO_4)_2 \cdot 6H_2O$ (primary source) into 50mL of reagent water in a 100mL volumetric flask. Acidify to pH <2 with 1:1 HCl and dilute to volume with reagent water.

Ferrous Iron Calibration Standards

Standard Level	TALS Reagent Name	Parent Standard	Aliquot (mL)	Final Volume (mL)	Final Concentration (mg/L)
1	FeCal7	1000mg/L	0.0050	100	0.050
2	FeCal6	1000mg/L	0.010	100	0.10
3	FeCal5	1000mg/L	0.030	100	0.30
4	FeCal4	1000mg/L	0.050	100	0.50
5	FeCal3	1000mg/L	0.10	100	1.0
6	FeCal2	1000mg/L	0.30	100	3.0
7	FeCal1	1000mg/L	0.50	100	5.0

Dilute to volume in reagent water.
Expiration: one month.

Ferrous Iron Matrix Spike Solution (40mg/L) – Dilute 4mL of the 1000mg/L Fe+2 Cal Stk to a final volume of 100mL with DI water.
Expiration: one month.

Ferrous Iron Laboratory Control Sample (2.0mg/L) – Dilute 5.0 mL of the 40mg/L matrix spike solution to a final volume of 100mL with DI water.
Expiration: one month.

Ferrous Iron ICV Stock Solution (1000mg/L) – Dissolve 0.7022g $Fe(NH_4)_2(SO_4)_2 \cdot 6H_2O$ (secondary source) into 50mL of reagent water in a 100mL volumetric flask. Acidify to pH <2 with 1:1 HCl and dilute to volume with reagent water.

Ferrous Iron Initial Calibration Verification (ICV) (3.0 mg/L) – Dilute 0.30mL of the 1000mg/L Fe+2 ICVSTK to a final volume of 100mL with DI water.
Expiration: one month.

4.0 Sample Preparation

4.1 Aqueous Samples

10mL of sample are filtered through a 0.45 micron filter prior to analysis. Add approximately 0.05mL (1 drop) of 1:1 HCl to adjust the sample to a pH 1.5–2.5. Use narrow range pH paper during adjustment to verify the proper pH is achieved.

Prepare QC samples in the same manner as field samples as follows:

- Continuing Calibration Verification- prepared as described above.
- Continuing Calibration Blank- reagent water acidified to a PH <2.0 with 1:1 HCl
- Method Blank- reagent water acidified to a PH <2.0 with 1:1 HCl
- Laboratory Control Sample – prepared as described above.
- Low-Level Laboratory Control Sample (Drinking Water only) – Lowest level standard of the initial calibration.
- Matrix Spike/Matrix Spike Duplicate – Add 0.50mL of Ferrous Iron Matrix Spike Solution to 10mL of the chosen sample
- .

4.2 Soil samples are not performed for this analysis.

5.0 Method-Specific QC Requirements

5.1 Linear Calibration Range required to be performed initially.

5.2 Reporting limit verification required for every batch. An RL check standard must be analyzed at the beginning of each sequence.

5.3 Analyze CCV immediately after ICAL (in addition to the ICV). Recovery must be within 5% of true value.

5.4 Any sample containing detection greater than 90% of the determined LDR must be diluted to until the raw result is less than 90% of the determined LDR.

6.0 Method-specific Modifications and/or Clarifications

6.1 The preparation of the Ferrous Iron Solution (1000mg/L) has been revised from the reference method to correspond to that outlined in the Konelab Application Notes.

Attachment 1d
SOP Summary
Hexavalent Chromium and Trivalent Chromium

1.0 Summary

The sample is acidified to pH 2 (+/-0.5) and 1,5-diphenylcarbazide (a color reagent) is added. The intensity of the purple color, measured at 540nm, is proportional to the concentration of hexavalent chromium present in the sample.

Trivalent Chromium is performed via the calculation below. The Total Chromium result is obtained via ICP and/or ICP/MS analysis in accordance with SOP SA-ME-070 or SA-ME-074.

Trivalent Chromium = Total Chromium – Hexavalent Chromium

2.0 Interferences

Iron in concentrations of greater than 1mg/L may produce a yellow color, but this is usually not significant when measured at 540nm. Hexavalent molybdenum and mercury salts react to form colored complexes with the color reagent, but the intensities of the colors produced are much lower than for chromium at the specified pH. Mercury and molybdenum concentrations of up to 200mg/L can be tolerated. Vanadium interferes more strongly, but concentrations of up to 10 times that of chromium can be tolerated.

All samples analyzed must be adjusted to a pH range of 1.5-2.5 with sulfuric acid prior to analysis to minimize interference and allow proper color development.

After acidification, soil digestates should be purged with helium to eliminate excess carbon dioxide gas. Bubble formation in the cuvettes can interfere with readings.

The recovery of soluble forms of hexavalent chromium may be low where the sample matrix contains substances that are readily oxidized by hexavalent chromium (a reducing matrix). The measurement pH and oxidation-reduction potential may provide information about the ability of hexavalent chromium to persist in the sample.

3.0 Standards and Reagents

3.1 Purchased Reagents

Phosphoric acid (H_3PO_4) – concentrated, A.C.S. grade
Storage: Acid resistant floors and good drainage; away from sunlight, heat and water.

Sulfuric Acid (H_2SO_4) – concentrated, A.C.S. grade
Storage: Acid resistant floors and good drainage; away from sunlight, heat, and water.

Acetone – concentrated, A.C.S. grade
Storage: Protect from heat, sparks, and flame in a flammable storage area.

1,5-diphenylcarbazide – reagent grade
Storage: Protect from light and store in desiccator.

3.2 Prepared Reagents

Sulfuric Acid (50% v/v) – Slowly add 50mL concentrated H_2SO_4 to about 40mL reagent water in a 100mL volumetric flask, cool and dilute to volume with reagent water. This solution must be prepared in a hood.

Color Reagent – Transfer 0.1250g of 1,5-diphenylcarbazide to 25mL volumetric flask. Add 10mL acetone; swirl to dissolve. Dilute to volume with acetone. Discard if reagent becomes discolored.
Storage: Store in the dark in a tightly capped.
Expiration: 1 Week

Diluent: 30mL of reagent water acidified to a pH <2.0 with 1:1 H_2SO_4. For use in KONE instrument.

3.3 Standards

Hexavalent Chromium Initial Calibration Verification Stock (1000mg/L) – Purchased from Sigma Aldrich.
Expiration: Opened: 1 year or the manufacturer's expiration date, whichever is shorter

Hexavalent Chromium Solution (10mg/L) – Purchased from CPI. Used to prepare calibration standards and matrix spike/ matrix spike duplicates (MS/MSD)
Storage: Opened: 1 year or the manufacturer's expiration date, whichever is shorter

Hexavalent Chromium Calibration Standards

Standard Level	TALS Reagent Name	Parent Standard	Aliquot (mL)	Final Volume (mL)	Final Concentration (mg/L)
1	CR6 CAL 6	10mg/L Cr6 Solution	0.10	100	0.010
2	CR6 CAL 5	10mg/L Cr6 Solution	0.25	100	0.025
3	CR6 CAL 4	10mg/L Cr6 Solution	0.50	100	0.050
4	CR6 CAL 3	10mg/L Cr6 Solution	1.0	100	0.10
5	CR6 CAL 2	10mg/L Cr6 Solution	3.0	100	0.30
6	CR6 CAL 1	10mg/L Cr6 Solution	5.0	100	0.50

Dilute to volume in reagent water. Expiration: 1 month from preparation

Hexavalent Chromium Laboratory Control Sample (LCS) (0.20mg/L) – Dilute 2mL of the 10mg/L Hexavalent Chromium Solution to 100mL with DI water. (Used as CCV and LCS)
Expiration: 1 month from preparation

Hexavalent Chromium ICV Intermediate Standard (10mg/L) – Dilute 1mL of the 1000mg/L CR6 ICV Stock standard to 100 mL with DI water.
Expiration: 1 month from preparation

Hexavalent Chromium Initial Calibration Verification (0.30mg/L) – Dilute 3mL of the 10mg/L CR6 ICV Intermediate standard to 100 mL with DI water.
Expiration: 1 month from preparation

4.0 Sample Preparation

4.1 Aqueous Samples

Aqueous samples are filtered through a 0.45 micron filter prior to analysis. Typically 20 mL of sample are filtered. Add approximately 0.05mL (1 drop) of concentrated H_3PO_4. Adjust the pH of 20mL of sample to pH 1.5–2.5 with 50% H_2SO_4. Use narrow range pH paper during adjustment to verify the proper pH is achieved.

Note: NCMs must be initiated for samples originating in WV that have not been field filtered.

Prepare QC samples in the same manner as field samples as follows:

- Continuing Calibration Verification- prepared as described above
- Continuing Calibration verification Blank – reagent water
- Method Blank – reagent water
- Laboratory Control Sample (LLCS) – prepared as described above.
- Low-Level Laboratory Control Sample (drinking water) – Lowest level standard of the initial calibration
- Matrix Spike/Matrix Spike Duplicate – Add 0.20mL of Hexavalent Chromium Solution (10mg/L) to 10mL of the chosen sample.

5.0 Method-Specific QC Requirements

SM3500-Cr B – Linear Dynamic Range (LDR) study initially. Reporting limit verification per batch. Any sample containing a detection greater than 90% of the determined LDR must be diluted to until the raw result is less than 90% of the determined LDR.

A CCV must be analyzed immediately following the initial calibration (in addition to the ICV). This CCV must recover within 5% of the true value.

An RL check sample must be analyzed at the beginning of each sequence.

6.0 Method-Specific Modifications and Clarifications

6.1 The SM3500-Cr B reference method states to adjust sample pH to 2 +/-0.5 with 0.2N sulfuric acid. The EPA 7196A reference method states to adjust sample pH to 2 +/-0.5 with 10%sulfuric acid. Adjusting the pH with such a weak solution requires adding >5% volume and therefore diluting the sample. As such, the laboratory has chosen to use 50% sulfuric to adjust the sample pH.

6.2 The EPA 7196A reference method states to add the color reagent, then adjust the sample pH to 1.5-2.5. The Konelab instrument automatically adds the color reagent; therefore, the order of these steps is reversed (i.e., the laboratory adjusts the pH then adds the color reagent). Correspondence with EPA indicates this is an acceptable modification to the method.

Attachment 1e
SOP Summary
Ortho-phosphate

1.0 Summary

Orthophosphate reacts with ammonium molybdate and antimony potassium tartrate under acidic conditions to form a complex which, when reduced with ascorbic acid, produces an intense blue color. The color is measured photometrically at 880nm and the absorbance is proportionate to the amount of orthophosphate in the sample.

2.0 Interferences

Samples that are blue in color may interfere. Samples with high concentrations of arsenic may interfere.

3.0 Reagents and Standards

3.1 Purchased Reagents

Antimony Potassium Tartrate [$K(SbO)C_4H_4O_6 \cdot ½\ H_2O$] – reagent grade

Ammonium Molybdate [$(NH_4)_6Mo_7O_{24} \cdot 4H_2O$] – reagent grade. Storage: Isolated from sources of heat and moisture.

Sulfuric Acid (H_2SO_4) – concentrated, A.C.S. grade
Storage: Acid resistant floors and good drainage. Away from sunlight, heat, and water.

Ascorbic Acid – reagent grade
Storage: Isolate from sources of heat and fire.

3.2 Prepared Reagents

Sulfuric acid solution 5N: Slowly add 70mL of concentrated H_2SO_4 to approximately 400mL of reagent water. Cool to room temperature and dilute to 500mL with reagent water.

Antimony Potassium Tartrate Solution – Dissolve 3.0g $K(SbO)C_4H_4O_6 \cdot ½\ H_2O$ in approximately 500mL of reagent water. Dilute to 1000mL with reagent water.
Storage: Store at 4°C in a dark, glass-stoppered bottle.

Ammonium Molybdate Solution – Dissolve 40g $(NH_4)_6Mo_7O_{24} \cdot 4H_2O$ in approximately 800mL of reagent water. Dilute to 1L with reagent water.
Storage: Store at 4°C in a plastic bottle.

Color Reagent 2 – Ascorbic Acid Solution (0.1M) – Dissolve 1.76g ascorbic acid in approximately 50mL of reagent water. Dilute to 100mL with reagent water.
Expiration: Prepare fresh daily.

Color Reagent 1 – Add 75mL Ammonium Molybdate Solution to 250mL 5N sulfuric acid. Mix well. Add 25mL Antimony Potassium Tartrate Solution to this solution and mix well.
Expiration: 6 months

Combined Color Reagent – Add 14mL of Color Reagent 1 to 6mL of Color Reagent 2. Mix well.
Expiration: Solution is only good for 4 hours from time of prep.

3.2 Standards

Phosphate Calibration Standard (1000mg/L) – Purchased from NSI Lab Solutions.
Storage: Away from sunlight.
Expiration: Opened: 1 year or the manufacturer's expiration date, whichever is shorter.

Ortho-phosphate Initial Calibration Verification (ICV) Stock (1000mg/L) – Purchased from Inorganic Ventures.
Storage: Away from sunlight.
Expiration: Opened: 1 year or the manufacturer's expiration date, whichever is shorter.

Ortho-phosphate Calibration Standards:

Standard Level	TALS Reagent Name	Parent Standard	Volume of Parent Standard (mL)	Final Volume (mL)	Final Concentration (mg/L)
7	PO4 CAL 1	PO4 100ppm	2	100	2.0
6	PO4 CAL 2	PO4 100ppm	1.6	100	1.6
5	PO4 LCS	PO4 100ppm	1.2	100	1.2
4	PO4 CAL 3	PO4 100ppm	0.8	100	0.80
3	PO4 CAL 4	PO4 100ppm	0.4	100	0.40
2	PO4 CAL 5	PO4 100ppm	0.2	100	0.20
1	PO4 CAL 1 PO4 RL	PO4 100ppm	0.050	100	0.050

Dilute to volume in reagent water.

Ortho-phosphate Laboratory Control Sample (1.2mg/L) – Dilute 1.2mL of 100mg/L Orthophosphate Matrix Spike Solution to a final volume of 100mL with reagent water. (Also used as CCV)
Expiration: 1 month

Ortho-phosphate Initial Calibration Verification (1.0mg/L) – Dilute 1.0mL of the 1000mg/L Ortho-phosphate ICV Stock to a final volume of 1000mL with reagent water.
Expiration: 1 month.

Ortho-phosphate Matrix Spike Solution (100mg/L) – Dilute 10mL of the 1000mg/L Phosphate Calibration Standard to a final volume of 100mL with reagent water.
Expiration: 1 month.

4.0 Sample Preparation

4.1 Aqueous Samples

Aqueous samples are routinely filtered. Filter samples through a 0.45 micron pp filter.

Note: Acidic samples must be neutralized with 1N NaOH prior to analysis.

4.1.1 QC Sample Preparation

-Continuing Calibration Verification – Prepared as described above
-Continuing Calibration Blank – reagent water
-Method Blank – reagent water
-Laboratory Control Sample – Prepared as outlined above
-Low-Level Laboratory Control Sample (Drinking Water only) – Lowest level standard of the initial calibration
-Matrix Spike/Matrix Spike Duplicate – Add 0.10mL of 100mg/L Matrix Spike Solution to 10mL of the chosen sample. The concentration of the MS/MSD must equal the concentration of the LCS.

Prepare all QC in the same manner as samples.

4.2 Soil Samples

Create a DI Leach batch in LIMs.

Weigh approximately 5g of sample into a 125mL screw-cap plastic bottle. Record the weight to the nearest 0.1g. Add 100mL of reagent water to each sample container and place in the tumbler.

Rotate for 2 hours +/- 30 minutes.

Remove the containers from the extractor and allow the leachates to settle. Filter the extract using a syringe filter with a 0.45um pore size filter and analyze the extracts as liquid samples.

Add one drop of phenolphthalein indicator solution to approximately 50mL of leachate. If a red color develops add 10N sulfuric acid dropwise until the color dissipates.

Note: Acidic samples must be neutralized with 1N NaOH prior to analysis.

4.2.1 QC Sample Preparation

Method Blank – Weigh approximately 20g of Teflon chips into a 125mL screw-cap plastic bottle. Record the weight to the nearest 0.1g.

Laboratory Control Sample – Weigh approximately 5g of Teflon chips into a 125mL screw-cap plastic bottle. Record the weight to the nearest 0.1g. Add 1.0mL of 100mg/L Matrix Spike Solution to the extraction bottle.

Matrix Spike – Weigh an additional 5g aliquot of one of the samples for the MS. Add 1.0mL of 100mg/L Matrix Spike Solution to the extraction bottle. One sample must be chosen from samples 1-10 and one sample from samples 11-20. The concentration of the MS must equal the concentration of the LCS.
Sample Duplicate – If precision is required, weigh one additional 20g aliquot of one of the samples for the SD.

Prepare all QC in the same manner as samples.

5.0 Method-Specific QC Requirements

SM4500-P F requires the MDL study to be analyzed over 3-5 days.

SM4500-P F recommends the matrix spike concentration be rotated.

Attachment 1f
SOP Summary
Sulfate, Total Sulfur, and Heat of Combustion (BTU)

1.0 Summary

Sulfate ion is precipitated in a strongly acidic medium with Barium Chloride. The resulting turbidity is measured photometrically at 405nm and compared with appropriate standard solutions.

Refer to SOP SA-GE-010 for information on the preparation procedures for Total Sulfur and Heat of Combustion (BTU). The attached Work Instruction provides the procedural and calculation information.

2.0 Interferences

Silica at a concentration above 500mg/L in the sample will interfere. Color and turbidity due to the sample matrix can cause positive interferences that must be accounted for by the analysis of sample blanks (i.e. samples analyzed without adding reagents). The absorbance or turbidity of the sample blank is subtracted from the absorbance or turbidity of the treated sample, and the concentration is determined from the corrected response.

3.0 Reagents and Standards

3.1 Purchased Reagents

Barium Chloride ($BaCl_2$) – reagent grade
Storage: Store in a cool, dry, ventilated area. Isolate from heat, moisture, and incompatibles.

Sodium Chloride (NaCl) – reagent grade
Storage: Store in a cool, dry, ventilated area.

Gelatin – (TALS – Gelatin)

Hydrochloric Acid (HCl) – concentrated, A.C.S. grade
Storage: Store in a cool, dry, ventilated area with acid resistant floors and good drainage. Store away from sunlight, heat, water, and incompatible materials.

3.2 Prepared Reagents

Precipitating Solution – In a 100mL volumetric flask, dissolve 1.0g $BaCl_2$, 2.0g NaCl, and 0.1g of Gelatin in approximately 30mL reagent water. Stir until completely dissolved. Carefully add 0.5mL concentrated HCl and dilute to volume with reagent water.
Expiration: 3 months

3.3 Purchased Standards

Sulfate Calibration Stock Solution (1000mg/L) – Purchased from Inorganic Ventures.
Expiration: Manufacturer's expiration date.

Sulfate Calibration Standards

Standard Level	TALS Reagent Name	Parent Standard	Volume of Parent Standard (mL)	Final Volume (mL)	Final Concentration (mg/L)
6	SO4 CAL 6	1000mg/L	3.5	100	35
5	SO4 CAL 5	1000mg/L	3.0	100	30
4	SO4 CAL 4	1000mg/L	2.5	100	25
3	SO4 CAL 3	1000mg/L	1.5	100	15
2	SO4 CAL 2	1000mg/L	1.0	100	10
1	SO4 CAL 1	1000mg/L	0.5	100	5

Dilute to volume in reagent water.

Sulfate Laboratory Control Sample (20mg/L) – Dilute 5mL of the 1000mg/L SO4 Cal stock to 250mL final volume.
Expiration: 3 months

Sulfate ICV Stock (1000 mg/L) – Purchased from NSI.
Expiration: Manufacturer's expiration date.

Sulfate Initial Calibration Verification (20mg/L) Add 2.0mL Sulfate Calibration Stock Solution to a 100mL volumetric flask. Dilute to mark with DI water.
Expiration: 3 months

Sulfate Matrix Spike Solution (1000mg/L) – This is the same as the SO4 Calibration Stock Solution
Expiration: Manufacturer's expiration date.

4.0 Sample Preparation

4.1 Aqueous Samples

Aqueous samples are routinely analyzed with no sample preparation.

4.1.1 QC Sample Preparation

Method Blank – reagent water

Laboratory Control Sample – Prepared as outlined above.

Low-Level Laboratory Control Sample (drinking water) – Lowest level standard of the initial calibration

Matrix Spike/Matrix Spike Duplicate – Add 0.2mL of Sulfate Matrix Spike Solution to 10mL of the chosen sample.

Prepare all QC in the same manner as samples

4.2 Soil Samples

Create a DI Leach batch in LIMS.

Weigh approximately 5g of sample into a 125mL screw-cap plastic bottle. Record the weight to the nearest 0.1g. Add 100mL of reagent water to each sample container and place in the tumbler.

Rotate for 2 hours +/- 15 minutes.

Remove the containers from the extractor and allow the leachates to settle. Filter the extract using a syringe filter with a 0.450-um pore size filter and analyze the extracts as liquid samples.

4.2.1 QC Sample Preparation
Method Blank – Weigh approximately 5g of Ottawa sand into a 125mL screw-cap plastic bottle. Record the weight to the nearest 0.1g.

Laboratory Control Sample – Weigh approximately 5g of Ottawa sand into a 125mL screw-cap plastic bottle. Record the weight to the nearest 0.1g. Add 2.0mL of Sulfate Cal Stock (1000mg/L) solution and 98mL of DI water.

Matrix Spike/Matrix Spike Duplicate – Weigh two additional 5g aliquots of one of the samples for the MS and MSD. Add 2.0mL of Sulfate Cal Stock (1000mg/L) solution and 98mL of DI water to each bottle.

Prepare all QC in the same manner as samples.

5.0 Method-Specific QC Requirements

A CCV must be analyzed every 3-4 sample analyses (EPA 375.4 Section 6.3.4 and EPA 9038 Section 7.3.4).

6.0 Method-Specific Modifications and Clarifications

6.1 The EPA 375.4 reference method states to measure turbidity every 30 seconds for 1-5 minutes after the addition of barium chloride, with the maximum turbidity reading used for determining the Sulfate concentration. The Konelab instrument performs parallel analyses of each sample - one for performing turbidity measurements and one for sulfate determination. The turbidity measurements are made 200 seconds after the color reagent is added and every 28 seconds after that for 240 seconds. The turbidity absorbances are presented on the raw data to indicate if the sample is settling out but do not affect how the sulfate result is determined. The sulfate determination is made 300 seconds after the color reagent is added. Although the Konelab instrument will allow multiple sulfate readings to be made, the instrument cannot select the result associated with the highest turbidity; rather, it will perform an average of the results obtained. As this instrument configuration cannot be changed to use the sulfate reading taken from the highest turbidity replicate.

6.2 The Sulfate Precipitating Solution is prepared in accordance with the Konelab Application Notes which differs from the information provided in the reference method.

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Calculation of Total Sulfur

Method: SW-846 5050 in conjunction with SW-846 9038

Summary of Procedure:

Total sulfur in a sample is determined by oxygen bomb combustion using SOP SA-GE-010: *Bomb Preparation*. The sample is oxidized by electrical ignition in a bomb containing oxygen under pressure. Under these conditions, sulfur is converted to inorganic sulfate and recovered in the DI water bomb washings. Sulfate content is quantitated by using the Konelab (SOP SA-GE-001: *Measurement of Analytes using the Konelab Analyzer)*.

Two fundamental conditions must apply before a sample can be considered to have combusted:

1. Some part of the sample must be heated to its ignition temperature to start the combustion. Then in burning, the sample must liberate sufficient heat to support its own combustion.
2. The combustion must produce enough turbulence within the bomb to bring oxygen into the fuel cup for burning the sample completely.

Calculation:

The calculation of Total Chlorine is performed by the LIMS system.

$$Total\,Sulfur, mg/kg\,dw = \frac{A \times B \times C}{D \times E}$$

Total Sulfur

where

A = mg S/L (mg SO4/L determined by Konelab divided by 3)
B = final volume of bomb washings in liters (0.10L)
C = 1000g/kg conversion factor.
D = sample weight in grams
E = decimal fraction of the percent solids

**NOTE-- Oil matrices will not have a percent solids, therefore the E variable is dropped from the calculation, and the total halogen is reported as mg/kg as is.

Quality Control:

This procedure is a calculation only; therefore, there are no QC items associated. Formal detection limit studies, as described in 40CFR Part 136B and SOP SA-QA-07: *Determination of Detection Limits*, are not required.

Formal demonstrations of capability (DOCs) are described in section 12.2 of SOP SA-GE-010.

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Calculation of Heat of Combustion

Method: ASTM D240-87

Summary of Procedure:

The gross heat of combustion in a sample is determined by oxygen bomb combustion using SOP SA-GE-010: *Bomb Preparation*. The sample is oxidized by electrical ignition in a bomb containing oxygen under pressure. Accurate time and temperatures are measured throughout the test period. Under these conditions, sulfur is converted to inorganic sulfate and recovered in the DI water bomb washings. The oxygen bomb washings are titrated for nitric acid formation then analyzed by Konelab (SOP SA-GE-001: *Measurement of Analytes using the Konelab Analyzer)* for sulfate content. Variables from the test period and analytical constants are used to calculate the gross heat of combustion (BTU/lb).

Two fundamental conditions must apply before a sample can be considered to have a heat of combustion:

1. Some part of the sample must be heated to its ignition temperature to start the combustion. Then in burning, the sample must liberate sufficient heat to support its own combustion.
2. The combustion must produce enough turbulence within the bomb to bring oxygen into the fuel cup for burning the sample completely.

Calculation:

A spreadsheet is used to determine the final heat of combustion value.

Energy (Water) Equivalent, W

$$W = \frac{[(H * m) + e1 + e3]}{t}$$

where:

W = energy (water) equivalent of the calorimeter in cal/ °C
H = heat of combustion of standard benzoic acid. (6318 cal/g)
m = weight (mass) of the benzoic acid standard in grams
e1 = correction for heat of formation of nitric acid in cal
e3 = correction for heat of combustion of firing wire in cal

Temperature Rise, t

$$t = tc - ta$$

where:

t = temperature rise in °C
tc = temperature at beginning of period in which the rate of temperature change with time has become constant in °C
ta = temperature at time of firing in °C

Gross Heat of Combustion (BTU/lb), H

$$H = \frac{[(t * W) - e1 - e2 - e3]}{m} * 1.8$$

where:

H =	**gross heat of combustion in BTU/lb**
t =	**temperature rise in °C**
W =	**energy equivalent in cal/ °C**
e1 =	**correction for heat of formation of nitric acid in cal**
e2 =	**correction for heat of formation of sulfuric acid in cal**
e3 =	**correction for heat of combustion of firing wire in cal**
m =	**weight (mass) of sample in grams**
1.8 =	**conversion factor from cal/g to BTU/lb**

Quality Control:

This procedure is a calculation only; therefore, there are no QC items associated. Formal detection limit studies, as described in 40CFR Part 136B and SOP SA-QA-07: *Determination of Detection Limits,* are not required.

Formal demonstrations of capability (DOCs) are described in section 12.2 of SOP SA-GE-010.

The spreadsheet used to determine the heat of combustion result has been verified and protected.

Attachment 2:
Sample Collection, Preservation, and Holding Time Table

Fraction	Matrix	Routine Sample Container	Routine Sample Size	Chemical Preservation	Thermal Preservation	Dechlorination Agent	Holding Time
Ammonia	Water	250mL plastic	40mL	1mL 1:1 H_2SO_4 pH <2	0-6°C[1]	Sodium Thiosulfate	28 Days
Ammonia	Soil	8oz Plastic	20g	None	0-6°C[1]	None	28 Days[2]
BTU	Water	4oz glass soil jar	1g	None	0-6°C[1]	None	28 Days
BTU	Soil	4oz glass soil jar	1g	None	0-6°C[1]	None	28 Days[2]
Chloride	Water	125mL plastic	10mL	None	0-6°C[1]	None	28 Days
Chloride	Soil	8oz Plastic	5g	None	0-6°C[1]	None	28 Days[2]
Chloride, Total	Water	125mL plastic	1g	None	0-6°C[1]	None	28 Days
Chloride, Total	Soil	8oz Plastic	1g	None	0-6°C[1]	None	28 Days[2]
Ferrous Iron	Water	125mL plastic	10mL	None	0-6°C[1]	None	15 Minutes[3]
Ferric Iron (Calc)	Water	125mL plastic	10mL	None	0-6°C[1]	None	15 Minutes[3]
Hexavalent Chromium	Water	125mL plastic	10mL	None	0-6°C[1]	None	24 Hours[4]

Fraction	Matrix	Routine Sample Container	Minimum Sample Size	Chemical Preservation	Thermal Preservation	Dechlorination Agent	Holding Time
Trivalent Chromium (Calc)	Water	125mL plastic	10mL	None	0-6°C[1]	None	24 Hours
Ortho-Phosphate	Water	125mL plastic	10mL	None	0-6°C[1]	None	48 Hours
Ortho-Phosphate	Soil	8oz Plastic	20g	None	0-6°C[1]	None	48 Hours[2]
Sulfate	Water	125mL plastic	10mL	None	0-6°C[1]	None	28 Days
Sulfate	Soil	8oz Plastic	5g	None	0-6°C[1]	None	28 Days[2]
Sulfur, Total	Water	125mL plastic	1g	None	0-6°C[1]	None	28 Days
Sulfur, Total	Soil	8oz Plastic	1g	None	0-6°C[1]	None	28 Days[2]

[1]Samples must be maintained at 0-6°C, with no frozen samples.
[2] Inclusive of digestion and analysis.
[3] Due to the nature of this analysis, this test is considered a field test. No definitive holding time is specified in the reference methods; however, the Methods Update Rule of 40CFR Part 136 identifies a 15 minute holding time for this method. Although these samples will be outside holding time upon receipt to the laboratory, an effort should be made to analyze as soon as possible after receipt.
[4] Clean Water Act (CWA) sampling requirements state that field filtration must be performed within 15minutes of sampling to prevent interconversion of chromium species. NCMs must be initiated for samples when the COC does not indicate field filtration was performed.

Note: NCMs must be initiated for samples collected in improper containers and containing improper or insufficient preservatives and/or de-chlorination agents.

Attachment 3a: Batch QC Frequency

Fraction	Method	MB Frequency[1]	LCS Frequency[1]	LCSD Frequency[1]	LLCS Frequency[1]	MS Frequency[1]	MSD Frequency[1]	SD Frequency[1]
Ammonia	EPA 350.1	1 per batch	1 per batch	1 per batch[2]	1 per batch[3]	10% of samples	1 per batch	None
Ammonia	SM4500-NH3 G	1 per batch	1 per batch	1 per batch[2]	1 per batch[3]	1 per batch	1 per batch	1 per batch
BTU	ASTM D240-87	None	1 per batch	None	None	None	None	1 per 10
Chloride	EPA 325.2	1 per batch	1 per batch	1 per batch[2]	1 per batch[3]	1 per batch	1 per batch	None
Chloride	EPA 9251	1 per batch	1 per batch	1 per batch[2]	None	10% of samples	10% of samples	None
Chloride	SM4500-Cl E	1 per batch	1 per batch	1 per batch[2]	1 per batch[3]	1 per batch	1 per batch	1 per batch
Chlorine	EPA 5050/EPA 9251	1 per batch	1 per batch	None	None	None	None	1 per 10
Ferrous Iron	SM3500-Fe B	1 per batch	1 per batch	1 per batch[2]	None	1 per batch	1 per batch	None
Ferric Iron (Calc)	SM3500-Fe B	NA	NA	NA	NA	NA	NA	NA
Hexavalent Chromium	SM3500-Cr B	1 per batch	1 per batch	1 per batch[2]	None	1 per batch	1 per batch	None
Hexavalent Chromium	SM3500-Cr D	1 per batch	1 per batch	1 per batch[2]	None	1 per batch	1 per batch	None
Hexavalent Chromium	EPA 7196A	1 per batch	1 per batch	1 per batch[2]	None	10% of samples	1 per batch	None
Ortho-phosphate	EPA 365.1	1 per batch	1 per batch	1 per batch[2]	1 per batch[3]	10% of samples	1 per batch	None
Ortho-phosphate	SM4500-P F	1 per batch	1 per batch	1 per batch[2]	1 per batch[3]	1 per batch	1 per batch	1 per batch
Sulfate	EPA 375.4	1 per batch	1 per batch	1 per batch[2]	1 per batch[3]	1 per batch	1 per batch	None
Sulfate	EPA 9038	1 per batch	1 per batch	1 per batch[2]	None	10% of samples	10% of samples	None
Total Sulfur	EPA 5050/EPA 9038	1 per batch	1 per batch	None	None	None	None	1 per 10
Trivalent Chromium (Calc)	SM3500-Cr B	NA	NA	NA	NA	NA	NA	NA
Trivalent Chromium (Calc)	SM3500-Cr D	NA	NA	NA	NA	NA	NA	NA
Trivalent Chromium (Calc)	EPA 7196A	NA	NA	NA	NA	NA	NA	NA

[1]Batch Definition = Up to 20 field samples prepared and/or analyzed together within 24 hours
[2]If insufficient sample provided for MS/MSD.
[3]For Drinking Water analyses only.

Attachment 3b: QC Summary

QC Item	Frequency	Criteria	Corrective Action
Initial Calibration (ICAL) Multi-point: Minimum 3 standards and 1 blank	Daily	Correlation ≥0.995 And each point on curve must be within +/- 10% of true value	Recalibrate Note: SM3500-Fe B and SM3500-Cr B do not allow quadratic curves
Second Source Initial Calibration Verification (ICV)	Immediately following the initial calibration; at a minimum, quarterly	Within ±10% of the true value	Recalibrate
Continuing Calibration Verification (CCV)	SM3500-Fe B, SM3500-Cr B: Immediately following ICAL Sulfate: At the beginning and end of the analysis, and after every 3-4 samples All Others: At the beginning and end of the analysis, and after every 10 samples	SM3500-Fe B, SM3500-Cr B: Within ±5% of the true value All Others: Within ±10% of the true value	Terminate the analysis, fix the problem and reanalyze the previous samples.
Initial Calibration Blank (ICB)	After ICV	SM3500-Fe B, SM3500-Cr B: <MDL Others: < ½ RL	Terminate the analysis, correct problem and reanalyze the previous 10 samples.
Continuing Calibration Blank (CCB)	After every CCV	SM3500-Fe B, SM3500-Cr B: <MDL	Terminate the analysis, correct problem and reanalyze the previous 10 samples.

QC Item	Frequency	Criteria	Corrective Action
		Others: < ½ RL	
Batch Frequency	<=20 field samples or 24 hours	Not Applicable	Not Applicable
Method Blank (MB)	One per batch	SM3500-Fe B, SM3500-Cr B: <MDL Others: < ½ RL	Evaluate according to SOP SA-QA-017
Laboratory Control Sample (LCS)	One per batch	Within limits listed in the MLG	Evaluate according to SOP SA-QA-017
Laboratory Control Sample Duplicate (LCSD)	One per batch, when insufficient sample provided for MS/MSD/SD	Within limits listed in the MLG	Evaluate according to SOP SA-QA-017
Low-Level Laboratory Control Sample (LLCS)	Drinking Water Only: One per batch	Within limits listed in the MLG	Evaluate according to SOP SA-QA-017
Reporting Limit Verification (RLV)	SM3500-Fe B, SM3500-Cr B: One per batch	Within limits listed in the MLG	Evaluate according to SOP SA-QA-017
Matrix Spike (MS)	Refer to QC Frequency Table	Within limits listed in the MLG	Evaluate according to SOP SA-QA-017
Matrix Spike Duplicate (MSD)	Refer to QC Frequency Table	Within limits listed in the MLG	Evaluate according to SOP SA-QA-017
Sample Duplicate (SD)	Refer to QC Frequency Table	Within limits listed in the MLG	Evaluate according to SOP SA-QA-017
Linear Calibration Range (LCR) (Also called Linear Dynamic Range, LDR)	Upon method/instrument set-up, and when LCR verification fails acceptance criteria.	Within ±10% of the true value	This determines the range at which the instrument is considered linear. The highest concentration of the calibration curve can not exceed the highest concentration of the LCR study.

QC Item	Frequency	Criteria	Corrective Action
Linear Calibration Range Verification (LCRV) (Also called Linear Dynamic Range Verification, LDRV)	Every 6 months	Within ±10% of the initial value	Re-perform the LCR study
Initial Demonstration of Capability (IDOC)	Initially, per analyst, per analyte/method combination	Refer to SOP SA-QA-006	Refer to SOP SA-QA-006 (Unsupervised work cannot begin until successful IDOC is obtained.)
Continuing Demonstration of Capability (CDOC)	Annually, per analyst, per analyte/method combination	Refer to SOP SA-QA-006	Refer to SOP SA-QA-006
Reporting Limit Verification (RLV)	Upon method/instrument set-up, per analyte/method/matrix combination. Then quarterly thereafter for DOD ELAP.	Refer to SOP SA-QA-007	Refer to SOP SA-QA-007
Method Detection Limit Study (MDL)	Upon method/instrument set-up, per analyte/method/matrix combination and annually thereafter EPA 365.1: Upon method/instrument set-up, per analyte/method/matrix combination and every 6 months thereafter	Refer to SOP SA-QA-007	Refer to SOP SA-QA-007

QC Item	Frequency	Criteria	Corrective Action
MDL Verification (MDLV)	Upon method/instrument set-up, per analyte/method/matrix combination. Then quarterly thereafter for methods/analytes on DOD ELAP	Refer to SOP SA-QA-007	Refer to SOP SA-QA-007

Attachment 4: Instrument Maintenance and Troubleshooting

Instrument Labeling

Each instrument must be labeled with its name or ID (e.g., MSA, ICP-D, etc.). Additionally, non-operational instruments must be isolated from service or marked as being out of service. Each piece of equipment has an "Operational / Not Operational" sticker that is used for this purpose.

Maintenance Log

A maintenance log must be established for each piece of equipment used in the laboratory. All maintenance that is performed on the instrument must be recorded in the log including:

- Type of maintenance performed (Note: This includes preventative/routine maintenance; non-routine maintenance; maintenance performed by an external vendor; updates to software versions; etc.)
- Analyst or technician performing the maintenance
- Date the maintenance was performed
- Detailed explanation of the reason for the maintenance
- resolution of the problem and return to control
- All service calls from instrument representatives
- Resolution of the problem (i.e., the Return to Control)

Preventive Maintenance

Refer to the instrument manufacturer's guides for troubleshooting items.

LABORATORY EQUIPMENT PREVENTIVE MAINTENANCE SCHEDULE								
EQUIPMENT ITEM	**Service Interval**							**SERVICE LEVEL**
	D	**W**	**M**	**Q**	**SA**	**A**	**AN**	
Sample Probe	**X**							Clean and inspect daily
Dispenser Rods			**X**					Clean and lube monthly
Fetcher Rods			**X**					Clean and lube monthly
Incubator Rods			**X**					Clean and lube monthly
Water Lines			**X**					Clean with isopropyl alcohol monthly

D = daily; W = Weekly; M = monthly; Q = Quarterly; SA = semi-annually; A = annually; AN = as needed

Contingency Plan

Maintenance contracts are carried for most instrumentation and close contact is maintained with service personnel to ensure optimal instrument functioning. An extensive spare parts inventory is maintained for routine repairs. Since instrumentation is standardized throughout the laboratory network, spare parts and components can be readily exchanged among the network.

In general, the laboratory has at least one backup unit for each critical unit. In the event of instrument failure, portions of the sample load may be diverted to duplicate instrumentation, the analytical technique switched to an alternate approved technique (such as manual colorimetric determination as opposed to automated colorimetric determination), or samples shipped to another properly certified or approved Eurofins Environment Testing location.

Attachment 5: Maximum Contaminant Levels (MCL) Table

Primary Drinking Water Regulations	
Contaminant	**MCL (mg/L)**
Nitrate (as N)	10
Nitrite (as N)	1

Secondary Drinking Water Regulations	
Contaminant	**MCL (mg/L)**
Chloride	250
Sulfate	250

Attachment 6: Glassware Cleaning Procedures

GLASSWARE CLEANING PROCEDURES - GENERAL LAB

MISCELLANEOUS APPARATUS

1. Scrub with hot tap water and Liquinox.
2. Rinse thoroughly with tap water.
3. Rinse at least three times with DI water.
4. Air-dry and store inverted in a cabinet or capped.

NOTE:

1. Do not use soap for cleaning COD digestion tubes.
2. Burets may be cleaned by filling with 10% KOH in methanol, allowing to stand for one hour, then rinsing thoroughly with DI water.
3. Evaporating dishes used for TDS determinations are cleaned by rinsing thoroughly with tap water, soaking for several hours in Nochromix, rinsing thoroughly with tap water, and rinsing three times with DI water. The dishes are then oven-dried.

TKN / TOTAL-P DIGESTION TUBES

1. Scrub with hot tap water and Liquinox.
2. Rinse thoroughly with tap water.
3. Rinse once with Nochromix.
4. Rinse at least three times with DI water.

CYANIDE DISTILLATION APPARATUS

1. Scrub with hot tap water and Liquinox.
2. Rinse thoroughly with tap water.
3. Rinse with 10% nitric acid, being certain to force at least 10mL through each dispersion tube.
4. Rinse thoroughly with DI water.

ODOR-FREE GLASSWARE

1. Scrub with hot tap water and Liquinox.
2. Rinse with acid cleaning solution.
3. Rinse thoroughly with odor-free water.
4. Glassware used in odor testing should be reserved for that purpose only.

FGE003:08.17.18:2

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18.0 Revision History

Summary of Changes for Revision 9:
- Removed references to TestAmerica from document.
- Removed references to WV criteria. The laboratory is not certified in WV as of 7/2021.
- Added calibration readback criteria of +/-10% to section 9.2.1.
- Revised blank criteria from <MDL to <1/2RL to be consistent with SM4020. <MDL is the criteria for SM3500 CR –B and Fe-B. Updates to Section 9.2.3 and Attachment 3.
- Updated definition of MDL in section 12.2 to be consistent with SOP SA-QA-007.
- Updated RLV and MDLV requirements.

Summary of Changes for Revision 8:
- Added section 6.2 to reference the Master Equipment Listing Maintained by the QA department.
- Updated section 10.1 that samples for Orthophosphate are filtered prior to analysis.
- Updated section 14.1 that RCRA hazardous and non-hazardous soil samples are disposed via the yardbox for incineration.
- Clarified requirements for Hexavalent Chromium in Attachment 2. Samples must be field filtered within 15minutes of sampling. An NCM must be initiated if no indication on COC of field filtering. WV Audit 2020
- Added pH verification and residual chlorine checks to Attachment 1a for Ammonia.
- Attachment 1c, section 3.3: Updated preparation of Ferrous Iron LCS solution.
- Attachment 1c, section 4.1: Updated sample preparation steps with more details.

Summary of Changes from Previous Revision 7:
- Minor editorial, grammatical, and/or formatting changes made.
- Revised soil sample preparation procedures for chloride. Attachment 1b, Section 4.2
- Revised Company logo and name where applicable

SOLID / RESIDUE DETERMINATIONS

(Methods: Various)

Approvals (Signature/Date):

Kimberly Chamberlain 11/18/2020

Kimberly Chamberlain Date
Quality Assurance Manager / Environmental Health & Safety Coordinator

11/18/2020

Adriana Geiger Date
Department Manager

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1.0 Scope and Application

This SOP contains the procedures for the determination of various residue analytes in water, soil, and waste samples.

The following analytes, matrices, methods, and technologies are included in this SOP:

Analyte	Matrix	Reference Method	Technology
Total Dissolved Solids	Water	EPA 160.1 and SM2540C	Gravimetric
Total Suspended Solids	Water	EPA 160.2 and SM2540D	Gravimetric
Total Solids	Water	EPA 160.3 and SM 2540B	Gravimetric
Volatile Dissolved Solids	Water and Waste (oil)	EPA 160.4 and SM2540E	Gravimetric
Volatile Suspended Solids	Water and Waste (oil)	EPA 160.4 and SM2540E	Gravimetric
Volatile Solids	Water and Waste (oil)	EPA 160.4 and SM2540E	Gravimetric
Fixed Solids	Water and Waste (oil)	EPA 160.4	Gravimetric
Ash Content	Water and Waste (oil)	SM 2540E	Gravimetric
Settleable Solids	Water	EPA 160.5 and SM2540F	Volumetric
Percent Moisture Fixed Solids Total Solids Total Volatile Solids Ash Content	Soil and Waste (oil)	SM2540G	Gravimetric
Percent Moisture Percent Solids (Used for Dry Weight Corrections)	Soil	EPA 3500B EPA 3500C EPA 3550B EPA 3550C	Gravimetric

The reporting limits (RL) and accuracy and precision criteria associated with this procedure are provided in the TALS Method Limit Groups (MLGs).

This SOP was written by and for Eurofins TestAmerica's Savannah laboratory.

2.0 Summary of Method

2.1 Total Solids (TS) – also called Total Residue

A well-mixed sample is transferred to a tared evaporating dish or pre-weighed vessel, and evaporated at 103°C-105°C. The residue that remains after the liquid has been evaporated is the total residue (or total solids) of the sample. The total solids are reported as milligrams of residue per liter of sample (mg/L).

The Total Solids is an approximate measure of the sum of the total suspended solids (TSS) and total dissolved solids (TDS). The total residue may not be equal to the sum of the TSS and TDS since the TDS is determined at 180°C and the total residue is determined at 103°C-105°C.

Note: The procedure used by the laboratory for the determination of percent solids/moisture as it pertains to dry weight determination is performed in a similar manner as the method procedure described above but the method-specified QC steps are not required. Refer to the applicable % Solids/% Moisture Work Instruction (in Attachment 15) for information on this procedure.

2.2 Volatile Solids (VS) and Ash Content/Fixed Solids

After the total solids has been determined, as outlined above, the residue is heated to 550 ± 50°C to determine the volatile solids. The portion of the residue that is lost during the 550°C heating step is defined as the volatile solids.

The residue remaining after heating to 550°C is the ash content or the fixed solids.

2.3 Total Suspended Solids (TSS) – also called Non-filterable Solids/Residue

A well-mixed sample is filtered through a tared glass fiber filter. The residue trapped on the filter pad is dried at 103°C-105°C. The weight of the residue (mg) divided by the volume of the sample filtered (L) is the total suspended solids. The total suspended solids are reported as milligrams of residue per liter of sample (mg/L).

Note: The TSS analysis is also referred to as "non-filterable residue" because the portion of the sample that is trapped on the filter pad is measured.

2.4 Volatile Suspended Solids (VSS)

The volatile suspended solids are determined by placing the filter containing the TSS residue into a muffle furnace at 550 ± 50°C. The portion of the residue that is volatilized at 550°C is the volatile suspended solid.

2.5 Total Dissolved Solids (TDS) – also called Filterable Solids/Residue

A well-mixed sample is filtered through a tared glass fiber filter. The filtrate (the liquid portion passing through the filter) is transferred to a tared evaporating dish or pre-weighed vessel and the liquid is evaporated between 115-125°C. The sample is then transferred to a new oven that is set to 180°C. The residue that remains after the sample has been heated at 180°C is the total dissolved solids portion of the sample. The total dissolved solids are reported as milligrams of residue per liter of sample (mg/L).

Note: The TDS analysis is also referred to as "filterable residue" because the dissolved solids in the portion of the sample that passes through the filter pad is measured.

2.6 Volatile Dissolved Solids (VDS)

The volatile dissolved solids are determined by placing the remaining TDS residue in a muffle furnace at 550°C ± 50°C. The weight that is lost after the sample has been heated at 550°C is the volatile dissolved solids portion of the sample.

Note: VDS determinations (and their associated TDS determination) must be performed using tared evaporating dishes ONLY, as the pre-weighed vessels are not capable of withstanding temperatures of 550 °C ± 50°C and therefore are not approved for VDS methods.

2.7 Settleable Solids – also called Settable Matter

A well-mixed sample is transferred to an Imhoff cone and measured volumetrically.

2.8 This SOP is based on the methods listed in Section 1.

3.0 Definitions

Refer to the Glossary Section of the *Quality Assurance Manual* (QAM) for a complete listing of applicable definitions and acronyms.

4.0 Interferences

4.1 Procedural Interferences

4.1.1 Interferences may be caused by contaminants in solvents, reagents, glassware, and other sample processing apparatus and can make quantification of the target analytes difficult.

4.1.2 All sample collection containers are single-use disposable containers which limits the potential for contamination. All non-disposable labware must be scrupulously cleaned in accordance with the posted Labware Cleaning Instructions to ensure it is free from contaminants and does not contribute artifacts.

4.1.3 High purity reagents and solvents are used to help minimize interference problems.

4.1.4 Instrument and/or method blanks are routinely used to demonstrate all reagents and apparatus are free from interferences under the conditions of the analysis.

4.1.5 Since the basis of the gravimetric procedure is the difference in the weight of the crucible containing the residue and the tare weight of the crucible, care must be taken to ensure that the crucible is not treated in such a manner to add or lose weight. Oils from the hand can be added to the dish by touching the dish with "ungloved" hands.

4.2 Matrix Interferences

4.2.1 For some settleable solids samples a separation of settleable and floating materials will occur. In these instances, the volume attributed to the floating materials is not included in the determination of the final result.

4.2.2 Highly mineralized water containing calcium, magnesium, chloride, or sulfate will require prolonged drying. A crust may form in some samples, trapping water underneath.

4.2.3 An excellent discussion on some of the problems that may be encountered when determining solids is found in Section 2540 of the *Standard Methods for the Examination of Water and Wastewater*, 20th Edition.

5.0 Safety

Employees must abide by the policies and procedures in the Eurofins TestAmerica Environmental Health and Safety Manual (EHSM), the Eurofins TestAmerica Savannah Addendum to the EHSM, and this document.

This procedure may involve hazardous materials, operations, and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user to follow appropriate safety, waste disposal, and health practices under the assumption that all samples and reagents are potentially hazardous.

The analyst must protect himself/herself from exposure to the sample matrix. Many of the samples that are tested may contain hazardous chemical compounds or biological organisms. The analyst must, at a minimum, wear protective clothing (lab coat), eye protection (safety glasses or face shield), disposable nitrile or latex gloves, and closed-toe, nonabsorbent shoes when handling samples.

5.1 Specific Safety Concerns or Requirements

All samples must be treated as if they are hazardous. The analyst must protect himself/herself from exposure to the sample matrix. Many of the samples that are tested for residue/solids may contain hazardous chemical compounds or biological organisms. The analyst must wear protective clothing (lab coat or apron), eye protection (glasses or face shield), and disposable gloves when handling these samples.

The toxicity or carcinogenicity of chemicals used in this method has not been precisely defined. Each chemical should be treated as a potential health hazard, and exposure to these chemicals should be minimized.

The analyst should handle samples that have been dried at or above 180°C with caution. This temperature can cause skin burns and damage to unprotected countertops. Gloves and tongs suitable for handling samples that have been placed in a muffle furnace must be used by the analyst to prevent the severe burns that can occur by contact with samples or containers at or near 550°C. The analyst is cautioned to make sure the surface that sample containers are placed on after removal from the oven or muffle furnace is capable of handling the heat without damage.

5.2 Primary Materials Used

The following is a list of the materials used in this procedure, which have a serious or significant hazard rating, and a summary of the primary hazards listed in their MSDS/SDS.

Note: This list does not include all materials used in the procedure. A complete list of materials used in this procedure can be found in the Reagents and Standards Section and the Equipment and Supplies Section of this SOP

Employees must review the information in the MSDS/SDS for each material before using it for the first time or when there are major changes to the MSDS/SDS. Electronic copies of MSDS/SDS can be found using the "MSDS" link on the Oasis homepage, on the EH&S webpage on Oasis, and through TALS file system shares.

6.0 Equipment and Supplies

6.1 Equipment and Instrumentation

Analytical Balance – Verify in accordance with SOP SA-AN-100: *Laboratory Support Equipment (Verification and Use)*

Top-loading Balance – Verify in accordance with SOP SA-AN-100: *Laboratory Support Equipment (Verification and Use)*

Portable Conductivity Meter- Verified daily prior to use

Vacuum Pump – with appropriate tubing and water trap

Drying Oven – capable of maintaining a constant temperature of 103-105°C and/or 180°C ± 2°C and/or 115-125 °C.

Muffle Furnace – capable of maintaining a constant temperature of 550 +/- 50°C.

Thermometers – Verify in accordance with SOP SA-AN-100: *Laboratory Support Equipment (Verification and Use)*. Note: To ensure full traceability, the thermometer ID and all applicable temperatures (i.e., start, stop, corrected, and uncorrected) must be documented in the Batch Data Types in TALS.

NIST-traceable timer – Verify in accordance with SOP SA-AN-100: *Laboratory Support Equipment (Verification and Use)*

6.2 Volumetric Containers and Dispensers

All volumetric labware must be verified in accordance with SOP SA-AN-100: *Laboratory Support Equipment (Verification and Use).* Refer to Attachment 16 for Labware Cleaning Procedures.

Volumetric Labware	Volume	Type (Quantitative / Qualitative)	Use	Verification Frequency	Laboratory Verification Criteria
Imhoff Cone	1L	QUANTITATIVE	Settable Solids Determination	Initially, prior to use	Accuracy = 2% Precision = 1%
Eppendorf-Style Mechanical Pipettes	Various	QUANTITATIVE	Preparing Dilutions, Standard and Reagent Preparation and Spiking	Initially, prior to first use, then quarterly thereafter (Daily for DOD)	Accuracy = 2% Precision = 1%
Graduated Cylinders (Class A)	Various	QUANTITATIVE	Preparing Dilutions, Standard and Reagent Preparation	None (Class A)	None (Class A)

6.3 Lab Supplies

Filtration Apparatus – consisting of a 47mm filter magnetic funnel and a 1000mL filtrate receiving flask with a sidearm. The filter funnel should extend into the receiving flask to ensure that no filtrate is suctioned into the water trap. (Note: This is especially important if TDS is to be determined on the filtrate.) A rubber or Neoprene stopper is used to attach the filter support to the receiving flask.

Constant weight, binderless glass fiber filter discs; 47-mm. Proweigh Double for TSS / VSS. Part number f93447MM-X (for TSS) and F93447VOL-X (for VSS)

Porcelain evaporating dishes - appropriate volume capacity. The evaporating dishes must be clearly marked so that samples transferred to the dishes can be clearly identified.

Pre-weighed Vessels – can be used in place of porcelain evaporating dishes for TS and TDS analyses only.

Weighing Bracket – for use with pre-weighed vessels, ensures accurate placement of vessel on center of balance platform

Modular Rack – for use transporting pre-weighed vessels to and from the oven

De-ionizing U-electrode and power supply – aids in eliminating static during weigh backs of pre-weighed vessels

Imhoff Cone Holder

Forceps – to handle the filter if TSS is being determined in conjunction with TDS

Aluminum Pans

Desiccator

Spatula or other utensil for transferring sample

Glass Rods

Nochromix – used to clean evaporating dishes and crucibles

Detergent – Liquinox or equivalent, used to wash non-disposable labware

6.4 Sample Collection Containers

All sample collection containers are single-use disposable containers which limits the potential for contamination. The routine samples containers supplied by the laboratory for these tests are listed below:

- 250mL, 500mL, or 1000mL plastic containers (dependent on the test being performed)
- 8oz plastic soil containers – used for SM2540G

All containers must be purchased with Certificate of Analysis attesting to purity. Refer to Attachment 2 for more information on container sizes.

7.0 Reagents and Standards

7.1 Expiration Dates

Expiration dates (time from initial use or receipt to final use) for standard and reagent materials must be set according to the guidance in this SOP. Note: These are maximum expiration dates and are not to be considered an absolute guarantee of standard or reagent quality. Sound judgment must be used when deciding whether to use a standard or reagent. If there is doubt about the quality of a standard or reagent material, a new material must be obtained or the standard or reagent material verified. Data quality must not be compromised to extend a standard's life.

The expiration date of any standard or reagent must not exceed the expiration date of the standard or reagent that was used to prepare it.

7.2 Reagents

Reagents must be prepared and documented in accordance with SOP SA-AN-041: *Reagent and Standard Materials Procedures.*

7.2.1 Laboratory Reagent Water – ASTM Type I

7.2.2 Desiccant – Drierite, blue to light purple color change
Storage: Store in a tightly closed container in a cool, dry location. Store away from incompatible materials.
Expiration: Unopened: Manufacturer's expiration date; Opened: 1year from date opened

7.2.3 Blank Matrix – Ottawa sand or equivalent. Used for the preparation of soil QC samples.
Storage: Room temperature
Expiration: Unopened: Manufacturer's expiration date; Opened: 1year from date opened

7.3 Standards

Standards must be prepared and documented in accordance with SOP SA-AN-041: *Reagent and Standard Materials Procedures.* Certificates of analysis or purity must be received with all purchased standards, and scanned and attached in TALS.

7.3.1 NSI Multi-analyte Solids Standard
Cat#QCI-171

7.3.2 Spectro Pures 1413us/cm for verification of the portable conductivity meter.
Cat#SP0696301A

8.0 Sample Collection, Preservation, Shipment, and Storage

8.1 Aqueous Samples

Aqueous samples are routinely collected in the following containers:
- 250mL, 500mL, or 1000mL plastic containers (dependent on the test being performed)

Samples must be iced at the time of collection and maintained at 0-6°C (less than 6°C but not frozen) until the time of analysis.

Refer to Attachment 2 for additional container and holding time information.

8.2 Soil Samples

Soil samples for SM2540G are routinely collected in 8oz plastic soil containers.

Percent solids samples used for the determination of dry weight are generally subsampled from the Metals or General Department sample portion. If the samples are for volatiles analyses only, then the soil samples are subsampled from the volatiles portion after the Volatiles Department has completed preparation of the sample.

Samples must be iced at the time of collection and maintained at 0-6°C (less than 6°C but not frozen) until the time of analysis.

Refer to Attachment 2 for holding time information.

8.3 Waste (Oil) Samples

Waste (oil) samples for Standard Methods 2540E and 2540G are routinely collected in 4oz amber glass soil containers.

Samples must be iced at the time of collection and maintained at 0-6°C (less than 6°C but not frozen) until the time of analysis.

Refer to Attachment 2 for holding time information.

9.0 Quality Control

SOP SA-QA-017: *Evaluation of Batch QC Data* and the SOP Summary in Attachment 3 provide requirements for evaluating QC data.

9.1 Batch QC

An analytical batch consists of up to 20 environment samples and the associated quality control (QC) items.

The default QC items performed for each analytical batch for this procedure are: a method blank and two separate sample duplicates; i.e., a minimum of 1 sample duplicate per 10 samples, equivalent to a 10 % frequency. The following methods also incorporate a laboratory control sample (LCS): EPA 160.1 and SM2540C, EPA 160.2 and SM2540D, and EPA 160.3 and SM2540B.

If there is insufficient sample to perform the sample duplicates, an NCM must be initiated. Note: Do not use field QC samples such as the equipment blank or field blank to perform the sample duplicates.

Batch QC must meet the criteria given in Attachment 3 of this SOP.

9.2 Method-Specific QC

For the VSS procedure, each lot of fiber filters must be tested prior to use to verify the suitability of the filter. This verification is performed by the vendor and a certificate is available from the vendor website.

9.3 Constant Weight Determination Procedures

For all residue determinations except percent solids/moisture, the constant weight must be achieved. If a constant weight cannot be achieved, the sample must undergo additional drying and weighing cycles until the constant weight criteria are achieved. An NCM must be initiated if constant weight is not achieved after three weighing.

Refer to Attachment 14 for these procedures and criteria.

9.4 Corrective Action for Out-of-Control Data

When the quality control parameters do not meet the criteria set forth in this SOP, corrective action must be taken in accordance with SOP SA-QA-005: *Preventive and Corrective Action Procedures* and the QC Summary Table in Attachment 3.

SOP SA-QA-005 provides contingencies for out-of-control data and gives guidance for exceptionally permitting departures from approved policies and procedures.

Nonconformance Memos must be initiated to document all instances where QC criteria are not met and all departures from approved policies and procedures.

10.0 Procedure

10.1 Field Sample Preparation

See Attachments 5 – 15 for details on field sample preparation.

10.2 QC Sample Preparation

See Attachments 5 – 15 for details on QC sample preparation.

10.3 Analysis

See Attachments 5 – 15 for details on sample analysis.

11.0 Calculations / Data Reduction

11.1 Data Reduction

Data must be evaluated in accordance with SOP SA-QA-002: *Data Generation and Review.*

11.1.1 These tests are empirical tests; therefore, there are no data reduction steps specific to these procedures.

11.1.2 Historical Data

Many of the laboratory's clients submit samples for repeat monitoring purposes. Prior to analysis, verify TALS Worksheet Notes and/or use the Historical Data Tracker feature to determine if historical data is available for review.

11.1.3 Chemical Relationships

When available, the following chemical relationships must be evaluated for each sample. If these relationships are not met the Department Manager must be contacted immediately.

- Total Solids (TS) ≥ TDS or TSS
- TDS ≥ individual anions

11.2 Calculations

11.2.1 The calculations associated with batch QC determinations are given in SOP SA-QA-017. Applicable calculations include accuracy (% recovery) and precision (%RPD).

11.2.2 The calculations associated with each analyte/method are contained in Attachments 5 through 15.

12.0 Method Performance

12.1 Reporting Limit Verification (RLV)

At a minimum, RLVs must be performed initially upon method set-up in accordance with SOP SA-QA-007: *Determination and Verification of Detection and Reporting Limits*. The reporting limit is based on the lowest discernable value achievable by the labware used in this procedure.

For analytes and methods certified by DOD ELAP, RLVs must also be performed quarterly thereafter. For all other analytes and methods, RLVs must also be performed annually thereafter. Exceptions may be made for project-specific non-routine analytes.

12.2 Method Detection Limit Study (MDL)

Method detection limits as generated in accordance with 40CFR Part 136 Appendix B are not applicable to this procedure. The reporting limit is based on the lowest discernable value achievable by the labware used in this procedure.

12.3 Method Detection Limit Verification (MDLV)

MDL verifications as outlined in SOP SA-QA-007: *Determination and Verification of Detection and Reporting Limits* are not applicable to this procedure. The reporting limit is based on the lowest discernable value achievable by the labware used in this procedure.

12.4 QC Limit Generation, Control Charting, and Trend Analysis

The control limits for the batch QC items (i.e., LCS/LCSD and SD) for this procedure are not specified by the reference methods; therefore, the laboratory defaults to in-house and/or laboratory-derived limits for the evaluation of batch QC items.

Although the laboratory must default to the method-defined QC limits, control charting is a useful tool and is performed to assess analyte recoveries over time to evaluate trends. Control charting must be performed periodically (at a minimum annually) in accordance with SOP SA-QA-017: *Evaluation of Batch QC Data*.

12.5 Demonstrations of Capability

Initial and continuing demonstration of capability must be performed in accordance with SOP SA-QA-006*: Training Procedures.*

Prior to performing this procedure unsupervised, each new analyst who performs this analysis must demonstrate proficiency per method by successful completion of an initial demonstration of capability.

The IDOC is performed by the analysis of 4 consecutive LCSs that meet the method criteria for accuracy and precision, or, when LCS are not available, by the analysis of client samples with statistically indistinguishable results when compared to another certified analyst. The IDOC must be documented and routed to the QA Department for filing.

Annual continuing demonstrations of capability (CDOCs) are also required per analyst per method/analyte combination. The CDOC requirement may be met by via the consecutive analysis of four LCS (where available) all in the same batch, by the analysis of four LCS analyzed in four consecutive batches (in different batches on different days), acceptable results on a PT study or by the analysis of client samples with statistically indistinguishable results when compared to another certified analyst. The CDOC must be documented and routed to the QA Department for filing.

12.6 Training Requirements

All training must be performed and documented in accordance with SOP SA-QA-006: *Training Procedures*.

Note: The SOPs listed in the Reference/Cross-Reference Section are applicable to this procedure. All employees performing this procedure must also be trained on these SOPs, and/or have a general understanding of these procedures, as applicable.

13.0 Pollution Control

It is Eurofins TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (e.g., examining recycling options, ordering chemicals based on quantity needed, preparing reagents based on anticipated usage and reagent stability, etc.). Employees must abide by the policies in Section 13 of the Environmental Health and Safety Manual and the Savannah Addendum to the EHSM.

This procedure has been evaluated for opportunities to minimize the waste generated. Where reasonably feasible, pollution control procedures have been incorporated.

14.0 Waste Management

Waste management practices must be conducted consistent with all applicable federal, state, and local rules and regulations. All waste (i.e., excess reagents, samples, and method process wastes) must be disposed of in accordance with Section 9 of the Eurofins TestAmerica Savannah Addendum to the EHSM. Waste description rules and land disposal restrictions must be followed.

14.1 Waste Streams Produced by the Method

The following waste streams are produced when this method is carried out:

- Non-hazardous acidic and alkaline wastewater and samples – Neutralize before disposal into drain/sewer system
- Excess aqueous samples – Dispose according to characterization on the sample disposal sheets. Neutralize non-hazardous samples before disposal into drain/sewer. Transfer hazardous samples (identified on disposal sheets) to the waste department for disposal.
- Acidic rinsings – Neutralize before disposal into drain/sewer system
- Excess soil and solid samples – Dispose according to characterization on sample disposal sheets. Transfer non-hazardous and RCRA hazardous samples to waste department for disposal via yard box. This yard box is incinerated by the waste disposal company. Transfer samples classified as PCB Hazardous or Foreign Soils to the waste department for disposal via drumming.

15.0 References / Cross-References

- SOP SA-AN-041: *Reagent and Standard Materials Procedures*
- SOP SA-AN-100: *Laboratory Support Equipment (Verification and Use)*
- SOP SA-GE-193: Measurement of Analytes via PC Titrate
- SOP SA-QA-002: *Data Generation and Review*
- SOP SA-QA-005: *Preventive and Corrective Action Procedures*
- SOP SA-QA-006*: Training Procedures*
- SOP SA-QA-015: *Homogenization, Compositing, and Segregation of Samples*
- SOP SA-QA-017: *Evaluation of Batch QC Data*
- Eurofins TestAmerica Savannah Quality Assurance Manual
- Eurofins TestAmerica Environmental Health and Safety Manual
- Eurofins TestAmerica Savannah Addendum to the Environmental Health and Safety Manual
- *Methods for Chemical Analysis of Water and Waste*; U.S. EPA Office of Research and Development: Cincinnati, OH, March, 1983.
- *Standard Methods for the Examination of Water and Wastewater*, Online Edition; American Public Health Association: Washington, D.C.
  - SM2540B: Total Solids Dried at 103–105°C; Approve 1997; Editorial Revisions 2011
  - SM2540C: Total Dissolved Solids Dried at 180°C; Approve 1997; Editorial Revisions 2011
  - SM2540D: Total Suspended Solids Dried at 103–105°C; Approve 1997; Editorial Revisions 2011
  - SM2540E: Fixed and Volatile Solids Ignited at 550°C; Approve 1997; Editorial Revisions 2011
  - SM2540F: Settleable Solids; Approve 1997; Editorial Revisions 2011
  - SM2540G: Total, Fixed, and Volatile Solids in Solid and Semisolid Samples; Approve 1997; Editorial Revisions 2011

- *Test Methods for Evaluating Solids Waste, Third Edition (Updates III and IV)*, SW-846; USEPA Office of Solids Waste and Emergency response, Washington, D.C.

16.0 Method Modifications and Clarifications

16.1 Clarification: EPA 160.5 and SM2540F do not specify batch QC requirements. To comply with NELAC and/or state-specific batch QC requirements, the laboratory performs a method blank and a sample duplicate with each batch.

16.2 Clarification: SM2450F gives volumetric and gravimetric procedures for the determination of settleable matter. The laboratory only performs the volumetric procedure.

16.3 Clarification: The gravimetric analytical methods call for drying samples for a minimum of 1 hour. The laboratory's standard procedure is to dry samples for a minimum of 4 hours. The filters are placed back in the oven for an additional hour or overnight to evaluate if a constant weight has been achieved.

16.4 Modification: Although samples comprised of a waste matrix are not addressed in the Standards Methods reference methods, the laboratory has adapted these methods to accommodate waste samples.

16.5 Clarification: With the exception of the Settleable Matter, the EPA reference methods do not specify holding times for these analyses; rather, these reference methods state to perform the test "as soon as possible". The Standard Methods reference methods state to perform the test "as soon as possible" preferably within 24 hours, but no longer than 7 days. As such, the laboratory has adopted a 7-day holding time for all these analyses.

16.6 Modification: The laboratory's procedures for Percent Solids/Percent Moisture used to determine dry weight correction are taken from EPA 3500B, EPA 3500C, EPA 3550B, and EPA 3550C. These procedures are performed in a similar manner as the EPA 160.3 and SM2540B and SM2540G method procedures; however, those method-specified QC steps are not required.

16.7 Modification: Although the associated reference methods (SM2540C, SM2540B, EPA 160.1, and EPA 160.3) do not cite an alternative to the use of porcelain evaporating dishes, new pre-weighed vessels (e.g., StableWeigh) have since been developed and may be used in place of porcelain evaporating dishes for TDS and TS analyses only.

17.0 Attachments

The following Tables, Diagrams, and/or Validation Data are included as Attachments:

Attachment 1: SOP Summary
Attachment 2: Sample Collection, Preservation, and Holding Time
Attachment 3: QC Summary
Attachment 4: Table Preventative Maintenance and Troubleshooting
Attachment 5: Total Solids (TS) Procedures – EPA 160.3 and SM2540B
Attachment 6: Volatile Solids (VS), Ash Content, and Fixed Solids Procedures – EPA 160.4 and SM2540E
Attachment 7: Total Suspended Solids (TSS) Procedures – EPA 160.2 and SM2540D
Attachment 8: Volatile Suspended Solids (VSS) Procedures – EPA 160.4 and SM2540E
Attachment 9: Total Dissolved Solids (TDS) Procedures – EPA 160.1 and SM2540C
Attachment 10: Volatile Dissolved Solids (VDS) Procedures – EPA 160.4 and SM2540E
Attachment 11: Settleable Solids Procedures – EPA 160.5 and SM2540F
Attachment 12: Percent Moisture and Total Solids Procedures – SM2540G
Attachment 13: Volatile Solids, Ash Content, and Fixed Solids Procedures – SM2540G
Attachment 14: Constant Weight Determination Procedures
Attachment 15: Percent Moisture and Percent Solids (for Dry Weight Correction)
Attachment 16: Glassware Cleaning Procedures

Attachment 1:
SOP Summary

Analyte	Matrix	Reference Method	Technology
Total Dissolved Solids	Water	EPA 160.1 and SM2540C	Gravimetric
Total Suspended Solids	Water	EPA 160.2 and SM2540D	Gravimetric
Total Solids	Water	EPA 160.3 and SM 2540B	Gravimetric
Volatile Dissolved Solids	Water and Waste (oil)	EPA 160.4 and SM2540E	Gravimetric
Volatile Suspended Solids	Water and Waste (oil)	EPA 160.4 and SM2540E	Gravimetric
Volatile Solids	Water and Waste (oil)	EPA 160.4 and SM2540E	Gravimetric
Fixed Solids	Water and Waste (oil)	EPA 160.4	Gravimetric
Ash Content	Water and Waste (oil)	SM 2540E	Gravimetric
Settleable Solids	Water	EPA 160.5 and SM2540F	Volumetric
Percent Moisture Fixed Solids Total Solids Total Volatile Solids Ash Content	Soil and Waste (oil)	SM2540G	Gravimetric
Percent Moisture Percent Solids (Used for Dry Weight Corrections)	Soil	EPA 3500B EPA 3500C EPA 3550B EPA 3550C	Gravimetric

Sample Preparation and Analysis Summary

Total Solids (TS) – also called Total Residue
A well-mixed sample is transferred to a tared evaporating dish or pre-weighed vessel, and evaporated at 103°C-105°C. The residue that remains after the liquid has been evaporated is the total residue (or total solids) of the sample. The total solids are reported as milligrams of residue per liter of sample (mg/L).

The Total Solids is an approximate measure of the sum of the total suspended solids (TSS) and total dissolved solids (TDS). The total residue may not be equal to the sum of the TSS and TDS since the TDS is determined at 180°C and the total residue is determined at 103°C-105°C.

Note: The procedure used by the laboratory for the determination of percent solids/moisture as it pertains to dry weight determination is performed in a similar manner as the method procedure described above but the method-specified QC steps are not required.

Volatile Solids (VS) and Ash Content/Fixed Solids
After the total solids has been determined, as outlined above, the residue is heated to 550 ± 50°C to determine the volatile solids. The portion of the residue that is lost during the 550°C heating step is defined as the volatile solids.

The residue remaining after heating to 550°C is the ash content or the fixed solids.

Total Suspended Solids (TSS) – also called Non-filterable Solids/Residue
A well-mixed sample is filtered through a tared glass fiber filter. The residue trapped on the filter pad is dried at 103°C-105°C. The weight of the residue (mg) divided by the volume of the sample filtered (L) is the total suspended solids. The total suspended solids are reported as milligrams of residue per liter of sample (mg/L).

Note: The TSS analysis is also referred to as "non-filterable residue" because the portion of the sample that is trapped on the filter pad is measured.

Volatile Suspended Solids (VSS)
The volatile suspended solids are determined by placing the filter containing the TSS residue into a muffle furnace at 550 ± 50°C. The portion of the residue that is volatilized at 550°C is the volatile suspended solid.

Total Dissolved Solids (TDS) – also called Filterable Solids/Residue
A well-mixed sample is filtered through a tared glass fiber filter. The filtrate (the liquid portion passing through the filter) is transferred to a tared evaporating dish or pre-weighed vessel and the liquid is evaporated at 115-125°C. The samples are then moved to an oven that is heated to 180°C± 2°C. The residue that remains after the sample has been heated at 180°C is the total dissolved solids portion of the sample. The total dissolved solids are reported as milligrams of residue per liter of sample (mg/L).

Note: The TDS analysis is also referred to as "filterable residue" because the dissolved solids in the portion of the sample that passes through the filter pad is measured.

Volatile Dissolved Solids (VDS)
The volatile dissolved solids are determined by placing the remaining TDS residue in a muffle furnace at 550°C ± 50°C. The weight that is lost after the sample has been heated at 550°C is the volatile dissolved solids portion of the sample.

Note: VDS determinations (and their associated TDS determination) must be performed using tared evaporating dishes ONLY as the pre-weighed vessels are not capable of withstanding temperatures of 550 °C ± 50°C therefore are not approved for VDS methods.

Settleable Solids – also called Settable Matter
A well-mixed sample is transferred to an Imhoff cone and measured volumetrically.

Attachment 2:
Sample Collection, Preservation, and Holding Time Table

Matrix	Fraction	Method	Routine Sample Container	Routine Sample Size	Minimum Sample Size	Chemical Preservation	Dechlorination Agent	Thermal Preservation	Holding Time[1]
Water	Total Dissolved Solids	EPA 160.1 SM2540C	250mL plastic	200mL	200mL	Not Applicable	Not Applicable	0-6 °C[2]	7 days
Water	Total Suspended Solids	EPA 160.2 SM2540D	1L plastic	1L	1L	Not Applicable	Not Applicable	0-6 °C[2]	7 days
Water	Total Solids	EPA 160.3 SM2540B	1L plastic	500mL	500mL	Not Applicable	Not Applicable	0-6 °C[2]	7 days
Water	Volatile Solids	EPA 160.4 SM2540E	1L plastic	1L	1L	Not Applicable	Not Applicable	0-6 °C[2]	7 days
Water	Settleable Solids	EPA 160.5 SM2540F	1L plastic	1L	1L	Not Applicable	Not Applicable	0-6 °C[2]	48 hours
Soil	Fixed Solids	SM2540G	8oz plastic soil jar	10g	10g	Not Applicable	Not Applicable	0-6 °C[2]	7 days
Waste / Oil	Fixed / Volatile Solids	SM2540E SM2540G	4oz amber glass soil jar	10g	10g	Not Applicable	Not Applicable	0-6 °C[2]	7 days

[1]From collection to analysis
[2]Less than 6 °C, with no frozen samples

Attachment 3:
QC Summary

Method	QC Item	Frequency	Criteria	Corrective Action
EPA 160.1 & SM 2540C (Total Dissolved Solids)	Method Blank (MB)	One per batch	<RL listed in the MLG	Re-analyze batch
	Laboratory Control Sample (LCS)	One per batch	Within %Recovery listed in the MLG	Re-analyze batch
	Laboratory Control Sample Duplicate (LCSD)	One per batch	Within %RPD listed in the MLG	Re-analyze batch
	Sample Duplicate (SD)	One per batch, or one for every 10 samples analyzed, whichever is greater	%RPD ≤ 5%	Flag associated data
	Initial Demonstration of Capability (IDOC)	Initially, per analyst per method	Within %RPD listed in the MLG	Re-perform DOC; Unsupervised work cannot proceed until successful IDOC is obtained.
	Continuing Demonstration of Capability (CDOC)	Annually, per analyst per method	Within %RPD listed in the MLG	Re-perform DOC

Method	QC Item	Frequency	Criteria	Corrective Action
EPA 160.2 & SM 2540D (Total Suspended Solids)	Method Blank (MB)	One per batch	<RL listed in the MLG	Re-analyze batch
	Laboratory Control Sample (LCS)	One per batch	Within %Recovery listed in the MLG	Re-analyze batch
	Laboratory Control Sample Duplicate (LCSD)	One per batch	Within %RPD listed in the MLG	Re-analyze batch
	Sample Duplicate (SD)	One per batch, or one for every 10 samples analyzed, whichever is greater	%RPD ≤ 5%	Flag associated data
	Initial Demonstration of Capability (IDOC)	Initially, per analyst per method	Within %RPD listed in the MLG	Re-perform DOC; Unsupervised work cannot proceed until successful IDOC is obtained.
	Continuing Demonstration of Capability (CDOC)	Annually, per analyst per method	Within %RPD listed in the MLG	Re-perform DOC

Method	QC Item	Frequency	Criteria	Corrective Action
EPA 160.3 & SM 2540B (Total Solids)	Method Blank (MB)	One per batch	<RL listed in the MLG	Re-analyze batch
	Laboratory Control Sample (LCS)	One per batch	Within %Recovery listed in the MLG	Re-analyze batch
	Laboratory Control Sample Duplicate (LCSD)	One per batch	Within %RPD listed in the MLG	Re-analyze batch
	Sample Duplicate (SD)	One per batch, or one for every 10 samples analyzed, whichever is greater	%RPD ≤ 5%	Flag associated data
	Initial Demonstration of Capability (IDOC)	Initially, per analyst per method	Within %RPD listed in the MLG	Re-perform DOC; Unsupervised work cannot proceed until successful IDOC is obtained.
	Continuing Demonstration of Capability (CDOC)	Annually, per analyst per method	Within %RPD listed in the MLG	Re-perform DOC

Method	QC Item	Frequency	Criteria	Corrective Action
EPA 160.4 & SM 2540E (Volatile Dissolved Solids, Volatile Suspended Solids, Volatile Solids, Fixed Solids, and Ash Content)	Method Blank (MB)	One per batch	<RL listed in the MLG	Re-analyze batch
	Sample Duplicate (SD)	One per batch, or one for every 10 samples analyzed, whichever is greater	Within %RPD listed in the MLG	Flag associated data
	Initial Demonstration of Capability (IDOC)	Initially, per analyst per method	Within %RPD listed in the MLG	Re-perform DOC; Unsupervised work cannot proceed until successful IDOC is obtained.
	Continuing Demonstration of Capability (CDOC)	Annually, per analyst per method	Within %RPD listed in the MLG	Re-perform DOC
	Filter Blank	One per each lot of filters, prior to use (for VSS analysis)	<RL listed in the MLG	Remove from service

Method	QC Item	Frequency	Criteria	Corrective Action
EPA 160.5 & SM 2540F (Settleable Solids)	Method Blank (MB)	One per batch	<RL listed in the MLG	Re-analyze batch
	Sample Duplicate (SD)	One per batch, or one for every 10 samples analyzed, whichever is greater	Within %RPD listed in the MLG	Initiate NCM if insufficient sample is available to perform a sample duplicate. Flag associated data
	Initial Demonstration of Capability (IDOC)	Initially, per analyst per method	Within %RPD listed in the MLG	Re-perform DOC; Unsupervised work cannot proceed until successful IDOC is obtained.
	Continuing Demonstration of Capability (CDOC)	Annually, per analyst per method	Within %RPD listed in the MLG	Re-perform DOC

Method	QC Item	Frequency	Criteria	Corrective Action
SM 2540G (Percent Moisture, Fixed Solids, Total Solids, Total Volatile Solids, Ash Content)	Method Blank* (MB)	One per batch	<RL listed in the MLG	Re-analyze batch
	Sample Duplicate (SD)	One per batch, or one for every 10 samples analyzed, whichever is greater	Within %RPD listed in the MLG	Initiate NCM if insufficient sample is available to perform a sample duplicate. Flag associated data
	Initial Demonstration of Capability (IDOC)	Initially, per analyst per method	Within %RPD listed in the MLG	Re-perform DOC; Unsupervised work cannot proceed until successful IDOC is obtained.
	Continuing Demonstration of Capability (CDOC)	Annually, per analyst per method	Within %RPD listed in the MLG	Re-perform DOC

***Note: Method Blank not performed for Total Solids, Fixed Solids, or % Moisture.**

Attachment 4:
Preventative Maintenance and Troubleshooting

Instrument Labeling

Each instrument must be labeled with its name or ID (e.g., MSA, ICP-D, etc.). Additionally, non-operational instruments must be isolated from service or marked as being out of service. Each piece of equipment has an "Operational / Not Operational" sticker that is used for this purpose.

Maintenance Log

A maintenance log must be established for each piece of equipment used in the laboratory. All maintenance that is performed on the instrument must be recorded in the log including:

- Type of maintenance performed (Note: This includes preventative/routine maintenance; non-routine maintenance; maintenance performed by an external vendor; updates to software versions; etc.)
- Analyst or technician performing the maintenance
- Date the maintenance was performed
- Detailed explanation of the reason for the maintenance
- resolution of the problem and return to control
- All service calls from instrument representatives
- Resolution of the problem (i.e., the Return to Control)

Desiccator Checks, Maintenance, and Use

The following checks must be performed daily:

- Desiccant is active.
- Hygrometer is in the low humidity zone.
- Door is making an air-tight seal.

The desiccator door must remain closed and seated whenever possible. When the desiccant turns from blue to light purple, discard desiccant and refill pan with fresh desiccant. The desiccant must be disposed in accordance with the Waste Disposal Section of the Eurofins TestAmerica Savannah Addendum to the Environmental Health and Safety Manual.

Attachment 5:

Total Solids (TS) Procedures
EPA 160.3 and SM2540B

Preparation of the Evaporating Dishes

If needed, thoroughly clean the 100mL porcelain evaporating dishes that will be required for the determination of VS (volatile solids). It may be necessary to place Nochromix in the dish for a short time to remove dissolved solids from a previous analysis.

Rinse the evaporating dishes thoroughly with reagent water and allow them to dry. The evaporating dishes should be placed in the oven at 180°C to facilitate the drying.

After the evaporating dish has remained in the oven at 180°C for a minimum of two hours, remove the dishes from the oven and place in the desiccator. Do not close the door of the desiccator completely until the dishes have cooled. After the dishes have cooled, close the door of the desiccator. The evaporating dishes may be stored in the desiccator until ready for use.

Note: If the evaporating dishes are placed into the desiccator while hot and the door is sealed, a vacuum may form as the dishes cool. If this occurs, the door of the desiccator may be difficult to open.

Note: No preparation is required if utilizing the 200mL pre-weighed vessels in place of the porcelain evaporating dishes. These may be used if only TS is determined.

Determination of Total Solids

Verify that the analytical balance is working within the proper parameters in accordance with SOP SA-AN-100: *Laboratory Support Equipment (Verification and Use)*.

Remove the evaporating dish from the desiccator and weigh it on the analytical balance. Record the evaporating dish ID and the weight of the dish to the nearest 0.0001g in the TALS AD batch.

Note: Remember never to touch dishes with bare hands as this can cause oils to deposit and affect the weight of the dish.

Note: If utilizing pre-weighed vessels in place of porcelain evaporating dishes, no initial weight is required. Record the vessel ID and vessel weight found stamped on each individual vessel in the TALS AD batch. Transport vessels to and from the oven using a modular rack.

Mix the sample well before pouring sample into a Class A graduated cylinder. Record the volume of sample in the TALS AD batch.

Note: The sample aliquot to be used in the determination of total residue may be estimated from the specific conductance. If the conductance is greater than 200umho/cm refer to the chart in Attachment 9 for appropriate sample volume. If the conductance is

less than 200umho/cm, use 200mL of sample. If the concentration of the suspended particulates of the sample is significant, a smaller aliquot should be used.

Carefully add the sample to the tared evaporating dish (up to 100mL) or a pre-weighed vessel (up to 200mL). A few milliliters of reagent water must be used to help transfer any particulates adhering to the side of the graduated cylinder to the sample container. Place the sample container into the drying oven maintained at 103°C-105°C to evaporate the liquid portion of the sample. This will generally require overnight in the oven.

After the liquid has evaporated, remove the sample containers from the oven, allow them to cool slightly, and place them in the desiccator. Remember not to place the sample containers on a surface that can cause dirt or other foreign objects to adhere to them. After the containers are cool, place them in the desiccator.

Allow the sample containers to remain in the desiccator until completely cool. Remove from the desiccator and weigh to constant weight using the procedure outlined in Attachment 14.

The Total Solids is calculated using the following equation:

$$TS, mg/L = \frac{(A - B)}{V} \otimes 1000 mg/g$$

Where:
A = weight of sample residue and evaporating dish (g)
B = weight of evaporating dish (g)
V = volume of sample used to determine the TS (L)

QC Sample Preparation and Analysis

For each batch of 20 or fewer samples prepare a minimum of one method blank, one laboratory control sample, and two separate sample duplicates.

For the method blank, follow the procedures outlined above and use 100mL or 200mL of reagent water to serve as the blank matrix.

For the laboratory control sample, follow the procedures outlined above and use 50mL of the Multi-analyte solids standard.

For the sample duplicate sample, follow the procedures outlined above and prepare and analyze one sample in duplicate per batch or 1 sample in duplicate for every 10 samples analyzed, whichever is greater.

Attachment 6:

Volatile Solids (VS), Ash Content, and Fixed Solids Procedures EPA 160.4 and SM2540E

Preparation of the Evaporating Dishes for Volatile Solids

Thoroughly clean the 100mL porcelain evaporating dishes that will be required for the determination of VS, Ash Content or Fixed Solids. It may be necessary to place Nochromix in the dish for a short time to remove dissolved solids from a previous analysis.

Rinse the evaporating dishes thoroughly with reagent water and allow them to dry. The evaporating dishes should be placed in the oven at 180°C to facilitate the drying.

After the evaporating dish has remained in the oven at 180°C for a minimum of two hours, remove the dishes from the oven and place them into the muffle furnace. Turn on the muffle furnace, and elevate the temperature 100°C per hour until it reaches 550°C ± 50°C. After one hour at 550°C, remove the dishes from the muffle furnace, allow them to cool for a few minutes, and place them in the desiccator. Do not close the door of the desiccator completely until the dishes have cooled. After the dishes have cooled, close the door of the desiccator. The evaporating dishes may be stored in the desiccator until ready for use.

Note: If the evaporating dishes are placed into the desiccator while hot and the door is sealed, a vacuum may form as the dishes cool. If this occurs, the door of the desiccator may be difficult to open.

Determination of Volatile Solids, Ash Content, and Fixed Solids

Once the Total Solids has been determined per Attachment 5, place the evaporating dish containing the sample residue into the muffle furnace. Turn on the muffle furnace, and elevate the temperature 100°C per hour until it reaches 550°C ± 50°C.

After one hour at 550°C, remove the evaporation dishes from the muffle furnace, allow them to cool for a few minutes, and place them in the desiccator. Remember not to place the evaporating dishes on a surface that can cause dirt, or other foreign objects, to adhere to the dishes or on a surface that will melt when hot dishes are placed on it.

Allow the evaporating dishes to remain in the desiccator until completely cool.

Note: Remember never to touch dishes with bare hands as this can cause oils to deposit and affect the weight of the dish.

Verify that the analytical balance is working within the proper parameters in accordance with SOP SA-AN-100: *Laboratory Support Equipment (Verification and Use)*.

Weigh the dishes to constant weight using the procedure outlined in Attachment 14. Record the weight of the dish in the TALs worksheet and calculate the residue to the nearest 0.0001g.

The Volatile Solids is calculated:

$$VS,mg/L = \frac{(R_{105} - R_{550})}{V} \otimes 1000mg/g$$

Where:
V = volume of sample analyzed
R_{550} = (weight of residue and dish at 550°C)g - (weight of dish)g
R_{105} = (weight of residue and dish at 105°C)g - (weight of dish)g

The Fixed Solids is calculated:

$$mg_fixed_solids/L = \frac{(R_{550})}{V} \otimes 1000mg/g$$

Where:
V = volume of sample analyzed
R_{550} = (weight of residue and dish at 550°C)g - (weight of dish)g

The Ash Content is calculated:

$$Ash_Content\% = \frac{R_{550}}{R_{105}} \otimes 100$$

Where:
R_{550} = (weight of residue and dish at 550°C)g - (weight of dish)g
R_{105} = (weight of residue and dish at 105°C)g - (weight of dish)g

QC Sample Preparation and Analysis

For each batch of 20 or fewer samples prepare a minimum of one method blank and two separate sample duplicates.

For the method blank, follow the procedures outlined above and use 100mL for porcelain dishes or 200mL for pre-weighed vessels of reagent water to serve as the blank matrix.

For the sample duplicate sample, follow the procedures outlined above and prepare and analyze one sample in duplicate per batch or 1 sample in duplicate for every 10 samples analyzed, whichever is greater.

Attachment 7:

Total Suspended Solids (TSS) Procedures
EPA 160.2 and SM2540D

Preparation of the Filters

Filters are purchased from an approved vendor and are pre-prepared according to method requirements. According to SM 2540 D, 47mm binderless glass fiber filters are washed 3 times in deionized water, dried at 103 - 105°C, and weighed to 0.1mg. The drying and weighing steps are then repeated to guarantee constant weight.

Determination of Total Suspended Solids

Calibrate and verify that the analytical balance is working within the proper parameters in accordance with SOP SA-AN-100: *Laboratory Support Equipment (Verification and Use)*.

The filters purchased as pre-weighed contain a number aluminum pan and filter tare weight. The barcode is scanned into the analytical batch to record the ID and filter tare weight.

Assemble the filtration apparatus consisting of a 47mm filter magnetic funnel and a 1000mL filtrate receiving flask with a sidearm. The filter funnel should extend into the receiving flask to ensure that no filtrate is suctioned into the water trap. (This is especially important if TDS is to be determined on the filtrate.) A rubber or Neoprene stopper is used to attach the filter support to the receiving flask.

Using forceps, transfer a filter from the numbered aluminum pan to the filter support with the rough side up. Clamp the filter into place and add a small aliquot (~5mL) of reagent water to the filtering apparatus to seat the filter.

Mix the sample well. Transfer an all or an aliquot of the well-mixed sample to a class A graduated cylinder. Record the sample identification, pan number, and volume of sample in the TALS AD batch.

Note: Use a sample size of 1mL to 1L that will produce a residue within the range of 2.5mg to 200mg, with a target residue of 100mg. 1L should be used for samples without historical data and samples that do not appear to contain suspended residue. Smaller aliquots can be used for samples suspected to contain significant suspended residue.

- If greater than 200mg of residue is obtained, the test must be re-performed using a smaller aliquot of sample. If the test cannot be re-performed using a smaller aliquot, then an NCM must be initiated to denote this situation.
- If less than 1L is used, and less than 2.5mg of residue is obtained, the test must be re-performed using a larger volume of sample (up to 1L). If the test cannot be re-performed using a larger volume, initiate an NCM to denote this situation.

Note: Wide bore pipettes can also be used to transfer the aliquot from the sample container to the filtration device. Narrow bore pipettes must not be used.

Turn on the pump and carefully pour the entire sample aliquot into the filter funnel. Use a small aliquot of reagent water to rinse the entire sample out of the graduated cylinder and onto the filter. Use three 10mL portions of reagent water to rinse down the sides of the filter funnel to transfer all of the particulates on to the filter.

Allow the pump to remain on for about a minute after all of the particulates have been transferred to the filter to remove the water trapped on the filter.

Turn off the pump and release the vacuum. Carefully remove the filter disc from the filter support with the forceps and place the filter in the aluminum pan. The filter should be placed on the edge of the aluminum pan so that the filter has minimum contact with the pan.
Dry the filter at 103°C-105°C for at least one hour, preferably overnight. Record the temperature of the oven corrected and uncorrected, oven ID, thermometer ID and the time that the samples are placed into the oven in the TALS AD batch.

Remove the filters and pans from the oven and place them into the desiccator. Record the oven temperature uncorrected and corrected, and time removed from the oven in the TALS batch.

Place an aluminum pan onto the analytical balance and tare. Using forceps, transfer the filter to the tared aluminum pan. Record the filter weight into the analytical batch and calculate the residue. Weigh the filter to constant weight using the procedure outlined in Attachment 14.

The Total Suspended Solids is calculated using the following equation:

$$TSS, mg/L = \frac{(A - B)}{V} \otimes 1000 mg/g$$

Where:
A = weight of pan, sample residue, and filter (g)
B = weight of pan and filter (g)
V = volume of sample used to determine the TSS (L)

QC Sample Preparation and Analysis

For each batch of 20 or fewer samples prepare a minimum of one method blank, one laboratory control sample, and two separate sample duplicates.

For the method blank, follow the procedures outlined above and use 1L of reagent water to serve as the blank matrix.

For the laboratory control sample, follow the procedures outlined above and use 50mL of the TSS Check Standard.

For the sample duplicate sample, follow the procedures outlined above and prepare and analyze one sample in duplicate per batch or 1 sample in duplicate for every 10 samples analyzed, whichever is greater.

Attachment 8:

Volatile Suspended Solids (VSS) Procedures
EPA 160.4 and SM2540E

Preparation of the Filters

Filters are purchased from an approved vendor and are pre-prepared according to method requirements. According to SM 2540 D, 47mm binderless glass fiber filters are washed 3 times in deionized water, dried at 550°C, and weighed to 0.1mg. The drying and weighing steps are then repeated to guarantee constant weight.

Note that these filters will be used to determine the Total Suspended Solids and the Volatile Suspended Solids.

Determination of Volatile Suspended Solids

Volatile suspended solids are the difference in the residue at 105°C (TSS) and the residue that remains after the sample is volatilized at 550°C. The associated TSS method blank must be taken through the ignition procedure described in this section. If the sample was analyzed in duplicate for TSS, both replicates must be taken though the ignition procedure.

After the TSS has been determined, carefully transfer the numbered aluminum pan, filter, and residue into the muffle furnace. If there is quite a bit of organic material in the TSS residue, potentially harmful gases may evolve as the sample ignites. The muffle furnace should be maintained under a hood or in a well ventilated area.

Note: Long handled tongs and proper gloves must be used when working with the muffle furnace.

Turn on the muffle furnace, and elevate the temperature 100°C per hour until it reaches 550°C ± 50°C. Allow the sample to remain in the muffle furnace for at least one hour at 550 ± 50°C. Record the time, temperature that is uncorrected and corrected, and oven ID and thermometer ID the TALs batch once the oven has reached 550 ± 50°C.

After one hour, remove the pans and filters from the furnace and place them on a non-reactive surface to cool slightly. Record the time, temperature that is uncorrected and corrected in the TALs analytical batch. Transfer the pans and filters to the desiccator and allow them to cool to ambient temperature.

Note: Be careful when placing the pans on any surface when they are taken out of the muffle furnace. Even though the heat will quickly dissipate, the pans/filters will be very hot and should not be placed on a surface that will melt or ignite.

Place an aluminum pan on the analytical balance and tare. Transfer the filter containing the residue into the tared aluminum pan. Weigh the filter to constant weight using the procedure outlined in Attachment 14.

Note: Caution must be taken when handling a filter that contains residue. The analyst must be careful not to lose any of the residue. If an obvious loss occurs, contact the department manager and repeat the analysis.

The Volatile Suspended Solids is calculated as follows:

$$VSS(mg/L) = = \frac{(R_{105} - R_{550})}{V} \otimes 1000mg/g$$

Where:
R_{105} = residue at 105°C (g)
R_{550} = residue at 550°C (g)

QC Sample Preparation and Analysis

For each batch of 20 or fewer samples prepare a minimum of one method blank and two separate sample duplicates.

For the method blank, follow the procedures outlined above and use 1000mL of reagent water to serve as the blank matrix.

For the sample duplicate sample, follow the procedures outlined above and prepare and analyze one sample in duplicate per batch or 1 sample in duplicate for every 10 samples analyzed, whichever is greater.

Attachment 9:

Total Dissolved Solids (TDS) Procedures
EPA 160.1 and SM2540C

Preparation of the Evaporating Dishes – Used for VDS procedure

Prepare the 100mL porcelain evaporating dishes that will be required for the determination of VDS (Volatile Dissolved Soils) if needed. It may be necessary to place Nochromix in the dish for a short time to remove dissolved solids from a previous analysis.

Rinse the evaporating dishes thoroughly with reagent water and allow them to dry. The evaporating dishes should be placed in the oven at 180°C to facilitate the drying.

After the evaporating dish has remained in the oven at 180°C for a minimum of two hours, remove the dishes from the oven and place in the desiccator. Do not close the door of the desiccator completely until the dishes have cooled. After the dishes have cooled, close the door of the desiccator. The evaporating dishes may be stored in the desiccator until ready for use.

Note: If the evaporating dishes are placed into the desiccator while hot and the door is sealed, a vacuum may form as the dishes cool. If this occurs, the door of the desiccator may be difficult to open.

Note: VDS determinations (and their associated TDS determination) must be performed using tared evaporating dishes ONLY, as the pre-weighed vessels are not capable of withstanding temperatures of 550 °C ± 50°C and therefore are not approved for VDS methods.

Determination of Total Dissolved Solids

Calibrate and verify that the analytical balance is working within the proper parameters in accordance with SOP SA-AN-100: *Laboratory Support Equipment (Verification and Use)*.

Note: If TSS is to be determined along with the TDS, the filter used in the filtration step should be prepared in accordance with Attachment 7.

If determining VSS, measure the weight of the pre-labeled, pre-cleaned porcelain dishes to the nearest 0.1mg on an analytical balance and record as the tare weight in the analytical batch.

Note: If utilizing pre-weighed vessels in place of porcelain evaporating dishes, for TDS, no initial weight is required. Record the vessel ID and vessel weight found stamped on each individual vessel in the TALS AD batch. Transport vessels to and from the oven using a modular rack.

A sample's conductivity can be used as a screening test for TDS. Measure the conductivity of the unfiltered samples. If the specific conductance of the sample is less than 200umho/cm, filter 200mL of the sample. The maximum capacity of the preweighed vessels is 200mL. If the specific conductance is greater than 200umho/cm, filter and

analyze an aliquot of sample as outlined in the table below. Record the specific conductivity in the TALS AD batch.

Conductivity (umho/cm)	Sample Volume (mL)
0-200	200
201-2000	50
2001-4000	25
4001-6000	10
>6000	5
>10,000	1

Note: The volume used for analysis must produce a TDS residue between 2.5mg and 200mg.

If greater than 200mg residue is obtained, the sample must be re-analyzed utilizing a larger dilution (i.e., a smaller sample volume).

If less than 2.5mg of residue was determined utilizing a reduced volume of sample (less than 200mL), repeat the test using a volume of sample up to 200mL. If a minimum of 2.5mg of residue is not achieved utilizing 200mL of sample document with an NCM.

Procedure:

Assemble the filtration apparatus.
Place a filter pad in the filter support with the rough side up. If determining TSS on the same sample, transfer a tared filter from the numbered aluminum pan to the filter support with the rough side up. Clamp the filter into place and add a small aliquot (~5mL) of reagent water to the filtering apparatus to seat the filter.

Mix the sample well before taking an aliquot for analysis. Transfer the aliquot, determined by specific conductance above, of the well mixed sample to a graduated cylinder. Do not use a narrow bore pipette to measure the aliquot if TSS is also being determined because a pipette may strain some of the particulates out of the sample aliquot. Record the sample identification, dish number, and volume of sample in the TALS AD batch.

Note: The volume needed for the TSS and the volume of sample needed for the TDS may be different. A sample with a low conductivity but containing a high concentration of suspended solids may require several filters to obtain the needed volume of sample for TDS. In this case, the TSS and TDS should not be analyzed from the same sample aliquot. The sample aliquot must be selected to give a TDS residue of less than or equal to 200mg.

Remove the evaporating dish from the desiccator and weigh it on the analytical balance. Record the evaporating dish ID and the weight of the dish to the nearest 0.0001g in the TALS AD batch.

Note: If utilizing pre-weighed vessels in place of porcelain dishes, no initial weight is required. Record the vessel ID and vessel weight found stamped on each individual vessel in the TALS AD batch. Transport vessels to and from the oven using a modular rack.

Turn on the pump and carefully pour the entire sample aliquot into the filter funnel. Use a small aliquot of reagent water to rinse the entire sample out of the graduated cylinder and on to the filter. Use another small portion of reagent water to rinse down the sides of the filter funnel to transfer all of the particulates onto the filter.

Allow the pump to remain on for a few moments after all of the particulates have been transferred to the filter to remove the water trapped on the filter.

Turn off the pump and release the vacuum. If TSS is being determined concurrently, carefully remove the filter disc from the filter support with the forceps and place the filter in an aluminum pan. Transfer the filtrate from the receiving flask to a properly labeled container.

Note: The entire filtrate must be used for the determination of TDS; that is, the sample filtrate and the reagent water used to rinse the graduated cylinder and the receiving flask are included in the aliquot for TDS. Since the reagent water contains very low concentrations of dissolved solids, the addition of small quantities of reagent water will have no net effect on the concentration of TDS in the sample.

Carefully add the filtered sample to the appropriate tared evaporating dish or pre-weighed vessel and place it in the drying oven maintained at 115°C to 125°C to evaporate the liquid portion of the sample. This will generally require overnight in the oven. Record the time, the temperature that is uncorrected and corrected, thermometer ID and the oven ID in the TALs batch.

After the sample has evaporated, transfer the sample to an oven than has been heated to 180°C ± 2°C and heat for a minimum of 2 hours. Record the time, the temperature that is uncorrected and corrected, thermometer ID and the oven ID in the TALs batch.

Remove the sample containers from the oven, allow them to cool slightly, and place them in the desiccator. Remember not to place the sample containers on a surface that can cause dirt or other foreign objects to adhere to the dishes.

Allow the sample containers to remain in the desiccator until completely cool. Remove from the desiccator and weigh the vessels.

The Total Dissolved Solids is calculated using the following equation:

$$TDS, mg/L = \frac{A - B}{V} \otimes 1000 mg/g$$

Where:
A = weight of sample residue and evaporating dish (g)
B = weight of evaporating dish (g)
V = volume of sample analyzed (L)

QC Sample Preparation and Analysis

For each batch of 20 or fewer samples prepare a minimum of one method blank, one laboratory control sample, and two separate sample duplicates.

For the method blank, follow the procedures outlined above and use 100mL for porcelain dishes and 200mL for pre-weighed vessels of reagent water to serve as the blank matrix.

For the laboratory control sample, follow the procedures outlined above and use 50mL of the multi-analyte solids standard..

For the sample duplicate sample, follow the procedures outlined above and prepare and analyze one sample in duplicate per batch or 1 sample in duplicate for every 10 samples analyzed, whichever is greater.

Attachment 10:

Volatile Dissolved Solids (VDS) Procedures
EPA 160.4 and SM2540E

Preparation of Evaporating Dishes

Note that the same evaporating dishes will be used to determine the TDS and the VDS. The pre-weighed vessels may not be used for TDS if VDS is required.

Thoroughly clean the 100mL and 200mL evaporating dishes that will be required for the determination of VDS. It may be necessary to place Nochromix in the dish for a short time to remove dissolved solids from a previous analysis.

Rinse the evaporating dishes thoroughly with reagent water and allow them to dry. The dishes should be placed in the oven at 180°C to facilitate the drying.

Remove the evaporating dishes from the oven and place them in the muffle furnace. Turn on the muffle furnace, and elevate the temperature 100°C per hour until it reaches 550°C ± 50°C.

After the evaporating dishes have remained in the muffle furnace at 550°C ± 50°C for a minimum of one hour, remove the dishes and place them in the desiccator. Do not seal the door of the desiccator completely until the dishes have cooled. After the evaporating dishes have cooled, close the door of the desiccator. The evaporating dishes may be stored in the desiccator until ready for use.

Note: If the evaporating dishes are placed into the desiccator while hot and the top or door is sealed, a vacuum may form as the dishes cool. The top or door may be difficult to open if this occurs. This can occur very readily after the evaporating dishes have been removed from the muffle furnace.

Note: VDS determinations (and their associated TDS determination) must be performed using tared evaporating dishes ONLY, as the pre-weighed vessels are not capable of withstanding temperatures of 550 °C ± 50°C and therefore are not approved for VDS methods.

Determination of Volatile Dissolved Solids

Determine the Total Dissolved Solids as described in Attachment 9. Use evaporating dishes that have been prepared as above.

Place the evaporating dishes in the muffle furnace. Turn on the muffle furnace, and elevate the temperature 100°C per hour until it reaches 550°C ± 50°C. Record the time, oven ID, thermometer ID and temperature that is corrected and uncorrected once the range of 550°C ± 50°C is reached in the TALS batch.

After the evaporating dishes have remained in the muffle furnace at 550°C ± 50°C for a minimum of one hour, remove the evaporating dishes from the muffle furnace and place

them on a non-reactive surface to cool. Record the time, temperature that is uncorrected and corrected in the TALs batch.
Caution: the dishes will be extremely hot when removed. Do not place them on a surface that will melt or ignite

Note: One option is to turn the muffle furnace off and let then dishes cool in the furnace. After the dishes have cooled, place them in desiccator for at least one hour or overnight.

Remove the dishes from the desiccator and weigh the dishes to constant weight using the procedure outlined in Attachment 14.

The Volatile Dissolved Solids is calculated:

$$Volatile\ \ dissolved\ \ solids,\ mg/L = \frac{R_{180}\ -\ R_{550}}{V} \otimes 1000 mg/g$$

Where:
R_{180} = (weight of sample residue + evaporating dish @ 180°C)g - (evaporating dish weight)g = residue at 180°C (g)
R_{550} = (weight of residue + evaporating dish @ 550°C)g - (weight of evaporating dish)g = residue at 550°C (g)
V = volume of sample analyzed

QC Sample Preparation and Analysis
For each batch of 20 or fewer samples prepare a minimum of one method blank and two separate sample duplicates.

For the method blank, follow the procedures outlined above and use 100mL of reagent water for porcelain dishes to serve as the blank matrix.

For the sample duplicate sample, follow the procedures outlined above and prepare and analyze one sample in duplicate per batch or one sample in duplicate for every 10 samples analyzed, whichever is greater.

Attachment 11:

Settleable Solids Procedures
EPA 160.5 and SM2540F

Sample Preparation and Analysis

Remove the samples from the refrigerator and allow them to come to room temperature.

Rinse the Imhoff cone well with reagent water. Wash with detergent, if needed.

Place the Imhoff cone in the holder and pour 1L of sample into the cone.

Allow the sample to settle for 45 minutes, using a NIST-traceable timer to measure elapsed time. Stir the sample gently with a glass rod to free matter stuck to the sides of the Imhoff cone.

Allow the sample to settle an additional 15 minutes, again using a NIST-traceable timer to measure elapsed time. Read the volume of settleable matter and report the results in mL/L. If less than 0.1mL of material has settled in the bottom, report the result as <0.1mL/L.

Ensure the following items are captured in the Batch Information screen in TALS:
Timer ID
Imhoff Cone ID
First Start Time
First End Time
Second start Time
Second End Time

For some samples a separation of settleable and floating materials will occur. In these instances, the volume attributed to the floating materials is not included in the determination of the final result.

QC Sample Preparation and Analysis

For each batch of 20 or fewer samples prepare a minimum of one method blank and two separate sample duplicates.

For the method blank, follow the procedures outlined above and use 1L of reagent water to serve as the blank matrix.

For the sample duplicate sample, follow the procedures outlined above and prepare and analyze one sample in duplicate per batch or 1 sample in duplicate for every 10 samples analyzed, whichever is greater.

Attachment 12:

Percent Moisture and Total Solids Procedures SM2540G

Preparation of the Evaporating Dishes or Crucibles

Thoroughly clean the evaporating dishes or crucibles that will be required for the determination of total solids. It may be necessary to place Nochromix in the dish for a short time to remove dissolved solids from a previous analysis.

Rinse the evaporating dishes or crucibles thoroughly with reagent water and allow them to dry. The dishes should be placed in muffle furnace to facilitate the drying.

After the evaporating dish has remained in the muffle furnace for a minimum of two hours, remove the dishes from the oven and place in the desiccator. Do not close the door of the desiccator completely until the dishes have cooled. After the dishes have cooled, close the door of the desiccator. The evaporating dishes and crucibles may be stored in the desiccator until ready for use.

Note: If the dishes are placed into the desiccator while hot and the door is sealed, a vacuum may form as the dishes cool. If this occurs, the door of the desiccator may be difficult to open.

Determination of Total Solids (or Percent Moisture)

Calibrate and verify that the top loading balance is working within the proper parameters in accordance with SOP SA-AN-100: *Laboratory Support Equipment (Verification and Use)*.

Label the drying dish with an identification number.

Measure the weight of the pre-labeled, pre-cleaned dishes to the nearest 0.1mg on an analytical balance. Record this information as the tare weight in the analytical balance.

Thoroughly mix the sample with a stainless steel spatula or glass rod. It is important to obtain a homogeneous mixture prior to sub-sampling so that the sub-sample will accurately reflect the composition of the sample. Leaves, rocks, and other foreign materials should not be included in the sub-sample.

Note: If there is any doubt as to how to treat a given sample, contact the department manager to determine the proper course of action. SOP SA-QA-015*: Homogenization, Compositing, and Segregation of Samples* provides guidance for homogenizing samples.

Add approximately a 25-50g aliquot of the well-mixed sample to the dish. Record the weight to the nearest 0.1mg in the analytical balance.

Place the sample in the drying oven, maintained at 103°C -105° for at least 12 hours (overnight).

Remove the evaporation dishes from the oven, allow them to cool slightly, and place them in the desiccator. Remember not to place the evaporating dishes on a surface that can cause dirt or other foreign objects to adhere to the dishes.

Allow the evaporating dishes to remain in the desiccator until completely cool.

Remove the dishes from the desiccator and weigh the dishes to constant weight using the procedure outlined in Attachment 14.

The Percent Solids is calculated using the following equation:

$$percent_total_solids = \frac{A - B}{W} x\,100$$

Where:
A = weight of sample residue and evaporating dish (g)
B = weight of evaporating dish (g)
W = weight of sample used to determine the total solids (g)
i.e., W = weight $_{wetsample + dish}$ – weight$_{dish}$

The Percent Moisture is calculated using the following equation:

$$percent_moisture = \frac{W - (A - B)}{W} x\,100$$

Where:
A = weight of sample residue and evaporating dish (g)
B = weight of evaporating dish (g)
W = weight of sample used to determine the total solids (g)
i.e., W = weight $_{wetsample + dish}$ – weight$_{dish}$

QC Sample Preparation and Analysis

For each batch of 20 or fewer samples prepare a minimum of one method blank and two separate sample duplicates.

For the method blank, follow the procedures outlined above and use 10g of Ottawa Sand that has been prepared for metals analysis to serve as the blank matrix.

For the sample duplicate sample, follow the procedures outlined above and prepare and analyze one sample in duplicate per batch or 1 sample in duplicate for every 10 samples analyzed, whichever is greater.

Attachment 13:
Volatile Solids, Ash Content, and Fixed Solids Procedures
SM2540G

Preparation of the Evaporating Dishes and Crucibles

Note that the same evaporating dishes will be used to determine the % moisture and total solids.

Thoroughly clean the evaporating dishes or crucibles that will be required for the determination of ash content and fixed solids. It may be necessary to place Nochromix in the dish for a short time to remove dissolved solids from a previous analysis.

Rinse the evaporating dishes thoroughly with reagent water and allow them to dry. The dishes should be placed in the oven at 105°C to facilitate the drying.

Remove the evaporating dishes from the oven and place them in the muffle furnace. Turn on the muffle furnace, and elevate the temperature 100°C per hour until it reaches 550°C ± 50°C.

After the evaporating dishes have remained in the muffle furnace at 550°C ± 50°C for a minimum of one hour, remove the dishes and place them in the desiccator. Do not seal the door of the desiccator completely until the dishes have cooled. After the evaporating dishes have cooled, close the door of the desiccator. The evaporating dishes may be stored in the desiccator until ready for use.

Note: If the evaporating dishes are placed into the desiccator while hot and the top or door is sealed, a vacuum may form as the dishes cool. The top or door may be difficult to open if this occurs. This can occur very readily after the evaporating dishes have been removed from the muffle furnace.

Determination of Volatile Solids, Ash Content, and Fixed Solids

Determine Percent Moisture, Total Solids (2540G) as described in Attachment 13. Use evaporating dishes that have been prepared as above.

Place the evaporating dishes in the muffle furnace. Turn on the muffle furnace, and elevate the temperature 100°C per hour until it reaches 550°C ± 50°C.

After the evaporating dishes have remained in the muffle furnace at 550°C ± 50°C for a minimum of one hour, remove the evaporating dishes from the muffle furnace and place them on a non-reactive surface to cool. Caution: the dishes will be extremely hot when removed. Do not place them on a surface that will melt or ignite

Note: One option is to turn the muffle furnace off and let then dishes cool in the furnace.

After the dishes have cooled, place them in desiccator for at least one hour or overnight.

Remove the dishes from the desiccator and weigh the dishes to constant weight using the procedure outlined in Attachment 14.

The Ash Content is calculated using the following equation:

$$percent_ash = \frac{A}{B} x\,100$$

Where:
A = weight of sample residue after heating to 550°C (g)
B = weight of sample residue after heating to 105°C (g)

The Ash content can also be reported as % Fixed Solids

The Volatile Solids is calculated using the following equation:

$$percent_volatile_solids = \frac{B - A}{B} x\,100$$

Where:
A = weight of sample residue after heating to 550°C (g)
B = weight of sample residue after heating to 105°C (g)

QC Sample Preparation and Analysis

For each batch of 20 or fewer samples prepare a minimum of one method blank and two separate sample duplicates.

For the method blank, follow the procedures outlined above and use 10g of Ottawa Sand that has been prepared for metals analysis to serve as the blank matrix.

For the sample duplicate sample, follow the procedures outlined above and prepare and analyze one sample in duplicate per batch or 1 sample in duplicate for every 10 samples analyzed, whichever is greater.

Attachment 14:

Constant Weight Determination Procedures

For all residue determinations except percent solids/moisture, the constant weight must be achieved. To verify constant weight has been achieved, replicate weighings of each sample must occur. Each sample must be placed back into the oven at the appropriate temperature and re-weighed after a minimum of 1 hour.

If the percent difference between the initial and subsequent weighing is less than 4% or the difference is less than 0.5mg from the previous weighing, the sample has been dried to a constant weight, and no further drying time is needed.

If the percent difference is greater than 4%, or if the difference is greater than 0.5mg, the sample must be dried and re-weighed until the “constant weight” criteria are achieved.

An NCM must be generated if constant weight cannot be achieved after a third weighing is performed.

Note: The “percent difference” and “difference” limits are separate criteria. Only one has to be met. The difference criteria will apply to low concentration samples and the percent difference criteria to higher concentration samples.

Note: When utilizing pre-weighed vessels, ensure a static-free environment when performing weigh-backs by using the De-ionizing U-Electrode in conjunction with the analytical balance. To ensure that the vessel is centered on the analytic balance platform, always use the weighing bracket.

Attachment 15:

Percent Solids/Moisture Procedures (Used for Dry Weight Correction) EPA 3500B, EPA 3500C, EPA 3550B, and EPA 3550C

Determination of Percent Solids (or Percent Moisture)

Calibrate and verify that the top loading balance is working within the proper parameters in accordance with SOP SA-AN-100: *Laboratory Support Equipment (Verification and Use)*.

Label an aluminum pan with an identification number. Create a new Moisture Batch in Analyst Desktop in TALS and enter the TALS IDs of the samples to be tested. Weigh the pan on the top loading balance. Record the weight (to the nearest 0.01g) of the aluminum pan in the column labeled "DishWeight" in the row of the corresponding sample.
****** Do NOT TARE the balance after weighing the dish.

Thoroughly mix the sample with a stainless steel spatula or glass rod. It is important to obtain a homogeneous mixture prior to sub-sampling so that the sub-sample will accurately reflect the composition of the sample. Leaves, rocks, and other foreign materials should not be included in the sub-sample.

Note: If there is any doubt as to how to treat a given sample, contact the department manager to determine the proper course of action. SOP SA-QA-015: Homogenization, *Compositing, and Segregation of Samples* provides guidance for homogenizing samples.

Add approximately a 10g aliquot of the well-mixed sample to the un-tared aluminum pan. The amount can vary by as much as +/- 50%. Record the weight to the nearest 0.01g in the column labeled "SampleMassWet."

Place the sample in the drying oven, maintained at 105°C +/- 5°C, for a minimum of 4 hours for % solids used for DW determination.

Remove the aluminum pan from the oven and allow to cool. Remember not to place the aluminum pan on a surface that can cause dirt or other foreign objects to adhere to the pan. Ensure that the surface that the pan is placed on can handle the temperature of the aluminum pan without damage.

Weigh the aluminum pan containing the sample residue on the top-loading balance and record the weight to the nearest 0.01g in the column labeled "SampleMassDry."

The TALS program automatically calculates the results for Percent Solids according to the equations listed below.

Use the following equation to calculate the Percent Solids:

$$Percent\ Solids = \frac{A - B}{C - D}\ x\ 100$$

Where:
A = weight of dry sample and aluminum pan
B = weight of aluminum pan
C = weight of wet sample and aluminum pan
D = weight of aluminum pan

Use the following equation to express the percent solids as a decimal equivalent ("Solids") for calculating sample results on a "dry weight basis":

$$\text{Solids} = \frac{\text{Percent Solids}}{100},$$

Or

$$Solids = \frac{A - B}{C - D}$$

Where:
A = weight of dry sample and aluminum pan
B = weight of aluminum pan
C = weight of wet sample and aluminum pan
D = weight of aluminum pan

Use the following equation to express the percent solids as percent moisture:

$$\text{Percent Moisture} = 100 - \text{Percent Solids}$$

Attachment 16:
Labware Cleaning Procedures

The graduated cylinder used for measuring initial sample volumes must be rinsed three times with reagent water between samples. Additionally, each day all non-disposable apparatus must be cleaned as outlined below:

GLASSWARE CLEANING PROCEDURES - GENERAL LAB

MISCELLANEOUS APPARATUS

1. Scrub with hot tap water and Liquinox.
2. Rinse thoroughly with tap water.
3. Rinse at least three times with DI water.
4. Air-dry and store inverted in a cabinet or capped.

NOTE:

1. Do not use soap for cleaning COD digestion tubes.
2. Burets may be cleaned by filling with 10% KOH in methanol, allowing to stand for one hour, then rinsing thoroughly with DI water.
3. Evaporating dishes used for TDS determinations are cleaned by rinsing thoroughly with tap water, soaking for several hours in Nochromix, rinsing thoroughly with tap water, and rinsing three times with DI water. The dishes are then oven-dried.

TKN / TOTAL-P DIGESTION TUBES

1. Scrub with hot tap water and Liquinox.
2. Rinse thoroughly with tap water.
3. Rinse once with Nochromix.
4. Rinse at least three times with DI water.

CYANIDE DISTILLATION APPARATUS

1. Scrub with hot tap water and Liquinox.
2. Rinse thoroughly with tap water.
3. Rinse with 10% nitric acid, being certain to force at least 10mL through each dispersion tube.
4. Rinse thoroughly with DI water.

ODOR-FREE GLASSWARE

1. Scrub with hot tap water and Liquinox.
2. Rinse with acid cleaning solution.
3. Rinse thoroughly with odor-free water.
4. Glassware used in odor testing should be reserved for that purpose only.

FGE003:08.17.18:2

Revision History

Summary of Changes Revision 12B:

- Updated Equipment Listing with current ovens utilized in the laboratory. ANAB Audit 2020.

Summary of Changes Revision 12A:

- Section 2.5 and Attachment 9 have been updated with oven conditions for the evaporation of TDS samples. Samples will be evaporated at 115-125C. ANAB Audit 2020
- Added Portable Conductivity Meter to Section 6.1.
- Updated section 7.3 with current standards utilized.
- Section 9.1 the LCS as a default QC item for EPA160.4 and SM2540E was removed.
- Added comments to section 9.3 that an NCM must be generated if constant weight is not achieved after three weighing.
- Updated disposal of soil material in section 14.1
- Removed the LCS and LCSD criteria in Attachment 3 for method EPA160.4, SM2540E and SM2540G.
- Removed use of PC titrate to measure conductivity in Attachment 5 and 9. The laboratory uses a portable conductivity meter which is calibrated each day of use.
- Removed prep of filters from Attachment 7 and Attachment 8. The lab purchased pre-certified filters for TSS and VSS.
- Updated Attachment 9 for TDS residue criteria. Sample must be reanalyzed if a minimum residue of 2.5mg is not achieved and less than 200mL of sample was utilized. An NCM must be initiated if a minimum residue of 2.5gm was not achieved when utilizing the volume of 200mL of sample. ANAB Audit 2020

Summary of Changes from Previous Revision 12:

- Updated SOP signatories to reflect current responsibilities and titles.
- Added part numbers for ProWeigh Double filters used for TSS and VSS analysis.
- Section 9.2- removed requirement for QA to login filter check for TSS. The ProWeigh Double filters utilized by the laboratory are tested for suitability by the vendor.
- Updated company name and logo where applicable.

MERCURY PREPARATION AND ANALYSIS

(Methods: EPA 245.1, EPA 7470A, and EPA 7471B)

Approvals (Signature/Date):

Kimberly Chamberlain 11/24/2020

Kimberly Chamberlain Date
Quality Assurance Manager /Technical Director
Environmental Health and Safety Coordinator

11/24/2020

Adriana Geiger Date
Department Manager

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1.0 Scope and Application

This SOP gives the procedures for the determination of mercury by cold vapor atomic absorption spectrophotometry (CVAA). The routine matrices performed by this procedure are waters and soils; however, this procedure may be adapted to accommodate other matrices as outlined in Section 16.1.

The reporting limits (RL), the method detection limits (MDL), and the accuracy and precision criteria associated with this procedure are provided in the TALS Method Limit Groups (MLGs).

This SOP was written by and for Eurofins TestAmerica's Savannah laboratory.

2.0 Summary of Method

This procedure is based on the absorption of characteristic radiation at 253.7nm by mercury vapor. Samples are digested with acids and potassium permanganate via the hot block digestion procedure. Upon analysis, the instrument introduces stannous chloride to the digestate to convert all forms of mercury to the same oxidation state. The mercury is then aerated from solution after passing through a mixing coil. The mixture passes through a gas/liquid separator and through a drying tube. The vapor is passed through a flow cell positioned in the light path of an atomic absorption spectrophotometer. Mercury concentration is measured as a function of absorbance.

This SOP is based on the following methods: EPA 7470A (liquids), EPA 7471B (solids), and EPA 245.1.

3.0 Definitions

Refer to the Glossary Section of the *Quality Assurance Manual* (QAM) for a complete listing of applicable definitions and acronyms.

4.0 Interferences

4.1 Procedural Interferences

4.1.1 Interferences may be caused by contaminants in solvents, reagents, glassware, and other sample processing apparatus and can make identification and/or quantification of the target analytes difficult.

4.1.2 All sample collection containers are single-use disposable containers which limits the potential for contamination. All non-disposable labware must be scrupulously cleaned in accordance with the posted Labware Cleaning Instructions to ensure it is free from contaminants and does not contribute artifacts.

4.1.3 High purity reagents and solvents are used to help minimize interference problems. Hydrochloric acid, nitric acid, and sulfuric acid must be verified prior to use in accordance with the Eurofins TestAmerica Solvent Lot Testing Program.

4.1.4 Instrument and/or method blanks are routinely used to demonstrate all reagents and apparatus are free from interferences under the conditions of the analysis.

4.2 Matrix Interferences

4.2.1 Matrix interferences may be caused by contaminants that are co-extracted from the sample matrix. The sample may require cleanup or dilution prior to analysis to reduce or eliminate the interferences.

4.2.2 Interfering contamination may occur when a sample containing low concentrations of analytes is analyzed immediately following a sample containing relatively high concentrations of analytes. As such, samples known to be clean should be analyzed first. To prevent carryover into subsequent samples, analysis of reagent blanks may be needed after the analysis of a sample containing high concentrations of analytes.

4.2.3 Potassium permanganate is added to eliminate the possibility of interference from sulfide and certain organic compounds.

4.2.4 High levels of residual chlorine (such as those produced when seawaters, brines, and industrial effluents high in chlorides are digested) are known to interfere with this analysis. Addition of extra potassium permanganate may be needed during the digestion of samples containing chloride. Also, the samples are not capped tightly during digestion so that excess chlorine can escape.

4.2.5 Interferences have been reported for waters containing sulfide, chloride, copper and tellurium. Organic compounds which have broad band UV absorbance (around 253.7 nm) are confirmed interferences. The concentration levels for interferents are difficult to define. This suggests that quality control procedures must be strictly followed.

4.2.6 Volatile materials (e.g., chlorine) which absorb at 253.7 nm will cause a positive interference. In order to remove any interfering volatile materials, the dead air space in the digestion vessel should be purged before addition of stannous chloride solution.

5.0 Safety

Employees must abide by the policies and procedures in the Eurofins TestAmerica Environmental Health and Safety Manual (EHSM), the Eurofins TestAmerica Savannah Addendum to the EHSM, and this document.

This procedure may involve hazardous materials, operations, and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user to follow appropriate safety, waste disposal, and health practices under the assumption that all samples and reagents are potentially hazardous.

The analyst must protect himself/herself from exposure to the sample matrix. Many of the samples that are tested may contain hazardous chemical compounds or biological organisms. The analyst must, at a minimum, wear protective clothing (lab coat), eye protection (safety glasses or face shield), disposable latex or nitrile gloves, and closed-toe, nonabsorbent shoes when handling samples.

5.1 Specific Safety Concerns or Requirements

Nitric and hydrochloric acids are extremely hazardous as oxidizers, corrosives, poisons, and are reactive. Inhalation of the vapors can cause coughing, choking, irritation of the nose, throat, and respiratory tract, breathing difficulties, and lead to pneumonia and pulmonary edema. Contact with the skin can cause severe burns, redness, and pain. Nitric acid can cause deep ulcers, and staining of the skin to a yellow or yellow-brown color. These acid vapors are irritating and can cause damage to the eyes. Contact with the eyes can cause permanent damage.

Sulfuric acid is a strong oxidizer and is a corrosive. It will react violently when combined with organic compounds, possibly producing fire. Inhalation can cause irritation of the nose, throat, mucus membranes, and upper respiratory tract. Contact with the eyes can cause blurred vision, redness, pain, and even blindness.

Samples that contain high concentrations of carbonates or organic matter, or samples that are at elevated pH can react violently when acids are added. Acids must be added to samples under a hood to avoid splash/splatter hazards and/or possibly toxic vapors that will be given off when the samples are acidified.

The making of aqua regia can produce toxic fumes and heat. This procedure must be performed under a fume hood.

The exhaust of the mercury analyzer must be vented or trapped so that mercury vapors do not enter the laboratory.

The preparation of the samples for mercury analysis uses a hot-block with a temperature of ~95ºC. The water and the steam produced can cause burns to unprotected skin. Employees must use appropriate PPE when working with sample digestions.

Mercury compounds are highly toxic if swallowed, inhaled, or absorbed through the skin. Analyses should be conducted in a laboratory exhaust hood. The analyst should use chemical resistant gloves when handling concentrated mercury standards.

The acidification of samples containing reactive materials may result in the release of toxic gases, such as cyanides or sulfides. Acidification of samples should be done in a fume hood.

5.2 Primary Materials Used

The following is a list of the materials used in this procedure, which have a serious or significant hazard rating, and a summary of the primary hazards listed in their MSDS/SDS.

NOTE: This list does not include all materials used in the procedure. A complete list of materials used in this procedure can be found in the Reagents and Standards Section and the Equipment and Supplies Section of this SOP.

Employees must review the information in the MSDS/SDS for each material before using it for the first time or when there are major changes to the MSDS/SDS. Electronic copies of MSDS/SDS can be found using the "MSDS" link on the Oasis homepage or on the EH&S webpage on Oasis.

Material	Hazards	Exposure Limit[1]	Signs and Symptoms of Exposure
Sulfuric Acid[2]	Corrosive Oxidizer Dehydrator Poison	1mg/m^3 TWA	Inhalation produces damaging effects on the mucous membranes and upper respiratory tract. Symptoms may include irritation of the nose and throat, and labored breathing. Symptoms of redness, pain, and severe burn can occur. Contact can cause blurred vision, redness, pain and severe tissue burns. Can cause blindness.
Nitric Acid[2]	Corrosive Oxidizer Poison	2ppm TWA 4ppm STEL	Nitric acid is extremely hazardous; it is corrosive, reactive, an oxidizer, and a poison. Inhalation of vapors can cause breathing difficulties and lead to pneumonia and pulmonary edema, which may be fatal. Other symptoms may include coughing, choking, and irritation of the nose, throat, and respiratory tract. Can cause redness, pain, and severe skin burns. Concentrated solutions cause deep ulcers and stain skin a yellow or yellow-brown color. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
Hydrochloric Acid[2]	Corrosive Poison	5ppm Ceiling	Inhalation of vapors can cause coughing, choking, inflammation of the nose, throat, and upper respiratory tract, and in severe cases, pulmonary edema, circulatory failure, and death. Can cause redness, pain, and severe skin burns. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.

Material	Hazards	Exposure Limit[1]	Signs and Symptoms of Exposure
Potassium Permanganate	Oxidizer	5mg/m^3 for Mn Compounds	Causes irritation to the respiratory tract. Symptoms may include coughing, shortness of breath. Dry crystals and concentrated solutions are caustic, causing redness, pain, severe burns, brown stains in the contact area and possible hardening of outer skin layer. Diluted solutions are only mildly irritating to the skin. Eye contact with crystals (dusts) and concentrated solutions causes severe irritation, redness, and blurred vision and can cause severe damage, possibly permanent.
Potassium Persulfate	Oxidizer	None	Causes irritation to the respiratory tract. Symptoms may include coughing, shortness of breath. Causes irritation to skin and eyes. Symptoms include redness, itching, and pain. May cause dermatitis, burns, and moderate skin necrosis.
[1]Exposure limit refers to the OSHA regulatory exposure limit.			
[2]Always add acid to water to prevent violent reactions.			

6.0 Equipment and Supplies

6.1 Equipment and Instrumentation

Top-loading Balance – Verify in accordance with SOP SA-AN-100: *Laboratory Support Equipment: Verification and Use*

Thermometers – Verify in accordance with SOP SA-AN-100: *Laboratory Support Equipment: Verification and Use*

Water bath or heating block capable of maintaining temperature of 95 ± 3°C

Leeman Hydra AA or other suitable automated mercury analyzer

Mercury Hollow Cathode Lamp

Absorption Cell

Nitrogen or argon gas supply and appropriate fittings

Air Pump

Pump (Aeration) tubing of appropriate sizes for use on the Hydra AA

Drying Tube – Purchased pre-packed from Leeman Labs

6.2 Analytical Data System / Software / Hardware

The Leeman software is used on a Windows-based PC to schedule and acquire data. The raw data from the Hg Analyzer is manually checked for validity by a minimum of 2 analysts. Raw instrument data are uploaded to the laboratory's LIMS (i.e., TALS) via the Environmental Information Systems Corporation Metals Analytical Review. Additionally, any quality control failures are flagged in TALS, as appropriate.
The QA department maintains a Master Equipment Listing which documents to Firmware and/or Software utilized by the instrumentation.

6.3 Volumetric Containers and Dispensers

All volumetric labware must be verified in accordance with SOP SA-AN-100: *Laboratory Support Equipment (Verification and Use).* Refer to Attachment 6 for Labware Cleaning Procedures.

Volumetric Labware	Volume	Type (Quantitative / Qualitative)	Use	Verification Frequency	Laboratory Verification Criteria
Disposable Medicine Cups	10mL	Qualitative	Used to contain sample aliquot for pH measurement	None	None
Autosampler Vials	10mL	Qualitative	Holding sample on instrument	None	None
Digitube Disposable Digestion Vials (Class A)	100mL	QUANTITATIVE	Preparing Digestions, Determination of Initial and Final Volumes	None, if received w/ COA	None, if received w/ COA
Eppendorf-Style Mechanical Pipettes	Various	QUANTITATIVE	Preparing Dilutions, Standard and Reagent Preparation and Spiking	Prior to 1st use, then quarterly thereafter (Daily for DOD)	Accuracy = 2% Precision = 1%
Glass Volumetric Flasks (Class A)	2L	QUANTITATIVE	Reagent Preparation	None (Class A)	None (Class A)
Glass Beakers (Class A)	2L	QUANTITATIVE	Reagent Preparation	None (Class A)	None (Class A)
Glass Disposable Pipettes	5mL	Qualitative	Preparing Dilutions, Standard and Reagent Preparation and Spiking	Per Lot	Accuracy = 2% Precision = 1%
Pump-Style Mechanical Pipettes	Various	QUANTITATIVE	To deliver acids/bases for pH adjustments	Prior to 1st use, then quarterly thereafter (Daily for DOD)	Accuracy = 2% Precision = 1%

6.3 Lab Supplies

pH paper

6.4 Sample Collection Containers

All sample collection containers are single-use disposable containers which limits the potential for contamination.

The routine sample collection containers supplied by the laboratory are:

Waters or Field-Filtered Dissolved Mercury Samples:
250mL plastic, nitric acid – purchased with Certificate of Analysis attesting to purity.

Soil Samples:
4oz plastic soil jar, unpreserved – purchased with Certificate of Analysis attesting to purity.

Wipe Samples:
40mL VOA vial, Acetic acid in water, pH 4.93 +/- 0.05 – purchased with Certificate of Analysis attesting to purity.

Leachate Samples (originally aqueous):
1L amber glass, unpreserved – purchased with Certificate of Analysis attesting to purity.

Leachate Samples (originally solid):
16oz glass soil jar, unpreserved – purchased with Certificate of Analysis attesting to purity.

Lab-Filtered Dissolved Mercury Samples
250mL plastic, unpreserved – purchased with Certificate of Analysis attesting to purity.

7.0 Reagents and Standards

The standards and reagents listed are those in use at the time this SOP was updated. Other standard and reagent stocks, vendors, and concentrations may be used provided they are fully documented and in compliance with method requirements.

7.1 Expiration Dates

Expiration dates (time from initial use or receipt to final use) for standard and reagent materials must be set according to the guidance in this SOP. Note: These are maximum expiration dates and are not to be considered an absolute guarantee of standard or reagent quality. Sound judgment must be used when deciding whether to use a standard or reagent. If there is doubt about the quality of a standard or reagent material, a new material must be obtained or the standard or reagent material verified. Data quality must not be compromised to extend a standard's life.

The expiration date of any standard or reagent must not exceed the expiration date of the standard or reagent that was used to prepare it.

7.2 Reagents

Reagents must be prepared and documented in accordance with SOP SA-AN-041: *Reagent and Standard Materials Procedures.*

Hydrochloric acid, nitric acid, and sulfuric acid must be verified prior to use in accordance with the Eurofins TestAmerica Solvent Lot Testing Program.

7.2.1 Blank Matrix – Teflon Chips, Ottawa Sand, or other suitable matrix. Used for the preparation of soil QC samples.

7.2.2 Laboratory Reagent Water – ASTM Type II

7.2.3 Nitric Acid (HNO_3), concentrated, reagent grade – stable under ordinary conditions of use and storage
Storage: Store in a cool, dry, ventilated storage area with acid resistant floors and good drainage. Store away from sunlight, heat, water, and incompatible materials.
Expiration:
Unopened: Manufacturer's expiration date
Opened: 5 years from date opened

7.2.4 Hydrochloric Acid (HCl), concentrated-reagent grade – stable under ordinary conditions of use and storage
Storage: Store in a cool, dry, ventilated storage area with acid resistant floors and good drainage. Store away from sunlight, heat, water, and incompatible materials.
Expiration:
Unopened: Manufacturer's expiration date.
Opened: 5 years from date opened

7.2.5 Aqua Regia (1:1): To 4 volumes of reagent water, carefully add 3 volumes of concentrated HCl and 1 volume of concentrated HNO3. Store this reagent away from incompatibles, combustibles, organics, and other readily oxidizable materials. This reagent will be stable for up to 3 months.

Note: This reagent must be prepared under a fume hood.

7.2.6 Potassium permanganate ($KMnO_4$), mercury free – stable under ordinary conditions of use and storage.
Storage: Store in a tightly closed container in a cool, dry, ventilated area. Keep away from heat and avoid storage on wood floors. Store away from incompatibles, combustibles, organics and other readily oxidizable materials.

Expiration:
Unopened: Manufacturer's expiration date
Opened: 5 years from date opened

7.2.7 Potassium permanganate, mercury-free, 5% solution (w/v) – Dissolve 50g of $KMnO_4$ in 1000mL of reagent water. Stable under ordinary conditions of use and storage.
Storage: Store in a tightly closed container in a cool, dry, ventilated area. Keep away from heat and avoid storage on wood floors. Store away from incompatibles, combustibles, organics and other readily oxidizable materials.
Expiration: 1 year from date prepared

7.2.8 Sodium Chloride (NaCl) – stable under ordinary conditions of use and storage.
Storage: Store in a tightly closed container in a cool, dry, ventilated area.
Expiration:
Unopened: Manufacturer's expiration date
Opened: 5 years from date opened

7.2.9 Hydroxylamine Sulfate ($(NH_2OH)•2H_2O$) – stable under ordinary conditions of use and storage.
Storage: Store in a tightly closed container in a cool, dry, ventilated area. Store away from incompatible materials.
Expiration:
Unopened: Manufacturer's expiration date
Opened: 5 years from date opened

7.2.10 Sodium chloride hydroxylamine sulfate solution – Dissolve 120g NaCl and 120g hydroxylamine sulfate in reagent water in a 1-L volumetric flask and dilute to volume. Stable under ordinary conditions of use and storage.
Storage: Store in a tightly closed container in a cool, dry, ventilated area. Store away from incompatible materials.
Expiration: 1 year from date prepared

7.2.11 Potassium persulfate ($K_2S_2O_8$) – stable under ordinary conditions of use and storage.
Storage: Store in a tightly closed container in a cool, dry, ventilated area. Keep away from heat and avoid storage on wood floors. Store away from incompatibles, combustibles, organics and other readily oxidizable materials.
Expiration:
Unopened: Manufacturer's expiration date
Opened: 5 years from date opened

7.2.12 Potassium persulfate, 5% solution (w/v) – Dissolve 50g potassium persulfate in 1000mL reagent water. Stable under ordinary conditions of use and storage.
Storage: Store in a tightly closed container in a cool, dry, ventilated area. Keep away from heat and avoid storage on wood floors. Store away from incompatibles, combustibles, organics and other readily oxidizable materials.
Expiration: 1 year from date prepared

7.2.13 Rinse Water, 5% HCl / 1%HNO_3 – to a clean 2-L bottle, add 1-L of reagent water. Carefully add 100mL of concentrated hydrochloric acid. Carefully add 20mL of concentrated nitric acid. Dilute to a final volume of 2L. Other volumes may be utilized providing the reagent proportions remain the same. Stable under ordinary conditions of use and storage.

Storage: Store in a cool, dry, ventilated storage area with acid resistant floors and good drainage. Store away from sunlight, heat, water, and incompatible materials.
Expiration: 1 year from date prepared

7.2.14 Stannous chloride ($SnCl_2.2H_2O$) – reagent grade, suitable for mercury determination. Stable if stored in tightly closed containers.
Storage: Store in a tightly closed container in a cool, dry, ventilated area. Keep away from incompatible materials. It will absorb air and form the insoluble oxychloride.
Expiration:
Unopened: Manufacturer's expiration date
Opened: 5 years from date opened

7.2.15 Stannous chloride ($SnCl_2•2H_2O$) solution – to a clean 2L volumetric flask, add 100g of stannous chloride. Add approximately 400mL of reagent water. Carefully add 500mL of concentrated hydrochloric acid. Add a stirring bar, and stir on a stir plate until the stannous chloride is dissolved. Remove the stirring bar and dilute to volume with reagent water. Stable under ordinary conditions of use and storage.
Storage: Store in a tightly closed container in a cool, dry, ventilated area. Keep away from incompatible materials.
Expiration: 1 year from date prepared

7.2.16 Sulfuric Acid (H_2SO_4), concentrated reagent grade – stable under ordinary conditions of use and storage.
Storage: Store in a cool, dry, ventilated storage area with acid resistant floors and good drainage. Store away from sunlight, heat, water, and incompatible materials.
Expiration:
Unopened: Manufacturer's expiration date
Opened: 5 years from date opened

7.2.17 Nitric Acid (HNO_3), 1:1 – Slowly add 250mL of concentrated nitric acid to 250mL of laboratory reagent water in a 1-L beaker. Mix well and transfer to a tightly closed container.
Storage: Store in a cool, dry, ventilated storage area with acid resistant floors and good drainage. Store away from sunlight, heat, water, and incompatible materials.
Expiration: 2 years from date prepared

7.3 Standards

Standards must be prepared and documented in accordance with SOP SA-AN-041: *Reagent and Standard Materials Procedures.* Certificates of analysis or purity must be received with all purchased standards, and attached to TALS.

7.3.1 Hg Stock Standard, 1000mg/L – currently purchased from SPEX.
Storage: Store in a cool, dry, ventilated storage area with acid resistant floors and good drainage. Store away from sunlight, heat, water, and incompatible materials.
Expiration: Stable under ordinary conditions of use and storage up to the manufacturer's expiration date.

7.3.2 Second Source Hg Stock Standard, 1000mg/L – currently purchased from Environmental Express.
Storage: Store in a cool, dry, ventilated storage area with acid resistant floors and good drainage. Store away from sunlight, heat, water, and incompatible materials.
Expiration: Stable under ordinary conditions of use and storage up to the manufacturer's expiration date.

7.3.3 Calibration Standards

7.3.3.1 Mercury Intermediate Standard, 500ug/L – Add 0.050mL of the purchased 1000mg/L Hg Stock Standard and 2.5mL of nitric acid to about 50mL of reagent water in a 100-mL volumetric flask and dilute to volume with reagent water.
Storage: Store in a cool, dry, ventilated storage area with acid resistant floors and good drainage. Store away from sunlight, heat, water, and incompatible materials.
Expiration: Stable under ordinary conditions of use and storage for up to 28 days. The expiration date cannot exceed the expiration of any of the components.

7.3.3.2 Mercury Calibration Standards – Prepare ICAL daily according to the matrix given in the instructions below:

Waters:
- Transfer 0.0, 0.02, 0.04, 0.1, 0.3, and 0.5mL portions of the Mercury Intermediate Standard to a series of 100mL disposable vials.
- Add reagent water from a graduated cylinder to each vial to make a final volume of 50mL. This results in calibration standard concentrations of 0.0, 0.2, 0.4, 1.0, 3.0, and 5.0ug/L mercury.
- Mix well. Digest according to the procedures in Section 10.1.1.

Soils:
- Add 0.50g blank matrix and 5.0mL to each of six 100mL disposable vials.
- Transfer 0.0, 0.02, 0.04, 0.1, 0.3, and 0.5mL portions of the Mercury Intermediate Standard to the vials.
- This results in calibration standard concentrations of 0.0, 0.020, 0.040, 0.10, 0.30, and 0.50mg/kg mercury.
- Digest according to the procedures in Section 10.1.2, omitting the initial addition of 5mL of water.

7.3.4 Initial Calibration Verification Standards (also used as QCS and IPC standards)

7.3.4.1 Second Source Intermediate Standard, 1.0mg/L – Add 0.1mL of the 1000mg/L Second Source Hg Stock Standard, and 2.5mL of nitric acid to about 50mL of reagent water in a 100mL volumetric flask and dilute to volume with reagent water.
Storage: Store in a cool, dry, ventilated storage area with acid resistant floors and good drainage. Store away from sunlight, heat, water, and incompatible materials.
Expiration: Stable under ordinary conditions of use and storage for up to 28 days. The expiration date cannot exceed the expiration date of any of the components.

7.3.4.2 Second Source Initial Calibration Verification (ICV) Standard, 3.0ug/L – Add 0.15mL of the 1.0mg/L Second Source Intermediate Standard to a 100mL disposable vial. Add enough reagent water from a graduated cylinder to give a final volume of 50mL. The ICV is now ready to be digested. Other final volumes may be used as long as the reagent ratios are kept the same.
Storage: Store in a cool, dry, ventilated storage area with acid resistant floors and good drainage. Store away from sunlight, heat, water, and incompatible materials.
Expiration: Stable under ordinary conditions of use and storage for up to 28 days. The expiration date cannot exceed the expiration date of any of the components.

7.3.4.3 Continuing Calibration Verification (CCV) solution - Prepare CCV daily according to the matrix given in the instructions below:

Waters:

- Add 50mL of DI water to two 100-mL digestion vials.
- Add 0.25mL of the 500ug/L Hg Intermediate Standard to each vial and mix.
- Prepare in accordance with Section 10.1.1.

Soils:

- Add 0.25mL of the 500ug/L Hg Intermediate Standard to a 100-mL vial.
- Prepare in accordance with Section 10.1.2.

7.3.5 QC Standards

7.3.5.1 Analytical Spike Standard, 73.26ug/L – Using a graduated cylinder, add 20mL of the 500ug/L Hg Intermediate Standard, and 5.0mL of nitric acid to about 50mL of reagent water and dilute to 136.5mL final volume with reagent water. Note: Since this standard is not digested the volume has been adjusted to compensate for the dilution that the other standards and samples undergo during the digestions.

8.0 Sample Collection, Preservation, Shipment, and Storage

8.1 Aqueous Samples

8.1.1 Total Mercury

Aqueous samples are routinely collected in 250mL plastic containers containing 3mL of a 1:3 nitric acid preservative. The preservative should be sufficient to achieve a sample pH of less than 2. Follow the procedures in 8.1.3 for checking and recording the pH.

Although no temperature preservation is required, samples are routinely iced at the time of collection at 0-6°C (less than 6°C, but not frozen). Samples are stored at room temperature until the time of digestion. Samples must be digested and analyzed within 28 days of sample collection. Digestates are stored at room temperature until the time of analysis.

NCMs must be initiated for samples collected in improper containers and containing improper or insufficient preservatives.

8.1.2 Dissolved Mercury

Field Filtered
Aqueous samples for dissolved metals are routinely filtered at the time of sampling and collected in 250mL plastic containers containing 3mL of a 1:3 nitric acid preservative. The preservative should be sufficient to achieve a sample pH of less than 2. Check and record the pH as directed in Section 8.1.3.

Lab Filtered
If the sample is to be filtered in the laboratory, the sample must be collected in 250mL plastic container with no preservatives. The sample must be stored at 0-6°C (less than 6°C but not frozen) until filtered. Once filtered, the laboratory will add nitric acid to obtain a pH of less than 2.

Although no temperature preservation is required, samples are routinely iced at the time of collection at 0-6°C (less than 6°C but not frozen). Samples are stored at room temperature until the time of digestion. Samples must be digested and analyzed within 28 days of sample collection. Digestates are stored at room temperature until the time of analysis.

NCMs must be initiated for samples collected in improper containers and containing improper or insufficient preservatives.

8.1.3 Preservation Checks – pH Verification

Place a piece of pH paper in a disposable medicine cup.

Pour a few drops of sample into the medicine cup and note the color change of the pH paper. Record results on the preservation sheet.

If the pH<2, proceed with digestion and analysis.

If the pH is >2, adjust the sample pH to less than 2 using 1:1 nitric acid. Record the volume of acid needed to adjust the sample to <2 and initiate an NCM.

If the volume of acid needed to adjust the pH to <2 exceeds 1% of the volume of the container (2.5mL per 250mL container), stop and notify the supervisor.

If the sample is a “Rush” and the pH>2, notify the supervisor or project manager immediately.

Allow the sample to sit for 24 hours and re-check the pH. If pH<2, proceed with digestion and analysis. If pH >2, stop and contact the supervisor immediately

Note: To avoid cross-contamination, use a separate medicine cup and piece of pH paper per sample. Do not dip the pH paper into the sample container. The pH paper dye may bleed into the sample and affect sample results.

8.2 Soil Samples

Soil samples are routinely collected in 4oz plastic soil containers.

Samples must be iced at the time of collection and maintained at 0-6°C (less than 6°C but not frozen) until the time of digestion. Samples must be digested and analyzed within 28 days of collection. Digestates are stored at room temperature until the time of analysis.

9.0 Quality Control

SOP SA-QA-017: *Evaluation of Batch QC Data* and the SOP Summary in Attachment 3 provide requirements for evaluating QC data.

9.1 Batch QC

9.1.1 EPA 245.1 – Drinking Water

A digestion batch consists of up to 20 environmental samples and the associated QC items extracted together within a 24 hour period.

The laboratory's default minimum QC items performed for each digestion batch are: a method blank, a laboratory control sample (LCS), a low-level LCS (LLCS), a matrix spike (MS) to be performed on a minimum of 10% of samples or one per batch – whichever is greater, and a matrix spike duplicate (MSD).

This frequency equates to the following:

- For a batch of 10 or fewer samples, the minimum QC items are a method blank, an LCS, an LLCS, 1 matrix spike, and 1 matrix spike duplicate (MSD).
- For a batch of 11-20 samples, the minimum QC items are a method blank, an LCS, an LLCS, 1 matrix spike (from sample 1-10), another matrix spike (from sample 11-20), and a matrix spike duplicate (MSD).

Note: Unless the client has specified which sample to use as the matrix spike, the matrix spike must be selected at random from the samples in the batch. Field QC (e.g., equipment blanks, trip blanks, and field blanks) must not be used for MS/MSD unless specifically requested to do so by the client or unless there is insufficient sample available for performing MS/MSD using an actual field sample.

9.1.2 EPA 245.1 – Clean Water Act

A digestion batch consists of up to 20 environmental samples and the associated QC items extracted together within a 24 hour period.

The laboratory's default minimum QC items performed for each digestion batch are: a method blank and a laboratory control sample (LCS), a matrix spike (MS) to be performed on a minimum of 10% of samples or one per batch – whichever is greater, and a matrix spike duplicate (MSD).

This frequency equates to the following:

- For a batch of 10 or fewer samples, the minimum QC items are a method blank, an LCS, 1 matrix spike, and 1 matrix spike duplicate.
- For a batch of 11-20 samples, the minimum QC items are a method blank, an LCS, 1 matrix spike (from sample 1-10), another matrix spike (from sample 11-20), and a matrix spike duplicate.

9.1.3 EPA 7470A and EPA 7471B

A digestion batch consists of up to 20 environmental samples and the associated QC items. The laboratory's default minimum QC items performed for each digestion batch are: a method blank, a laboratory control sample (LCS), a matrix spike (MS), and a matrix spike duplicate (MSD).

The routine container supplied for this method is a 250mL (water) or 4oz (soil) container. 50mL or 1g is required for digestion. Reduced sample initial volumes or weights may be necessary to achieve the required batch QC frequency; however, the minimum digestion amount to be used is 10mL or 0.2g.

Note: Spike amounts must be adjusted to compensate for these reduced initial volumes or weights. Since final volumes are not easily reduced, elevated reporting limits will be provided when reduced initial volumes and weights are used

9.1.4 If there is insufficient sample volume to perform the required matrix spike(s), the LCS must be prepared in duplicate (i.e., LCS/LCSD). An NCM must be initiated on all affected samples to denote this situation. Insufficient sample volume or weight is defined as receiving less than a total of 50mL or 1g.

Note: If an LCS and LCSD are performed, both QC items must be evaluated and reported. Acceptable recoveries (as well as %RPD) for both LCS and LCSD are required.

9.1.5 Batch QC must meet the criteria given in Attachment 3 of this SOP.

9.2 Instrument QC

9.2.1 Initial Calibration (ICAL)

The instrument must be calibrated in accordance with SOP SA-QA-016: *Evaluation of Calibration Curves.* This SOP provides requirements for establishing the calibration curve and gives the applicable formulas.

Instrument calibration is performed by analyzing a series of known standards. The calibration curve must consist of a minimum of 5 standards and a blank. The lowest level calibration standard must be at or below the reporting limit, and the remaining standards will define the working range of the analytical system.

The initial calibration standards must be prepared daily for each sample matrix prepared on that day.

The initial calibration standard concentrations currently in use in the laboratory are as follows:

Standard Level	Water Concentration (ug/L)	Soil/Solid Concentration (mg/kg)
1	0.0	0.0
2	0.2	0.020
3	0.4	0.040
4	1.0	0.10
5	3.0	0.30
6	5.0	0.50

Refer to Section 7.3 for the standard preparation instructions. Other standard concentrations may be used provided they support the reporting limit and are fully documented in accordance with SOP SA-AN-041.

The correlation coefficient (r) of the regression curve must be greater than 0.995 for the initial calibration curve to be acceptable.

9.2.2 Second Source Initial Calibration Verification (ICV)

The calibration curve must be verified initially – prior to any sample analyses – in accordance with SOP SA-QA-016 with a standard obtained from a second source.

For EPA 245.1, the ICV must be within 5% of the true value to be acceptable.
For EPA 7470A and EPA 7471B, the ICV must be within 10% of the true value to be acceptable.

Note: The ICV is utilized to satisfy the EPA 245.1 requirement to analyze a QCS and IPC.

The initial calibration verification standard concentration currently in use in the laboratory is equivalent to level 5 of the ICAL. Refer to Section 7.3 for the standard preparation instructions. Another standard concentration may be used provided it is mid-level and fully documented in accordance with SOP SA-AN-041.

9.2.3 Initial Calibration Blank (ICB) / Continuing Calibration Blank (CCB)

The instrument must be shown to be free from contamination by the analysis of calibration blanks. Initial calibration blanks are analyzed immediately following the ICV. Continuing calibration blanks are analyzed immediately following each CCV.

The absolute value of the initial and continuing calibration blanks must be <½RL to be acceptable.

9.2.4 Continuing Calibration Verification

The initial calibration curve must be verified initially and after every 10 samples with a mid-level standard.

For EPA 245.1, all CCVs must be within 10% of the true value to be acceptable.
For EPA 7470A and EPA 7471B, all CCVs must be within 10% of the true value to be acceptable.

The continuing calibration verification standard concentration currently in use in the laboratory is equivalent to the mid-point of the calibration curve, or 2.5ug/L. Refer to Section 7.3 for the standard preparation instructions. Another standard concentration may be used provided it is mid-level and fully documented in accordance with SOP SA-AN-041.

Note: The CCV is utilized to satisfy the EPA 245.1 requirement to analyze an IPC.

9.2.5 Reporting Limit Standard

Certain project plans require the analysis of a reporting limit standard. If required, the reporting limit standard must be analyzed daily after the ICB, to verify the accuracy of the calibration curve at the reporting limit. The limits of recovery for the RL standard are 50-150%.

9.2.6 Quality Control Standard (QCS)

EPA 245.1 requires a second source QCS to be performed quarterly, at a minimum. The QCS must recover within 10% to be acceptable.

The laboratory uses the ICV to satisfy this QCS requirement.

9.2.7 Instrument Performance Check (IPC)

EPA 245.1 requires an IPC to be performed daily, after each calibration and after every tenth sample. The initial IPC must recover within 5% to be acceptable. Subsequent IPCs must recover within 10% to be acceptable.

The laboratory uses the ICV to satisfy the initial IPC requirement and the CCV to satisfy any subsequent IPC requirements.

9.3 Corrective Action for Out-of-Control Data

When the quality control parameters do not meet the criteria set forth in this SOP, corrective action must be taken in accordance with SOP SA-QA-005: *Preventive and Corrective Action Procedures* the QC Summary Table in Attachment 3. SOP SA-QA-005 provides contingencies for out-of-control data and gives guidance for exceptionally permitting departures from approved policies and procedures. Nonconformance Memos must be initiated to document all instances where QC criteria are not met and all departures from approved policies and procedures.

10.0 Procedure

10.1 Sample Preparation

Remove the samples from the refrigerator, if refrigeration is required, and allow them to come to room temperature.

Soil samples must be homogenized prior to preparation in accordance with SOP SA-QA-015: *Compositing, Homogenization, and Segregation of Samples*.

10.1.1 Water Samples

10.1.1.1 Mix the sample thoroughly. Verify the sample pH as instructed in Section 8.1.3 and adjust as needed.

Note: If the pH is greater than 2 the sample must be held for 24 hours after pH adjustment and the pH re-verified.

10.1.1.2 Add 50mL of sample to a labeled 100mL digestion vial. For dilutions and high level samples, add an aliquot of the sample to the vial and dilute to volume with reagent water.

10.1.1.3 Add 1.25mL HNO_3, 2.5mL H_2SO_4, and 7.5mL of $KMnO_4$ solution to each sample, mixing after each addition of reagent.

The purple color of $KMnO_4$ must persist for at least 15 minutes. If not, an additional 7.5mL of $KMnO_4$ solution to the sample and mix. Up to three 7.5mL aliquots of KMnO4 (total of 22.5mL additional) may be added to the sample. If additional KMnO4 is added to the sample(s), then the same amount must be added to the MB also.

If the color does not persist after the additional 22.5mL of $KMnO_4$ is added, stop the digestion and start over with a smaller aliquot of sample.

Note: If a sample requires additional $KMnO_4$ solution to be added, then an NCM must be initiated to denote this situation.

10.1.1.4 Add 4mL of potassium persulfate to each sample, cap, shake well, **loosen cap**, and place the samples in a water bath or block digestion apparatus at 95 ± 3°C for at least 2 hours.

NOTE: To verify temperature of the hotblock a digestion tube filled with DI water must be placed at random locations across each block used for digestion. This position must be changed daily. A single thermometer is transferred from block to block and a minimum of 10 minutes allowed for the thermometer to equilibrate before taking the reading. This temperature reading, uncorrected and corrected, is then recorded in the batch information.

10.1.1.5 Remove the samples and allow them to cool to room temp. Add 3mL of sodium chloride-hydroxylamine sulfate solution to each bottle to neutralize excess $KMnO_4$.

10.1.1.6 Adjust the final volume to 75mL with reagent water.

If more than 15mL of $KMnO_4$ was added to the sample (Section 10.1.1.3), a dilution factor must be applied to the sample. Calculate the dilution factor (DF) as:

DF = (75mL + KMnO4 added) / 75mL.

10.1.2 Soil Samples

10.1.2.1 Homogenize the sample thoroughly. Weigh between 0.50-0.60g wet weight of sample into a 100mL digestion vial.

If the sample is a non-soil solid or will potentially form particles that can become air-borne when the sample is broken up, homogenize the sample under the hood. Tare the digestion vessel, take it to the hood, and add the sample to the vessel. Adjust the weight to 0.50-0.60g, adding the sample to the vessel under the hood.

10.1.2.2 Add 5.0mL DI water and 10mL of 1:1 aqua regia. Heat for at least 2 minutes in a water bath or digestion block at 95°C± 3°C.

10.1.2.3 Allow the samples to cool to room temperature.

10.1.2.4 Add 20mL DI water and 15mL $KMnO_4$ solution to sample, cap, and shake well.

The purple color of $KMnO_4$ must persist for at least 15 minutes. If not, an additional 7.5mL of $KMnO_4$ solution to the sample and mix. Up to two 7.5mL aliquots of $KMnO_4$ (total of 15mL additional) may be added to the sample. If additional KMnO4 is added to the sample(s), then the same amount must be added to the MB also.

If the color does not persist after the additional 15mL of $KMnO_4$ is added, stop the digestion and start over with a smaller aliquot of sample.

Note: If a sample requires additional KMnO4 solution to be added, then an NCM must be initiated to denote this situation.

10.1.2.5 **Loosen cap**, and place the samples in a water bath or block digestion apparatus at 95 ± 3°C for at least 30 minutes.

10.1.2.6 Allow the samples to cool to room temperature.

10.1.2.7 Add 6mL of sodium chloride-hydroxylamine sulfate solution to each bottle to neutralize excess $KMnO_4$. Dilute the sample to a final volume of 75mL.

10.2 Batch QC Sample Preparation

10.2.1 Water Samples

10.2.1.1 Method Blank – Add 50mL of reagent water to a 100mL disposable vial. Prepare in accordance with Section 10.1.1.

10.2.1.2 Laboratory Control Samples (LCS/LCSD) – Add 50mL of DI water to a 100-mL digestion vial. Add 0.25mL of the 500ug/L Hg Intermediate Standard to the vial and mix. Prepare in accordance with Section 10.1.1.

10.2.1.3 Matrix Spikes (MS/MSD) – Add 50mL of the sample selected for the batch matrix spike to a 100mL digestion vial. Add 0.10mL of the 500ug/L Hg Intermediate Standard to the sample. Prepare in accordance with Section 10.1.1.

Note: For EPA 245.1 Clean Water Act, EPA 7470A and EPA 7471B the matrix spike must be prepared in duplicate.

10.2.1.4 Low Level Laboratory Control Sample (LLCS) – Add 50mL of reagent water to a 100mL digestion vial. Add 0.02mL of the 500ug/L Hg Intermediate Standard to the sample and mix. Prepare in accordance with Section 10.1.1.

Note: The LLCS is only required for EPA 245.1 drinking water samples.

10.2.2 Soil Samples

10.2.2.1 Method Blank – Add 0.50-0.60g of blank matrix to a 100mL disposable vial. Prepare in accordance with Section 10.1.2 for soils.

10.2.2.2 Laboratory Control Samples (LCS/LCSD) – Add 0.50-0.60g of blank matrix to a 100mL disposable vial. Add 0.25mL of the 500ug/L Hg Intermediate Standard. Prepare in accordance with Section 10.1.2 for soil samples.

10.2.2.3 Matrix Spikes (MS/MSD) – Add 0.50-0.60g of the soil sample selected for the batch matrix spike to a 100mL disposable vial. Add 0.1mL of the 500ug/L Hg Intermediate Standard to the sample. Prepare in accordance with Section 10.1.2.

10.3 Analysis

10.3.1 Instrument Start-up and Operating Conditions

The instrument conditions listed in this SOP are provided for guidance purposes. The actual conditions used by the laboratory may be slightly different from those listed here and must be documented in the instrument maintenance log, data system, and/or run log.

Instrument maintenance must be performed in accordance with Attachment 4 of this SOP.

Before analysis begins, inspect the system (pump tubes, mixing coil, gas/liquid separator) to see if any parts need to be cleaned or replaced.

Inspect the drying tube. Clean or replace as needed with a pre-made drying tube from Leeman Labs that has been inspected for discoloration. Caution should be used if moisture is visible in the tubing that follows the drying tube.

Fill the rinse tank with rinse water.

If the lamp is not already on and warmed up, turn on the lamp. The lamp must warm up for a minimum of 2 hours.

If the lamp is already on and warmed up, make sure the platens have the appropriate tension and turn on the pump. Allow a minimum of 20 minutes of pump time for the pump tubes to break in each day.

Autosampler setup

Fill the standard tubes with the appropriate standards for the protocol being followed. (Refer to Section 10.3.4 for more information on method-specific analytical sequence and standards required.)

Fill the labeled sample test tubes with the samples and calibration verification standards in the proper order.

The method blank will be analyzed first. The LCS will follow immediately after the method blank. The samples, matrix spikes, and duplicates will then follow with a maximum of 10 analyses between CCVs/CCBs.

Enter the sample/QC IDs into the autosampler table giving each rack a unique name.

Load the rack(s) onto the autosampler.

10.3.2 Initial and Continuing Calibration

Calibrate the instrument using the standards and criteria described given in Section 9.2.1. Once the calibration has been established and verified with an ICV in accordance with Section 9.2.2, sample analysis may proceed.

Verify the calibration curve with a continuing calibration verification using the standards and criteria described given in Section 9.2.4.

Calibration of the Mercury Analyzer
Call up the required protocol. Open a new data folder.

Go to CALIBRATION, RESET, and reset the calibration for a new calibration.

Go to CALIBRATION, STANDARDS, and ensure that calibration standards are entered at the proper concentrations.

Analyze the standards, beginning with standard 1 (Blank), proceeding from lowest to highest concentration.

When all calibration standards have been analyzed, go to CALIBRATION, LINE CALIBRATION. If calibration is within acceptable limits, accept the linear calibration and print the calibration curve.

10.3.3 Sample Analysis

The digestate must be analyzed using the same volume as that used for the calibration standards. Samples known to be relatively clean should be analyzed first. Samples suspected of containing high concentrations should be analyzed last. Instrument blanks may be analyzed after suspected high concentration samples to allow the detector response to stabilize.

The default procedure is to include QC items (method blank, LCS, MS/MSD, and SD) in determining the maximum number of samples between CCVs/CCBs.

Sample Analysis
Go to AUTOSAMPLER, SETUP. Enter the Rack IDs and the cup numbers to be analyzed.

Carryover from high concentration samples usually affects only the next one to two samples in the sequence. The two samples following an off-scale sample that is greater than 10ug/L must be reanalyzed to verify the presence or absence of mercury and the quantitation of mercury. It is the responsibility of the analyst to clearly demonstrate that all mercury results are accurate and free from carry-over contamination.

10.3.4 Example Analytical Sequence

An example analytical sequence is listed below.

Analytical Sequence for samples immediately following an initial calibration:

Description	Comments
Blank	
Initial Calibration	
ICV	Second Source
ICB	
RL Standard	Required for EPA 245.1 (Not a requirement for EPA 7470A and EPA 7471B if calibrated to RL)
Samples & Batch QC Items	Up to 9 analyses, including QC.
CCV	
CCB	
Samples & Batch QC Items	Up to 10 analyses, including QC.
CCV	
CCB	

Analytical Sequence for samples not immediately following an initial calibration:

Description	Comments
CCV	
CCB	
Samples & Batch QC Items	Up to 10 analyses, including QC.
CCV	
CCB	
Samples & Batch QC Items	Up to 10 analyses, including QC.
CCV	
CCB	

11.0 Calculations / Data Reduction

11.1 Data Reduction

Data must be evaluated in accordance with SOP SA-QA-002: *Data Generation and Review*. See Section 9.2.9 for guidance on the evaluation of MS/MSD.

11.1.1 Dilutions

If the concentration of a sample is above the calibration range of the mercury analyzer, the sample digestate must be diluted and reanalyzed. The amount of digestate needed to prepare the desired dilution is determined from the following equation:

$$V_{digest} = \frac{V_{fv}}{DF}$$

Where:

V_{digest} = volume of sample digestate used to make the dilution
V_{fv} = final volume of diluted sample
DF = dilution factor

Note: Samples should be diluted with digested blank solution.

Note: This calculation assumes all applicable unit correction factors are applied.

The dilution factor is calculated as follows:

$$DF = \frac{V_{fv}}{V_{digest}}$$

Where:

V_{digest} = volume of sample digestate used to make the dilution
V_{fv} = final volume of diluted sample
DF = dilution factor

Note: This calculation assumes all applicable unit correction factors are applied.

If a sample exceeds the calibration range of the instrument by more than a factor of 10 (i.e., if the final, calculated result is greater than 50ug/L Hg at the instrument) the samples should be re-digested and reanalyzed with a smaller amount. This is a good check for possible positive bias of the sample by incomplete digestion of organic compounds. Initial weights or volumes of <0.2g or <1.0mL should be avoided, if possible, so that a representative sample can be achieved. If, due to the level of mercury in the samples, greater dilutions are required, consult with the Department Manager for further instructions.

11.1.2 Historical Data

Many of the laboratory's clients submit samples for repeat monitoring purposes. Prior to analysis, verify the TALS Worksheet Notes and/or use the TALS Historical Data Tracker feature to determine if historical data is available for review.

11.1.3 Chemical Relationships

When available, the following chemical relationships must be evaluated for each sample. If these relationships are not met, the Department Supervisor must be contacted immediately.

- Total Results are ≥ Dissolved results (e.g. metals)

11.1.4 Drinking Water Compliance Evaluation

Public water suppliers (PWS) are governed by EPA-specified Maximum Contaminant Levels (MCL) above which indicates noncompliance. The MCL associated with this procedure is 0.002mg/L.

11.2 Calculations

11.2.1 The calculations associated with batch QC determinations are given in SOP SA-QA-017. Applicable calculations include accuracy (% recovery) and precision (%RPD). Evaluate MS/MSD results using the guidance in Section 9.2.9.

11.2.2 The calculations associated with initial and continuing calibrations and are given in SOP SA-QA-016. Applicable calculations include determination for: calibration factor, standard deviation, relative standard deviation, relative response factor, and relative standard deviation.

11.2.3 The calculation to determine final concentration is given as follows:

Regression Curve:

$$FinalConcentration = CONC_{Sample} \otimes \frac{F}{I \times dw} \otimes D$$

Where:

$CONC_{Sample}$= Concentration of the sample
F = Final volume/weight
I = Initial volume/weight
dw = % Solids decimal equivalent
D = Dilution factor

Note: All dry weight corrections are performed automatically in TALS.

Note: This calculation assumes all applicable unit correction factors are applied.

12.0 Method Performance

12.1 Reporting Limit Verification (RLV)

At a minimum, RLVs must be performed initially upon method set-up in accordance with SOP SA-QA-007: *Determination and Verification of Detection and Reporting Limits*.

For analytes and methods certified by DOD ELAP, RLVs must also be performed quarterly thereafter. For all other analytes and methods, RLVs must also be performed annually thereafter. Exceptions may be made for project-specific non-routine analytes.

12.2 Method Detection Limit (MDL) Study

The MDL is the lowest concentration that can be detected for a given analytical method and sample matrix with 99% confidence that the analyte is present. MDLs reflect a calculated (statistical) value determined under ideal laboratory conditions in a clean matrix and may not be achievable in all environmental matrices. The current MDLs associated with this procedure are given in the Method Limit Group (MLG) in TALS.

At a minimum, MDL Studies must be performed initially upon method set-up in accordance with SOP SA-QA-007: *Determination and Verification of Detection and Reporting Limits*.

Note: MDL Studies are not required for non-routine analytes provided results are not reported below the RL (i.e., MDL equals RL in TALS).

12.3 Method Detection Limit Verification (MDLV)

At a minimum, MDLVs must be performed initially upon method set-up in accordance with SOP SA-QA-007: *Determination and Verification of Detection and Reporting Limits*.

For analytes and methods certified by DOD ELAP, MDLVs must also be performed quarterly thereafter. For all other analytes and methods, MDLVs must also be performed annually thereafter.

Note: MDLVs are not required for non-routine analytes provided results are not reported below the RL (i.e., MDL equals RL in TALS).

12.4 Determination of the Instrument Detection Limit (IDL)

The instrument detection limit (IDL) is the concentration of analyte that can be statistically distinguished from the background noise of the instrument. The IDL limit must be determined annually, at a minimum, for each analyte in accordance with SOP SA-QA-007: *Determination and Verification of Detection and Reporting Limits (RLs, MDLs, and IDLs)*.

The IDL is defined as three times the average of the standard deviation of seven replicate analyses of the IDL solution performed over three non-consecutive days. The IDL may be elevated above the background noise (blank levels). The current IDL associated with this procedure is given in the Equipment Limit Group (ELG) in TALS.

12.5 QC Limit Generation, Control Charting, and Trend Analysis

The control limits for the batch QC items (LCS, MS/MSD, Analytical Spike) for this procedure are specified in the reference method and cannot be broadened; therefore, the laboratory defaults to the method-defined limits and does not utilize in-house or laboratory-derived limits for the evaluation of batch QC items.

Although the laboratory must default to the method-defined QC limits, control charting is a useful tool and is performed to assess analyte recoveries over time to evaluate trends. Control charting must be performed periodically (at a minimum annually) in accordance with SOP SA-QA-017: *Evaluation of Batch QC Data.*

12.6 Demonstrations of Capability

Initial and continuing demonstration of capability must be performed in accordance with SOP SA-QA-006*: Training Procedures.*

Prior to performing this procedure unsupervised, each new analyst who performs this analysis must demonstrate proficiency per method/analyte combination by successful completion of an initial demonstration of capability. The IDOC is performed by the analysis of 4 consecutive LCSs that meet the method criteria for accuracy and precision. The IDOC must be documented and routed to the QA Department for filing.

Annual continuing demonstrations of capability (CDOCs) are also required per analyst per method/analyte combination. The CDOC requirement may be met by the consecutive analysis of four LCS all in the same batch, by the analysis of four LCS analyzed in four consecutive batches (in different batches on different days), via acceptable results on a PT study, or analysis of client samples with statistically indistinguishable results when compared to another certified analyst. The CDOC must be documented and routed to the QA Department for filing.

12.7 Training Requirements

All training must be performed and documented in accordance with SOP SA-QA-006: *Training Procedures*.

Note: The SOPs listed in the Reference/Cross-Reference Section are applicable to this procedure. All employees performing this procedure must also be trained on these SOPs.

13.0 Pollution Control

It is Eurofins TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (e.g., examining recycling options, ordering chemicals based on quantity needed, preparing reagents based on anticipated usage and reagent stability, etc.). Employees must abide by the policies in Section 13 of the Environmental Health and Safety Manual and the Savannah Addendum to the EHSM.

This procedure has been evaluated for opportunities to minimize the waste generated. Where reasonably feasible, pollution control procedures have been incorporated.

14.0 Waste Management

Waste management practices must be conducted consistent with all applicable federal, state, and local rules and regulations. All waste (i.e., excess reagents, samples, and method process wastes) must be disposed of in accordance with Section 9 of the Eurofins TestAmerica Savannah Addendum to the EHSM. Waste description rules and land disposal restrictions must be followed.

14.1 Waste Streams Produced by the Method

The following waste streams are produced when this method is carried out:

- Excess aqueous samples – Dispose according to characterization on the sample disposal sheets. Neutralize non-hazardous samples before disposal into drain/sewer. Transfer hazardous samples (identified on disposal sheets) to the waste department for disposal.
- Excess soil and solid samples –Transfer RCRA and non-hazardous samples to the yard box for disposal vis incineration. If samples are classified as PCB or Foreign-soils the samples are transferred to the waste coordinator for drumming.
- Acidic sample digestions – Neutralize before disposal into drain/sewer system.
- Excess oil samples – Transfer to waste department for storage/disposal.

14.2 Waste Disposal

The table below summarizes the disposal process:

Material	Treatment	Destination
Aqueous Samples (Non-Haz)	Neutralization	Sink
Aqueous Samples (Haz)	Neutralization	RCRA Water Drum
Solid Samples (Non-Haz)	None	Yard Box
Solid Samples (Haz)	None	Yard Box
Standards	Neutralization	RCRA Water Drum
Nitric Acid	Neutralization	Sink
Sulfuric Acid	Neutralization	Sink
Hydrochloric Acid	Neutralization	Sink
Hydroxyl amine	None	Sink
Sodium chloride	None	Sink
Hydroxyl amine-sodium	React with KMnO4 until	RCRA Water Drum

chloride solution	KMnO4 is colorless	
Stannous chloride	None	RCRA Water Drum
Potassium permanganate	None	Lab pack
5% Potassium permanganate	React with hydroxylamine-sodium chloride until KMnO4 is colorless	RCRA Water
Potassium persulfate	None	Sink

15.0 References / Cross-References

- SOP SA-AN-100: *Laboratory Support Equipment (Verification and Use)*
- SOP SA-AN-041: *Reagent and Standard Materials Procedures*
- SOP SA-QA-002: *Data Generation and Review*
- SOP SA-QA-005: *Preventive and Corrective Action Procedures*
- SOP SA-QA-006*: Training Procedures*
- SOP SA-QA-007: *Determination and Verification of Detection and Reporting Limits (RLs, MDLs, and IDLs)*
- SOP SA-QA-015: *Homogenization, Compositing, and Segregation of Samples*
- SOP SA-QA-016: *Evaluation of Calibration Curves*
- SOP SA-QA-017: *Evaluation of Batch QC Data*
- Eurofins TestAmerica Savannah Quality Assurance Manual
- Eurofins TestAmerica Environmental Health and Safety Manual
- Eurofins TestAmerica Savannah Addendum to the Environmental Health and Safety Manual
- Method OB 10/90: Extraction and Analysis of Organics in Biological Tissue; U.S Environmental Protection Agency Environmental Services Division, Region IV Analytical Support Branch Athens, GA
- *Test Methods for Evaluating Solid Waste, Third Edition*; U.S. EPA Office of Solid Waste and Emergency Response: Washington, D.C., February 2007 (SW-846 Update IV).
    - Method 7000B, Revision 2: Flame Atomic Absorption Spectrophotometry; February 2007.
    - Method 7470A, Revision 1: Mercury in Liquid Waste (Manual Cold-Vapor Technique); September 1994.
    - Method 7471B, Revision 2: Mercury in Solid or Semisolid Waste (Manual Cold-Vapor Technique); February 2007.
- *Methods for Analysis of Water and Waste*; U.S. EPA Office of Research and Development: Cincinnati, OH, March 1983.
    - Method 245.1, Revision 3.0, EMMC Version: Determination of Mercury in Water by Cold Vapor Atomic Adsorption Spectrometry; 1994.
- *Methods for the Determination of Metals in Environmental Samples*; US EPA Office of Research and Development. Washington, DC.
    - Method 245.6, Revision 2.3, Determination of Mercury in Tissues by Cold Vapor Atomic Absorption Spectrometry, 1991

16.0 Method Modifications

16.1 Incorporation of Non-Routine Matrices

This procedure may be modified to analyze other matrices (e.g., waste (oil), wipe, tissue, and TCLP/SPLP leachate samples) upon client request. This will need to be arranged by the Project Manager at the initiation of the project.

Waste (oil), wipe, and tissue matrices are non-routine, and the laboratory is not currently NELAC certified for these matrices. The laboratory uses its routine soil RLs (converted for initial and final volumes, etc.) and soil QC limits to evaluate wipe, waste, and tissue samples. Soil DOCs can be used to satisfy analyst demonstrations of capability for these types of non-routine matrices. Teflon chips or blank sand are used as the blank matrix for tissues unless a "true" tissue matrix is required by the project. The laboratory uses its routine soil RLs (converted for initial and final volumes, etc.) and soil QC limits to evaluate TCLP/SPLP leachate samples. Water DOCs can be used to satisfy analyst demonstrations of capability for TCLP/SPLP matrices.

16.1.1 Collection and Handling Procedures for Non-Routine Matrices

Waste (oil) samples are collected in 4oz plastic soil containers. Waste (oil) samples must be iced at the time of collection and maintained at 4°C (less than 6°C but not frozen) until the time of digestion. Waste (oil) samples must be digested and analyzed within 28 days of collection. Waste (oil) digestates are stored at room temperature until the time of analysis.

Wipe samples are routinely collected in 40mL VOA vials containing acetic acid in water, pH 4.93 +/- 0.05. Wipe samples must be iced at the time of collection and maintained at 4°C (less than 6°C but not frozen) until the time of digestion. Wipe samples must be digested and analyzed within 28 days of collection. Wipe digestates are stored at room temperature until the time of analysis. Refer to the Work Instruction on *Wipe Tests: Sampling and Analysis* for additional information on wipe procedures.

Tissue samples are routinely collected in plastic containers with the size dependent upon the type of tissue being collected. Plastic jars or plastic baggies can be used. Upon receipt, tissue samples must be placed in the freezer at -10° to -20°C if extraction/digestion cannot be completed that day. Per the EPA Region 4 guidance document used for tissue analyses, tissue samples can be stored frozen for up to 6 months, and must be digested and analyzed within 28 days of thawing.

For TCLP and SPLP samples, once the TCLP/SPLP extraction procedure has been performed, the leachate is transferred to a plastic container and refrigerated at 4°C (less than 6°C with no frozen samples). TCLP/SPLP leachates must be stored at 4°C (less than 6°C with no frozen samples) until the time of preparation and/or analysis. The leachate sample must be digested and analyzed within 28 days of completion of the TCLP/SPLP extraction.

16.1.2 Preparation and Analytical Procedures for Non-Routine Matrices

16.1.2.1 Waste (oil) samples are prepared in the same manner as routine soil samples, as outlined in Section 10.1 of this SOP. Waste (oil) QC samples are prepared in the same manner as routine soil samples, as outlined in Section 10.2 of this SOP.

16.1.2.2 Tissue samples are calculated on an "as is" basis and are prepared as outlined below. Tissue QC samples are prepared in the same manner as routine soil QC as outlined in Section 10.2 of this SOP.

Weigh between 1.0g and 1.2g of the sample and place into a 100mL disposable vial. Add 2mL H_2SO_4 and 0.5mL HNO_3 to each sample and digest in the waterbath or heating block for at least 30 minutes at 80°C +/- 3ºC or until the tissue is completely dissolved.

After samples are cooled, add 7.5mL of $KMnO_4$ (more $KMnO_4$ may be added if required), 4mL potassium persulfate solution, 25mL DI water and put samples back into water bath for an additional at least 90 minutes at 30°C +/- 3ºC. Be sure the purple color of $KMnO_4$ persists for at least 15 minutes prior to placing samples in the water bath or block digestion apparatus. If not, add 7.5mL of $KMnO_4$ solution up to three additional times.

Remove the samples and allow them to cool. Add 3mL of sodium chloride-hydroxylamine sulfate solution to each bottle to neutralize excess $KMnO_4$. Add 26mL DI water and shake well. This should give a final volume of 68mL.

Note: If additional volume(s) of $KMnO_4$ were added, compensate for the addition(s) by adding less DI water so that the final volume will remain constant.

16.1.2.3 Wipe samples are prepared as outlined in the Work Instruction, *Wipe Tests: Sampling and Analysis* (FQA088).

16.1.2.4 Waste (oil), tissue, and wipe samples are analyzed in the same manner as routine matrices as outlined in Section 10.3.

16.2 Other Considerations

16.2.1 The EPA Manual for the Certification of Laboratories Analyzing Drinking Water requires a LFB at the MRL to be performed each day. The laboratory meets this requirement by preparing an LCS at the RL in each EPA 245.1 batch of drinking water samples. The EPA DW Manual does not specify criteria for the low-level LCS (LLCS). The laboratory requires detection of this LLCS to be acceptable.

16.2.2 Calibration standards are assigned a hold time of 28 days and may be used to quantify samples prepped on the same day as the ICAL in cases where re-analysis is required.

16.2.3 The referenced liquid preparation methods call for an initial volume of 100mL of sample. The reference methods utilize BOD bottles that required 100mL of sample to reach the required detection limit. The newer instrumentation utilized by the laboratory requires less than 10mL of the final digestion for analysis. Therefore, this SOP utilizes a 50mL initial volume and the reagents added for digestion are lowered proportionately.

16.2.4 EPA 245.1 specifies a 100mL initial volume but allows for reduced volumes as long as the reagent ratios and quality control are met. The laboratory uses 50mL as its default initial volume, and reagent ratios have been adjusted accordingly. All method detection limits, demonstrations of capability, and PTs have been performed in this same manner.

16.2.5 EPA 245.1 specifies the use of stannous chloride/sulfuric acid suspension used to reduce mercury to elemental mercury. EPA 7470A and EPA 7471B specify stannous sulfate to be used to reduce elemental mercury to mercury; however, these methods also state stannous chloride dissolved in hydrochloric acid can be used as an option. In accordance with the instrument manufacturer's recommendations, the laboratory uses stannous chloride dissolved in hydrochloric acid as the stannous chloride/sulfuric acid suspension tend to clog the instrument's lines. All method detection limits, demonstrations of capability, and PTs have been performed in this same manner.

16.2.6 EPA 245.1 specifies that a Method Detection Limit (MDL) study to be performed annually and when a new analyst begins. The laboratory performs an MDL study initially, with MDL Verifications performed quarterly, and when a significant change is made to the equipment. The laboratory does not use analyst-specific MDLs; therefore, new analysts are required only to perform an initial demonstration of capability (IDOC) as described in Section 12.4.

16.2.7 EPA 245.1 specifies control criteria for the method blank as >10% the analyte concentration in the sample or 2.2 times the MDL, whichever is greater. The laboratory requires the method blank to be <1/2RL. The RL for this procedure is routinely within a factor of within ~3-5x the MDL; therefore, the 1/2RL criteria used for the method blank satisfies the method requirement.

16.2.8 EPA 245.1 requires a Linear Dynamic Range (LDR) study to be performed initially, with an LDR verification performed annually thereafter. An initial LDR study is performed in accordance with method requirements, and a re-evaluation of the calibration curve as samples is performed to satisfy the annual verification requirement. The laboratory dilutes all samples with concentrations above the high point of the calibration curve to bring them within the linear calibration range of the instrument.

16.2.9 EPA 7470A states non-aqueous samples must be analyzed as soon as possible. EPA 7471B states non-aqueous samples must be analyzed as soon as possible but should be held no longer than 28 days. Therefore, the laboratory defaults to a 28-day holding time for these methods.

16.2.10 EPA 245.1 does not contain a method-defined batch precision requirement. The laboratory's default QC items incorporate an MSD to satisfy the Clean Water Act requirements and those clients who batch require precision to be reported. Additionally, if insufficient sample volume is provided to perform the MS/MSD, the LCS is routinely prepared in duplicate (i.e., LCS/LCSD).

16.2.10 EPA 7471B requires the use of aqua regia prepared at a specific molarity. Due to safety concerns involving the use of such concentrated acid, the laboratory uses a more dilute solution than what is prescribed in the method.

16.2.11 EPA 245.1 includes the parenthetical phrase "without heating" in reference to preparation of calibration standards. The laboratory does not interpret this as an outright prohibition against the practice and believes it is acceptable to digest calibration standards, as is done for many other mercury methods. All method detection limits, demonstrations of capability, and PTs have been performed in this same manner.

17.0 Attachments

The following Tables, Diagrams, and/or Validation Data are included as Attachments:

Attachment 1: SOP Summary
Attachment 2: Sample Collection, Preservation, and Holding Time Table
Attachment 3: QC Summary
Attachment 4: Instrument Maintenance and Troubleshooting
Attachment 5: Labware Cleaning Posting

Attachment 1:
SOP Summary

Sample Preparation and Analysis Summary

This procedure is based on the absorption of characteristic radiation at 253.7nm by mercury vapor. After digestion, to convert all forms of mercury to the same oxidation state, the mercury ions are reduced to mercury by the addition of stannous chloride and aerated from solution after passing through a mixing coil. The mixture passes through a gas/liquid separator and through a drying tube. The vapor is passed through a flow cell positioned in the light path of an atomic absorption spectrophotometer. Mercury concentration is measured as a function of absorbance.

Analytical Sequence

Analytical Sequence for samples immediately following an initial calibration:

Description	Comments
Blank	
Initial Calibration	
ICV	Second Source
ICB	
RL Standard	Required for EPA 245.1 and SM4110B (Not a requirement for EPA 7470A and EPA 7471B if calibrated to RL)
Samples & Batch QC Items	Up to 9 analyses, including QC.
CCV	
CCB	
Samples & Batch QC Items	Up to 10 analyses, including QC.
CCV	
CCB	

Analytical Sequence for samples not immediately following an initial calibration:

Description	Comments
CCV	
CCB	
Samples & Batch QC Items	Up to 10 analyses, including QC.
CCV	
CCB	
Samples & Batch QC Items	Up to 10 analyses, including QC.
CCV	
CCB	

Attachment 2:
Sample Collection, Preservation, and Holding Time Table

Matrix	Routine Sample Container	Routine Sample Size	Minimum Sample Size	Dechlorination Agent	Chemical Preservation	Thermal Preservation	Holding Time[1]
Water	250mL plastic	50mL	10mL	Not Applicable	Nitric Acid pH<2	None[2]	28 days from collection
Soil	4oz plastic soil jar	10g	0.2g	Not Applicable	None	None[2]	28 days from collection

[1] Inclusive of digestion and analysis.
[2] Thermal preservation is not required; however, samples are routinely maintained at less than 6°C with no frozen samples.

Attachment 3:
QC Summary

QC Item	Frequency	Criteria	Corrective Action
Initial Calibration	Daily	1 blank and 5 standards Correlation coefficient ≥ 0.995	Recalibrate
Initial Calibration Verification Standard (ICV)	At the beginning of the analysis	EPA 7470A = within ± 10% EPA 7471B = within ± 10% EPA 245.1 = within ± 5%	Recalibrate
Instrument Performance Check (IPC) (Note: ICV and CCV are used to satisfy IPC.)	EPA 245.1 only: After each ICAL and every 10 samples	Initial IPC = within 5% Subsequent IPC = within 10%	Recalibrate
2nd Source Quality Control (QCS) (Note: ICV is used to satisfy QCS.)	EPA 245.1 only: Quarterly	within 10%	Recalibrate
Continuing Calibration Verification (CCV)	At the beginning and end of the analysis and every 10 samples.	EPA 7470A = within ± 10% EPA 7471B = within ± 10% EPA 245.1 = within ±10%	Terminate the analysis. Correct the problem and reanalyze all samples since the last compliant CCV.
Calibration Blank (ICB/CCB)	After ICV and every CCV	Absolute value of the calibration blank must be < ½ RL	Terminate the analysis. Correct the problem and reanalyze all samples since the last compliant CCB.
RL Standard	After every calibration but not before the ICV. This standard is not a requirement of SW846, if a multi-point calibration encompassing the RL level is used.	50-150% of true value	Recalibrate

QC Item	Frequency	Criteria	Corrective Action
Method Blank (MB)	One per batch of twenty or fewer samples	Result < ½ RL	Refer to SOP SA-QA-017
Low-Level Laboratory Control Sample (LLCS)	EPA 245.1 Drinking Water Only: One per batch of twenty or fewer samples	Qualitatively identified	If the "regular" LCS meets criteria, initiate NCM and report data If the "regular" LCS does not meet criteria, redigest and reanalyze batch
Laboratory Control Sample (LCS)	One per batch of twenty or fewer samples	MLG Limits	Refer to SOP SA-QA-017
Matrix Spike (MS)	EPA 245.1: One MS per 10% of samples (i.e., 2 MS per batch of 20 samples) Other Methods: One MS per 5% of samples (i.e., 1 MS per batch of 20 samples)	MLG Limits	Refer to SOP SA-QA-017
Matrix Spike Duplicate (MSD)	1 per batch	MLG Limits	Refer to SOP SA-QA-017
Post Digestion Spikes (PDS)	When MS/MSD is unacceptable	Refer to Section 9.2.9 for guidance	Refer to Section 9.2.9 for guidance
Initial Demonstration of Capability (IDOC)	Initially, per analyst, per analyte/method/matrix combination	Refer to SOP SA-QA-006	Refer to SOP SA-QA-006 Note: Unsupervised work must not begin until acceptable IDOC is obtained.
Continuing Demonstration of Capability	Annually, per analyst, per analyte/method combination	Refer to SOP SA-QA-006	Refer to SOP SA-QA-006

QC Item	Frequency	Criteria	Corrective Action
(CDOC)			
Reporting Limit Verification (RLV)	Upon method/instrument set-up, per analyte/method/matrix combination. Then quarterly thereafter (for DOD ELAP) or annually thereafter (for non-DOD ELAP)	Refer to SOP SA-QA-007	Refer to SOP SA-QA-007
Method Detection Limit Study (MDL)	Upon method/instrument set-up, per analyte/method/matrix combination	Refer to SOP SA-QA-007	Refer to SOP SA-QA-007
MDL Verification (MDLV)	Upon method/instrument set-up, per analyte/method/matrix combination. Then quarterly thereafter (for DOD ELAP) or annually thereafter (for non-DOD ELAP)	Refer to SOP SA-QA-007	Refer to SOP SA-QA-007
Instrument Detection Limit (IDL)	Upon method/instrument set-up, and then quarterly thereafter	Refer to SOP SA-QA-007	Refer to SOP SA-QA-007

Attachment 4:
Instrument Maintenance and Troubleshooting

Instrument Labeling

Each instrument must be labeled with its name or ID (e.g., MSA, ICP-D, etc.). Additionally, non-operational instruments must be isolated from service or marked as being out of service. Each piece of equipment has an "Operational / Not Operational" sticker that is used for this purpose.

Maintenance Log

A maintenance log must be established for each piece of equipment used in the laboratory. All maintenance that is performed on the instrument must be recorded in the log including:

- Type of maintenance performed (Note: This includes preventative/routine maintenance; non-routine maintenance; maintenance performed by an external vendor; updates to software versions; etc.)
- Analyst or technician performing the maintenance
- Date the maintenance was performed
- Detailed explanation of the reason for the maintenance
- Resolution of the problem and return to control
- All service calls from instrument representatives
- Resolution of the problem (i.e., the Return to Control)

Preventive Maintenance

Refer to the instrument manufacturer's guides for trouble-shooting items.

LABORATORY EQUIPMENT PREVENTIVE MAINTENANCE SCHEDULE								
EQUIPMENT ITEM	**Service Interval**							**SERVICE LEVEL**
	D	**W**	**M**	**Q**	**SA**	**A**	**AN**	
Pump Tubing	**X**							Inspect daily. Replace as needed.
Optical cells							**X**	Clean as needed.
Drying Tube							**X**	Replace as needed.
Sample Probe			**X**					Inspect monthly. Clean or replace as needed.
Mercury Lamp							**X**	Replace as needed.

D = Daily; W = Weekly; M = Monthly; Q = Quarterly; SA = Semi-Annually; A = Annually; AN = as needed

Troubleshooting

Troubleshooting should be documented as outlined above. If possible, troubleshooting is best performed in a step-wise manner to systematically isolate instrument components. Refer to the instrument manufacturer's guides for specific information and strategies. Enlist assistance from technical and/or department management as needed.

Contingency Plan

Maintenance contracts are carried for most instrumentation and close contact is maintained with service personnel to ensure optimal instrument functioning. An extensive spare parts inventory is maintained for routine repairs, consisting of Hg lamps, drying tubes, flow cells, tubing, and other common instrumentation components. Since instrumentation is standardized throughout the laboratory network, spare parts and components can be readily exchanged among the network.

In general, the laboratory has at least one backup unit for each critical unit. In the event of instrument failure, portions of the sample load may be diverted to duplicate instrumentation, the analytical technique switched to an alternate approved technique (such as manual colorimetric determination as opposed to automated colorimetric determination), or samples shipped to another properly certified or approved Eurofins TestAmerica location.

Attachment 5:
Labware Cleaning Posting

GLASSWARE CLEANING PROCEDURES METALS DEPARTMENT
Graduated Cylinders 1. Scrub with hot, soapy H_2O and brush. 2. Rinse thoroughly with tap H2O. 3. Rinse with 10% HNO_3 4. Rinse thoroughly with DI H_2O.
Volumetric Flasks 1. Empty contents of flask 2. Squirt a small amount of cleaning detergent directly into volumetric flask 3. Fill flask 1/3 full with **HOT** H_2O 4. Replace top and shake flask. 5. Empty flask and rinse with **HOT** H_2O until no soap remains in flask. 6. Add approximately 10mL concentrated HNO_3 to 50mL, 100mL, and 250mL flasks ---- replace top and shake well. For 500mL or 1000mL flasks use 25mL and for 10mL flasks use 2 - 5mL of concentrated HNO_3. 7. Rinse 3 times with DI H_2O, filling flask 1/3 full and replacing top. Store until needed. *** NEVER PLACE VOLUMETRIC FLASKS OR TOPS IN SINK OR DISHPAN WITH OTHER DIRTY DISHES.**
***Dispose of all acid waste in accordance with the Eurofins TestAmerica Savannah Addendum to the Corporate Safety Manual.**

18.0 Revision History

Summary of Changes for Revision 15A

- Updated all references to an 8oz sample container to 4oz as this is the volume referenced in LIMS.
- Updated waste disposal procedures for soil samples.

Summary of Changes from Previous Revision 15

- Minor editorial and/or grammatical changes.
- Updated SOP signatories to reflect current responsibilities and titles.
- Updated company logo and name where applicable.
- Updated CCV criteria for method 7471B and 7470A to 10%D.
- Removed Attachment 5 for prep of mercury standards.

LIQUID PREPARATION PROCEDURES FOR ICP AND ICP/MS

(Methods: EPA 200.7, 200.8, 3005A, 3010A, SM3030C, and Filtration)

Approvals (Signature/Date):

08/31/2021

Adriana Geiger — Date
Department Manager

08/31/2021

Kim Chamberlain — Date
Quality Assurance Manager / Environmental Health & Safety Coordinator

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1.0 Scope and Application

This SOP gives the procedures for the preparation of metals in water and leachate samples prior to analysis by ICP (SOP SA-ME-070: *Elements by ICP*) or ICP/MS (SOP SA-ME-074: *Elements by ICP/MS*).

A complete target analyte list, the reporting limits (RL), the method detection limits (MDL), and the accuracy and precision criteria associated with this procedure are provided in the LIMS Method Limit Groups (MLGs).

This SOP was written by and for Eurofins TestAmerica's Savannah laboratory.

2.0 Summary of Method

2.1 Total Recoverable Metals: A known volume of sample, usually 50mL, is transferred to a digestion vessel. The sample is refluxed with dilute nitric acid and hydrochloric acid at approximately 95°C. After the sample has evaporated to approximately 10-20mL, the sample is brought up to the original volume with reagent water. The laboratory utilizes two versions of the Total Recoverable preparation procedure. One version is equivalent to the EPA 3005A procedure and the second procedure is equivalent to the EPA 200.7 and 200.8 procedures.

2.2 Total Metals and TCLP/SPLP Leachates: A known volume of sample, usually 50mL, is transferred to a digestion vessel. The sample is refluxed with nitric acid at approximately 95°C. After the sample has digested, as evidenced by a clear, pale yellow color, HCl is added and the sample is brought up to the original volume with reagent water. This procedure is equivalent to EPA 3010A, and is utilized as the total metals preparation procedure for the EPA 200.7 and EPA 200.8.

2.3 Acid Extractable Metals: A known volume of sample, usually 50mL, preserved with nitric acid, is transferred to a digestion vessel. Hydrochloric acid is added and the sample is heated on a hot block for 15 minutes. This procedure is equivalent to Standard Methods 3030C.

2.4 Silica Samples for EPA 200.7 and EPA 6010C: Liquid samples are filtered. The filtered liquid samples are analyzed by ICP.

2.5 Drinking water samples for EPA 200.7 and EPA 200.8 with a turbidity concentration of less than 1NTU may be analyzed with no digestion. The exception to this rule is silver, which requires sample digestion prior to analysis. If the sample turbidity is >1NTU, then the EPA 200.7 or the EPA Method 200.8 preparation procedure is used.

2.6 Samples filtered for the determination of dissolved metals do not require digestion if the following criteria are met:
1) The sample has a low COD (<20mg/L);
2) The sample has a turbidity <1NTU ;
3) The sample is colorless with no significant odor ; ***and***
4) The sample is of one liquid phase and is free of suspended particulates or precipitates after acidification.

Note: Generally the lab will digest samples for dissolved metals, even if the above criteria are met, as the presence of organics in the native sample may lead to false positives for arsenic and selenium.

2.7 This SOP is based on the following methods: EPA 200.7, EPA 200.8, EPA 3005A, EPA 3010A, and Standard Methods 3030C.

3.0 Definitions

Refer to the Glossary Section of the *Quality Assurance Manual* (QAM) for a complete listing of applicable definitions and acronyms.

4.0 Interferences

4.1 Procedural Interferences

4.1.1 Interferences may be caused by contaminants in solvents, reagents, glassware, and other sample processing apparatus and can make identification and/or quantification of the target analytes difficult.

4.1.2 All sample collection containers are single-use disposable containers which limits the potential for contamination. All non-disposable labware must be scrupulously cleaned in accordance with the posted Labware Cleaning Instructions to ensure it is free from contaminants and does not contribute artifacts.

4.1.3 High purity reagents and solvents are used to help minimize interference problems. Hydrochloric acid and nitric acid must be verified prior to use in accordance with the Eurofins TestAmerica Solvent Lot Testing Program.

4.1.4 Instrument and/or method blanks are routinely used to demonstrate all reagents and apparatus are free from interferences under the conditions of the analysis.

4.2 Matrix Interferences

4.2.1 Matrix interferences may be caused by contaminants that are co-extracted from the sample matrix. The sample may require cleanup or dilution prior to analysis to reduce or eliminate the interferences.

4.2.2 Interfering contamination may occur when a sample containing low concentrations of analytes is analyzed immediately following a sample containing relatively high concentrations of analytes. As such, samples known to be clean should be analyzed first. To prevent carryover into subsequent samples, analysis of reagent blanks may be needed after the analysis of a sample containing high concentrations of analytes.

4.2.3 Turbidity Matrix Interferences

4.2.3.1 The presence of floating debris and coarse sediments which settle out rapidly will give a low reading. Air bubbles will affect the results positively.

4.2.3.2 The presence of true color, that is the color of water which is due to dissolved substances that absorb light, will cause turbidities to be low, although this effect is generally not significant with drinking waters.

4.2.3.3 Light absorbing materials such as activated carbon in significant concentrations can cause low readings.

5.0 Safety

Employees must abide by the policies and procedures in the Eurofins TestAmerica Environmental Health and Safety Manual (EHSM), the Eurofins TestAmerica Savannah Addendum to the EHSM, and this document.

This procedure may involve hazardous materials, operations, and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user to follow appropriate safety, waste disposal, and health practices under the assumption that all samples and reagents are potentially hazardous.

The analyst must protect himself/herself from exposure to the sample matrix. Many of the samples that are tested may contain hazardous chemical compounds or biological organisms. The analyst must, at a minimum, wear protective clothing (lab coat), eye protection (safety glasses or face shield), disposable latex or nitrile gloves, and closed-toe, nonabsorbent shoes when handling samples.

5.1 Specific Safety Concerns or Requirements

Nitric acid and hydrochloric acid are extremely hazardous as oxidizers, corrosives, poisons, and are reactive. Inhalation of the vapors can cause coughing, choking, irritation of the nose, throat, and respiratory tract, breathing difficulties, and lead to pneumonia and pulmonary edema. Contact with the skin can cause severe burns, redness, and pain. Nitric acid can cause deep ulcers, and staining of the skin to a yellow or yellow-brown color. These acid vapors are irritating and can cause damage to the eyes. Contact with the eyes can cause permanent damage.

Samples that contain high concentrations of carbonates or organic matter, or samples that are at elevated pH can react violently when acids are added. Acids must be added to samples under a hood to avoid splash/splatter hazards and/or possibly toxic vapors that will be given off when the samples are acidified.

5.2 Primary Materials Used

The following is a list of the materials used in this procedure, which have a serious or significant hazard rating, and a summary of the primary hazards listed in their MSDS/SDS.

NOTE: This list does not include all materials used in the procedure. A complete list of materials used in this procedure can be found in the Reagents and Standards Section and the Equipment and Supplies Section of this SOP.

Employees must review the information in the MSDS/SDS for each material before using it for the first time or when there are major changes to the MSDS/SDS. Electronic copies of MSDS/SDS can be found using the "MSDS" link on the Oasis homepage and on the EH&S webpage on Oasis.

Material	Hazards	Exposure Limit[1]	Signs and Symptoms of Exposure
Hydrochloric Acid[2]	Corrosive Poison	5ppm Ceiling	Inhalation of vapors can cause coughing, choking, inflammation of the nose, throat, and upper respiratory tract, and in severe cases, pulmonary edema, circulatory failure, and death. Can cause redness, pain, and severe skin burns. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
Nitric Acid[2]	Corrosive Oxidizer Poison	2ppm TWA 4ppm STEL	Nitric acid is extremely hazardous; it is corrosive, reactive, an oxidizer, and a poison. Inhalation of vapors can cause breathing difficulties and lead to pneumonia and pulmonary edema, which may be fatal. Other symptoms may include coughing, choking, and irritation of the nose, throat, and respiratory tract. Can cause redness, pain, and severe skin burns. Concentrated solutions cause deep ulcers and stain skin a yellow or yellow-brown color. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
[1]Exposure limit refers to the OSHA regulatory exposure limit.			
[2]Always add acid to water to prevent violent reactions.			

6.0 Equipment and Supplies

6.1 Equipment and Instrumentation

Thermometers – Verify in accordance with SOP SA-AN-100: *Laboratory Support Equipment (Verification and Use)*

Digestion Block – capable of maintaining a sample digestion temperature of 95±5°C. The temperature of the digestion block must be monitored and recorded for each batch. The temperature is measured in a beaker or digestion vessel containing reagent water.

Turbidimeter – Hach Model 2100AN.

6.2 Volumetric Containers

All volumetric labware must be verified in accordance with SOP SA-AN-100: *Laboratory Support Equipment (Verification and Use).* Refer to Attachment 6 for Labware Cleaning Procedures.

Volumetric Labware	Volume	Type (Quantitative / Qualitative)	Use	Verification Frequency	Laboratory Verification Criteria
Volumetric Flasks (Class A)	Various	QUANTITATIVE	Preparing Standards and Dilutions	None (Class A)	None (Class A)
Graduated Cylinders (Class A)	Various	QUANTITATIVE	Preparing Standards and Dilutions	None (Class A)	None (Class A)
Eppendorf-Style Mechanical Pipettes	Various	QUANTITATIVE	Preparing Standards and Dilutions	Initially, prior to first use, then quarterly thereafter (Daily for DOD)	Accuracy = 2% Precision = 1%
Pump-Style Pipettes	Various	Qualitative	Acid/Base Delivery for pH Adjustment	Initially	Accuracy = 10% Precision = 10%
Autosampler Tubes	14mL	Qualitative	Digest Analysis	None	None
Digestion Vials	50mL	QUANTITATIVE	Initial and Final Volumes	None (Class A)	None (Class A)
Digestion Vials	100mL	QUANTITATIVE	Initial and Final Volumes	None (Class A)	None (Class A)
Disposable Medicine Cups	300mL	Qualitative	Used to contain aliquot for pH measurement	None	None

6.3 Lab Supplies

pH paper

Detergent – Alconox or equivalent, used for washing non-disposable labware.

0.45um polypropylene filters and syringes

6.4 Sample Collection Containers

All sample collection containers are single-use disposable containers which limits the potential for contamination.

The routine sample collection containers supplied by the laboratory are 250mL plastic containers purchased with Certificate of Analysis attesting to purity.
Note: Drinking Water samples collected for the Lead and Copper Rule require 1 liter containers.

7.0 Reagents and Standards

7.1 Expiration Dates

Expiration dates (time from initial use or receipt to final use) for standard and reagent materials must be set according to the guidance in this SOP. Note: These are maximum expiration dates and are not to be considered an absolute guarantee of standard or reagent quality. Sound judgment must be used when deciding whether to use a standard or reagent. If there is doubt about the quality of a standard or reagent material, a new material must be obtained or the standard or reagent material verified. Data quality must not be compromised to extend a standard's life.

The expiration date of any standard or reagent must not exceed the expiration date of the standard or reagent that was used to prepare it.

7.2 Reagents

Reagents must be prepared and documented in accordance with SOP SA-AN-041: *Reagent and Standard Materials Procedures.*

Hydrochloric Acid and Nitric Acid must be verified prior to use in accordance with the Eurofins TestAmerica Solvent Lot Testing Program.

Laboratory Reagent Water – ASTM Type I

Nitric Acid (HNO_3): reagent grade. Stable under ordinary conditions of use and storage.
Storage: Store in a cool, dry, ventilated storage area with acid resistant floors and good drainage. Store away from sunlight, heat, water, and incompatible materials.
Expiration:

Unopened: Manufacturer's expiration date
Opened: 5 years from date of opening

Hydrochloric Acid (HCl): reagent grade. Stable under ordinary conditions of use and storage.
Storage: Store in a cool, dry, ventilated storage area with acid resistant floors and good drainage. Store away from sunlight, heat, water, and incompatible materials.
Expiration:

Unopened: Manufacturer's expiration date
Opened: 5 years from date of opening

7.3 Standards

Standards must be prepared and documented in accordance with SOP SA-AN-041: *Reagent and Standard Materials Procedures.* Certificates of analysis or purity must be received with all purchased standards, and scanned and attached in LIMS.

7.3.1 Turbidity Standards

Turbidity Standard, 1.0 NTU, Hach pre-prepared 1L
TALS Name: TURB 1.0_XXXX
Storage: Gen. Instrument Lab – Store in a cool, dry area away from heat or light
Expiration: Manufacturer's expiration date, or 30 days from opening

7.3.2 Silica/Silicon Standards

Silicon (Si) stock solutions are usually purchased for this procedure. The following conversion is used to adjust any volumes or concentrations appropriately:

$$Si = \frac{SiO_2}{2.14}$$

7.3.2.1 Stock SiO_2 Standard, 10000mg/L Si / 21400mg/L SiO_2 – purchased from CPI. Store at room temperature. This standard must be used by the manufacturer's expiration date. Storage: Store in a cool, dry, ventilated storage area. Store away from sunlight, heat, water, and incompatible materials.

7.3.2.2 Intermediate SiO_2 Standard, 467mg/L Si / 1000mg/L SiO_2 – Add 20mL to 30mL of reagent water to a clean, plastic 100mL volumetric flask. Add the volume of the Stock SiO_2 Standard given in the table below to the volumetric flask. Dilute to volume with reagent water.

Element	Conc. Stock SiO_2 Std (mg/L)	Volume Stock SiO_2 Std (mL)	Final Volume (mL)	Final Conc. (mg/L)
Silica (SiO_2) [Silicon (Si)]	21400 [10000]	4.67	100	1000 [467]

Storage: Store in a cool, dry, ventilated storage area. Store away from sunlight, heat, water, and incompatible materials.

Expiration: Prepare this solution every 180 days or sooner as needed or required. The expiration date must not exceed the expiration date of any of the components.

7.3.3 ICP/MS Spiking Solutions

7.3.3.1 ICP/MS Spiking Solution 1 – Purchased from CPI International (4400-060117RHO2). The concentrations of the analytes in this solution are listed on the accompanying certificate of analysis.
Storage: Store this solution at room temperature.
Expiration: Replace this solution by the manufacturer's expiration date or sooner if needed or required.

7.3.3.2 ICP/MS Spiking Solution 2 – Add 10mL of hydrochloric acid to a 100mL volumetric flask containing about 50mL of laboratory reagent water. Add the appropriate volume of each single element stock standard to the flask. Dilute to volume with reagent water, and mix thoroughly. Transfer the spiking solution to a labeled storage container.

Element	Parent Standard Concentration (mg/L)	Volume Added (mL)	Final Volume (mL)	Final Concentration (mg/L)
Mercury (Hg)	1000	0.25	500	0.50

Storage: Store in a cool, dry, ventilated storage area with acid resistant floors and good drainage. Store away from sunlight, heat, water, and incompatible materials.
Expiration: Due to mercury requirements, prepare this solution every 28 days or sooner as needed. The expiration date must not exceed the expiration date of any of the components.

7.3.3.3 ICP/MS Silver Spiking Solution – Add 10mL of hydrochloric acid to a 100mL volumetric flask containing about 50mL of laboratory reagent water. Add the appropriate volume of each single element stock standard to the flask. Dilute to volume with reagent water, and mix thoroughly. Transfer the spiking solution to a labeled storage container.

Element	Parent Standard Concentration (mg/L)	Volume Added (mL)	Final Volume (mL)	Final Concentration (mg/L)
Silver (Ag)	1000	2.5	500	5.0

8.0 Sample Collection, Preservation, Shipment, and Storage

8.1 Aqueous Samples

8.1.1 Total Metals

Aqueous samples are routinely collected in 250mL plastic containers containing 3mL of a 1:3 nitric acid preservative. The preservative should be sufficient to achieve a sample pH of less than 2.

Although no temperature preservation is required, samples are routinely iced at the time of collection at 0-6°C (less than 6°C but not frozen). Samples are stored at room temperature until the time of digestion. Samples must be digested and analyzed within 180 days of sample collection. If mercury is requested the samples must be digested and analyzed within 28 days of collection. Digestates are stored at room temperature until the time of analysis.

Note: Drinking water samples for the Lead and Copper Rule require 1 liter of sample to be collected.

NCMs must be initiated for samples collected in improper containers and containing improper or insufficient preservatives.

8.1.2 Dissolved Metals

Aqueous samples for dissolved metals are routinely filtered at the time of sampling and collected in 500mL plastic containers containing 3mL of a 1:3 nitric acid preservative. The preservative should be sufficient to achieve a sample pH of less than 2.

Although no temperature preservation is required, samples are routinely iced at the time of collection at 0-6°C (less than 6°C but not frozen). Samples are stored at room temperature until the time of digestion. Samples must be digested and analyzed within 180 days of sample collection. If mercury is requested the samples must be digested and analyzed within 28 days of collection. Digestates are stored at room temperature until the time of analysis.

Note: If the sample is to be filtered in the laboratory, the sample must be collected in 500mL plastic container with no preservatives. The sample must be stored at 0-6°C (less than 6°C but not frozen) until filtered. Once filtered, the laboratory will add nitric acid to obtain a pH of less than 2.

NCMs must be initiated for samples collected in improper containers and containing improper or insufficient preservatives.

8.1.3 Silica Samples

Aqueous samples for silica are routinely collected in 500mL plastic containers.

Samples must be iced at the time of collection and maintained at 0-6°C (less than 6°C but not frozen) until the time of filtration and analysis. Samples must be filtered and analyzed within 180 days of collection.

NCMs must be initiated for samples collected in improper containers.

Note: Acid preserved samples routinely collected for ICP analyses and acid digested samples should not be used for the analysis of silica. Also note that glass containers should be avoided.

8.1.4 Preservation Checks – pH Verification

For each sample, prior to sample preparation,

- Place a piece of pH paper in a disposable medicine cup.
- Pour a few drops of sample into the medicine cup and note the color change of the pH paper.
- If the pH is greater than 2, initiate a Nonconformance Memo, and perform the additional steps listed below:
  1. Add ~0.5mL of concentrated nitric acid. Mix well, and re-check the pH. If the pH is still > 2, add additional aliquots of concentrated nitric acid, until the pH is < 2.

2. Set the sample aside, and re-check the pH after 24 hours. If pH is still greater than 2 after 24 hours, then repeat Steps 1 and 2, until the pH remains < 2 after 24 hours.

Note: Initial preservation checks for samples that require pH adjustment are captured in the PRESERV_CHK batch, with the final pH check (i.e., the re-check that occurs after 24 hours) recorded in the prep batch. Preservation checks for samples that do not require pH adjustment are captured in the prep batch.

Note: To avoid cross-contamination, use a separate medicine cup and piece of pH paper per sample. Do not dip the pH paper into the sample container. The pH paper dye may bleed into the sample and affect sample results.

Note: Samples that are not at pH <2 upon arrival in the lab may contain cyanide or sulfide or may be highly buffered. Working under a hood minimizes the hazard that may be caused by the evolution of hydrogen cyanide or hydrogen sulfide upon acidification of the sample. Be aware that acid/base neutralization reaction may be violent and evolve a significant amount of heat.

Note: The Sample Receiving Department performs an initial determination of sufficient preservation upon sample receipt to the laboratory. Samples received from the client that are not properly preserved are marked with a black dot sticker on the lid of the container, prior to transfer to the Metals Department. This sticker serves as a method of informing the preparations analyst that the sample was not preserved properly and that preservation steps are needed.

8.2 TCLP/SPLP Leachate Samples

Once the TCLP/SPLP extraction procedure has been performed, the leachate is transferred to a plastic container. The leachate must be acidified with nitric acid to pH <2, upon receipt to the Metals Department. The leachate sample must be digested and analyzed within 180 days of completion of the TCLP/SPLP extraction. If mercury is requested, the leachate sample must be digested and analyzed within 28 days of completion of the TCLP/SPLP extraction.

9.0 Quality Control

SOP SA-QA-017: *Evaluation of Batch QC Data* and the SOP Summary in Attachment 3 of the associated analytical SOPs provide requirements for evaluating QC data.

9.1 Batch QC

A digestion batch consists of up to 20 environmental samples and the associated QC items digested together within a 24 hour period.

EPA 200.7 and EPA 200.8 – Drinking Water

The laboratory's default minimum QC items performed for each digestion batch are: a method blank and a laboratory control sample (LCS), a low-level LCS (LLCS), a matrix spike (MS) to be performed on a minimum of 10% of samples or one per batch – whichever is greater, and a matrix spike duplicate (MSD).

This frequency equates to the following:

- For a batch of 10 or fewer samples, the minimum QC items are a method blank, an LCS, an LLCS, 1 matrix spike, and 1 matrix spike duplicate.
- For a batch of 11-20 samples, the minimum QC items are a method blank, an LCS, an LLCS, 1 matrix spike (from sample 1-10), another matrix spike (from sample 11-20), and a matrix spike duplicate (MSD).

EPA 200.7 and EPA 200.8 – Clean Water Act

The laboratory's default minimum QC items performed for each digestion batch are: a method blank and a laboratory control sample (LCS), a matrix spike (MS) to be performed on a minimum of 10% of samples or one per batch – whichever is greater, and a matrix spike duplicate (MSD).

This frequency equates to the following:

- For a batch of 10 or fewer samples, the minimum QC items are a method blank, an LCS, 1 matrix spike, and a matrix spike duplicate.
- For a batch of 11-20 samples, the minimum QC items are a method blank, an LCS, 1 matrix spike (from sample 1-10), another matrix spike (from sample 11-20), and a matrix spike duplicate.

EPA Methods 3005A and 3010A and Standard Methods 3030C

The laboratory's default minimum QC items performed for each digestion batch are: a method blank, a laboratory control sample (LCS), a matrix spike (MS), and a matrix spike duplicate (MSD) or a sample duplicate.

The routine container supplied for this method is a 250mL container. 50mL is required for digestion. Reduced sample initial volumes may be necessary to achieve the required batch matrix spike frequency; however, the minimum digestion volume to be used for the matrix spike samples is 25mL. Note: Final volumes and spike amounts must be adjusted to compensate for these reduced initial volumes.

If there is insufficient sample volume to perform the required matrix spike(s), the LCS must be prepared in duplicate (i.e., LCSD). An NCM must be initiated on all affected samples to denote this situation. Insufficient sample volume is defined as receiving less than a total of 100mL.

Note: If an LCS and LCSD are performed, both QC items must be evaluated and reported. Acceptable recoveries (as well as %RPD) for both LCS and LCSD are required.

9.2 Instrument QC

Details on instrument QC are given in the associated analytical SOPs listed in Section 1.

9.2.1 Turbidimeter Instrument QC

Refer to SOP SA-GE-206 for additional information on the initial calibration of the turbidimeter. The Metals Department uses this turbidimeter only to assess whether drinking water samples can be analyzed undigested; therefore, a reduced QC frequency is applied for this department.

The calibration curve must be verified initially and at the end of the analysis with a low-level standard at 1.0 NTU. The CCV must be within 10% of the true value to be acceptable.

9.3 Corrective Action for Out-of-Control Data

When the quality control parameters do not meet the criteria set forth in this SOP, corrective action must be taken in accordance with SOP SA-QA-005: *Preventive and Corrective Action Procedures* the QC Summary Table in Attachment 3 of the associated analytical SOP. SOP SA-QA-005 provides contingencies for out-of-control data and gives guidance for exceptionally permitting departures from approved policies and procedures. Nonconformance Memos must be initiated to document all instances where QC criteria are not met and all departures from approved policies and procedures.

10.0 Procedure

10.1 Sample and QC Sample Preparation

Unless otherwise requested, groundwater and surface waters are to be prepared using the total recoverable metals procedure given in Section 10.1.3. TCLP/SPLP samples must be digested for total metals (Section 10.1.4). Note that there are different spiking solutions for routine and TCLP/SPLP matrix spikes.

NOTE: The block digestion apparatus must be at 95 ± 3°C during prep. To verify temperature of the hotblock a digestion tube filled with DI water must be placed at random locations across each block used for digestion. This position must be changed daily. A single thermometer is transferred from block to block and a minimum of 10 minutes allowed for the thermometer to equilibrate before taking the reading. This temperature reading, uncorrected and corrected, is then recorded in the batch information.

10.1.1 Dissolved Metals

10.1.1.1 Results reported as “dissolved metals” are defined as the concentrations of analytes in a liquid sample that will pass through a 0.45um membrane filter prior to acidification/preservation. The filtration step can occur in the field or in the laboratory. In either case, the sample must be unpreserved prior to filtration and then filtered using a 0.45um filter.

10.1.1.2 Field filtered samples are usually preserved in the field, immediately after filtration. Unpreserved samples sent to the laboratory for lab filtration must be preserved immediately after filtration.

10.1.1.3 If the laboratory filters the samples, the reagent water used to prepare the method blank and laboratory control samples associated with the "dissolved metals" samples must be filtered through the same type of filter used to filter the field samples.

Note: The LCS must be spiked prior to filtration.

10.1.1.4 A comparison between total and dissolved results is good practice, and logic should follow that total results should be equal or greater than dissolved results, especially if the dissolved metals are lab filtered. Comparable hits with less than 20% RPD should be considered equal. Any other discrepancies should be discussed with the Department Manager or Supervisor, Technical Manager, or the Project Manager.

10.1.1.5 The laboratory's standard practice is to digest all non-silica liquid samples; however, samples filtered for the determination of dissolved metals do not require digestion if the sample:

- has a low COD (<20mg/L);
- has a turbidity <1 NTU;
- is colorless with no significant odor; ***and***
- is of one liquid phase and free of suspended particulates or precipitates after acidification

10.1.2 Turbidity Determination for Drinking Water Samples

Drinking water samples with a turbidity concentration of less than 1NTU may be analyzed without digestion. The exception to this rule is silver, which requires sample digestion prior to analysis. The turbidity of drinking water samples is checked using the procedures outlined in this section. If the turbidity of the sample is not checked, the digestion procedure must be performed.

10.1.2.1 Turn on turbidimeter and allow it to warm up 15-30 minutes.

10.1.2.2 Verify the instrument is operating properly by the analysis of a continuing calibration verification sample initially, after every 25 samples, and at the end of the run with the Turbidity Standard at 1.0 NTU.

10.1.2.3 Analyze a DI water blank (CCB). The DI water blank must be less than 1NTU to be acceptable. If the DI water blank is greater the 1NTU, check to be certain the sample cell is clean. If the blank is significantly above 1NTU, the sample cell may be scratched and may need to be replaced.

10.1.2.4 Determine the turbidities for all samples by filling the cell with sample and placing the cell in the sample well of the turbidimeter. Record values for samples with a turbidity less than 1NTU in the drinking water prep batch worksheet in LIMS. (Samples with a turbidity reading greater than 1NTU or samples being analyzed for silver, MUST be digested). Be certain to rinse the cells between samples with DI water to avoid contamination.

10.1.3 Total Recoverable Metals (EPA 3005A)

This digestion procedure is used to prepare aqueous samples for total recoverable metals determination by ICP and ICP/MS.

10.1.3.1 Transfer a 50mL aliquot (or an appropriate volume diluted to 50mL with reagent water) of a well-mixed sample to a 50mL digestion block vial.

Note: If there is not sufficient volume to use a 50mL aliquot, the lab can use a smaller volume of sample, proportional volumes of reagents, and adjust the final digestate volume back to the original volume of the sample used. For example, if 25mL of sample is digested, one half of the routine volumes of reagent are used and the final volume of the digestate is brought back to 25mL. When a smaller aliquot is used, the digestion analyst must be careful not to allow the sample digest to evaporate completely.

10.1.3.2 Add 50mL of reagent water to a digestion block vial to serve as the method blank. This QC sample is taken through all digestion and sample preparation steps to monitor for contamination that may be due to glassware, reagents, or sample handling.

10.1.3.3 Add 0.50mL of the appropriate spiking solutions to a 50mL aliquot of reagent water to serve as the laboratory control spike (LCS). If a duplicate laboratory control spike (LCSD) is required, spike a second 50mL aliquot of reagent water with 0.50mL of the appropriate spiking solutions.

Analytical Method	Matrix	QC	Spiking Solutions
EPA 6010C	Aqueous	LCS/MS/MSD	ICP/MS Spike 1 ICP/MS Spike 2 ICP/MS Ag Spike
EPA 6020A	Aqueous	LCS/MS/MSD	ICP/MS Spike 1 ICP/MS Spike 2 ICP/MS Ag Spike

10.1.3.4 Add 0.50mL of the appropriate spiking solutions to each of two 50mL aliquots of the client sample designated as the matrix spike samples (MS and MSD).

10.1.3.5 Add 2.5mL of concentrated HCl and 1.0mL of concentrated HNO_3 to each sample. Gently heat the digestion vessel until the sample refluxes. The sample must not be heated to boiling; that is, bubbles are not formed in the liquid in the bottom of the digestion vessel. The sample/acid solution is refluxing when the liquid evaporates and drops of liquid condense on the sides of the digestion vessel and fall back into the digestion vessel. Evaporate the sample until the volume is approximately 15mL. Do not allow any portion of the vessel bottom to become dry at any time during the digestion.

Note: If a volume of sample smaller than 50mL is digested, the amount of acid should be reduced proportionately.

10.1.3.6 Wash down the inside of the digestion vessel with reagent water. Dilute the sample digestate to 50mL with reagent water.

Note: The digestate may be diluted to a volume less than the original volume if sample pre-concentration is required to meet lower reporting limits. The pre-concentration must be limited to a factor of four.

10.1.3.7 The sample is now ready for analysis via the appropriate analytical SOP.

10.1.4 Total Metals (EPA 3010A, EPA 200.7, and EPA 200.8)

This digestion procedure is used for the preparation of aqueous samples for total metals determination by ICP and ICP/MS.

10.1.4.1 Transfer a 50mL aliquot (or an appropriate volume diluted to 50mL with reagent water) of a well-mixed sample to a 50mL digestion block vial.

Note: If there is not sufficient volume to use a 50mL aliquot, the lab can use a smaller volume of sample, proportional volumes of reagents, and adjust the final digestate volume back to the original volume of the sample used. For example, if 25mL of sample is digested, one half of the routine volumes of reagent are used and the final volume of the digestate is brought back to 25mL. When a smaller aliquot is used, the digestion analyst must be careful not to allow the sample digest to evaporate completely.

10.1.4.2 Add 50mL of reagent water to a digestion block vial to serve as the method blank. This QC sample is taken through all digestion and sample preparation steps to monitor for contamination that may be due to glassware, reagents, or sample handling.

10.1.4.3 Add 0.50mL of the appropriate spiking solutions to a 50mL aliquot of reagent water to serve as the laboratory control spike (LCS). If a duplicate laboratory control spike (LCSD) is required, spike a second 50mL aliquot of reagent water with 0.50mL of the appropriate spiking solutions.

Analytical Method	Matrix	QC	Spiking Solutions
EPA 6010C EPA 200.7	Aqueous	LCS/MS/MSD	ICP/MS Spike 1 ICP/MS Spike 2 ICP/MS Ag Spike
EPA 6020A EPA 200.8	Aqueous	LCS/MS/MSD	ICP/MS Spike 1 ICP/MS Spike 2 ICP/MS Ag Spike

10.1.4.4 Add 0.50mL of the appropriate spiking solutions to two 50mL aliquots of the client sample designated as the matrix spike samples (MS and MSD).

10.1.4.5 Add 1.5mL of concentrated HNO_3 to each sample. Gently heat the digestion vessel until the sample refluxes. The sample must not be heated to boiling; that is, bubbles are not formed in the liquid in the bottom of the digestion vessel. The sample/acid solution is refluxing when the liquid evaporates and drops of liquid condense on the sides of the digestion vessel and fall back into the digestion vessel. Evaporate the sample until the volume is approximately 5-10mL. Do not allow any portion of the vessel bottom to become dry at any time during the digestion.

Note: If a volume of sample smaller than 50mL is digested, the amount of acid should be reduced proportionately.

10.1.4.6 Remove the digestion vessels from the digestion block and cool the digestion vessels to room temperature. Add another 1.5mL portion of concentrated HNO_3. Continue heating the sample on the digestion block. Again, at the proper temperature, the sample should gently reflux in the digestion vessel. Do not allow the sample to boil.

10.1.4.7 Continue heating the sample and adding additional 1.5mL portions of concentrated HNO_3 until the digestate is light in color or does not change in appearance after subsequent additions of HNO_3. If a sample requires more than 6mL of acid to digest, contact the Department Manager for guidance.

10.1.4.8 Evaporate the digestate until the volume is approximately 5-10mL.

10.1.4.9 Add 2.5mL of concentrated HCl and warm the sample digestate for 15 minutes.

10.1.4.10 Wash down the inside of the digestion vessel with reagent water. Dilute the sample digestate to 50mL with reagent water.

Note: The digestate may be diluted to a volume less than the original volume if sample pre-concentration is required to meet lower reporting limits. The pre-concentration should be limited to a factor of four.

10.1.4.11 The sample is now ready for analysis via the appropriate analytical SOP.

10.1.5 Total Metals and TCLP/SPLP Samples (EPA 3010A)

This digestion procedure is used for the preparation of TCLP/SPLP leachate samples for total metals determination by ICP. Note that the LCS is spiked with the routine analytes to allow the TCLP/SPLP samples to be digested along with aqueous samples.

Note: The TCLP/SPLP leachate MS/MSD must be spiked prior to acidification. This is to be performed as close to 8 hours upon receipt of the leachate to the Metals Prep area as possible. Refer to Section 10.1.5.4 for more information.

10.1.5.1 Transfer a 5mL aliquot (diluted to 50mL) for TCLP or a 50mL aliquot for SPLP of a well-mixed sample to a clean 50mL digestion vessel. Larger volumes may be digested at the discretion of the lab. The volume of spike solution added should be adjusted proportionately.

10.1.5.2 Add 5mL of extraction fluid diluted to 50mL for TCLP or 50mL for SPLP to a digestion vessel to serve as the method blank. This QC sample is taken through all digestion and sample preparation steps to monitor for contamination that may be due to glassware, reagents, or sample handling. A blank for each type of extraction fluid must be digested and analyzed.

10.1.5.3 Add 1.0mL of the appropriate spiking solutions to a 5mL aliquot of extraction fluid diluted to 50mL for TCLP or to a 50mL aliquot of extraction fluid for SPLP. This is designated as the laboratory control spike (LCS). If required, perform a LCSD by following the same procedure as the LCS. The routine ICP/MS spiking solution is used for the TCLP/SPLP LCS because of frequent client requests for reporting more analytes from the TCLP/SPLP leachate than are currently regulated. Preparing an LCS with all of the target analytes eliminates re-digestion and provides QC for the requested analytes.

Note: If both Extraction Fluid 1 and Extraction Fluid 2 are included in the batch, use Extraction Fluid 1 for the LCS and LCSD. Refer to SOP SA-EX-015: *Toxicity Characteristic Leaching Procedure (TCLP) and Synthetic Precipitation Leaching Procedure (SPLP)* for information on the TCLP/SPLP extraction fluids.

Analytical Method	Matrix	QC	Spiking Solutions
EPA 6010C	TCLP/SPLP	LCS	ICP/MS Spike 1 ICP/MS Spike 2 ICP/MS Ag Spike

10.1.5.4 The TCLP MS/MSD is prepared as follows: add 4.0mL of the ICPMS Calibration Stock and 0.4mL of the ICPMS Hg intermediate calibration standard to each of two 20mL aliquots of leachate.

The SPLP MS/MSD is prepared as follows: add 4.0mL of the ICPMS Calibration Stock and 0.04mL of the ICPMS Hg intermediate calibration standard to each of two 100mL aliquots of the leachate.

Note: The TCLP/SPLP digestion batch consists of twenty or fewer field samples and the associated QC items. A TCLP/SPLP digestion batch must not exceed 20 field samples. Every TCLP/SPLP digestion batch will have a method blank (MB), a laboratory control sample (LCS), and a matrix spike (MS).

10.1.5.5 Add 1.5mL of concentrated HNO_3 to each sample. Gently heat the digestion vessel until the sample refluxes. The sample is not heated to boiling; that is, bubbles are not formed in the liquid in the bottom of the digestion vessel. The sample/acid solution is refluxing when the liquid evaporates and drops of liquid condense on the sides of the digestion vessel and fall back into the digestion

vessel. Evaporate the sample until the volume is approximately 5mL. Do not allow any portion of the vessel bottom to become dry at any time during the digestion.

Note: If a volume of sample smaller than 50mL is digested, the amount of acid should be reduced proportionately.

10.1.5.6 Remove the digestion vessels from the digestion block and cool the digestion vessels to room temperature. Add another 1.5mL portion of concentrated HNO_3. Continue heating the sample on the digestion block. Again, at the proper temperature, the sample should gently reflux in the digestion vessel. Do not allow the sample to boil.

10.1.5.7 Continue heating the sample and adding additional 1.5mL portions of concentrated HNO_3 until the digestate is light in color or does not change in appearance after subsequent additions of HNO_3. If a sample requires more than 6mL of acid to digest, contact the Department Manager for guidance.

10.1.5.8 Evaporate the digestate until the volume is approximately 5-10mL.

10.1.5.9 Add 2.5mL of concentrated HCl and warm the sample digestate for 15 minutes.

10.1.5.10 Wash down the inside of the digestion vessel with reagent water. Dilute the sample digestate to 50mL with reagent water.

10.1.5.11 The sample is now ready for analysis by ICP.

10.1.6 Acid-Extractable Metals (SM3030C)

This digestion procedure is used for the preparation of aqueous samples for acid-extractable metals by ICP and ICP/MS.

10.1.6.1 Transfer a 50mL aliquot (or an appropriate volume diluted to 50mL with reagent water) of a well-mixed sample to a 50mL digestion block vial.

Note: If there is not sufficient volume to use a 50mL aliquot, the lab can use a smaller volume of sample, proportional volumes of reagents, and adjust the final digestate volume back to the original volume of the sample used. For example, if 25mL of sample is digested, one half of the routine volumes of reagent are used and the final volume of the digestate is brought back to 25mL. When a smaller aliquot is used, the digestion analyst must be careful not to allow the sample digest to evaporate completely.

10.1.6.2 Add 50mL of reagent water to a beaker that has been designated as the method blank. This QC sample is taken through all digestion and sample preparation steps to monitor for contamination that may be due to glassware, reagents, or sample handling.

10.1.6.3 Add 0.50mL of the appropriate spiking solutions to a 50mL aliquot of reagent water designated as the laboratory control spike (LCS). If a duplicate laboratory control spike (LCSD) is required, spike a second 50mL aliquot of reagent water with 0.50mL of the appropriate spiking solutions.

Analytical Method	Matrix	QC	Spiking Solutions
EPA 6010C	Aqueous	LCS/MS/MSD	ICP/MS Spike 1 ICP/MS Spike 2 ICP/MS Ag Spike
EPA 6020A	Aqueous	LCS/MS/MSD	ICP/MS Spike 1 ICP/MS Spike 2 ICP/MS Ag Spike

10.1.6.4 Add 0.50mL of the appropriate spiking solutions to each of two 50mL aliquots of the client sample designated as the matrix spikes sample (MS and MSD).

10.1.6.5 Add 1.25mL of concentrated HCl to each sample and QC item.

Note: If a volume of sample smaller than 50mL is digested, the amount of acid should be reduced proportionately.

10.1.6.6 Heat for 15 minutes on a hot block.

10.1.6.7 The sample is now ready for filtration and final volume adjustment which occurs immediately prior to analysis by ICP or ICP/MS.

10.1.7 Total Recoverable Metals (EPA 200.8 and EPA 200.7)

This digestion procedure is used for the preparation of water and drinking water samples for total recoverable metal determination by ICP and ICP/MS.

10.1.7.1 Transfer a 50mL aliquot (or an appropriate volume diluted to 50mL with reagent water) of a well-mixed sample to a clean 50mL block digestion vial.

Note: If there is not sufficient volume to use a 50mL aliquot, the lab can use a smaller volume of sample and bring the final digestate volume back to the original volume of the sample used. That is, if 25mL of sample is digested, the final volume of the digestate should be brought back to 25mL. If a smaller aliquot is used, the digestion analyst must be careful not to allow the sample digest to evaporate completely.

10.1.7.2 Add 50mL of reagent water to a block digestion vial that has been designated as the method blank. This QC sample is taken through all digestion and sample preparation steps to monitor for contamination that may be due to glassware, reagents, or sample handling.

10.1.7.3 Add 0.50mL of the appropriate spiking solutions to a 50mL aliquot of reagent water designated as the laboratory control spike (LCS). If a duplicate laboratory control spike (LCSD) is required, spike a second 50mL aliquot of reagent water with 0.50mL of the appropriate spiking solutions.

Analytical Method	Matrix	QC	Spiking Solutions
200.7 DW and TR	Aqueous	LCS/MS/MSD	ICP/MS Spike 1 ICP/MS Spike 2 ICP/MS Ag Spike
200.8 DW and TR	Aqueous	LCS/MS/MSD	ICP/MS Spike 1 ICP/MS Spike 2 ICP/MS Ag Spike

10.1.7.4 Add 0.50mL of the appropriate spiking solutions to each of two 50mL aliquots of the client sample designated as the matrix spikes sample (MS and MSD).

10.1.7.5 Add 0.5mL of concentrated HNO_3 and 0.25mL of concentrated HCl to each sample. Gently heat the block digestion vial and reduce the sample volume to about 10mL without boiling; that is, bubbles are not formed in the liquid in the bottom of the block digestion vial. Do not allow any portion of the vessel bottom to become dry at any time during the digestion.

Note: If a volume of sample smaller than 50mL is digested, the amount of acid should be reduced proportionately.

10.1.7.6 Wash down the inside of the block digestion vial with reagent water. Dilute the sample digestate to 50mL with reagent water. Transfer the digest to a labeled storage container, usually a 125mL plastic vial. Allow any undissolved material to settle out or centrifuge the sample to remove particulate matter. A portion of the sample may be filtered, if necessary, to remove particulates but care must be taken to avoid contamination of the sample during filtration.

Note: The digestate may be diluted to a volume less than the original volume if sample concentration is required to meet lower reporting limits. The pre-concentration should be limited to a factor of four.

10.1.7.7 The sample is now ready for analysis by ICP and ICP/MS.

10.1.8 Silica Samples

10.1.8.1 Samples are prepared by filtration of an unpreserved aliquot through a 0.45um syringe filter. The sample must be kept refrigerated until time of analysis. Note: The same number of syringe filters should be used for the method blank, LCS, and the samples.

10.1.8.2 The method blank is prepared by filtering an amount of reagent water equal to the volume of sample that is filtered.

10.1.8.3 The LCS is prepared by spiking 10mL of reagent water with 0.10mL of SiO_2 Intermediate Standard, and passing it through a 0.45um syringe filter. The SiO_2 spike concentration in the LCS is 10mg/L.

10.1.8.5 The matrix spike and/or matrix spike duplicate (MS/MSD) are prepared by adding 0.10mL of SiO_2 Intermediate Standard solution to two separate 10mL aliquots of the sample to be spiked and filtering through a 0.45um filter. The SiO_2 spike concentration in the MS/MSD is 10mg/L.

10.2 Analysis

Details on sample analysis are given in the associated analytical SOPs listed in Section 1.

11.0 Calculations / Data Reduction

11.1 Data Reduction

Data must be evaluated in accordance with SOP SA-QA-002: *Data Generation and Review*.

11.1.1 Historical Data

Many of the laboratory's clients submit samples for repeat monitoring purposes. Prior to analysis, verify LIMS Worksheet Notes and/or use the Historical Data Tracker feature to determine if historical data is available for review.

11.1.2 Chemical Relationships

When available, the following chemical relationships must be evaluated for each sample. If these relationships are not met the Department Manager must be contacted immediately.

- Total results are ≥ Dissolved results

11.2 Calculations

Details on sample calculations are given in the associated analytical SOPs listed in Section 1.

12.0 Method Performance

12.1 Reporting Limit Verification (RLV)

At a minimum, RLVs must be performed initially upon method set-up in accordance with SOP SA-QA-007: *Determination and Verification of Detection and Reporting Limits*.

For analytes and methods certified by DOD ELAP, RLVs must also be performed quarterly thereafter. For analytes and methods certified by NELAC, RLVs must also be performed annually thereafter. Exceptions may be made for project-specific non-routine analytes.

12.2 Method Detection Limit (MDL) Study

The MDL is the lowest concentration that can be detected for a given analytical method and sample matrix with 99% confidence that the analyte is present. MDLs reflect a calculated (statistical) value determined under ideal laboratory conditions in a clean matrix and may not be achievable in all environmental matrices. The current MDLs associated with this procedure are given in the Method Limit Group (MLG) in LIMS.

At a minimum, MDL Studies must be performed initially upon method set-up in accordance with SOP SA-QA-007: *Determination and Verification of Detection and Reporting Limits*.

Note: MDL Studies are not required for non-routine analytes provided results are not reported below the RL (i.e., MDL equals RL in LIMS).

12.3 Method Detection Limit Verification (MDLV)

At a minimum, MDLVs must be performed initially upon method set-up in accordance with SOP SA-QA-007: *Determination and Verification of Detection and Reporting Limits*.

For analytes and methods certified by DOD ELAP, MDLVs must also be performed quarterly thereafter. For analytes and methods certified by NELAC, MDLVs must also be performed annually thereafter.

Note: MDLVs are not required for non-routine analytes provided results are not reported below the RL (i.e., MDL equals RL in LIMS).

12.4 Determination of the Instrument Detection Limit (IDL)

The instrument detection limit (IDL) is the concentration of analyte that can be statistically distinguished from the background noise of the instrument. The IDL limit must be determined annually, at a minimum, for each analyte in accordance with SOP SA-QA-007: *Determination and Verification of Detection and Reporting Limits.*

The IDL is defined as three times the standard deviation of seven replicate analyses of a blank solution analyzed over three non-consecutive days. The IDL may be elevated above the background noise (blank levels). The current IDL associated with this procedure is given in the Equipment Limit Group (ELG) in LIMS.

12.5 QC Limit Generation, Control Charting, and Trend Analysis

The control limits for the batch QC items (LCS and MS/MSD) for this procedure are specified in the reference method and cannot be broadened; therefore, the laboratory defaults to the method-defined limits and does not utilize in-house or laboratory-derived limits for the evaluation of batch QC items.

Although the laboratory must default to the method-defined QC limits, control charting is a useful tool and is performed to assess analyte recoveries over time to evaluate trends. Control charting must be performed periodically (at a minimum annually) in accordance with SOP SA-QA-017: *Evaluation of Batch QC Data.*

12.6 Demonstrations of Capability

Initial and continuing demonstration of capability must be performed in accordance with SOP SA-QA-006*: Training Procedures.*

Prior to performing this procedure unsupervised, each new analyst who performs this analysis must demonstrate proficiency per method/analyte combination by successful completion of an initial demonstration of capability. The IDOC is performed by the analysis of 4 consecutive LCSs that meet the method criteria for accuracy and precision. The IDOC must be documented and routed to the QA Department for filing.

Annual continuing demonstrations of capability (CDOCs) are also required per analyst per method/analyte combination. The CDOC requirement may be met by the consecutive analysis of four LCS all in the same batch, by the analysis of four LCS analyzed in four consecutive batches (in different batches on different days), via acceptable results on a PT study, or analysis of client samples with statistically indistinguishable results when compared to another certified analyst. The CDOC must be documented and routed to the QA Department for filing.

12.7 Training Requirements

All training must be performed and documented in accordance with SOP SA-QA-006: *Training Procedures*.

Note: The SOPs listed in the Reference/Cross-Reference Section are applicable to this procedure. All employees performing this procedure must also be trained on these SOPs, and/or have a general understanding of these procedures, as applicable.

13.0 Pollution Control

It is Eurofins TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (e.g., examining recycling options, ordering chemicals based on quantity needed, preparing reagents based on anticipated usage and reagent stability, etc.). Employees must abide by the policies in Section 13 of the Environmental Health and Safety Manual.

This procedure has been evaluated for opportunities to minimize the waste generated. Where reasonably feasible, pollution control procedures have been incorporated.

14.0 Waste Management

Waste management practices must be conducted consistent with all applicable federal, state, and local rules and regulations. All waste (i.e., excess reagents, samples, and method process wastes) must be disposed of in accordance with Section 9 of the Eurofins TestAmerica Savannah Addendum to the EHSM. Waste description rules and land disposal restrictions must be followed.

14.1 Waste Streams Produced by the Method

The following waste streams are produced when this method is carried out:

- Excess aqueous samples – Dispose according to characterization on the sample disposal program. Neutralize non-hazardous samples before disposal into drain/sewer. Transfer hazardous samples (identified on disposal program) to the waste department for disposal.
    - Samples within the prep lab must be placed in boxes to prevent possible loss or misplacement, and may be stacked and kept for a short time. Boxes up to a height of 3 stacked boxes may be kept, or for a duration of 1 month. Once either of these limits are reached samples must be scanned to the storage area.
- Acidic sample digestions: Neutralize before disposal into drain/sewer system.

Dispose of all metals standards with concentrations exceeding the TCLP threshold limits as a RCRA aqueous hazardous waste. The TCLP constituents and the respective threshold limits are:

Analyte	Disposal Limit (mg/L)
Arsenic	5.0
Barium	100
Cadmium	1.0
Chromium	5.0
Lead	5.0
Selenium	1.0
Silver	5.0
Mercury	0.20

Neutralize the standard by passing with sodium bicarbonate before transferring the waste to the RCRA aqueous hazardous waste drum.

All other metals standards may be disposed, using copious amounts of water, down a sink that flows to the neutralization pit.

15.0 References / Cross-References

- SOP SA-AN-100: *Laboratory Support Equipment (Verification and Use)*
- SOP SA-AN-041: *Reagent and Standard Materials Procedures*
- SOP SA-QA-002: *Data Generation and Review*
- SOP SA-QA-005: *Preventive and Corrective Action Procedures*
- SOP SA-QA-006*: Training Procedures*
- SOP SA-QA-007: *Determination and Verification of Detection and Reporting Limits*
- SOP SA-QA-015: *Homogenization, Compositing, and Segregation of Samples*
- SOP SA-QA-017: *Evaluation of Batch QC Data*
- SOP SA-GE-206: *Turbidity*
- Eurofins TestAmerica Savannah Quality Assurance Manual
- Eurofins TestAmerica Environmental Health and Safety Manual (CW-E-M-001)
- Eurofins TestAmerica Savannah Addendum to the Environmental Health and Safety Manual
- Methods 3005A and 3010A: *Test Methods for Evaluating Solid Waste*, Third Edition, SW-846; vs. EPA Office of Solid Waste and Emergency Response: Washington, DC.
- Methods 200.7 and 200.8 (Drinking Water): Methods for the Determination of Metals in Environmental Samples, May 1994, Supplement 1. (EPA 600/R-94/111).
- Standard Methods for the Examination of Water and Wastewater, Online Edition; American Public Health Association: Washington, DC 2004.

16.0 Method Modifications

16.1 The EPA 200.7 and EPA 200.8 reference methods were written specifically for drinking water and source water samples; however, the laboratory may perform other types of water samples using this procedure.

16.2 The EPA Manual for the Certification of Laboratories Analyzing Drinking Water requires a LFB at the MRL to be performed each day. The laboratory meets this requirement by preparing an LCS at the RL in each EPA 200.7 and EPA 200.8 drinking water batch. The EPA DW Manual does not specify criteria for the low-level LCS; therefore, the laboratory defaults to qualitative identification of the LL-LCS to be acceptable.

16.3 There is no method-defined batch precision requirement for some of these methods. The EPA does require precision for all samples analyzed under the Clean Water Act, and the laboratory routinely performs an MSD to support clients who require precision to be reported. If insufficient sample volume is provided to perform the MS/MSD, the laboratory performs the LCS in duplicate (i.e., LCS/LCSD).

16.4 The turbidimeter is calibrated and maintained by the General Chemistry Department for use with turbidity analyses for EPA 180.1 and SM2130B. The Metals Department uses this turbidimeter only to assess whether drinking water samples can be analyzed undigested; therefore, a reduced QC frequency applied for this department. Refer to SOP SA-GE-206 for additional information on the calibration and verification of this meter.

17.0 Attachments

The following Tables, Diagrams, and/or Validation Data are included as Attachments:

Attachment 1: SOP Summary
Attachment 2: Sample Collection, Preservation, and Holding Time Table
Attachment 3: QC Summary
Attachment 4: Instrument Maintenance and Troubleshooting
Attachment 5: Turbidity Form
Attachment 6: Glassware Cleaning Procedures

Attachment 1
SOP Summary

Sample Preparation Summary
Total Metals and TCLP/SPLP leachates: A known volume, usually 50mL, of sample is transferred to a digestion vessel. The sample is refluxed with nitric acid at approximately 95°C. After the sample has digested, as evidenced by a clear, pale yellow color, HCl is added and the sample is brought up to the original volume with reagent water. The laboratory utilizes two versions of the Total Recoverable preparation procedure. One version is equivalent to the EPA 3005A procedure and the second procedure is equivalent to the EPA 200.7 and 200.8 procedures.

Total Recoverable Metals: A known volume, usually 50mL, of sample is transferred to a digestion vessel. The sample is refluxed with dilute nitric acid and hydrochloric acid at approximately 95°C. After the sample has evaporated to approximately 10-20mL, the sample is brought up to the original volume with reagent water. This procedure is equivalent to EPA Method 3005A, and the EPA Methods 200.7 and 200.8 prep procedures for total recoverable metals.

Acid Extractable Metals: A known volume, usually 50mL of sample preserved with nitric acid, is transferred to a digestion vessel. Hydrochloric acid is added and the sample is heated on a hot block for 15 minutes. The sample is filtered and the final volume adjusted to the original volume. This procedure is equivalent to Standard Methods 3030C.

Silica samples for EPA Methods 200.7 and 6010C: Liquid samples are filtered. The filtered liquid samples are analyzed by ICP.

Drinking water samples for EPA Methods 200.7 and 200.8 with a turbidity concentration of less than 1NTU may be analyzed with no digestion. The exception to this rule is silver, which requires sample digestion prior to analysis. If the sample turbidity is >1NTU, then the EPA Method 200.7 preparation procedure or the EPA Method 200.8 preparation procedure is used.

Samples filtered for the determination of dissolved metals do not require digestion if the sample:
5) has a low COD(<20mg/L);
6) has a turbidity <1NTU ;
7) is colorless with no significant odor ; ***and***
8) is of one liquid phase and free of suspended particulates or precipitates after acidification.

Sample Analysis Summary

ICP: Sample digestates are aspirated and nebulized into a spray chamber. A stream of argon gas carries the sample aerosol through the innermost of three concentric tubes and injects it into the middle of the donut-shaped plasma. The sample elements are dissociated, atomized, and excited to a higher energy level. As the elements fall to a lower energy level, radiation characteristic of the elements present in the plasma is emitted. The light is directed through an entrance slit, dispersed by the diffraction grating, and projected on to the photomultiplier tube (PMT). The PMTs, located behind the exit slits, convert the light energy to an electrical current. This signal is then digitized and processed by the data system. Background correction is required for trace element determination.

ICP/MS: Sample digestates are aspirated and nebulized into a spray chamber. A stream of argon gas carries the sample aerosol through the innermost of three concentric tubes and injects it into the middle of the donut-shaped plasma. The sample elements are dissociated, atomized, and excited to a higher energy level. The ions that are produced are entrained in the plasma gas and introduced, by means of an interface, into a mass spectrometer. The ions are sorted according to their mass to charge ratios and quantified with a channel mass spectrometer.

Analytical Sequence

Refer to the appropriate analytical SOP for information on the analytical sequence.
ICP: SOP SA-ME-070: *Elements by ICP*
ICP/MS: SOP SA-ME-074: *Elements by ICP/MS*

Attachment 2:
Sample Collection, Preservation, and Holding Time Table

Matrix	Routine Sample Container	Routine Sample Size	Minimum Sample Size	Dechlorination Agent	Chemical Preservation	Thermal Preservation	Holding Time[2]
Water	250mL Plastic	50mL	25mL	Not Applicable	1:1 HNO_3 to pH<2	Not Applicable	Mercury: 28 days from collection Other Metals: 180 days from collection
Water (Lead & Copper Rule Only)	1L Plastic	50mL	25mL	Not Applicable	1:1 HNO_3 to pH<2	Not Applicable	180 days from collection
Water (Silica)	250mL Plastic	50mL	25mL	Not Applicable	Not Applicable	0-6 °C[1]	180 days from collection
Leachate	100mL or 250mL Plastic	50mL	25mL	Not Applicable	1:1 HNO_3 to pH<2	Not Applicable	Mercury: 28 days from leaching Other Metals: 180 days from leaching Note: SM3030C has a 72 hour HT.

[1]Samples are collected on ice and maintained at <6 °C with no frozen samples.
[2] Inclusive of digestion and analysis.

Note: If dissolved metals are requested, the sample must be filtered prior to the acid being added to the sample.

Attachment 3: QC Summary

QC Item	Frequency	Criteria	Corrective Action
Batch Definition	Digested together w/in 24-hr timeframe; not to exceed 20 field samples	Not Applicable	Not Applicable
Method Blank (MB)	One per batch	Refer to analytical SOP	Refer to analytical SOP
Lab Control Sample (LCS)	One per batch	Refer to analytical SOP	Refer to analytical SOP
Laboratory Control Sample Duplicate (LCSD)	One per batch, if insufficient sample provided for MS/MSD	LIMS MLG	Redigest and reanalyze batch
Low-Level Laboratory Control Sample (LLCS) – spiked at the RL	EPA 200.7 and EPA 200.8 DW: One per batch	Qualitatively identified	If the "regular" LCS meets criteria, initiate NCM and report data If the "regular" LCS does not meet criteria, redigest and reanalyze batch
Matrix Spike (MS)	EPA 200.7 and EPA 200.8: 10% of samples prepared; i.e., 2 separate matrix spikes per batch of twenty samples Other Methods: 5% of samples prepared; i.e., 1 matrix spike per batch of twenty samples	LIMS MLG	Flag and report data
Matrix Spike Duplicate (MSD) or Sample Duplicate (SD)	One per batch	LIMS MLG	Flag and report data
Initial Demonstration of Capability	Initially, per analyst, per analyte/method/matrix	Refer to SOP SA-QA-006	Refer to SOP SA-QA-006

QC Item	Frequency	Criteria	Corrective Action
(IDOC)	combination		Note: Unsupervised work must not begin until acceptable IDOC is obtained.
Continuing Demonstration of Capability (CDOC)	Annually, per analyst, per analyte/method combination	Refer to SOP SA-QA-006	Refer to SOP SA-QA-006
Reporting Limit Verification (RLV)	Upon method/instrument set-up, per analyte/method/matrix combination. Then quarterly thereafter (for DOD ELAP) or annually thereafter (for NELAC)	Refer to SOP SA-QA-007	Refer to SOP SA-QA-007
Method Detection Limit Study (MDL Study)	Upon method/instrument set-up, per analyte/method/matrix combination	Refer to SOP SA-QA-007	Refer to SOP SA-QA-007
MDL Verification (MDLV)	Upon method/instrument set-up, per analyte/method/matrix combination. Then quarterly thereafter (for DOD ELAP) or annually thereafter (for NELAC)	Refer to SOP SA-QA-007	Refer to SOP SA-QA-007

Attachment 4:
Instrument Maintenance and Troubleshooting

Instrument Labeling

Each instrument must be labeled with its name or ID (e.g., MSA, ICP-D, etc.). Additionally, non-operational instruments must be isolated from service or marked as being out of service. Each piece of equipment has an "Operational / Not Operational" sticker that is used for this purpose.

Maintenance Logbook

A maintenance logbook must be available and in use for each piece of equipment. Note: This requirement includes analytical instruments as well as any support equipment/meters used to generate a final result (e.g., pH meters, turbidimeters, and spectrophotometers). The maintenance logbook can be a hardcopy notebook or an electronic notebook.

All maintenance performed must be recorded in the logbook in real time. Items to be captured include:

- Type of maintenance performed (Note: This includes preventative/routine maintenance; non-routine maintenance; maintenance performed by an external vendor; updates to software versions; etc.)
- Name of person performing the maintenance
- Date the maintenance was performed
- Detailed explanation of the reason for any non-routine maintenance (i.e., the source of the problem)
- Resolution of the problem (i.e., the Return to Control)

Note: All maintenance performed by external service providers should be accompanied by work orders, etc., which need to be scanned and filed with the associated instrument records.

Note: Monthly review of maintenance logbooks is required.

Preventive Maintenance

Refer to the instrument manufacturer's guides for trouble-shooting items.

The temperature of the hot plate or digestion block must be monitored with each batch. If the temperature required for sample preparation cannot be maintained, the heating device must be removed from service and repaired or replaced.

Troubleshooting

Troubleshooting should be documented as outlined above. If possible, troubleshooting is best performed in a step-wise manner to systematically isolate instrument components. Refer to the instrument manufacturer's guides for specific information and strategies. Enlist assistance from technical and/or department management as needed.

Contingency Plan

Maintenance contracts are carried for most instrumentation and close contact is maintained with service personnel to ensure optimal instrument functioning. An extensive spare parts inventory is maintained for routine repairs, consisting of thermocouples, digestion tubes, and turbidimeter cells, and other common instrumentation components. Since instrumentation is standardized throughout the laboratory network, spare parts and components can be readily exchanged among the network.

In general, the laboratory has at least one backup unit for each critical unit. In the event of instrument failure, portions of the sample load may be diverted to duplicate instrumentation, the analytical technique switched to an alternate approved technique (such as manual colorimetric determination as opposed to automated colorimetric determination), or samples shipped to another properly certified or approved Eurofins TestAmerica location.

Attachment 5:
Turbidity Form

Turbidity for Metals Drinking Water Samples

Date:

Analyst Initials:

CCV LIMS Standard Number:

CCV True Value: NTU

CCV criteria: ±10% of True Value
CCB criteria: <1NTU
If sample turbidity is <1NTU and silver is not requested, the sample does not require digestion.

Job Number	Turbidity (NTU)	Comments
CCV		
CCB		
CCV		
CCB		

FME158:09.19.07:1

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Attachment 6:
Glassware Cleaning Procedures

GLASSWARE CLEANING PROCEDURES METALS DEPARTMENT
Graduated Cylinders 1. Scrub with hot, soapy H_2O and brush. 2. Rinse thoroughly with tap H2O. 3. Rinse with 10% HNO_3 4. Rinse thoroughly with DI H_2O.
Volumetric Flasks 1. Empty contents of flask 2. Squirt a small amount of cleaning detergent directly into volumetric flask 3. Fill flask 1/3 full with **HOT** H_2O 4. Replace top and shake flask. 5. Empty flask and rinse with **HOT** H_2O until no soap remains in flask. 6. Add approximately 10mL concentrated HNO_3 to 50mL, 100mL, and 250mL flasks ---- replace top and shake well. For 500mL or 1000mL flasks use 25mL and for 10mL flasks use 2 - 5mL of concentrated HNO_3. 7. Rinse 3 times with DI H_2O, filling flask 1/3 full and replacing top. Store until needed. *** NEVER PLACE VOLUMETRIC FLASKS OR TOPS IN SINK OR DISHPAN WITH OTHER DIRTY DISHES.**
***Dispose of all acid waste in accordance with the Eurofins TestAmerica Savannah Addendum to the Corporate Safety Manual.**

18.0 Revision History

Summary of Changes Revision 22A:
- Review completed and no changes implemented.

Summary of Changes from Revision 22:
- Added reference to SOP SA-GE-206: Turbidity as per SC DHEC certification renewal 07.10.2020.

Summary of Changes from Previous Revision:
- Minor editorial, grammatical, and/or formatting changes made.
- Updated SOP signatories to reflect current responsibilities and titles.
- Replaced all references to TALS to LIMS.
- Updated reference to Turbidity standard to the 1.0NTU standard in sections 7.3.1 and 10.1.2.

SOIL PREPARATION PROCEDURES FOR ICP AND ICP/MS

(Methods: 3050B and DI Leach)

Approvals (Signature/Date):

05/20/2021

Adriana Geiger — Date
Department Manager

05/20/2021

Kimberly Chamberlain — Date
Quality Assurance Manager / Technical Director / Environmental Health & Safety Coordinator

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1.0 Scope and Application

This SOP gives the procedures for the digestion soil samples for the determination of total metals prior to analysis by ICP (SOP SA-ME-070: *Elements by ICP*) or ICP/MS (SOP SA-ME-074: *Elements by ICP/MS)*.

Note: The routine matrix for this procedure is soil; however, this procedure may be adapted to accommodate other matrices as outlined in Section 16.1.

A complete target analyte list, the reporting limits (RL), the method detection limits (MDL), and the accuracy and precision criteria associated with this procedure are provided in the LIMS Method Limit Groups (MLGs).

This SOP was written by and for Eurofins TestAmerica's Savannah laboratory.

2.0 Summary of Method

A known weight (approximately 1g) of the well-mixed sample is transferred to a suitable digestion vessel. The sample is digested with aliquots of nitric acid and hydrogen peroxide to break down the organics present in the sample. After the sample has been digested, as evidenced by a clear, pale yellow digestate, HCl is added to give an approximate acid concentration of 10% HCl and 5% HNO_3. Then the sample digest is diluted to 100mL with reagent water.

A smaller weight of sample may be digested and the sample brought to a final volume that is proportional to the 1g sample to 100mL final volume ratio. For example, if 0.50g is digested, the final volume of the digestate must be 50mL to achieve the same reporting limits.

Note: For silica samples for EPA 6010C or EPA 6010D, solid samples are extracted with reagent water using a DI Leach procedure and then filtered. The filtered solid extracts are analyzed by ICP.

This SOP is based on the following methods: SW-846 Method 3050B. Silica soil preparation guidance is taken from ASTM Method D3987-85.

3.0 Definitions

Refer to the Glossary Section of the *Quality Assurance Manual* (QAM) for a complete listing of applicable definitions and acronyms.

4.0 Interferences

4.1 Procedural Interferences

4.1.1 Interferences may be caused by contaminants in solvents, reagents, glassware, and other sample processing apparatus and can make identification and/or quantification of the target analytes difficult.

4.1.2 All sample collection containers are single-use disposable containers which limits the potential for contamination. All non-disposable labware must be scrupulously cleaned in accordance with the posted Labware Cleaning Instructions to ensure it is free from contaminants and does not contribute artifacts.

4.1.3 High purity reagents and solvents are used to help minimize interference problems. Hydrochloric acid, hydrogen peroxide, and nitric acid must be verified prior to use in accordance with the Eurofins TestAmerica Solvent Lot Testing Program.

4.1.4 Instrument and/or method blanks are routinely used to demonstrate all reagents and apparatus are free from interferences under the conditions of the analysis.

4.2 Matrix Interferences

4.2.1 Matrix interferences may be caused by contaminants that are co-extracted from the sample matrix.

4.2.2 Interfering contamination may occur when a sample containing low concentrations of analytes is analyzed immediately following a sample containing relatively high concentrations of analytes. As such, samples known to be clean should be analyzed first. To prevent carryover into subsequent samples, analysis of reagent blanks may be needed after the analysis of a sample containing high concentrations of analytes.

5.0 Safety

Employees must abide by the policies and procedures in the Eurofins TestAmerica Environmental Health and Safety Manual (EHSM), the Eurofins TestAmerica Savannah Addendum to the EHSM, and this document.

This procedure may involve hazardous materials, operations, and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user to follow appropriate safety, waste disposal, and health practices under the assumption that all samples and reagents are potentially hazardous.

The analyst must protect himself/herself from exposure to the sample matrix. Many of the samples that are tested may contain hazardous chemical compounds or biological organisms. The analyst must, at a minimum, wear protective clothing (lab coat), eye protection (safety glasses or face shield), disposable nitrile or latex gloves, and closed-toe, nonabsorbent shoes when handling samples.

5.1 Specific Safety Concerns or Requirements

Nitric and hydrochloric acids are extremely hazardous as oxidizers, corrosives, poisons, and are reactive. Inhalation of the vapors can cause coughing, choking, irritation of the nose, throat, and respiratory tract, breathing difficulties, and lead to pneumonia and pulmonary edema. Contact with the skin can cause severe burns, redness, and pain. Nitric acid can cause deep ulcers and staining of the skin to a yellow or yellow-brown color. These acid vapors are irritating and can cause damage to the eyes. Contact with the eyes can cause permanent damage. Procedures that utilize the addition of concentrated acids must be performed under a fully functioning fume hood.

Samples that contain high concentrations of carbonates or organic matter, or samples that are at elevated pH can react violently when acids are added. Acids must be added to samples under a hood to avoid splash/splatter hazards and/or possibly toxic vapors that will be given off when the samples are acidified.

Hydrogen peroxide is a strong oxidizer that can cause a fire when it comes into contact with materials. Contact with the skin or eyes can cause irritation and burns.

5.2 Primary Materials Used

The following is a list of the materials used in this procedure, which have a serious or significant hazard rating, and a summary of the primary hazards listed in their MSDS/SDS.

Note: This list does not include all materials used in the procedure. A complete list of materials used in this procedure can be found in the Reagents and Standards Section and the Equipment and Supplies Section of this SOP

Employees must review the information in the MSDS/SDS for each material before using it for the first time or when there are major changes to the MSDS/SDS. Electronic copies of MSDS/SDS can be found using the “SDS” link on the Oasis homepage, and on the EH&S webpage on Oasis.

Material	Hazards	Exposure Limit[1]	Signs and Symptoms of Exposure
Hydrochloric Acid[2]	Corrosive Poison	5ppm Ceiling	Inhalation of vapors can cause coughing, choking, inflammation of the nose, throat, and upper respiratory tract, and in severe cases, pulmonary edema, circulatory failure, and death. Can cause redness, pain, and severe skin burns. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns or permanent eye damage.
Nitric Acid[2]	Corrosive Oxidizer Poison	2ppm TWA 4ppm STEL	Nitric acid is extremely hazardous; it is corrosive, reactive, an oxidizer, and a poison. Inhalation of vapors can cause breathing difficulties and lead to pneumonia and pulmonary edema, which may be fatal. Other symptoms may include coughing, choking, and irritation of the nose, throat, and respiratory tract. Can cause redness, pain, and severe skin burns. Concentrated solutions cause deep ulcers and stain skin a yellow or yellow-brown color. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
Hydrogen Peroxide	Corrosive Oxidizer	1ppm TWA	Danger! Strong oxidizer. Contact with other material may cause a fire. Corrosive. Light sensitive. May be harmful if swallowed. May cause central nervous system effects. Eye contact may result in permanent eye damage. May cause severe respiratory tract irritation with possible burns. Causes eye and skin irritation and possible burns. May cause severe digestive tract irritation with possible burns.
[1]Exposure limit refers to the OSHA regulatory exposure limit.			
[2]Always add acid to water to prevent violent reactions.			

6.0 Equipment and Supplies

6.1 Equipment and Instrumentation

Top-loading Balance – Verify in accordance with SOP SA-AN-100: *Laboratory Support Equipment (Verification and Use)*

Thermometers – Verify in accordance with SOP SA-AN-100: *Laboratory Support Equipment (Verification and Use)*

Digestion Block – capable of maintaining a sample temperature of 95±5°C – The temperature of the digestion block must be monitored and recorded for each batch. The temperature is measured using an external thermometer placed in a digestion vessel containing reagent water.

Agitation/Leaching Equipment – capable of holding the sample containers and rotating around a central axis at 23-35rpm.

6.2 Lab Supplies

0.45um Syringe Filters

6.3 Volumetric Labware

Volumetric Containers – various sizes; Class A, where applicable. Verify in accordance with SOP SA-AN-100: *Laboratory Support Equipment (Verification and Use)*. Refer to Attachment 5 for Labware Cleaning Procedures.

Volumetric Labware	Volume	Type (Quantitative / Qualitative)	Use	Verification Frequency	Laboratory Verification Criteria
Eppendorf-Style Pipettes	Various	QUANTITATIVE	Standard Preparation and Spiking	Initially, prior to first use, then quarterly thereafter (Daily for DOD)	Accuracy = 2% Precision = 1%
Digestion Tubes	50-100mL	QUANTITAIVE	Final Sample Volumes	None (if purchased w/ COA attesting Class A criteria)	None (if purchased w/ COA attesting Class A criteria)
Pump-Style Pipettes	Various	Qualitative	To deliver acids	Initially, upon 1st use	Accuracy = 10% Precision = 10%

6.4 Sample Collection Containers

All sample collection containers are single-use disposable containers which limits the potential for contamination.

The routine sample collection containers supplied by the laboratory are:

8oz plastic soil jar – purchased with Certificate of Analysis attesting to purity.

7.0 Reagents and Standards

7.1 Expiration Dates

Expiration dates (time from initial use or receipt to final use) for standard and reagent materials must be set according to the guidance in this SOP. Note: These are maximum expiration dates and are not to be considered an absolute guarantee of standard or reagent quality. Sound judgment must be used when deciding whether to use a standard or reagent. If there is doubt about the quality of a standard or reagent material, a new material must be obtained or the standard or reagent material verified. Data quality must not be compromised to extend a standard's life.

The expiration date of any standard or reagent must not exceed the expiration date of the standard or reagent that was used to prepare it.

7.2 Reagents

Reagents must be prepared and documented in accordance with SOP SA-AN-041: *Reagent and Standard Materials Procedures.*

Hydrochloric acid, hydrogen peroxide, and nitric acid must be verified prior to use in accordance with the Eurofins TestAmerica Solvent Lot Testing Program.

7.2.1 Blank Matrix – Teflon chips or equivalent. Used for the preparation of soil QC samples.
LIMS Name: P_TEFLONCH_xxxxx
Storage: Room Temperature
Expiration: Unopened: Manufacturer's expiration date; Opened: 5 years from date opened

7.2.2 Laboratory Reagent Water – ASTM Type I; the conductivity must be checked daily in accordance with SOP SA-AN-100: *Laboratory Support Equipment (Verification and Use)*.

7.2.3 Nitric acid (HNO_3) – reagent grade.(Macron Chemicals)
LIMS Name: ME_HNO3_XXXX
Storage: room temperature in the acid cabinet. Store in a cool, dry, ventilated storage area with acid resistant floors and good drainage. Store away from sunlight, heat, water, and incompatible materials. Stable under ordinary conditions of use and storage.
Expiration:
Unopened: Manufacturer's expiration date or 5 years, whichever is sooner
Opened: Manufacturer's expiration date or 5 years, whichever is sooner

7.2.5 Hydrochloric acid (HCl) – reagent grade. (JT Baker)
LIMS Name: ME_HCL_XXXX
Storage: room temperature in the acid cabinet. Store in a cool, dry, ventilated storage area with acid resistant floors and good drainage. Store away from sunlight, heat, water, and incompatible materials. Stable under ordinary conditions of use and storage.
Expiration:
Unopened: Manufacturer's expiration date or 5 years, whichever is sooner
Opened: Manufacturer's expiration date or 5 years, whichever is sooner

7.2.7 Hydrogen peroxide (H_2O_2), 30% – reagent grade.
LIMS Name: ME_PEROX_XXXX
Storage: refrigerated between 0°C and 6°C. Separate from incompatibles.
Expiration:
Unopened: Manufacturer's expiration date
Opened: Manufacturer's expiration date

7.3 Standards

Standards must be prepared and documented in accordance with SOP SA-AN-041: *Reagent and Standard Materials Procedures.* Certificates of analysis or purity must be received with all purchased standards, and scanned and attached in LIMS.

7.3.1 Stock Standards – purchased from CPI, Inorganic Ventures, or equivalent vendor, at the concentrations listed below.

Aluminum – 10000mg/L; LIMS Reagent Name: Alspx10000
Antimony – 1000mg/L; LIMS Reagent Name: Sbcpi1000
Arsenic – 1000mg/L; LIMS Reagent Name: Ascpi1000
Barium – 1000mg/L; LIMS Reagent Name: Bacpi1000
Beryllium – 1000mg/L; LIMS Reagent Name: Becpi1000
Boron – 1000mg/L; LIMS Reagent Name: Bcpi1000
Cadmium – 1000mg/L; LIMS Reagent Name: Cdcpi1000
Calcium – 10000mg/L; LIMS Reagent Name: Caspx10000
Chromium – 1000mg/L; LIMS Reagent Name: Crcpi1000
Cobalt – 1000mg/L; LIMS Reagent Name: Cocpi1000
Copper – 1000mg/L; LIMS Reagent Name: Cucpi1000
Iron – 10000mg/L; LIMS Reagent Name: Fecpi10000
Lead – 1000mg/L; LIMS Reagent Name: Pbcpi1000
Magnesium – 10000mg/L; LIMS Reagent Name: Mgspx10000
Manganese – 1000mg/L; LIMS Reagent Name: Pbcpi1000
Mercury – 1000mg/L; LIMS Reagent Name: Hgspex1000
Molybdenum – 1000mg/L; LIMS Reagent Name: Mocpi1000
Nickel – 1000mg/L; LIMS Reagent Name: Nicpi1000
Potassium – 10000mg/L; LIMS Reagent Name: Kcpi10000
Selenium – 1000mg/L; LIMS Reagent Name: Secpi1000
Silver – 1000mg/L; LIMS Reagent Name: Agcpi1000
Sodium – 10000mg/L; LIMS Reagent Name: Nacpi10000

Strontium – 1000mg/L; LIMS Reagent Name: SrABS1000
Thallium – 1000mg/L; LIMS Reagent Name: Tlcpi1000
Tin – 1000mg/L; LIMS Reagent Name: Sncpi1000
Titanium – 1000mg/L; LIMS Reagent Name: Ticpi1000
Vanadium – 1000mg/L; LIMS Reagent Name: Vcpi1000
Zinc – 1000mg/L; LIMS Reagent Name: Zncpi1000

Storage: room temperature. Store away from sunlight, heat, water, and incompatible materials.
Expiration: opened and unopened containers are given the manufacturer's expiration date

7.3.2 Silica/Silicon Standards

Silicon (Si) stock solutions are usually purchased for this procedure. The following conversion is used to adjust any volumes or concentrations appropriately:

$$Si = \frac{SiO_2}{2.14}$$

7.3.2.1 Stock SiO_2 Standard, 10000mg/L Si / 21400mg/L SiO_2 – purchased from CPI.
LIMS Name: Sicpi10000_xxxxx
Storage: Store at room temperature in a cool, dry, ventilated storage area with acid resistant floors and good drainage. Store away from sunlight, heat, water, and incompatible materials.
Expiration: This standard must be used by the manufacturer's expiration date.

Silicon (Si) stock solutions are usually purchased for this procedure. The following conversion is used to adjust any volumes or concentrations appropriately:

$$Si = \frac{SiO_2}{2.14}$$

7.3.2.2 Intermediate SiO_2 Standard, 467mg/L Si / 1000mg/L SiO_2 – Add 20mL to 30mL of reagent water to a clean, plastic 100mL volumetric flask. Add the volume of the Stock SiO_2 Standard given in the table below to the volumetric flask. Dilute to volume with reagent water. Store the standard at room temperature. This standard must be used by its parent standard's expiration date or within 6 months of preparation, whichever comes first.

Element	Parent Concentration (mg/L)	Volume Added (mL)	Final Volume (mL)	Final Concentration (mg/L)
Silica (SiO_2) [Silicon (Si)]	21400 [10000]	4.67	100	1000 [467]

LIMS Name: P_SiO2_INT_xxxxx
Storage: Store in a cool, dry, ventilated storage area with acid resistant floors and good drainage. Store away from sunlight, heat, water, and incompatible materials.
Expiration: Prepare this solution every 180 days or sooner as needed or required. The expiration date must not exceed the expiration date of any of the components.

7.3.5 Spiking Solution 1 – Add approximately 50mL reagent water to a clean 100-mL volumetric flask. Add 2mL of concentrated nitric acid and 0.5mL of concentrated hydrochloric acid to the volumetric flask. The standard will have an acid concentration of 2% HNO_3 and 0.5% HCl when diluted to volume.

Add the volumes of the stock standards given in the following table to the volumetric flask:

Element	Parent Concentration (mg/L)	Volume Added (mL)	Final Volume (mL)	Final Concentration (mg/L)
Aluminum (Al)	10000	5	100	500
Antimony (Sb)	1000	5		5
Arsenic (As)	1000	1		10
Barium (Ba)	1000	1		10
Beryllium (Be)	1000	0.5		5
Boron (B)	1000	2		20
Cadmium (Cd)	1000	0.5		5
Calcium (Ca)	10000	5		500
Chromium (Cr)	1000	1		10
Cobalt (Co)	1000	0.5		5
Copper (Cu)	1000	1		10
Iron (Fe)	10000	5		500
Lead (Pb)	1000	5.0		50
Magnesium (Mg)	10000	5		500
Manganese (Mn)	1000	5		50
Molybdenum (Mo)	1000	1		10
Nickel (Ni)	1000	1		10
Potassium (K)	10000	5		500
Selenium (Se)	1000	1		10
Sodium (Na)	10000	5		500
Strontium (Sr)	1000	1		10
Thallium (Tl)	1000	0.4		4.0
Tin (Sn)	1000	2		20
Titanium (Ti)	1000	1		10
Vanadium (V)	1000	1		10
Zinc (Zn)	1000	1		10

Dilute to volume with reagent water.

LIMS Name: MS_LCS1_wk_xxxxx
Storage: room temperature Expiration: 6 months of preparation or sooner based on the manufacturer's expiration date.

7.3.6 Spiking Solution 2 – Add approximately 50mL reagent water to a clean 100mL volumetric flask. Add 10mL of concentrated hydrochloric acid to the volumetric flask. The standard will have an acid concentration of 10% HCl when diluted to volume.

Add the volumes of the stock standards given in the following table to the volumetric flask:

Element	Parent Concentration (mg/L)	Volume Added (mL)	Final Volume (mL)	Final Concentration (mg/L)
Mercury (Hg)	1000	0.05	100	0.5

Dilute to volume with reagent water.

LIMS Name: MS_lcs2_wk_xxxxx
Storage: room temperature
Expiration: 28 days of preparation or sooner based on the manufacturer's expiration date.

7.3.7 Spiking Solution 3 – Add approximately 50mL reagent water to a clean 100mL volumetric flask. Add 10mL of concentrated hydrochloric acid to the volumetric flask. The standard will have an acid concentration of 10% HCl when diluted to volume.

Add the volumes of the stock standards given in the following table to the volumetric flask:

Element	Parent Concentration (mg/L)	Volume Added (mL)	Final Volume (mL)	Final Concentration (mg/L)
Silver (Ag)	1000	0.5	100	5

Dilute to volume with reagent water.

LIMS Name: MS_Ag_LCS_SPKxxxxx
Storage: room temperature
Expiration: 6 months of preparation or sooner based on the manufacturer's expiration date.

8.0 Sample Collection, Preservation, Shipment, and Storage

8.1 Soil Samples

Soil samples are routinely collected in 8oz plastic soil containers.

Samples must be iced at the time of collection and maintained at 0-6°C (less than 6°C but not frozen) until the time of digestion/leaching. Samples for mercury must be digested/leached and analyzed within 28 days of collection. All other samples must be digested and analyzed within 6 months of collection. Digestates may be stored at room temperature until the time of analysis.

9.0 Quality Control

SOP SA-QA-017: *Evaluation of Batch QC Data* and the SOP Summary in Attachment 3 provide requirements for evaluating QC data.

9.1 Batch QC

A digestion batch consists of up to 20 environmental samples and the associated QC items. The default QC items performed for each digestion batch are: a method blank, a laboratory control sample (LCS), a matrix spike (MS), and a matrix spike duplicate (MSD) or sample duplicate (SD).

The routine container supplied for this method 8oz (soil) container. 1g is required for digestion. It is unlikely that insufficient sample would be provided to meet the batch QC frequency. If insufficient sample is provided, reduced sample initial weights may be used to achieve the required batch matrix spike frequency; however, the minimum extraction weight to be used for the matrix spike samples is 0.5g.

Note: Final volumes and spike amounts must be adjusted to compensate for these reduced initial volumes.

Note: Unless the client has specified which sample to use as the matrix spike, the matrix spike must be selected at random from the samples in the batch. Field QC (e.g., equipment blanks, trip blanks, and field blanks) must not be used for MS/MSD unless specifically requested to do so by the client or unless there is insufficient sample available for performing MS/MSD using an actual field sample.

If there is insufficient sample to perform the required matrix spike(s) and/or sample duplicates, the LCS must be prepared in duplicate (i.e., LCS/LCSD). An NCM must be initiated on all affected samples to denote this situation. Insufficient sample is defined as receiving less than a total of 2g.

Batch QC must meet the criteria given in Attachment 3 of this SOP.

9.2 Instrument QC

The instrument QC requirements for the analytical procedures associated with this digestion procedure are given in SOP SA-ME-070: *Elements by ICP* or SOP SA-ME-074: *Elements by ICP/MS.*

9.3 Corrective Action for Out-of-Control Data

When the quality control parameters do not meet the criteria set forth in this SOP, corrective action must be taken in accordance with SOP SA-QA-005: *Preventive and Corrective Action Procedures* the QC Summary Table in Attachment 3. SOP SA-QA-005 provides contingencies for out-of-control data and gives guidance for exceptionally permitting departures from approved policies and procedures. Nonconformance Memos must be initiated to document all instances where QC criteria are not met and all departures from approved policies and procedures.

10.0 Procedure

10.1 Sample Preparation

10.1.1 EPA 3050B Sample Preparation

10.1.1.1 Remove the samples from the refrigerator and allow them to come to room temperature.

10.1.1.2 Soil samples must be homogenized prior to preparation in accordance with SOP SA-QA-015: *Homogenization, Compositing, and Segregation of Samples*.

10.1.1.3 Weigh 1.0-1.2g (wet weight) of each homogeneous sample into a 100mL digestion vessel. The lab may weigh a larger aliquot equal to 1.0g of sample on a dry weight basis, if required.

Note: A smaller weight of sample may be digested and the sample brought to a final volume that is proportional to the 1g sample to 100mL final volume ratio. For example, if 0.50g is digested, the final volume of the digestate must be 50mL to achieve the same reporting limits; if 0.1g is digested, the final volume of the digestate must be 10mL. If the sample weight to final volume ratio is less than 1:100, the reporting limits will be higher than those listed in the LIMS MLGs. Adjust the volumes of spikes and reagents proportionately to compensate for the sample weight/final volume. For example, if 0.5g is digested, use ½ of the spike and reagent volumes listed above.

10.1.1.4 Add 5mL of reagent water, 5mL of concentrated HNO_3, and 2.5 mL of HCL to each digestion vessel and swirl the vessel to mix the contents. Perform this step under a fume hood.

10.1.1.3.1 Place the digestion vessels on the digestion block. The water in the digestion block must be 95°C +/- 5°C. Carefully heat the vessel until a gentle reflux is achieved. The sample is not heated to boiling; that is, bubbles are not formed in the liquid in the bottom of the digestion vessel. The sample/acid solution is refluxing when the liquid evaporates and drops of liquid condense on the sides of the digestion vessel and fall back into the digestion vessel. Do not allow the samples to boil. Reflux for 10-15 minutes.

NOTE: To verify temperature of the hotblock a digestion tube filled with DI water must be placed at random locations across each block used for digestion. This position must be changed daily. A single thermometer is transferred from block to block and a minimum of 10 minutes allowed for the thermometer to equilibrate before taking the reading. This temperature reading, uncorrected and corrected, is then recorded in the batch information.

10.1.1.6 Remove the digestion vessels from the digestion block, and allow them to cool to room temperature. Add 5mL of concentrated HNO_3 to each sample. Return the digestion vessels to the digestion block. Carefully heat the digestion vessels until a gentle reflux is achieved. Reflux the samples for at least 30 minutes. Do not allow the samples to boil.

If brown fumes are given off during reflux, remove the vessels from the digestion block, allow to cool, add a second 5mL portion of HNO_3, and reflux for at least 30 more minutes. Repeat this step until no brown fumes are given off.

10.1.1.7 Evaporate the sample digestate to approximately 5mL. Do not allow the bottom of the digestion vessels to go dry during the evaporation. Remove the samples from the digestion block, and allow the samples to cool to room temperature before continuing onto the next step.

Note: If the sample is still warm when the 30% hydrogen peroxide (H_2O_2) is added in the next step, the sample may "boil over" and the entire process must be started over.

10.1.1.8 Add 2mL of reagent water to each digestion vessel. Slowly and carefully add 3mL of 30% H_2O_2 to each digestion vessel. It is very important to add the hydrogen peroxide slowly to prevent loss of sample due to vigorous effervescence. Return the digestion vessels to the digestion block, and heat until the effervescence subsides. Cool the digestion vessels after the effervescence subsides.

10.1.1.9 Continue to add 30% H_2O_2 in 1-3mL aliquots to the sample digestate until the effervescence is minimal or until the general appearance of the digestate is unchanged. Warm the sample digestate after each addition of H_2O_2 on the digestion block.

Note: Do not add more than 10mL of hydrogen peroxide to each sample.

10.1.1.10 After the last addition of peroxide, return the samples to the digestion block and reduce the volume of the digestate to approximately 5mL without boiling and without allowing the bottom of the digestion vessel to go dry. Remove samples, allow to cool, and add 10mL of concentrated HCl to each sample digestate. Return the digestion vessels to the digestion block, and reflux the sample digestates for 10-15 minutes.

10.1.1.11 Remove samples from the digestion block, allow to cool, and wash down the inside of the digestion vessels with reagent water. Dilute the sample digestate to 100mL with reagent water.

10.1.1.4 Silica (DI Leach) Sample Preparation

Solid and waste samples for silica are extracted by performing a DI leaching procedure. Note: Any QC samples which need the DI leach procedure (i.e. LCS, MS/MSD, IDOCs/CDOCs, MDL studies, etc.) need to be appropriately spiked prior to the leaching procedure.

10.1.1.5 Remove the samples from the refrigerator and allow them to come to room temperature.

10.1.1.6 Soil samples must be homogenized prior to preparation in accordance with SOP SA-QA-015: *Homogenization, Compositing, and Segregation of Samples*.

10.1.1.7 To a 125mL plastic container weigh a 4.5-5.5g portion of homogenized sample. Note: Different initial weights may be used as long as the extraction fluid to sample ratio remain the same.

10.1.1.8 Add 100mL of reagent water to each sample container (or a volume equal to 20 times the weight of the samples used).

10.1.1.9 Agitate (leach) continuously for 18 ± 2 hours at 23-35rpm.

10.1.1.10 After the extraction period, allow the sample to settle and filter a portion for analysis through a 0.45um filter. The sample may be allowed to settle, centrifuged, decanted, or filtered through coarse filter paper before the final filtration to remove the solid phase. The method blank and laboratory control sample must be treated the same as the samples (i.e., same volumes and same number of filter pads) so that an adequate background, contamination, and recovery checks can be made.

10.2 QC Sample Preparation

10.2.1.1 EPA Method 3050B QC Sample Preparation

10.2.1.2 Method Blank – Weigh a 1.0-1.2g aliquot of blank matrix into a labeled digestion vessel and prepare as listed in Section 10.1.1.

10.2.1.3 Laboratory Control Sample – Weigh a 1.0-1.2g aliquot of blank matrix into a labeled digestion vessel, add 1.0mL of each spiking solution (Spike I, Spike 2 and Spike 3) and prepare as listed in Section 10.1.1.

10.2.1.4 Matrix Spike / Matrix Spike Duplicate – Weigh out 2 separate 1.0 -1.2-g aliquots of the sample chosen for the matrix spike into a labeled digestion vessel. Add 1.0mL of each spiking solution (Spike I, Spike 2, and Spike 3) and prepare as listed in Section 10.1.1.

10.2.2 Silica (DI Leach) QC Sample Preparation

10.2.2.1 Method Blank – Weigh a 4.5-5.5g aliquot of blank matrix into a labeled 125mL plastic container and continue as outlined in Section 10.1.2.4 through Section 10.1.2.6.

10.2.2.2 Laboratory Control Sample – Weigh a 4.5-5.5g aliquot of blank matrix into a labeled 125mL plastic container, add 1.0mL of the SiO_2 Intermediate Standard and continue as outlined in Section 10.1.2.4 through Section 10.1.2.6.

10.2.2.3 Matrix Spike / Matrix Spike Duplicate – Weigh out 2 separate 4.5-5.5g aliquots of the sample chosen for the matrix spike into a labeled 125mL plastic container. Add 1.0mL of the SiO_2 Intermediate Standard and continue as outlined in Section 10.1.2.4 through Section 10.1.2.6.

10.3 Analysis

The analytical procedures associated with this digestion procedure are given in SOP SA-ME-070: *Elements by ICP* or SOP SA-ME-074: *Elements by ICP/MS.*

11.0 Calculations / Data Reduction

11.1 Data Reduction

Data must be evaluated in accordance with SOP SA-QA-002: *Data Generation and Review*.

The data reduction procedures associated with this digestion procedure are given in SOP SA-ME-070: *Elements by ICP* or SOP SA-ME-074: *Elements by ICP/MS.*

11.1.1 Historical Data

Many of the laboratory's clients submit samples for repeat monitoring purposes. Prior to analysis, verify LIMS Worksheet Notes and/or use the Historical Data Tracker feature to determine if historical data is available for review.

11.1.2 Chemical Relationships

When available, the following chemical relationships must be evaluated for each sample. If these relationships are not met the Department Manager must be contacted immediately.

- Total Results are ≥ Dissolved results (e.g. metals)

11.2 Calculations

The calculations for the determination of metals by ICP and ICP/MS are given in the associated analytical SOPs (SOP SA-ME-070 or SOP SA-ME-074).

12.0 Method Performance

12.1 Reporting Limit Verification (RLV)

At a minimum, RLVs must be performed initially upon method set-up in accordance with SOP SA-QA-007: *Determination and Verification of Detection and Reporting Limits*.

For analytes and methods certified by DOD ELAP, RLVs must also be performed quarterly thereafter. For analytes and methods certified by NELAC, RLVs must also be performed annually thereafter. Exceptions may be made for project-specific non-routine analytes.

12.2 Method Detection Limit (MDL) Study

The MDL is the lowest concentration that can be detected for a given analytical method and sample matrix with 99% confidence that the analyte is present. MDLs reflect a calculated (statistical) value determined under ideal laboratory conditions in a clean matrix and may not be achievable in all environmental matrices. The current MDLs associated with this procedure are given in the Method Limit Group (MLG) in LIMS.

At a minimum, MDL Studies must be performed initially upon method set-up in accordance with SOP SA-QA-007: *Determination and Verification of Detection and Reporting Limits*.

12.3 Method Detection Limit Verification (MDLV)

At a minimum, MDLVs must be performed initially upon method set-up in accordance with SOP SA-QA-007: *Determination and Verification of Detection and Reporting Limits*.

For analytes and methods certified by DOD ELAP, MDLVs must also be performed quarterly thereafter. For analytes and methods certified by NELAC, MDLVs must also be performed annually thereafter.

Note: MDLVs are not required for non-routine analytes provided results are not reported below the RL (i.e., MDL equals RL in LIMS).

12.4 QC Limit Generation, Control Charting, and Trend Analysis

The control limits for the batch QC items (LCS and MS/MSD) for this procedure are specified in the reference method and cannot be broadened; therefore, the laboratory defaults to the method-defined limits and does not utilize in-house or laboratory-derived limits for the evaluation of batch QC items.

Although the laboratory must default to the method-defined QC limits, control charting is a useful tool and is performed to assess analyte recoveries over time to evaluate trends. Control charting must be performed periodically (at a minimum annually) in accordance with SOP SA-QA-017: *Evaluation of Batch QC Data.*

12.5 Demonstrations of Capability

Initial and continuing demonstration of capability must be performed in accordance with SOP SA-QA-006*: Training Procedures.*

Prior to performing this procedure unsupervised, each new analyst who performs this analysis must demonstrate proficiency per method/analyte combination by successful completion of an initial demonstration of capability. The IDOC is performed by the analysis of 4 consecutive laboratory QC check samples that meet the method criteria for accuracy and precision. The IDOC must be documented and routed to the QA Department for filing.

Annual continuing demonstrations of capability (CDOCs) are also required per analyst per method/analyte combination. The CDOC requirement may be met by the consecutive analysis of four LCS all in the same batch, by the analysis of four LCS analyzed in four consecutive batches (in different batches on different days), via acceptable results on a PT study, or analysis of client samples with statistically indistinguishable results when compared to another certified analyst. The CDOC must be documented and routed to the QA Department for filing.

12.6 Training Requirements

All training must be performed and documented in accordance with SOP SA-QA-006: *Training Procedures*.

Note: The SOPs listed in the Reference/Cross-Reference Section are applicable to this procedure. All employees performing this procedure must also be trained on these SOPs, and/or have a general understanding of these procedures, as applicable.

13.0 Pollution Control

It is Eurofins TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (e.g., examining recycling options, ordering chemicals based on quantity needed, preparing reagents based on anticipated usage and reagent stability, etc.). Employees must abide by the policies in Section 13 of the Environmental Health and Safety Manual.

This procedure has been evaluated for opportunities to minimize the waste generated. Where reasonably feasible, pollution control procedures have been incorporated.

14.0 Waste Management

Waste management practices must be conducted consistent with all applicable federal, state, and local rules and regulations. All waste (i.e., excess reagents, samples, and method process wastes) must be disposed of in accordance with Section 9 of the Eurofins TestAmerica Savannah Addendum to the EHSM. Waste description rules and land disposal restrictions must be followed.

14.1 Waste Streams Produced by the Method

The following waste streams are produced when this method is carried out:

- Excess soil and solid samples – Transfer RCRA hazardous and non-hazardous samples to waste department for disposal via yardbox. The samples must be transferred in LIMs to the designated yardbox. PCB or Foreign Soil samples must be disposed per waste stream with waste coordinator.

- Samples within the prep lab must be placed in boxes to prevent possible breakage, and may be stacked and kept for a short time.

- Acidic sample digestions – Neutralize before disposal into drain/sewer system.

- Excess oil samples – Transfer to waste department for storage/disposal.

Neutralize the standard by passing with sodium bicarbonate before transferring the waste to the RCRA aqueous hazardous waste drum.

All other metals standards may be disposed, using copious amounts of water, down a sink that flows to the neutralization pit.

15.0 References / Cross-References

- SOP SA-AN-041: *Reagent and Standard Materials Procedures*
- SOP SA-AN-100: *Laboratory Support Equipment (Verification and Use)*
- SOP SA-QA-002: *Data Generation and Review*
- SOP SA-QA-005: *Preventive and Corrective Action Procedures*
- SOP SA-QA-006*: Training Procedures*
- SOP SA-QA-007: *Determination and Verification of Detection and Reporting Limits (RLs, MDLs, and IDLs)*
- SOP SA-QA-015: *Homogenization, Compositing, and Segregation of Samples*
- SOP SA-QA-016: *Evaluation of Calibration Curves*
- SOP SA-QA-017: *Evaluation of Batch QC Data*
- Eurofins TestAmerica Savannah Quality Assurance Manual
- Eurofins TestAmerica Environmental Health and Safety Manual
- Eurofins TestAmerica Savannah Addendum to the Environmental Health and Safety Manual
- *Test Methods for Evaluating Solid Waste*, Third Edition, SW-846; EPA Office of Solid Waste and Emergency Response: Washington, DC. (including Updates III and IV)
    - Method 3050B Revision 2: Acid Digestion of Sediments, Sludges, and Soils
- ASTM Method D3987-85: *Shake Extraction for Solid Waste with Water*

16.0 Method Modifications/Clarifications

16.1 Incorporation of Other Matrices

16.1.1 This procedure may be modified to analyze other matrices (e.g., wipe, waste, tissue, and filter samples) based on the needs of the client. This will need to be arranged by the Project Manager at the initiation of the project.

16.1.2 Wipe, waste, filter, and tissue matrices are non-routine, and the laboratory is not currently NELAC certified for these matrices. The laboratory uses its routine soil RLs (converted for initial and final volumes, etc.) and soil QC limits to evaluate wipe, waste, filter, and tissue samples. Soil DOCs can be used to satisfy analyst demonstrations of capability for these types of non-routine matrices. Teflon chips, Ottawa sand, or equivalent is used as the blank matrix for these non-routine matrices unless specifically requested otherwise by the project.

16.1.3 Waste samples may be collected in 8oz plastic soil jars. However, it should be noted that an alternate container may be required as some organic wastes (oils) may not be conducive to plastic. Tissue samples may be collected in 8oz plastic soil jars or plastic bags. Wipes and filters may be collected in a variety of different containers.

16.1.4 Wipe, waste, and filter samples must be iced at the time of collection and maintained at 0-6°C (less than 6°C but not frozen) until the time of digestion/leaching. Samples for mercury must be digested/leached and analyzed within 28 days of collection. All other samples must be digested and analyzed within 6 months of collection. Digestates may be stored at room temperature until the time of analysis.

16.1.5 Tissue samples must be iced at the time of collection and maintained at -10°C to -20°C for up to 6 months. Samples for mercury must be digested/leached and analyzed within 28 days of thawing. All other samples must be digested and analyzed within 6 months of thawing. Digestates may be stored at room temperature until the time of analysis.

16.1.6 Wipe, waste, filter, and tissue matrices are prepared in the same manner as soil samples with the following exceptions:

- The initial amount for wipe or filter samples is 1 wipe or 1 filter.
- The initial amount for tissue and waste samples is 1.0-1.2g.

16.2 Other Considerations

16.2.1 As allowed by EPA 3050B, an alternative determinative technique has been employed for samples analyzed by ICP/MS. The preparation procedures in the method are identical for ICP and ICP/MS samples up to the addition of concentrated HCl. SW-846 Method 3050B only requires this step for samples to be analyzed by ICP. The laboratory has adopted this procedure for both ICP and ICP/MS samples. All field samples and quality control samples (including MDLs, DOCs, and PT samples) have been prepared using this alternative technique.

16.2.2 The analyte list in Section 1.0 of EPA 3050B has been expanded to include all analytes currently performed by the laboratory.

16.2.3 EPA 3050B requires only a method blank, matrix spike, and matrix spike duplicate with each preparation batch; however, the analytical methods also require the analysis of a laboratory control sample taken through the entire preparation process. For this reason the laboratory includes the LCS as part of the required batch QC for SW-846 Method 3050B. The analytical methods also allow a sample duplicate to be performed in lieu of a matrix spike duplicate. The laboratory's procedures allow for this option; however, the MSD is performed as the default.

16.2.4 EPA 3050B requires a method blank be analyzed prior to use of nitric acid, hydrochloric acid, and hydrogen peroxide with results less than the MDL for all analytes of interest. The laboratory does not perform this analysis in advance. The vendor certificate of analysis is reviewed and approved prior to use. Method blanks are also analyzed with each batch as a check on the cleanliness of the reagents.

16.2.5 EPA 3050B includes a final filtration step prior to establishing the final volume. The laboratory does not routinely encounter interferences that could be eliminated by this step. Additionally, this filtration step introduces another source of contamination. Therefore, this step has not been included in this procedure.

16.2.6 EPA 3050B includes information on performing an alternate digest to obtain better recoveries for antimony. The laboratory has adjusted its default procedure to incorporate additional HNO_3 at the beginning of the digestion process to accommodate this analyte.

16.2.7 EPA 3050B requires digestates to be covered with a ribbed watch glass and heated until the volume has been reduced to approximately 5 mL or heated at 95 °C ± 5°C without boiling for two hours. The laboratory does not follow this practice.

16.2.8 Although not a SW-846 requirement, the laboratory requires that soil samples be submitted on ice to satisfy other state, regulatory and/or client requirements.

17.0 Attachments

The following Tables, Diagrams, and/or Validation Data are included as Attachments:

Attachment 1: SOP Summary
Attachment 2: Sample Collection, Preservation, and Holding Time Table
Attachment 3: QC Summary
Attachment 4: Instrument Maintenance and Troubleshooting
Attachment 5: Labware Cleaning Procedures

Attachment 1:
SOP Summary

Sample Preparation Summary
A known weight (approximately 1g) of the well-mixed sample is transferred to a suitable digestion vessel. The sample is digested with aliquots of nitric acid and hydrogen peroxide to break down the organics present in the sample. After the sample has been digested, as evidenced by a clear, pale yellow digestate, HCl is added to give an approximate acid concentration of 10% HCl and 5% HNO_3. Then the sample digest is diluted to 100mL with reagent water.

A smaller weight of sample may be digested and the sample brought to a final volume that is proportional to the 1g sample to 100mL final volume ratio. For example, if 0.50g is digested, the final volume of the digestate must be 50mL to achieve the same reporting limits.

Silica samples for EPA Method 6010C: Solid samples are extracted with reagent water using a DI Leach procedure and then filtered. The filtered solid extracts are analyzed by ICP.

Sample Analysis Summary
Prior to analysis by ICP, the sample must be digested using the sample preparation method appropriate to the matrix. Sample digestates are aspirated and nebulized into a spray chamber. A stream of argon gas carries the sample aerosol through the innermost of three concentric tubes and injects it into the middle of the donut-shaped plasma. The sample elements are dissociated, atomized, and excited to a higher energy level. As the elements fall to a lower energy level, radiation characteristic of the elements present in the plasma is emitted. The light is directed through an entrance slit, dispersed by the diffraction grating, and projected on to the photomultiplier tube (PMT) or charge coupled device (CCD) detector. The PMTs and CCD detectors convert the light energy to an electrical current. This signal is then digitized and processed by the data system. Background correction is required for trace element determination.

Prior to analysis by ICP-MS, the sample must be solubilized or digested using the sample preparation method appropriate to the matrix. Sample digestates are aspirated and nebulized into a spray chamber. A stream of argon gas carries the sample aerosol through the innermost of three concentric tubes and injects it into the middle of the donut-shaped plasma. The sample elements are dissociated, atomized, and excited to a higher energy level. The ions that are produced are entrained in the plasma gas and introduced, by means of an interface, into a mass spectrometer. The ions are sorted according to their mass to charge ratios and quantified with a channel mass spectrometer.

Attachment 2:
Sample Collection, Preservation, and Holding Time Table

Matrix	Routine Container	Routine Sample Size	Minimum Sample Size	Chemical Preservation	Thermal Preservation	Dechlorination Agent	Holding Time [1]
Soil	4oz or 8oz soil jar	1g 5g (Silica)	0.5g 2.5g (Silica)	None	<0-6°C	None	6 months (except for Hg which is 28 days)

[1] Inclusive of digestion and analysis.

Attachment 3:
QC Summary

QC Item	Frequency	Criteria	Corrective Action
Batch Definition	Up to 20 field samples, prepared together w/in 24-hour time period	Not Applicable	Not Applicable
Method Blank (MB)	One per batch	Refer to analytical SOP	Refer to analytical SOP
Lab Control Sample (LCS)	One per batch	Refer to analytical SOP	Refer to analytical SOP
Lab Control Sample Duplicate (LCSD)	One per batch, if insufficient sample provided for MS/MSD/SD	Refer to analytical SOP	Refer to analytical SOP
Matrix Spike (MS)	One per batch	Refer to analytical SOP	Refer to analytical SOP
Matrix Spike Duplicate (MSD) Or Sample Duplicate (SD)	One per batch	Refer to analytical SOP	Refer to analytical SOP
Initial Demonstration of Capability (IDOC)	Initially, per analyst, per method/analyte combination	Refer to SOP SA-QA-006	Refer to SOP SA-QA-006 (Unsupervised work cannot begin until acceptable IDOC is obtained.)
Continuing Demonstration of Capability (CDOC)	Annually, per analyst, per method/analyte combination	Refer to SOP SA-QA-006	Re-perform CDOC
Reporting Limit Verification (RLV)	Upon method/instrument set-up, per analyte/method/matrix combination. Then quarterly thereafter (for	Refer to SOP SA-QA-007	Refer to SOP SA-QA-007

QC Item	Frequency	Criteria	Corrective Action
	DOD ELAP) or annually thereafter (for NELAC)		
Method Detection Limit Study (MDL Study)	Upon method/instrument set-up	Refer to SOP SA-QA-007	Refer to SOP SA-QA-007
MDL Verification (MDLV)	Upon method/instrument set-up, per analyte/method/matrix combination. Then quarterly thereafter (for DOD ELAP) or annually thereafter (for NELAC)	Refer to SOP SA-QA-007	Refer to SOP SA-QA-007

Attachment 4:
Instrument Maintenance and Troubleshooting

Instrument Labeling

Each instrument must be labeled with its name or ID (e.g., MSA, ICP-D, etc.). Additionally, non-operational instruments must be isolated from service or marked as being out of service. Each piece of equipment has an "Operational / Not Operational" sticker that is used for this purpose.

Maintenance Log

A maintenance log must be established for each piece of equipment used in the laboratory. All maintenance that is performed on the instrument must be recorded in the log including:

- Type of maintenance performed (Note: This includes preventative/routine maintenance; non-routine maintenance; maintenance performed by an external vendor; updates to software versions; etc.)
- analyst or technician performing the maintenance
- date the maintenance was performed
- detailed explanation of the reason for the maintenance
- resolution of the problem and return to control
- all service calls from instrument representatives

Preventive Maintenance

Refer to the instrument manufacturer's guides for trouble-shooting items.

The temperature of the hot plate or digestion block must be monitored with each batch. If the temperature required for sample preparation cannot be maintained, the heating device must be removed from service and repaired or replaced.

Troubleshooting

Troubleshooting should be documented as outlined above. If possible, troubleshooting is best performed in a step-wise manner to systematically isolate instrument components. Refer to the instrument manufacturer's guides for specific information and strategies. Enlist assistance from technical and/or department management as needed.

Contingency Plan

In general, the laboratory has at least one backup unit for each critical unit. In the event of instrument failure, portions of the sample load may be diverted to duplicate instrumentation, the analytical technique switched to an alternate approved technique (such as manual colorimetric determination as opposed to automated colorimetric determination), or samples shipped to another properly certified or approved Eurofins TestAmerica location.

Attachment 5: Labware Cleaning Procedures

GLASSWARE CLEANING PROCEDURES METALS DEPARTMENT
Graduated Cylinders 1. Scrub with hot, soapy H_2O and brush. 2. Rinse thoroughly with tap H2O. 3. Rinse with 10% HNO_3 4. Rinse thoroughly with DI H_2O.
Volumetric Flasks 1. Empty contents of flask 2. Squirt a small amount of cleaning detergent directly into volumetric flask 3. Fill flask 1/3 full with **HOT** H_2O 4. Replace top and shake flask. 5. Empty flask and rinse with **HOT** H_2O until no soap remains in flask. 6. Add approximately 10mL concentrated HNO_3 to 50mL, 100mL, and 250mL flasks ---- replace top and shake well. For 500mL or 1000mL flasks use 25mL and for 10mL flasks use 2 - 5mL of concentrated HNO_3. 7. Rinse 3 times with DI H_2O, filling flask 1/3 full and replacing top. Store until needed. *** NEVER PLACE VOLUMETRIC FLASKS OR TOPS IN SINK OR DISHPAN WITH OTHER DIRTY DISHES.**
***Dispose of all acid waste in accordance with the Eurofins TestAmerica Savannah Addendum to the Corporate Safety Manual.**

FME023:03.11.09:3

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18.0 Revision History

Summary of Changes for Revision 15A:

- Added Vendor name to Hydrochloric and Nitric Acid reagents.
- Revised Attachment 2 for sample containers.

Summary of Changes from Revision 15:

- Minor editorial, grammatical, and/or formatting changes made.
- Updated SOP signatories to reflect current responsibilities and titles.
- Updated company name where applicable.
- Replaced references of TALS to LIMS.
- Updated section 14.1 for current disposal practices.

ELEMENTS BY ICP

(Methods: EPA 200.7, EPA 6010C, EPA 6010D, SM2340B)

Approvals (Signature/Date):

Kimberly Chamberlain 11/25/2020

Kimberly Chamberlain Date
Quality Assurance Manager/Technical Director
Environmental Health and Safety Coordinator

11/25/2020

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1.0 Scope and Application

This SOP gives the procedures for the determination of metals (elements) by inductively coupled plasma (ICP) atomic emission spectroscopy.

The routine matrices for this procedure are waters and soils; however, this procedure may be adapted to accommodate other matrices as outlined in Section 16.1.

A complete target analyte list, the reporting limits (RL), the method detection limits (MDL), and the accuracy and precision criteria associated with this procedure are provided in the TALS Method Limit Groups (MLGs).

This SOP was written by and for Eurofins TestAmerica's Savannah laboratory.

2.0 Summary of Method

Prior to analysis by ICP, the sample must be digested/filtered using the sample preparation method appropriate to the analyte/matrix combination. Sample digestates/filtrates are aspirated and nebulized into a spray chamber. A stream of argon gas carries the sample aerosol through the innermost of three concentric tubes and injects it into the middle of the donut-shaped plasma. The sample elements are dissociated, atomized, and excited to a higher energy level. As the elements fall to a lower energy level, radiation characteristic of the elements present in the plasma is emitted. The light is directed through an entrance slit, dispersed by the diffraction grating, and projected on to the photomultiplier tube (PMT) or onto a charge-coupled device (CCD). The PMTs and CCDs, located behind the exit slits, convert the light energy to an electrical current. This signal is then digitized and processed by the data system. Background correction is required for trace element determination.

Note: Drinking water samples (EPA 200.7) only require digestion if the determination of silver (Ag) is requested or if the turbidity is greater than or equal to 1.0 NTU.

This SOP is based on the following methods: EPA 200.7, EPA 6010C, and EPA 6010D. The procedure for the determination hardness via the calculation method (i.e., SM2340B) is provided in Attachment 9.

3.0 Definitions

Refer to the Glossary Section of the *Quality Assurance Manual* (QAM) for a complete listing of applicable definitions and acronyms.

4.0 Interferences

4.1 Procedural Interferences

4.1.1 Interferences may be caused by contaminants in solvents, reagents, glassware, and other sample processing apparatus and can make identification and/or quantification of the target analytes difficult.

4.1.2 All sample collection containers are single-use disposable containers which limits the potential for contamination. All non-disposable labware must be scrupulously cleaned in accordance with the posted Labware Cleaning Instructions to ensure it is free from contaminants and does not contribute artifacts.

4.1.3 High purity reagents and solvents are used to help minimize interference problems. Hydrochloric acid and nitric acid must be verified prior to use in accordance with the Eurofins TestAmerica Solvent Lot Testing Program.

4.1.4 Instrument and/or method blanks are routinely used to demonstrate all reagents and apparatus are free from interferences under the conditions of the analysis.

4.1.5 Physical interferences are effects associated with the sample nebulization and transport processes. Changes in viscosity can cause significant inaccuracies, especially in samples containing high concentrations of dissolved solids or high acid concentrations. Typically, physical interferences are mitigated by the instrument's peristaltic pump and the use of internal standard.

4.2 Matrix Interferences

4.2.1 Matrix interferences may be caused by contaminants that are co-extracted from the sample matrix. The sample may require dilution prior to analysis to reduce or eliminate the interferences.

4.2.2 Interfering contamination may occur when a sample containing low concentrations of analytes is analyzed immediately following a sample containing relatively high concentrations of analytes. As such, samples known to be clean should be analyzed first. To prevent carryover into subsequent samples, analysis of reagent blanks may be needed after the analysis of a sample containing high concentrations of analytes.

4.2.3 Spectral interferences are caused by the overlap of a spectral line from another element, unresolved overlap of molecular band spectra, background contribution from continuous phenomena, and stray light from the line emissions of highly concentrated elements.

4.2.3.1 Spectral overlap may be compensated for by the use of inter-element correction factors.

4.2.3.2 Background contribution and stray light can be compensated for by a background correction adjacent to the analyte line.

4.2.3.3 The total recoverable sample digestion procedure is suitable for the determination of silver in aqueous samples containing concentrations up to 0.1mg/L and soil samples containing concentrations up to 50mg/kg. For samples containing higher concentrations of silver, succeeding smaller aliquots should be prepared until the digestate contains less than these levels of silver.

Similarly, soil samples digested via the total recoverable metals digestion procedure with a tin concentration that exceeds 1%, must be re-digested using a smaller aliquot.

Alternatively, samples may be digested using the total metals digestion procedure.

5.0 Safety

Employees must abide by the policies and procedures in the Eurofins TestAmerica Environmental Health and Safety Manual (EHSM), the Eurofins TestAmerica Savannah Addendum to the EHSM, and this document.

This procedure may involve hazardous materials, operations, and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user to follow appropriate safety, waste disposal, and health practices under the assumption that all samples and reagents are potentially hazardous.

The analyst must protect himself/herself from exposure to the sample matrix. Many of the samples that are tested may contain hazardous chemical compounds or biological organisms. The analyst must, at a minimum, wear protective clothing (lab coat), eye protection (safety glasses or face shield), disposable latex or nitrile gloves, and closed-toe, nonabsorbent shoes when handling samples.

5.1 Specific Safety Concerns or Requirements

The ICP plasma emits strong UV light and is harmful to vision. All analysts must avoid looking directly at the plasma.

The plasma generates high temperatures. Analysts must ensure that all equipment is shut down and cooled off before performing maintenance and troubleshooting in the plasma area.

Nitric and hydrochloric acids are extremely hazardous as oxidizers, corrosives, poisons, and are reactive. Inhalation of the vapors can cause coughing, choking, irritation of the nose, throat, and respiratory tract, breathing difficulties, and lead to pneumonia and

pulmonary edema. Contact with the skin can cause severe burns, redness, and pain. Nitric acid can cause deep ulcers, and staining of the skin to a yellow or yellow-brown color. These acid vapors are irritating and can cause damage to the eyes. Contact with the eyes can cause permanent damage.

Samples that contain high concentrations of carbonates or organic matter, or samples that are at elevated pH can react violently when acids are added. Acids must be added to samples under a hood to avoid splash/splatter hazards and/or possibly toxic vapors that will be given off when the samples are acidified.

5.2 Primary Materials Used

The following is a list of the materials used in this procedure, which have a serious or significant hazard rating, and a summary of the primary hazards listed in their MSDS/SDS.

NOTE: This list does not include all materials used in the procedure. A complete list of materials used in this procedure can be found in the Reagents and Standards Section and the Equipment and Supplies Section of this SOP

Employees must review the information in the MSDS/SDS for each material before using it for the first time or when there are major changes to the MSDS/SDS. Electronic copies of MSDS/SDS can be found using the "MSDS" link on the Oasis homepage, on the EH&S webpage on Oasis, and via TALS File System Shares.

Material	Hazards	Exposure Limit[1]	Signs and Symptoms of Exposure
Hydrochloric Acid[2]	Corrosive Poison	5ppm Ceiling	Inhalation of vapors can cause coughing, choking, inflammation of the nose, throat, and upper respiratory tract, and in severe cases, pulmonary edema, circulatory failure, and death. Can cause redness, pain, and severe skin burns. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
Nitric Acid[2]	Corrosive Oxidizer Poison	2ppm TWA 4ppm STEL	Nitric acid is extremely hazardous; it is corrosive, reactive, an oxidizer, and a poison. Inhalation of vapors can cause breathing difficulties and lead to pneumonia and pulmonary edema, which may be fatal. Other symptoms may include coughing, choking, and irritation of the nose, throat, and respiratory tract. Can cause redness, pain, and severe skin burns. Concentrated solutions cause deep ulcers and stain skin a yellow or yellow-brown color. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
[1]Exposure limit refers to the OSHA regulatory exposure limit.			
[2]Always add acid to water to prevent violent reactions.			

6.0 Equipment and Supplies

6.1 Equipment and Instrumentation

Varian 730 ES or other suitable inductively coupled plasma emission spectrometer. Data is evaluated and quantitated using TALS

Top-loading Balance – Verify in accordance with SOP SA-AN-100: *Laboratory Support Equipment (Verification and Use)*

6.2 Lab Supplies

Argon gas supply and appropriate fittings

Nitrogen Gas

Cooling water supply

Peristaltic Pump
pH paper

6.3 Analytical Data System/ Software / Hardware

The Quality Assurance Department maintains the Master Equipment Listing for the laboratory. This is a listing of all instrumentation utilized by the laboratory and includes details about the instrumentation including the software or firmware.

6.4 Volumetric Containers and Dispensers

All volumetric labware must be verified in accordance with SOP SA-AN-100: *Laboratory Support Equipment (Verification and Use).* Refer to Attachment 4 for Labware Cleaning Procedures.

Volumetric Labware	Volume	Type (Quantitative / Qualitative)	Use	Verification Frequency	Laboratory Verification Criteria
Autosampler Vials	14mL	Qualitative	Sample Introduction to Instrument	None	None
Mechanical Eppendorf-style Pipettes (w/ tips)	Various	QUANTITATIVE	Dilution and Standard Preparation	Initially, prior to first use then quarterly thereafter (Daily, for DOD)	Accuracy = 2% Precision = 1%
Volumetric Flasks (Class A)	Various	QUANTITATIVE	Standard Preparation	None (Class A)	None (Class A)
Digestion Vials	100mL	Qualitative	Standard Preparation and Storage	None	None

6.5 Sample Collection Containers

All sample collection containers are single-use disposable containers which limits the potential for contamination.

The routine sample collection containers provided by the laboratory are as follows:

Waters: 250mL Plastic – purchased with Certificate of Analysis attesting to purity.
Solids: 8oz Plastic or Glass Jar – purchased with Certificate of Analysis attesting to purity.

7.0 Reagents and Standards

The standards and reagents listed are those in use at the time this SOP was updated. Other standard and reagent stocks, vendors, and concentrations may be used provided they are fully documented and in compliance with method requirements.

7.1 Expiration Dates

Expiration dates (time from initial use or receipt to final use) for standard and reagent materials must be set according to the guidance in this SOP. Note: These are maximum expiration dates and are not to be considered an absolute guarantee of standard or reagent quality. Sound judgment must be used when deciding whether to use a standard or reagent. If there is doubt about the quality of a standard or reagent material, a new material must be obtained or the standard or reagent material verified. Data quality must not be compromised to extend a standard's life.

The expiration date of any standard must not exceed the expiration date of the standard that was used to prepare it.

Unless listed elsewhere in this SOP, the expiration dates given below apply.

7.1.1 The expiration date for unopened standards and reagents is the manufacturer's expiration date.

7.1.2 The expiration date for opened stock reagents is the manufacturer's expiration date or 5 years from the date opened, whichever is sooner.

7.1.3 The expiration date for opened stock standards is the manufacturer's expiration date.

7.1.4 The expiration date for prepared reagents is 6 months from the date prepared or the expiration date of the parent reagent, whichever is sooner.

7.1.5 The expiration date for prepared standards is 6 months from the date prepared or the expiration date of the parent standard, whichever is sooner.

7.2 Reagents

Reagents must be prepared and documented in accordance with SOP SA-AN-041: *Reagent and Standard Materials Procedures.*

Hydrochloric acid and nitric acid must be verified prior to use in accordance with the Eurofins TestAmerica Solvent Lot Testing Program.

7.2.1 Laboratory Reagent Water – ASTM Type I. The conductivity must be monitored in accordance with SOP SA-AN-100: *Laboratory Support Equipment (Verification and Use).*

7.2.2 Nitric acid (HNO_3) – reagent grade. Store in a cool, dry, ventilated storage area with acid resistant floors and good drainage. Store away from sunlight, heat, water, and incompatible materials. Stable under ordinary conditions of use and storage.

7.2.3 Hydrochloric acid (HCl) – reagent grade. Store in a cool, dry, ventilated storage area with acid resistant floors and good drainage. Store away from sunlight, heat, water, and incompatible materials. Stable under ordinary conditions of use and storage.

7.3 Standards

Standards must be prepared and documented in accordance with SOP SA-AN-041: *Reagent and Standard Materials Procedures.* Certificates of analysis or purity must be received with all purchased standards, and scanned and attached in TALS.

Refer to Attachments 5 and 6 for standard preparation information.

8.0 Sample Collection, Preservation, Shipment, and Storage

8.1 Aqueous Samples

Aqueous samples are routinely collected in 250mL plastic containers containing 1.5mL 1:1 nitric acid preservative or 500mL plastic containers containing 3.0mL 1:1 nitric acid preservative. The preservative must be sufficient to achieve a sample pH of less than 2.

If dissolved metals are requested, the sample must be filtered in the field. This filtration step must occur prior to the addition of the preservative.

If the laboratory is requested to filter the sample, then the sample must be collected in a 250mL plastic container without preservative. The samples are filtered upon receipt and are then acidified with 1.25mL concentrated nitric acid to a pH<2. Note: Samples must sit for 24 hours after the addition of the acid to verify pH is maintained, prior to digestion.

Note: The reference methods state that filtration must occur immediately; therefore, if the laboratory is requested to filter the samples – as opposed to a field filtration - appropriate narration must be included in the final report to denote this method deviation.

Samples are stored at room temperature until the time of digestion. Samples must be digested within 6 months of collection. Digestates are stored at room temperature until the time of analysis and must be analyzed within 6 months of collection.

8.2 Soil Samples

Soil samples are routinely collected in 8oz plastic or glass containers.

Samples must be iced at the time of collection and maintained at 4°C (less than 6°C but not frozen) until the time of digestion. Samples must be digested within 6 months of collection. Digestates are stored at room temperature until the time of analysis and analyzed within 6 months of collection.

9.0 Quality Control

SOP SA-QA-017: *Analytical Batching and Evaluation of QC Data* and the SOP Summary in Attachment 4 provide requirements for evaluating QC data.

9.1 Batch QC

9.1.1 EPA 200.7 – Drinking Water

A digestion batch consists of up to 20 environmental samples and the associated QC items digested together within a 24 hour period.

The laboratory's default minimum QC items performed for each digestion batch are: a method blank, a laboratory control sample (LCS), a low-level LCS (LLCS), a matrix spike (MS) to be performed on a minimum of 10% of samples or one per batch – whichever is greater, and a matrix spike duplicate (MSD).

This frequency equates to the following:

- For a batch of 10 or fewer samples, the default QC items are a method blank, an LCS, an LLCS, 1 matrix spike, and 1 matrix spike duplicate (MSD).
- For a batch of 11-20 samples, the minimum QC items are a method blank, an LCS, an LLCS, 1 matrix spike (from sample 1-10), another matrix spike (from sample 11-20), and a matrix spike duplicate (MSD).

9.1.2 EPA 200.7 – Clean Water Act

A digestion batch consists of up to 20 environmental samples and the associated QC items digested together within a 24 hour period.

The laboratory's default minimum QC items performed for each digestion batch are: a method blank, a laboratory control sample (LCS), a matrix spike (MS) to be performed on a minimum of 10% of samples or one per batch – whichever is greater, and a matrix spike duplicate (MSD).

This frequency equates to the following:

- For a batch of 10 or fewer samples, the default QC items are a method blank, an LCS, 1 MS, and 1 MSD.
- For a batch of 11-20 samples, the default QC items are a method blank, an LCS, 1 MS (from sample 1-10), another MS (from sample 11-20), and an MSD.

9.1.3 EPA 6010C

A digestion batch consists of up to 20 environmental samples and the associated QC items. The laboratory's default minimum QC items performed for each digestion batch are: a method blank, a laboratory control sample (LCS), a matrix spike (MS), and a matrix spike duplicate (MSD).

9.1.4 If there is insufficient sample provided to perform the required matrix spikes, the LCS must be prepared induplicate (i.e., LCS/LCSD). An NCM must be initiated to denote this situation.

Insufficient sample volume is defined as receiving less than a total of 50mL or 2g.

The routine container supplied for this method is a 250mL (water) or 8oz (soil) container. 50mL or 1g is required for digestion. Reduced sample initial volumes may be necessary to achieve the required batch matrix spike frequency; however, the minimum extraction volume to be used for the matrix spike samples is 25mL or 0.5g. Note: Final volumes and spike amounts must be adjusted to compensate for these reduced initial volumes.

Note: Unless the client has specified which sample to use as the matrix spike, the matrix spike must be selected at random from the samples in the batch. Field QC (e.g., equipment blanks, trip blanks, and field blanks) must not be used for MS/MSD unless specifically requested to do so by the client or unless there is insufficient sample available for performing MS/MSD using an actual field sample.

9.1.5 Batch QC must meet the criteria given in Attachment 3 of this SOP.

Note: If an LCS and LCSD are performed, both QC items must be evaluated and reported. Acceptable recoveries (as well as %RPD) for both LCS and LCSD are required.

9.1.6 Preparation SOPs

Refer to the following SOPs for specifics on the preparation process:

Matrix	SOP
Water Samples	SA-ME-050
Soil Samples	SA-ME-051

9.2 Instrument QC

9.2.1 Initial Calibration (ICAL)

The instrument must be calibrated in accordance with SOP SA-QA-016: *Evaluation of Calibration Curves.* This SOP provides requirements for establishing the calibration curve and gives the applicable formulas.

Instrument calibration is performed by analyzing a series of known standards. The calibration curve must consist of at least one standard (the high standard) and a blank. If a multi-point curve is requested, the calibration curve must consist of a minimum of 3 standards and a blank. The initial calibration standard concentrations currently in use in the laboratory for a multi-point curve are included in Attachment 5.

Note: Refer to Attachment 5 for the standard preparation instructions. Other standard concentrations may be used provided they support the reporting limit and are fully documented in accordance with SOP SA-AN-041.

The correlation coefficient (r) of the regression curve must be greater than or equal to 0.998 for the initial calibration curve to be acceptable.

If a multi-point calibration curve is used, the upper limit of the linear range is the concentration of the High Standard.

9.2.1.1 Determination of Linear Range of the ICP

If the instrument is not calibrated over its entire linear range for a particular element, a linear range standard must be analyzed immediately following the calibration to validate the linear range.

This standard must recover within +/- 10% of the true value and any sample that is greater than 90% of the concentration in the linear range standard must be diluted and reanalyzed.

9.2.2 High Standard Read Back

The highest concentration calibration standard must be reanalyzed as an “unknown” after the instrument is calibrated. The results for the re-analysis of the highest concentration calibration standard must be within ± 5% of the true value for each target analyte. If the result for any target analyte is outside of this range, the ICP may need to be "profiled" and the standardization/calibration repeated.

9.2.3 Second Source Initial Calibration Verification (ICV)

The calibration curve must be verified initially – prior to any sample analyses – in accordance with SOP SA-QA-016 with a standard obtained from a second source.

The ICV must be within 10% of the true value to be acceptable for EPA 6010C. The ICV must be within 5% of the true value to be acceptable for EPA 200.7, and must also have a %RSD ≤3% of the 3 replicates analyzed to be acceptable for EPA 200.7.

The initial calibration verification standard concentration currently in use in the laboratory is given in Attachment 5. Another standard concentration may be used provided it is mid-level and fully documented in accordance with SOP SA-AN-041.

9.2.4 Initial Calibration Blank (ICB) / Continuing Calibration Blank (CCB)

The instrument must be shown to be free from contamination by the analysis of calibration blanks. Initial calibration blanks are analyzed immediately following the initial calibration. Continuing calibration blanks must be analyzed immediately following each continuing calibration verification (CCV).

The absolute value of the initial and continuing calibration blanks must be <½RL to be acceptable.

9.2.5 Continuing Calibration Verification

The initial calibration curve must be verified every 10 samples with a mid-level standard.

The CCV must be within 10% of the true value to be acceptable and must also have a %RSD ≤5% of the 3 replicates.

The continuing calibration verification standard concentration currently in use in the laboratory is equivalent to half the concentration of the High Standard. Refer to Attachment 5 for the standard preparation instructions. Another standard concentration may be used provided it is mid-level and fully documented in accordance with SOP SA-AN-041.

9.2.6 Internal Standard (ISTD)

This procedure is an internal standard (ISTD) procedure. Yttrium is used as the primary internal standard; however, Scandium may also be used as an alternate ISTD.

The internal standard must be added to all standards, samples, and QC items prior to analysis. This is accomplished by means of an additional channel on the peristaltic pump and connected to the sample line with a 'T' or 'Y' connector fitting. This ensures constant concentration of the internal standard and eliminates the possibility of human spiking error. The concentration of the internal standard must be the same in all calibration samples, field samples, and QC samples. A concentration of 7mg/L is used for Yttrium and Scandium.

Any sample containing ISTD recoveries greater than 130% must be diluted and re-analyzed. Although ISTD recoveries less than 70% are extremely rare, the analyst should consider further dilution if this situation occurs.

9.2.6.1 Ionization Effects

High concentrations of some elements, such as Na, K, Ca, and Mg, can produce interferences from their ionization effects. The introduction of additional ions in abundance, such as Lithium, will minimize this interference. Therefore, Lithium is added to the internal standard solution, and is continually pumped at a constant rate.

See Attachment 5 for details on the preparation of the internal standard solution.

9.2.7 Reporting Limit Check Standard

A reporting limit (RL) check standard is used to demonstrate that the ICP is capable of detecting the target compounds at or below the reporting limit. EPA 200.7 requires this check only when utilizing a single point calibration. EPA 6010C requires this check for both single point and multi-point calibrations.

The concentrations in the RL check standard must be at levels that are less than or equal to the reporting limit for the samples being analyzed. The determined concentration must be detected within ±50% (for EPA 200.7), ±30% (for EPA 6010C) or ±20% with a RSD < 20% (for EPA 6010D) of the true concentration. The RL check standard must be analyzed at the beginning and end of an analysis run (i.e., all samples must be bracketed by this check standard), except for EPA 6010D and DOD QSM V5 which do not require a closing LLCCV.

Note: This RL Check Standard is referred to as the LLICV and LLCCV in EPA 6010C/6010D.

9.2.8 Interference Check Standards

The purpose of the Interference Check Standard is to prove that the instrument software is adequately correcting for common interferences through the use interelement correction factors. The concentrations of the target analytes must be within 20% of the true concentrations to be acceptable. The analyst must pay particular attention to false positives and false negatives for elements not present in the interference check solutions.

Note: Non-spiked analytes (aside from known contaminants) must be ≤ LLOQ, or RL.

9.2.9 Serial Dilution

A dilution must be prepared and analyzed on one sample per batch to determine if matrix interferences are present. Compare the results of the diluted and un-diluted aliquots of sample digestate for analytes that are present in the native sample at a concentration ≥50x IDL.

If the results of the dilution are within ±10% of the results of the undiluted sample, no matrix interference is present. If the results differ by greater than the amounts outlined above, a matrix interference should be suspected and the sample digestate should be subjected to a post-digestion spike.

9.2.10 Post-Digestion Spike

A post-digestion spike is performed on one sample per analytical batch to determine if matrix interferences are present. This post-digestion spike is evaluated if the serial dilution fails or if the analyte concentration is not at least 50 times the instrument detection limit. The sample selected as the post-digestion spike should be the same sample selected for serial dilution.

The post-digestion spike must be within 25% of the true value to be acceptable. If these criteria are not met, then the data are qualified.

9.2.11 % RSD of Multiple Exposures

Replicate analyses of a sample should agree within 30% RSD for samples with detections greater than the reporting limit. Replicates that are greater 30% RSD should be investigated for matrix interferences or differences in viscosity of the digest.

9.2.12 Interelement Correction Factors (IEC)

Interelement correction factors (IEC) for all elements must be determined every six months using the manufacturer's guidance.

The laboratory may combine the linearity study with the IEC study, thereby eliminating redundancy. (Single element linearity check solutions for each analyte should be closely evaluated for all non-spiked elements, and applicable correction factors should be applied according to the instrument's software. The analyst must be careful not to correct for any contamination which may be present in the actual solution.)

At a minimum the IECs must be verified at the beginning of each analytical sequence through the analysis of interferent check solutions ICSA and ICSAB. Note: Some protocols and project plans require the analysis of the ICSA and ICSAB standard at the beginning and end of an analysis sequence. The analyst must be aware of the requirements of the project before beginning the analysis.

Note: The reference methods require performance of single element spectral interference checks initially, when an instrument is set up, and at least every 6 months thereafter, to verify interelement correction factors. The laboratory combines this with the linear range study performed every 6 months to meet this requirement.

9.3 Corrective Action for Out-of-Control Data

When the quality control parameters do not meet the criteria set forth in this SOP, corrective action must be taken in accordance with SOP SA-QA-005: *Preventive and Corrective Action Procedures* the QC Summary Table in Attachment 3. SOP SA-QA-005 provides contingencies for out-of-control data and gives guidance for exceptionally permitting departures from approved policies and procedures. Nonconformance Memos must be initiated to document all instances where QC criteria are not met and all departures from approved policies and procedures.

10.0 Procedure

10.1 Sample Preparation

The sample preparation procedures are given in the following SOPs:

Matrix	SOP #
Water Samples	SA-ME-050
Soil Samples	SA-ME-051

10.2 QC Sample Preparation

Batch QC sample preparation procedures are given in the SOPs listed above. Additional QC items are listed below.

10.2.1 Serial Dilution

Dilute the digestate by a factor of five, and analyze the dilution using the same procedures used for the un-diluted aliquot.

10.2.2 Post Digestion Spike

Transfer 10mL of a digestate to a suitable vial. Spike the sample with 0.10mL of MS Cal Stk.

10.3 Analysis

All soil digestates and all water digestates containing particulates are filtered through a 0.45um filter prior to analysis.

Note: If digestates are filtered prior to analysis, then the applicable method blank and LCS/LCSD must also be filtered. This information must be captured via the Batch Data Types in TALS.

10.3.1 Instrument Operating Conditions

Turn the ICP on and allow it to become thermally stable before beginning to analyze the calibration standards. It will take about an hour for the instrument to warm up. If the optics were turned off, allow 2 hours warm up time. After the warm-up period, perform a torch alignment sequence to properly profile the instrument.

Instrument maintenance must be performed in accordance with Attachment 4 of this SOP.

10.3.2 Internal Standard (ISTD)

Prior to analysis, internal standard must be added to all standards, samples, and QC items. The concentration of the internal standard must be the same in all calibration samples, field samples, and QC samples.

10.3.3 Initial and Continuing Calibration

Calibrate the instrument using the standards and criteria described in Section 9.2.1. Once the calibration has been established and verified with a high level standard and an ICV in accordance with Sections 9.2.2 and 9.2.3, sample analysis may proceed.

Verify the calibration curve with a continuing calibration verification using the standards and criteria described given in Section 9.2.5.

10.3.4 Sample Analysis

Samples that are known to be relatively clean should be analyzed first. Samples suspected of containing high concentrations should be analyzed last. Instrument blanks may be analyzed after suspected high concentration samples to allow the complete removal of trace analytes. The instrument blanks would be included in the sample count in determining the maximum number of samples between CCVs/CCBs.

The default procedure is to include QC items (method blank, LCS, MS/MSD, and SD) in determining the maximum number of samples between CCVs/CCBs.

10.3.5 Example Analytical Sequence

An example analytical sequence is listed below.

Analytical Sequence for samples immediately following an initial calibration:

Description	Comments
Instrument Warm-up	
Torch Alignment	If torch is changed
Initial Calibration	
High Calibration Standard	Re-analyzed as a sample
ICV	Second Source
ICB	
Reporting Limit Check Standard	
ICP Interference Check Solution A (ICSA)	
ICP Interference Check Solution AB (ICSAB)	
CCV	
CCB	10-analysis count begins after analysis of the CCB
Samples & Batch QC Items	Up to 10 analyses, including QC
CCV	
CCB	10-analysis count begins after analysis of the CCB

Samples & Batch QC Items	Up to 10 analyses, including QC
CCV	
CCB	10-analysis count begins after analysis of the CCB
Samples & Batch QC Items	Up to 10 analyses, including QC
CCV	
CCB	10-analysis count begins after analysis of the CCB
Reporting Limit Check Standard	

Note: The analytical sequence continues as outlined above and must end with the analysis of the CCV and CCB. The 10 samples include all QC samples/standards with the exception of CCVs and CCBs.

11.0 Calculations / Data Reduction

11.1 Data Reduction

Data must be evaluated in accordance with SOP SA-QA-002: *Data Generation and Review*.

11.1.1 Dilutions

If the concentration of a sample is above the calibration range (or 90% of the linear range for single point curves) of the instrument the sample digestate must be diluted and reanalyzed.

Dilutions must be prepared in reagent water containing 5% hydrochloric acid and 1% nitric acid by volume.

Note: Samples digested via the total recoverable metals digestion procedure with a silver concentration >0.1mg/L or >50mg/kg must be diluted and re-digested. Smaller volume aliquots should be prepared until the results are below these values. Alternatively, samples may be digested using the total metals digestion procedure.

Note: Soil samples digested via the total recoverable metals digestion procedure with a tin concentration that exceeds 1%, must be re-digested using a smaller aliquot. Alternatively, samples may be digested using the total metals digestion procedure.

11.1.2 Historical Data

Many of the laboratory's clients submit samples for repeat monitoring purposes. Prior to analysis, the analyst must verify TALS Worksheet Notes and/or use the TALS Historical Data Tracker feature to determine if historical data is available for review.

11.1.3 Chemical Relationships

The analyst must be aware of the following chemical relationships:

- Total Results should be ≥ Dissolved results

If this relationship is not met, further evaluation is needed to rule out a laboratory error.

11.1.4 Evaluation of Batch QC and Samples for Matrix Interferences

Matrix interference is evaluated using three criteria: the recoveries of the matrix spike (MS) and matrix spike duplicate (MSD), the results of the serial dilution, and the recovery of the post digestion spike. This laboratory uses internal standard calibration and quantification which minimizes the need to use the method of standard additions (MSA) to quantify sample digests with known or suspect matrix interferences. MSA is used when the recoveries of the MS/MSD are less than 50% and there is no obvious reason for the poor recoveries (e.g., viscosity of digest, high dissolved solids, undigested solids, known high concentrations of inter-element interferences, etc.).

The primary indicator of matrix interference is the results of the MS/MSD. If the recoveries and precision of the MS/MSD are within acceptance limits, matrix interferences are not indicated and unacceptable results for the serial dilution and post-digestion spikes are likely due to poor preparation rather than matrix interference.

Use the following table to evaluate for the indication of matrix interferences.

MS	MSD	Serial dilution[1]	Post Digestion Spike[1]	Action
OK	OK	OK	OK	Report
OK	OK	Out	OK	Unlikely to occur; suspect preparation of serial dilution or post digestion spike
OK	OK	OK	Out	
OK	Out	OK	OK	Suspect preparation-qualify results
Out	OK	OK	OK	
Out	Out	Out	OK	Matrix interference confirmed-qualify results if recoveries >50% If MS/MSD recoveries <50% and there is no obvious reason, perform MSA as directed in section 11.1.5.
Out	Out	OK	Out	
Out	Out	Out	Out	

[1]Performed on un-spiked digest of the sample designated as the MS/MSD

11.1.5 Evaluation of Post-Digestion Spikes

Result of Post-Digestion Spikes	Action
Within 75-125% limits	None
>125% recovery	Re-spike and reanalyze if multiple analytes >125%. Repeat analysis of un-spiked sample and re-evaluate recoveries. Qualify results for affected analytes.
<75% recovery but >50% recovery	Qualify results for affected analytes. Dilute digestate and repeat spike for affected analytes. ***Treat all samples associated with spike in the same manner as the spiked sample (i.e., spike or dilute samples).*** If recoveries are not 75-125%, all associated samples may be re-analyzed by single point MSA. Note: High levels of target analytes may inhibit spike recovery. Consult the Department Manager in events where high levels of targets appear to be interfering.
<50% recovery	Dilute and re-spike. Elevate RL accordingly (for all associated samples). Spike and evaluate all associated samples. Spike and evaluate all associated samples by single point MSA. Qualify results for affected analytes.

Note: The >50% recovery of the post digestion spike is a benchmark below which samples may be biased high if corrected for spike recovery.

11.2 Calculations

11.2.1 The calculations associated with batch QC determinations are given in SOP SA-QA-017. Applicable calculations include accuracy (% recovery) and precision (%RPD and % difference).

11.2.2 The calculations associated with initial and continuing calibrations are given in SOP SA-QA-016. Applicable calculations include determination for correlation coefficient, slope, intercept, and percent recoveries.

11.2.3 The calculation to determine final concentration is given as follows:

$$FinalConcentration = CONC_{Sample} \otimes \frac{F}{I \times dw} \otimes D$$

Where:

$CONC_{Sample}$= Concentration of the sample
F = Final volume/weight
I = Initial volume/weight
D = Dilution factor
dw = % Solids decimal equivalent

Note: All dry weight corrections are performed automatically in TALS.

Note: This calculation assumes all applicable unit correction factors are applied.

11.2.4 Method of Standard Additions

The concentrations of both sample aliquots are measured and the sample concentration is calculated as follows:

$$C_x = \frac{S_2 V_s C_s}{(S_1 - S_2) V_x}$$

Where:

S_1 = Absorbance or concentration of the spiked aliquot
S_2 = Absorbance or concentration of the un-spiked aliquot
V_s = Volume of spike solution
V_x = Volume of sample aliquots
C_s = Spike solution concentration

Note: This calculation assumes all applicable unit correction factors are applied.

12.0 Method Performance

12.1 Reporting Limit Verification (RLV)

At a minimum, RLVs must be performed initially upon method set-up in accordance with SOP SA-QA-007: *Determination and Verification of Detection and Reporting Limits*.

For analytes and methods certified by DOD ELAP, RLVs must also be performed quarterly thereafter. For all other analytes and methods, RLVs must also be performed annually thereafter. Exceptions may be made for project-specific non-routine analytes.

12.2 Method Detection Limit (MDL) Study

The MDL is the lowest concentration that can be detected for a given analytical method and sample matrix with 99% confidence that the analyte is present. MDLs reflect a calculated

(statistical) value determined under ideal laboratory conditions in a clean matrix and may not be achievable in all environmental matrices. The current MDLs associated with this procedure are given in the Method Limit Group (MLG) in TALS.

At a minimum, MDL Studies must be performed initially upon method set-up in accordance with SOP SA-QA-007: *Determination and Verification of Detection and Reporting Limits*.

Note: MDL Studies are not required for non-routine analytes provided results are not reported below the RL (i.e., MDL equals RL in TALS).

12.3 Method Detection Limit Verification (MDLV)

At a minimum, MDLVs must be performed initially upon method set-up in accordance with SOP SA-QA-007: *Determination and Verification of Detection and Reporting Limits*.

For analytes and methods certified by DOD ELAP, MDLVs must also be performed quarterly thereafter. For all other analytes and methods, MDLVs must also be performed annually thereafter.

Note: MDLVs are not required for non-routine analytes provided results are not reported below the RL (i.e., MDL equals RL in TALS).

12.4 Determination of the Instrument Detection Limit (IDL)

The instrument detection limit (IDL) is the concentration of analyte that can be statistically distinguished from the background noise of the instrument. The IDL limit must be determined quarterly, at a minimum, for each analyte in accordance with SOP SA-QA-007: *Determination and Verification of Detection and Reporting Limits (RLs, MDLs, and IDLs)*.

The IDL is defined as three times the average of the standard deviation of a minimum of seven replicate analyses of blank solution performed three times over non-consecutive days. The IDL may be elevated above the background noise (blank levels). The current IDL associated with this procedure is given in the Equipment Limit Group (ELG) in TALS.

12.5 QC Limit Generation, Control Charting, and Trend Analysis

The control limits for the batch QC items (LCS, MS/MSD) for this procedure are specified in the reference method and cannot be broadened; therefore, the laboratory defaults to the method-defined limits and does not utilize in-house or laboratory-derived limits for the evaluation of batch QC items.

Although the laboratory must default to the method-defined QC limits, control charting is a useful tool and is performed to assess analyte recoveries over time to evaluate trends. Control charting must be performed periodically (at a minimum annually) in accordance with SOP SA-QA-017: *Evaluation of Batch QC Data*.

12.6 Lower Limit of Quantitation Check Sample (LLQC)

EPA 6010C requires a lower limit of quantitation check sample (LLQC) analysis to demonstrate the required reporting limit capability. The laboratory shall prepare and analyze this solution on a yearly basis, in accordance with all applicable preparatory SOPs (i.e. the solution must be digested.) It is recommended that the laboratory utilize the RL Check solution (Section 9.2.7) by simply carrying it through the entire preparatory procedure for each of the prep methods associated with EPA 6010C. Digesting this solution on the same prep batch as the yearly MDL studies is preferred. The LLQC must recover within 70-130% to be acceptable, or a new (higher) reporting limit must be established.

12.7 Demonstrations of Capability

Initial and continuing demonstration of capability must be performed in accordance with SOP SA-QA-006*: Training Procedures.*

Prior to performing this procedure unsupervised, each new analyst who performs this analysis must demonstrate proficiency per method/analyte combination by successful completion of an initial demonstration of capability. The IDOC is performed by the analysis of 4 consecutive LCSs that meet the method criteria for accuracy and precision. The IDOC must be documented and routed to the QA Department for filing.

Annual continuing demonstrations of capability (CDOCs) are also required per analyst per method/analyte combination. The CDOC requirement may be met by the consecutive analysis of four LCS all in the same batch, by the analysis of four LCS analyzed in four consecutive batches (in different batches on different days), via acceptable results on a PT study, or analysis of client samples with statistically indistinguishable results when compared to another certified analyst. The CDOC must be documented and routed to the QA Department for filing.

12.8 Training Requirements

All training must be performed and documented in accordance with SOP SA-QA-006: *Training Procedures*.

Note: The SOPs listed in the Reference/Cross-Reference Section are applicable to this procedure. All employees performing this procedure must also be trained on these SOPs.

13.0 Pollution Control

It is Eurofins TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (e.g., examining recycling options, ordering chemicals based on quantity needed, preparing reagents based on anticipated usage and reagent stability, etc.). Employees must abide by the policies in Section 13 of the Environmental Health and Safety Manual (EHSM) and the Savannah Addendum to the EHSM.

This procedure has been evaluated for opportunities to minimize the waste generated. Where reasonably feasible, pollution control procedures have been incorporated.

14.0 Waste Management

Waste management practices must be conducted consistent with all applicable federal, state, and local rules and regulations. All waste (i.e., excess reagents, samples, and method process wastes) must be disposed of in accordance with Section 9 of the Eurofins TestAmerica Savannah Addendum to the EHSM. Waste description rules and land disposal restrictions must be followed.

14.1 Waste Streams Produced by the Method

The following waste streams are produced when this method is carried out:

- Excess aqueous samples – Dispose according to characterization on the sample disposal sheets. Neutralize non-hazardous samples before disposal into drain/sewer. Transfer hazardous samples (identified on disposal sheets) to the waste department for disposal.

- Excess soil and solid samples – Dispose according to characterization on sample disposal sheets. Transfer non-hazardous samples to TCLP container for characterization in hazardous waste department. Transfer hazardous samples (identified on disposal sheets) to waste department for disposal.

- Acidic sample digestions – Neutralize before disposal into drain/sewer system.

Dispose of all metals standards with concentrations exceeding the TCLP threshold limits as a RCRA aqueous hazardous waste. The TCLP constituents and the respective threshold limits are:

Analyte	Disposal Limit (mg/L)
Arsenic	5.0
Barium	100
Cadmium	1.0
Chromium	5.0
Lead	5.0
Selenium	1.0
Silver	5.0
Mercury	0.20

Neutralize the standard by passing with sodium bicarbonate before transferring the waste to the RCRA aqueous hazardous waste drum.

All other metals standards may be disposed, using copious amounts of water, down a sink that flows to the neutralization pit.

15.0 References / Cross-References

- SOP SA-AN-100: *Laboratory Support Equipment (Verification and Use)*
- SOP SA-AN-041: *Reagent and Standard Materials Procedures*
- SOP SA-QA-002: *Data Generation and Review*
- SOP SA-QA-005: *Preventive and Corrective Action Procedures*
- SOP SA-QA-006*: Training Procedures*
- SOP SA-QA-007: *Determination and Verification of Detection and Reporting Limits*
- SOP SA-QA-015: *Homogenization, Compositing, and Segregation of Samples*
- SOP SA-QA-016: *Evaluation of Calibration Curves*
- SOP SA-QA-017: *Evaluation of Batch QC Data*
- Eurofins TestAmerica Savannah Quality Assurance Manual
- Eurofins TestAmerica Environmental Health and Safety Manual
- Eurofins TestAmerica Savannah Addendum to the Environmental Health and Safety Manual
- *Methods for Chemical Analysis of Water and Waste*; U.S EPA Office of Research and Development: Cincinnati, OHIO, March 1983.
- EPA 200.7, Revision 4.4, EMMC Version: Determination of Metals and Trace Elements in Water and Wastes by Inductively Coupled Plasma-Atomic Emission Spectrometry; 1994
- *Methods for the Determination of Metals in Environmental Samples*; US EPA Office of Research and Development. Washington, DC.
- *Test Methods for Evaluating Solid Waste, Third Edition*; U.S. EPA Office of Solid Waste and Emergency Response: Washington, D.C., November 1986 (SW-846 IV).
- Method 6010C, Revision 3: Inductively Coupled Plasma-Atomic Emission Spectrometry; February 2007.
- Method 6010D, Revision 4: Inductively Coupled Plasma-Optical Emission Spectrometry; July 2014.
- *Standard Methods for the Examination of Water and Wastewater, Online Edition;* American Public Health Association: Washington, DC.
- SM2340B: Hardness by Calculation (Approved 1997, Editorial Revision 2011)

16.0 Method Modifications/Clarifications

16.1 Incorporation of Non-Routine Matrices

This procedure may be modified to analyze other matrices (e.g., wipe, waste, tissue, filter, and TCLP/SPLP leachate samples) upon client request. This will need to be arranged by the Project Manager at the initiation of the project.

Wipe, waste, filter, and tissue matrices are non-routine, and the laboratory is not currently NELAC certified for these matrices. The laboratory uses its routine soil RLs (converted for initial and final volumes, etc.) and soil QC limits to evaluate wipe, waste, filter, and tissue samples. Soil DOCs can be used to satisfy analyst demonstrations of capability for these types of non-routine matrices. The laboratory uses its routine soil RLs (converted for initial and final volumes, etc.) and soil QC limits to evaluate TCLP/SPLP leachate samples. Water DOCs can be used to satisfy analyst demonstrations of capability for TCLP/SPLP matrices. Teflon chips, Ottawa sand, or equivalent is used as the blank matrix for wipes, wastes, filters, and tissues unless specifically requested otherwise by the project.

16.1.1 Collection and Handling Procedures for Non-Routine Matrices

Waste (oil) samples are collected in 500mL plastic containers. Waste (oil) samples are stored at room temperature until the time of digestion. Wipe samples are collected in 20mL scintillation vials. Wipe and filter samples must be iced at the time of collection and maintained at 4°C (less than 6°C but not frozen) until time of digestion. Waste (oil), wipe, and filter samples must be digested within 6 months of collection.

Tissue samples are collected in plastic containers with the size dependent upon the type of tissue being collected. Plastic jars or plastic baggies can be used. Upon receipt, tissue samples must be placed in the freezer at -10° to -20°C for up to 6 months if digestion cannot be completed that day. Tissue samples must be digested within 6 months of thawing.

Digestates for waste (oil), wipe, tissue, and filter samples are stored at room temperature until the time of analysis and must be analyzed within 6 months of collection.

Once the TCLP/SPLP extraction procedure has been performed, the TCLP/SPLP leachate must be transferred to a 500mL plastic container and preserved with 1.0mL nitric acid to a pH <2. Preserved TCLP/SPLP leachates are stored at room temperature until the time of digestion. The leachate sample must be digested within 6 months of completion of the TCLP/SPLP extraction. Digestates are stored at room temperature until the time of analysis and must be analyzed within 6 months of completion of the TCLP/SPLP extraction.

16.1.2 Preparation and Analytical Procedures for Non-Routine Matrices

Wipe, waste, filter, and tissue samples are prepared in the same manner as routine soil samples as outlined in SOP SA-ME-051. TCLP/SPLP matrices are prepared in the same manner as routine water samples as outlined in SOP SA-ME-050. Refer to the applicable preparation SOPs for more information.

Wipe, waste, filter, tissue, and TCLP/SPLP matrices are analyzed in the same manner as routine samples as outlined in this SOP.

16.2 Other Considerations

16.2.1 The EPA Manual for the Certification of Laboratories Analyzing Drinking Water requires a LFB at the MRL to be performed each day. The laboratory meets this requirement by preparing a low-level LCS (LLCS), spiked at the RL, in each EPA 200.7 batch of drinking water samples. The EPA DW Manual does not specify criteria for the low-level LCS; therefore, the laboratory requires this standard to be qualitatively identified to be acceptable.

16.2.2 The stock standards utilized for the Silica procedure are routinely purchased as Silicon (Si). Both Silica (SiO_2) and Silicon (Si) can be reported using this procedure. The following equation is used to convert between Silicon (Si) and Silica (SiO_2):

$$Si = \frac{SiO_2}{2.14}$$

16.2.3 The laboratory's default QC items incorporate an MSD to satisfy the Clean Water Act requirements and those clients who batch require precision to be reported. Additionally, if insufficient sample volume is provided to perform the MS/MSD, the LCS is routinely prepared in duplicate (i.e., LCS/LCSD).

16.2.4 Linear Range determinations are performed initially upon instrument set-up and when significant instrument changes are made. Linear Range verifications are performed quarterly as required by DOD QSM. This frequency is greater than that defined by the reference methods.

16.2.5 EPA Method 200.7 requires that IEC studies be performed annually. The laboratory defaults to a 6-month frequency. This frequency is greater than that defined by the reference method and is consistent with the requirement outlined in EPA Method 6010C.

16.2.6 The reference methods do not specify a %RSD criteria for replicate exposures of the CCV. The laboratory has incorporated an RSD criteria of 5% for all CCVs, as required by the DOD QSM.

16.2.7 The reference methods require IDLs to be performed annually. The laboratory performs IDLs quarterly as required by the DOD QSM.

16.2.8 The reference methods state that, for dissolved metals analysis, filtration must occur in the field. The laboratory may be requested to filter the samples upon receipt. If this occurs, then appropriate narration must be included in the final report to denote this method deviation.

17.0 Attachments

The following Tables, Diagrams, and/or Validation Data are included as Attachments:

Attachment 1: SOP Summary
Attachment 2: Sample Collection, Preservation, and Holding Time Table
Attachment 3: QC Summary
Attachment 4: Instrument Maintenance and Troubleshooting
Attachment 5: Standard Preparation Tables
Attachment 6: Silica/Silicon Standard Preparation
Attachment 7: Linear Range Determination
Attachment 8: Element Wavelengths
Attachment 9: Hardness Calculation Work Instruction
Attachment 10: Labware Cleaning Procedures

Attachment 1:
SOP Summary

Sample Preparation Summary

Prior to analysis by ICP, the sample must be digested using the sample preparation method appropriate to the matrix. Samples should be prepared according to the appropriate matrix-specific SOP.

Matrix	SOP #
Aqueous Samples	SA-ME-050
Soil Samples	SA-ME-051

Sample Analysis Summary

Sample digestates are aspirated and nebulized into a spray chamber. A stream of argon gas carries the sample aerosol through the innermost of three concentric tubes and injects it into the middle of the donut-shaped plasma. The sample elements are dissociated, atomized, and excited to a higher energy level. As the elements fall to a lower energy level, radiation characteristic of the elements present in the plasma is emitted. The light is directed through an entrance slit, dispersed by the diffraction grating, and projected on to the photomultiplier tube (PMT) or on to a charge-coupled device (CCD). The PMTs and CCDs located behind the exit slits, convert the light energy to an electrical current. This signal is then digitized and processed by the data system. Background correction is required for trace element determination.

Analytical Sequence

Description	Comments
Instrument Warm-up	
Torch Alignment	If torch is changed
Initial Calibration	
High Calibration Standard	Re-analyzed as a sample
ICV	Second Source
ICB	
Reporting Limit Check Standard	
ICP Interference Check Solution A (ICSA)	
ICP Interference Check Solution AB (ICSAB)	
CCV	
CCB	10-analysis count begins after analysis of the CCB
Samples & Batch QC Items	Up to 10 analyses, including QC
CCV	
CCB	10-analysis count begins after analysis of the CCB
Samples & Batch QC Items	Up to 10 analyses, including QC
CCV	
CCB	10-analysis count begins after analysis of the CCB
Samples & Batch QC Items	Up to 10 analyses, including QC
CCV	
CCB	10-analysis count begins after analysis of the CCB
Reporting Limit Check Standard	Method criterion

Note: The analytical sequence continues as outlined above and must end with the analysis of the CCV and CCB. The 10 samples include all QC samples/standards with the exception of CCVs and CCBs.

Attachment 2:
Sample Collection, Preservation, and Holding Time Table

Matrix	Routine Sample Container	Routine Sample Size	Minimum Sample Size	Dechlorination Agent	Thermal Preservation[1]	Chemical Preservation[1]	Holding Time[2]
Water	250mL or 500mL plastic	50mL	25mL	Not Applicable	None required	1:1 Nitric Acid to pH<2	6 months
Water (Silica)	250mL or 500mL plastic	10mL	5mL	Not Applicable	4°C	Not Applicable	6 months
Soil	4oz plastic or glass	1g	0.50g	Not Applicable	4°C	Not Applicable	6 months
Soil (Silica)	4oz plastic or glass	5g	2.5g	Not Applicable	4°C	Not Applicable	6 months

[1]Samples requiring dissolved metals must be filtered prior to preservation.
[2] Inclusive of digestion and analysis.
[3]Samples must be collected on ice and maintained at 0-6 °C with no frozen samples.

Attachment 3:
QC Summary

QC Item	Frequency	Criteria	Corrective Action
Initial Calibration (ICAL)	Daily	Single Point: 1 standard and 1 blank Multi-point: Minimum 3 standards and 1 blank Correlation coefficient ≥0.998	Recalibrate
High Standard	Immediately after every calibration	Recoveries within ± 5% of expected values	New initial calibration
Initial Calibration Verification Standard (ICV)	At the beginning of the analysis	EPA 6010C: Within ±10% EPA 200.7: Within ± 5%, </= 3%RSD	Recalibrate
Continuing Calibration Verification Standard (CCV)	At the beginning and end of the analysis, and every 10 samples	Within ±10% of true value </=5%RSD	Terminate the analysis, correct the problem and reanalyze the previous 10 samples.
Calibration Blank (ICB/CCB)	After ICV and every CCV	Absolute value <½RL	Terminate the analysis, correct problem and reanalyze the previous 10 samples
Interference Check Standards (ICSA/ICSAB)	At the beginning of an analysis run	± 20% of the true values Pay attention to false positives and false negatives for elements not present in the solutions.	Terminate the analysis, correct the problem, recalibrate, and reanalyze all samples since the last ICS that was in control.
Batch Definition	Up to 20 field samples digested together within a 24-hr time period	Not Applicable	Not Applicable
Method Blank	One per batch of twenty samples or less	|result| <½RL or result <10% of the analyte level in the sample	Refer to SOP SA-QA-017

QC Item	Frequency	Criteria	Corrective Action
(MB)			
Laboratory Control Sample (LCS)	One per batch	TALS MLG	Refer to SOP SA-QA-017
Laboratory Control Sample Duplicate (LCSD)	One per batch, if insufficient sample provided for MS/MSD	TALS MLG	Refer to SOP SA-QA-017
Low-Level Laboratory Control Sample (LLCS) – at the RL	EPA 200.7 DW Only: One per batch of twenty samples or less for drinking water	Qualitatively detected	If the "regular" LCS meets criteria, initiate NCM and report data If the "regular" LCS does not meet criteria, redigest and reanalyze batch
Matrix Spike (MS)	EPA 200.7: 10% of samples prepared; i.e., 2 separate matrix spikes per batch EPA 6010C: 5% of samples prepared; i.e., 1 matrix spike per batch	TALS MLG	Refer to SOP SA-QA-017
Matrix Spike Duplicate (MSD)	One per batch	TALS MLG	Refer to SOP SA-QA-017
Serial Dilution (1/5 Dilution)	One per batch	Refer to Section 9.2.9	Refer to Section 9.2.9
Post Digestion Spike (PDS)	One per batch	Refer to Section 9.2.10	Refer to Section 9.2.10
Reporting Limit Check	At the beginning and end of an analysis run (Required for EPA	EPA 200.7: +/-50% of the true concentration	Opening:

QC Item	Frequency	Criteria	Corrective Action
Solution (also known as LLICV and LLCCV for EPA 6010C/6010D)	200.7 when using a single point calibration. Required for EPA 6010C when using both single point and multi-point calibrations.6010D and DOD do not require a closing analysis)	EPA 6010C: +/-30% of the true concentration 6010D +/-20% of TV; RSD < 20%	Stop the analysis, fix the problem, and reanalyze the affected samples. Capping: Qualify affected samples; reanalyze if required by project plan
%RSD (CV) of Multiple Exposures	Evaluate for all Calibration, QC, and samples	Conc. >= RL - Warning: <=20% - Acceptance: <=30%	-if CV>20 but <=30, review data for possible interferences; -if interference present, reanalyze digest -if no interference present, report average -if CV>=30%, reanalyze digest, report the result that has the lower precision value or dilute the digestion and reanalyze
		Conc. < RL -use professional judgment	-use professional judgment
Linear Range	Determined at least every 6 months in accordance with Attachment 7	% difference </=10%	-reanalyze at a lower concentration
Interelement Correction Factors (IEC)	Determined every 6 months. Verified at the beginning of an analysis run	Refer to ICSA, ICSAB criteria	See ICSA, ICSAB corrective action
Lower Limit of Quantitation Check Sample	Annually	EPA 6010C: 70-130%	- reanalyze at a higher concentration - elevate RL accordingly
Initial Demonstration of Capability (IDOC)	Initially, per analyst, per analyte/method/matrix combination	Refer to SOP SA-QA-006	Refer to SOP SA-QA-006 Note: Unsupervised work must not begin

QC Item	Frequency	Criteria	Corrective Action
			until acceptable IDOC is obtained.
Continuing Demonstration of Capability (CDOC)	Annually, per analyst, per analyte/method combination	Refer to SOP SA-QA-006	Refer to SOP SA-QA-006
Reporting Limit Verification (RLV)	Upon method/instrument set-up, per analyte/method/matrix combination. Then quarterly thereafter (for DOD ELAP) or annually thereafter (for non-DOD ELAP)	Refer to SOP SA-QA-007	Refer to SOP SA-QA-007
Method Detection Limit Study (MDL)	Upon method/instrument set-up, per analyte/method/matrix combination	Refer to SOP SA-QA-007	Refer to SOP SA-QA-007
MDL Verification (MDLV)	Upon method/instrument set-up, per analyte/method/matrix combination. Then quarterly thereafter (for DOD ELAP) or annually thereafter (for non-DOD ELAP)	Refer to SOP SA-QA-007	Refer to SOP SA-QA-007
Instrument Detection Limit (IDL)	Upon method/instrument set-up, and then quarterly thereafter	Refer to SOP SA-QA-007	Refer to SOP SA-QA-007

Attachment 4:
Preventative Maintenance and Troubleshooting

Preventive Maintenance

EQUIPMENT ITEM	Service Interval							SERVICE LEVEL
	D	W	M	Q	SA	A	AN	
ICAP Service Schedule								
Pump Tubing							X	Inspect and replace as needed (Recommend daily)
Nebulizer							X	Clean as needed
Filters							X	Clean or replace as needed (Recommend monthly)
Chiller Water Filter							X	Clean or replace as needed (Recommend every six months)
Injector Tip / Torch							X	Clean or replace as needed (Recommend Daily inspection)
Tubing Connectors							X	Replace as needed

D = daily; W = Weekly; M = monthly; Q = Quarterly; SA = semi-annually; A = annually; AN = as needed

Troubleshooting

Troubleshooting should be documented as outlined above. If possible, troubleshooting is best performed in a step-wise manner to systematically isolate instrument components. Refer to the instrument manufacturer's guides for specific information and strategies. Enlist assistance from technical and/or department management as needed.

Maintenance Log

A maintenance log must be established for each piece of equipment used in the laboratory. All maintenance that is performed on the instrument must be recorded in the log including:

- Type of maintenance performed (Note: This includes preventative/routine maintenance; non-routine maintenance; maintenance performed by an external vendor; updates to software versions; etc.)
- Analyst or technician performing the maintenance
- Date the maintenance was performed
- Detailed explanation of the reason for the maintenance
- Resolution of the problem and return to control
  all service calls from instrument representatives
- Resolution of the problem (i.e., the Return to Control)

Note: An electronic Maintenance Logbook has been established for these instruments.

Instrument Labeling

Each instrument must be labeled with its name or ID (e.g., MSA, ICP-D, etc.). Additionally, non-operational instruments must be isolated from service or marked as being out of service. Each piece of equipment has an "Operational / Not Operational" sticker that is used for this purpose.

Attachment 5:
Standard Preparation

Note: All standards must be stored at room temperature and have an expiration date of 6 months from date prepared.

Continuing Calibration Verification (CCV)

Final Volume (mL): 2000

Solvent: 5% HCL / 1 % HNO3

Purchased Standards	Concentration (mg/L)	Vendor *	Volume Used (mL)	Final Concentration (mg/L)
Single Element Stocks				
Ag	1000	CPI	1.0	0.50
Al	10000	SPEX	1.0	5.0
B	1000	CPI	10.0	5.0
Fe	10000	CPI	1.0	5.0
Sr	1000	ABSOLUTE	5.0	2.5
Ti	1000	CPI	1.0	0.50
Na	10000	CPI	0.50	2.5
Sb	1000	CPI	1	0.5
Si	1000	CPI	10	5.0
Multi Element Mixes				
CAL STD 2				
As	50	CPI	20	0.50
Mo	50			0.50
Pb	50			0.50
Sb	50			0.50
Se	500			5.0
Tl	500			5.0
CAL STD 3				
Ba	500	CPI	20	5.0
Be	50			0.50
Cd	50			0.50
Co	50			0.50
Cr	500			5.0
Cu	500			5.0
Mn	500			5.0
Ni	250			2.5
Zn	250			2.5

CAL STD 5				
Ca	500	CPI	20	5.0
K	1000			10
Mg	500			5.0
Na	500			5.0
Sn	500			5.0
V	500			5.0

Internal Standard (ISTD)

Final Volume (mL): 10000

Solvent: 30% HNO3

Purchased Standards	Concentration (mg/L)	Vendor *	Amount Used	Final Concentration (mg/L)
Y	10000	CPI	7.0mL	7.0
Lithium Carbonate (powder)	18.78% Li	Mallinckrodt	5.0g	94.65
Sc	10000	CPI	7.0mL	7.0

MDL & IDL Intermediate
Final Volume:
(mL) 100
Solvent: 5% HCL / 1 % HNO3

Purchased Standards	Concentration (mg/L)	Vendor *	Volume Used (mL)	Final Concentration (mg/L)
Single Element Stocks				
Ag	1000	CPI	0.040	0.40
Al	10000	SPEX	0.10	10
As	1000	CPI	0.20	2.0
B	1000	CPI	0.40	4.0
Ba	1000	CPI	0.020	0.20
Be	1000	CPI	0.0050	0.10
Ca	10000	SPEX	0.10	10
Cd	1000	CPI	0.040	0.40
Co	1000	CPI	0.10	1.0
Cr	1000	CPI	0.10	1.0
Cu	1000	CPI	0.10	1.0
Fe	10000	CPI	0.10	10
K	10000	CPI	0.20	20
Mg	10000	SPEX	0.10	10
Mn	1000	CPI	0.020	0.20
Mo	1000	CPI	0.10	1.0
Na	10000	CPI	2.04	204
Ni	1000	CPI	0.10	1.0
Pb	1000	CPI	0.10	1.0
Sb	1000	CPI	0.20	2.0
Se	1000	CPI	0.20	2.0
Sn	1000	CPI	0.20	2.0
Sr	1000	CPI	0.040	0.40
Ti	1000	CPI	0.050	0.50
Tl	1000	CPI	0.20	2.0
V	1000	CPI	0.060	0.60
Zn	1000	CPI	0.10	1.0

ICP-MS LCS/MATRIX SPIKE SOLUTION

Preparation of the ICP-MS Matrix Spiking / Post Digestion Spiking Solution

Element	Parent Concentration (mg/L)	Volume Added (mL)	Final Volume (mL)	Final Concentration (mg/L)
Aluminum (Al)	10000	5.0	100	500
Antimony (Sb)	1000	0.5		5
Arsenic (As)	1000	1.0		10
Boron (B)	1000	2.0		20
Barium (Ba)	1000	1.0		10
Beryllium (Be)	1000	0.5		5.0
Cadmium (Cd)	1000	0.5		5.0
Calcium (Ca)	10000	5.0		500
Cobalt (Co)	1000	1.0		5.0
Chromium (Cr)	1000	1.0		10
Copper (Cu)	1000	1.0		10
Iron (Fe)	10000	5.0		500
Lead (Pb)	1000	0.50		5.0
Magnesium (Mg)	10000	5.0		500
Manganese (Mn)	1000	5.0		50
Mercury (Hg)	1000	0.050		0.50
Molybdenum Mo)	1000	1.0		10
Nickel (Ni)	1000	1.0		10
Potassium (K)	10000	5.0		500
Selenium (Se)	1000	1.0		10
Silver (Ag)	1000	0.50		5.0
Sodium (Na)	10000	5.0		500
Strontium (Sr)	1000	1.0		10
Thallium (Tl)	1000	0.4		4.0
Tin (Sn)	1000	1.0		10
Titanium (Ti)	1000	1.0		10
Vanadium (V)	1000	1.0		10
Zinc (Zn)	1000	1.0		10

Separate mixtures can be utilized for stability considerations.

RL ICP INTERMEDIATE

Final Volume (mL): 100

Solvent: 5% HCL / 1 % HNO3

Purchased Standards	Concentration (mg/L)	Vendor *	Volume Used (mL)	Final Concentration (mg/L)
Single Element Stocks				
Ag	1000	CPI	0.10	1.0
Al	10000	SPEX	0.20	20
As	1000	CPI	0.20	2.0
B	1000	CPI	1.0	10
Ba	1000	CPI	0.10	1.0
Be	1000	CPI	0.040	0.4
Ca	10000	SPEX	0.50	50
Cd	1000	CPI	0.050	0.5
Co	1000	CPI	0.10	1.0
Cr	1000	CPI	0.10	1.0
Cu	1000	CPI	0.20	2.0
Fe	10000	CPI	0.050	5.0
K	10000	CPI	1.0	100
Mg	10000	SPEX	0.50	50
Mn	1000	CPI	0.10	1.0
Mo	1000	CPI	0.10	1.0
Na	10000	CPI	1.0	100
Ni	1000	CPI	0.40	4.0
Pb	1000	CPI	0.10	1.0
Sb	1000	CPI	0.20	2.0
Se	1000	CPI	0.20	2.0
Sn	1000	CPI	0.50	5.0
Sr	1000	CPI	0.10	1.0
Ti	1000	CPI	0.10	1.0
Tl	1000	CPI	0.25	2.5
V	1000	CPI	0.10	1.0
Zn	1000	CPI	0.20	2.0

RL ICP Working
Final Volume (mL): 100
Solvent: 5% HCL / 1 % HNO3

Purchased Standards	Concentration (mg/L)	Vendor *	Volume Used (mL)	Final Concentration (mg/L)
From RL ICP Intermediate				
Ag	1.0	CPI	1.0	0.010
Al	20	SPEX		0.20
As	2.0	CPI		0.020
B	10	CPI		0.10
Ba	1.0	CPI		0.010
Be	0.4	CPI		0.0040
Ca	50	SPEX		0.50
Cd	0.5	CPI		0.0050
Co	1.0	CPI		0.010
Cr	1.0	CPI		0.010
Cu	2.0	CPI		0.020
Fe	5.0	CPI		0.050
K	100	CPI		1.0
Mg	50	SPEX		0.50
Mn	1.0	CPI		0.010
Mo	1.0	CPI		0.010
Na	100	CPI		1.0
Ni	4.0	CPI		0.040
Pb	1.0	CPI		0.010
Sb	2.0	CPI		0.020
Se	2.0	CPI		0.020
Sn	5.0	CPI		0.050
Sr	1.0	CPI		0.010
Ti	1.0	CPI		0.010
Tl	2.5	CPI		0.025
V	1.0	CPI		0.010
Zn	2.0	CPI		0.020

ICV (different vendor used for ICV)

Final Volume (mL): 100

Solvent: 5% HCL / 1 % HNO3

NOTE: Must be made fresh daily.

Purchased Standards	Concentration (mg/L)	Vendor *	Volume Used (mL)	Final Concentration (mg/L)
Single Element Stocks				
Na	1000	Ultra	0.09	9.0
Sn	1000	Ultra	0.50	5.0
Sr	1000	Ultra	0.05	5.0
Multi Element Stocks				
Ultra26				
As	100	Ultra	1.0	1.0
Be	100			1.0
Ca	100			1.0
Cd	100			1.0
Co	100			1.0
Cr	100			1.0
Cu	100			1.0
Fe	100			1.0
Mg	100			1.0
Mn	100			1.0
Mo	100			1.0
Ni	100			1.0
Pb	100			1.0
Sb	100			1.0
Se	100			1.0
Tl	100			1.0
V	100			1.0
Zn	100			1.0
Ultra 26 cont				
Ag	100	Ultra	1.0	1.0
Al	100			1.0
B	100			1.0
Ba	100			1.0
K	1000			10
Na	100			1.0
Si	50			0.50

ICSA

Final Volume (mL): 100

Solvent: 5% HCL / 1 % HNO3

Purchased Standards	Concentration (mg/L)	Vendor *	Volume Used (mL)	Final Concentration (mg/L)
Multi Element Mixes				
Interferents A				
Al	5000	SPEX	10	500
Ca	5000			500
Mg	5000			500
Fe	2000			200

ICSAB
Final Volume (mL): 100
Solvent: 5% HCL / 1 % HNO3

Purchased Standards	Concentration (mg/L)	Vendor *	Volume Used (mL)	Final Concentration (mg/L)
Multi Element Mixes				
Interferents A				
Al	5000	SPEX	10	500
Ca	5000			500
Mg	5000			500
Fe	2000			200
Altany C				
Ag	20	SPEX	1.0	0.20
As	10			0.10
Ba	50			0.50
Be	50			0.50
Cd	100			1.0
Co	50			0.50
Cr	50			0.50
Cu	50			0.50
Mn	50			0.50
Ni	100			1.0
Pb	5			0.050
Sb	60			0.60
Se	5			0.050
Tl	10			0.10
V	50			0.50
Zn	100			1.0
Trace AB				
Mo	100	CPI	1.0	1.0
Sn	100			1.0

Trace AB
Final Volume (mL): 100
Solvent: 5% HCL / 1 % HNO3

Purchased Standards	Concentration (mg/L)	Vendor *	Volume Used (mL)	Final Concentration (mg/L)
Single Element Stocks				
Mo	1000	CPI	10	100
Sn	1000	CPI	10	100

High Std
Final Volume (mL): 1000
Solvent: 5% HCL / 1 % HNO3

Purchased Standards	Concentration (mg/L)	Vendor *	Volume Used (mL)	Final Concentration (mg/L)
Single Element Stocks				
Ag	1000	CPI	1.0	1.0
Al	10000	SPEX	1.0	10
B	1000	CPI	10	10
Fe	10000	CPI	1.0	10
Na	10000	CPI	0.50	5.0
Sr	1000	ABSOLUTE	5.0	5.0
Ti	1000	CPI	1.0	1.0
Sb	1000	CPI	1.0	1.0
Si	1000	CPI	10	10
Multi Element Mixes				
CAL STD 2				
As	50	CPI	20	1.0
Mo	50			1.0
Pb	50			1.0
Sb	50			1.0
Se	500			10
Tl	500			10

CAL STD 3				
Ba	500	CPI	20	10
Be	50			1.0
Cd	50			1.0
Co	50			1.0
Cr	500			10
Cu	500			10
Mn	500			10
Ni	250			5.0
Zn	250			5.0

CAL STD 5				
Ca	500	CPI	20	10
K	1000			20
Mg	500			10
Na	500			10
Sn	500			10
V	500			10

Attachment 6:
Silica/Silicon Standard Preparation

Silicon (Si) stock solutions are usually purchased for this procedure. The following conversion is used to adjust any volumes or concentrations appropriately:

$$Si = \frac{SiO_2}{2.14}$$

- Stock SiO_2 Standard, 10000mg/L Si / 21400mg/L SiO_2 – purchased from CPI. Store at room temperature. This standard must be used by the manufacturer's expiration date.

- Stock Second Source SiO_2 Initial Calibration Verification (ICV), 50mg/L Si / 107mg/L SiO_2 QC7 Standard, purchased from Ultra. Store at room temperature. This standard must be used by the manufacturer's expiration date.

  Note: The ICV must be prepared from a stock standard that is obtained from a different source than the stock standard used to prepare the calibration standard. The second source must be from a separate vendor, unless a second vendor is unavailable, in which case a separate lot from the same vendor may be used.

- Intermediate SiO_2 Standard, 467mg/L Si / 1000mg/L SiO_2 – Add 20mL to 30mL of reagent water to a clean, plastic 100mL volumetric flask. Add the volume of the Stock SiO_2 Standard given in the table below to the volumetric flask. Dilute to volume with reagent water. Store the standard at room temperature. This standard must be used by its parent standard's expiration date or within 6 months of preparation, whichever comes first.

Element	Conc. Stock SiO_2 Std (mg/L)	Volume Stock SiO_2 Std (mL)	Final Volume (mL)	Final Conc. (mg/L)
Silica (SiO_2) [Silicon (Si)]	21400 [10000]	4.67	100	1000 [467]

- Initial Calibration Standards

  - Preparation of the Calibration Blank (ICB, CCB) – The calibration blank is reagent water. The calibration blank is used as the initial calibration blank (ICB) and the continuing calibration blank (CCB).

  - High Level SiO_2 Calibration Standard, 10mg/L SiO_2 – Add 20mL to 30mL of reagent water to a clean, plastic 100-mL volumetric flask. Add the volume of the Intermediate SiO_2 Standard given in the table, below, to the volumetric flask. Dilute to a final volume of 100mL with reagent water. Store the standard at room temperature. This standard must be used by its parent standard's expiration date or within 6 months of preparation, whichever comes first.

Element	Conc. SiO_2 Intermediate Std (mg/L)	Volume SiO_2 Intermediate Std (mL)	Final Volume (mL)	Final Conc. (mg/L)
Silica (SiO_2)	1000	1.0	100	10

- Mid Level SiO_2 Calibration Standard (also used as CCV solution), 5.0mg/L SiO_2 – Add 20mL to 30mL of reagent water to a clean, plastic 100mL volumetric flask. Add the volume of the stock standard given in the table, below, to the volumetric flask. Dilute to volume with reagent water. Store the standard at room temperature. This standard must be used by its parent standard's expiration date or within 6 months of preparation, whichever comes first.

Element	Conc. SiO_2 Intermediate Std (mg/L)	Volume SiO_2 Intermediate Std (mL)	Final Volume (mL)	Final Conc. (mg/L)
Silica (SiO_2)	1000	0.50	100	5

- Low Level SiO_2 Calibration Standard (also used as RL solution), 0.50mg/L – Add 20mL to 30mL of reagent water to a clean, plastic 100mL volumetric flask. Add the volume of the stock standard given in the table, below, to the volumetric flask. Dilute to volume with reagent water. Store the standard at room temperature. This standard must be used by its parent standard's expiration date or within 6 months of preparation, whichever comes first.

Element	Conc. SiO_2 Intermediate Std (mg/L)	Volume SiO_2 Intermediate Std (mL)	Final Volume (mL)	Final Conc. (mg/L)
Silica (SiO_2)	1000	0.050	100	0.50

- Second Source SiO_2 ICV Standard, 0.50mg/L SiO_2 / 1.07mg/L Si – Add 20mL to 30mL of reagent water to a clean, plastic 100mL volumetric flask. Add the volume of the stock standard given in the table to the volumetric flask. Dilute to volume with reagent water. Store the standard at room temperature. This standard must be used by its parent standard's expiration date or within 6 months of preparation, whichever comes first.

Element	Conc. of Stock Std (currently from Element) (mg/L)	Volume Stock Std (mL)	Final Volume of Cal Std mL)	Final Conc. (mg/L)
Silica (SiO_2) [Silicon (Si)]	107 [50]	1.0	100	1.07 [0.50]

Stock ICP Interference A Check Standard – purchased Interference A Stock Standard, from SPEX. Store the standard at room temperature. This standard must be used by its manufacturer's expiration date.

Note: This solution does not contain any SiO_2. Concentrations are as follows:

Aluminum (Al)	5000mg/L
Calcium (Ca)	5000mg/L
Magnesium (Mg)	5000mg/L
Iron (Fe)	2000mg/L

- ICP Interference A Check Standard, Working – Add 20mL to 30mL of reagent water to a clean 100-mL plastic volumetric flask. Add 10mL of the Stock ICP Interference A Check Standard to the volumetric flask. Dilute to a final volume of 100mL with reagent water. Store the standard at room temperature. This standard must be used by its parent standard's expiration date or within 6 months of preparation, whichever comes first.

  Note: This solution does not contain any SiO_2. Final concentrations are as follows:

Aluminum (Al)	500mg/L
Calcium (Ca)	500mg/L
Magnesium (Mg)	500mg/L
Iron (Fe)	200mg/L

- ICP Interference Check Solution AB – Add 20mL to 30mL of reagent water to a clean 100-mL plastic volumetric flask. Add 10mL of Stock ICP Interference A Check Standard and 0.50mL of the Intermediate SiO_2 Standard to the volumetric flask. Dilute to a final volume of 100mL with reagent water. Store the standard at room temperature. This standard must be used by its parent standard's expiration date or within 6 months of preparation, whichever comes first.

  The final concentrations of this solution should be as follows:

Aluminum (Al)	500mg/L
Calcium (Ca)	500mg/L
Magnesium (Mg)	500mg/L
Iron (Fe)	200mg/L
Silica (SiO_2)	5.0mg/L

- Linearity Check Solution, 100mg/L – P Add 20mL to 30mL of reagent water to a clean 100-mL plastic volumetric flask. Add 10mL of Intermediate SiO_2 Standard to the volumetric flask. Dilute to a final volume of 100mL with reagent water. Store the standard at room temperature. This standard must be used by its parent standard's expiration date or within 6 months of preparation, whichever comes first.

Note: Historically, the Linear Dynamic Range for silica has been 100mg/L; however, the instrument is dynamic. The concentration of this standard may need to be adjusted based how the instrument is performing. Prepare other volumes and/or concentrations according to the following equation:

$$Vs = \frac{Vlc \otimes Clc}{Cs}$$

where
V_s = volume of stock standard (mL)
C_s = concentration of stock standard (mg/L)
V_{lc} = volume of linearity check standard to prepare (mL)
C_{lc} = concentration of linearity check standard to prepare (mg/L)

Attachment 7:
Linear Dynamic Range Determination and Verification

Initial Linear Dynamic Range Determination:
The linear dynamic range (LDR) of each instrument must be determined initially, upon instrument set-up, and whenever significant instrument changes are made following the procedure outlined below:

- Prepare and analyze individual standards at concentrations (a minimum of three are recommended) that are expected to define the linear dynamic range of the instrument.

  Note: The calibration standards and the LDR standards must be matrix matched; that is, they must have the same percentage of hydrochloric and nitric acids.

- Compare the concentration of each standard with its true concentration as follows:

$$PercentDifference = \left| \frac{C_{cal} - C_{true}}{C_{true}} \right| \otimes 100$$

  Where:
  C_{cal} = concentration determined from analysis
  C_{true} = true concentration of the standard

- Continue analyzing increasing concentrations of standards (recommend increasing by a factor of two) until the percent difference is greater than 10%. The highest standard that meets the 10% criteria establishes the initial LDR of the instrument.

Note: The laboratory routinely utilizes a “working” LDR that is equivalent to the concentration of the High Standard of the calibration curve. This working LDR allows for consistency between instruments and must not be greater than the LDR established by the initial LDR study.

Linear Dynamic Range Verification:
The working LDR must be verified quarterly by analyzing a standard at this concentration. If the percent difference is less than or equal to 10%, the working LDR is confirmed at that concentration. If the percent difference is greater than 10%, repeat the initial LDR Study as outlined above.

Evaluation of Samples with Relation to the Linear Dynamic Range:
Samples with concentrations greater than 90% of the working LDR must be diluted to bring the concentration below 90% of the working LDR.

Attachment 8:
Element Wavelengths

Element	Wavelength ICP-E (nm)	Wavelength ICP-F (nm)
Aluminum (Al)	308.215	308.215
Antimony (Sb)	206.834	206.834
Arsenic (As)	188.98	188.98
Barium (Ba)	389.178	389.178
Beryllium (Be)	313.042	313.042
Boron (B)	249.678	249.678
Cadmium (Cd)	226.502	226.502
Calcium (Ca)	370.602	370.602
Chromium (Cr)	267.716	267.716
Cobalt (Co)	228.615	228.615
Copper (Cu)	324.754	324.754
Iron (Fe)	271.441	271.441
Lead (Pb)	220.353	220.353
Magnesium (Mg)	279.078	279.078
Manganese (Mn)	257.610	257.610
Molybdenum (Mo)	202.032	202.032
Nickel (Ni)	231.604	231.604
Potassium (K)	766.491	766.491
Selenium (Se)	196.026	196.026
Silver (Ag)	328.068	328.068
Sodium (Na)	330.237	330.237
Strontium (Sr)	216.596	216.596
Thallium (Tl)	190.794	190.794
Tin (Sn)	189.925	189.925
Titanium (W)	334.941	334.941
Vanadium (V)	292.401	292.401
Zinc (Zn)	206.200	206.200

Note: Other wavelengths may be used with DM and TM approval.

Attachment 9:
Hardness Calculation Work Instruction

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Work Instruction
FME157:05.23.07:0
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CALCULATION OF HARDNESS

(as Calcium Carbonate)

Method: SM2340B

Summary of Procedure:

An aqueous sample is analyzed for calcium and magnesium using ICP by methods EPA 200.7 or 6010B (SOP ME70: *Elements by ICP)*, or using ICP/MS by methods EPA 200.8 or 6020 (SOP ME74*: Elements by ICP/MS)*. The concentrations of these elements are converted to equivalents of calcium carbonate using the equation below and added together to give the final result.

Calculation:

Determine the concentration of calcium and magnesium as described in SOP ME70 or ME74.

Calculate the hardness as calcium carbonate using the following equation:

Hardness (as mg/L calcium carbonate) = (4.118 X [Mg]) + (2.497 X [Ca])

where:
[Mg] = Concentration of magnesium (mg/L)
[Ca] = Concentration of calcium (mg/L)

Quality Control:

Report the method blank as the default QC unless otherwise requested.

Formal detection limit studies, as described in 40CFR Part 136B and SOP QA07: *Determination of Detection Limits,* are not required for SM2340B.

This procedure is a calculation based on another analytical procedure; therefore, demonstration of capability (DOC) is accomplished by successful completion of analytical DOCs. (i.e. If the analyst has a successful DOC on file for Ca and Mg by EPA 200.7/6010B or EPA 200.8/6020, proficiency has been shown for SM2340B by default.)

Attachment 10: Labware Cleaning Procedures

GLASSWARE CLEANING PROCEDURES METALS DEPARTMENT
Graduated Cylinders 1. Scrub with hot, soapy H_2O and brush. 2. Rinse thoroughly with tap H2O. 3. Rinse with 10% HNO_3 4. Rinse thoroughly with DI H_2O.
Volumetric Flasks 1. Empty contents of flask 2. Squirt a small amount of cleaning detergent directly into volumetric flask 3. Fill flask 1/3 full with **HOT** H_2O 4. Replace top and shake flask. 5. Empty flask and rinse with **HOT** H_2O until no soap remains in flask. 6. Add approximately 10mL concentrated HNO_3 to 50mL, 100mL, and 250mL flasks ---- replace top and shake well. For 500mL or 1000mL flasks use 25mL and for 10mL flasks use 2 - 5mL of concentrated HNO_3. 7. Rinse 3 times with DI H_2O, filling flask 1/3 full and replacing top. Store until needed. * **NEVER PLACE VOLUMETRIC FLASKS OR TOPS IN SINK OR DISHPAN WITH OTHER DIRTY DISHES.**
***Dispose of all acid waste in accordance with the Eurofins TestAmerica Savannah Addendum to the Corporate Safety Manual.**

17.0 Revision History

Summary of Changes

- Added section 6.3 to address the Master Equipment Listing maintained by the QA department.

Summary of Changes from Previous Revision 18:

- Minor editorial, grammatical, and/or formatting changes made.
- Updated SOP signatories to reflect current responsibilities and titles.
- Updated company name and logo as applicable.
- Updated forms for spelling and logo changes.

ELEMENTS BY ICP-MS

(Methods: EPA 200.8, EPA 6020A, and EPA 6020B)

Approvals (Signature/Date):

Kimberly Chamberlain 11/25/2020

Kimberly Chamberlain Date
Quality Assurance Manager / Environmental Health & Safety Coordinator

11/25/2020

Adriana Geiger Date
Department Manager

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1.0 Scope and Application

This SOP gives the procedures for the determination of various metals by inductively coupled plasma mass spectrometry (ICP-MS).

Note: The routine matrices for this procedure are waters and soils; however, this procedure may be adapted to accommodate other matrices as outlined in Section 16.1.

A complete target analyte list, the reporting limits (RL), the method detection limits (MDL), and the accuracy and precision criteria associated with this procedure are provided in the TALS Method Limit Groups (MLGs).

This SOP was written by and for Eurofins TestAmerica's Savannah laboratory.

2.0 Summary of Method

Prior to analysis by ICP-MS, the sample must be solubilized or digested using the sample preparation method appropriate to the matrix. Sample digestates are aspirated and nebulized into a spray chamber. A stream of argon gas carries the sample aerosol through the innermost of three concentric tubes and injects it into the middle of the donut-shaped plasma. The sample elements are dissociated, atomized, and excited to a higher energy level. The ions that are produced are entrained in the plasma gas and introduced, by means of an interface, into a mass spectrometer. The ions are sorted according to their mass to charge ratios and quantified with a channel mass spectrometer.

This SOP is based on the following methods: EPA Method 200.8, EPA Method 6020A, and EPA Method 6020B.

3.0 Definitions

Refer to the Glossary Section of the *Quality Assurance Manual* (QAM) for a complete listing of applicable definitions and acronyms.

4.0 Interferences

4.1 Procedural Interferences

4.1.1 Interferences may be caused by contaminants in solvents, reagents, glassware, and other sample processing apparatus and can make identification and/or quantification of the target analytes difficult.

4.1.2 All sample collection containers are single-use disposable containers which limits the potential for contamination. All non-disposable labware must be scrupulously cleaned in accordance with the posted Labware Cleaning Instructions to ensure it is free from contaminants and does not contribute artifacts.

4.1.3 High purity reagents and solvents are used to help minimize interference problems. Hydrochloric acid and nitric acid must be verified prior to use in accordance with the Eurofins TestAmerica Solvent Lot Testing Program.

4.1.4 Instrument and/or method blanks are routinely used to demonstrate all reagents and apparatus are free from interferences under the conditions of the analysis.

4.2 Matrix Interferences

4.2.1 Matrix interferences may be caused by contaminants that are co-extracted from the sample matrix. The sample may require cleanup such as filtration or dilution prior to analysis to reduce or eliminate the interferences.

4.2.2 Interfering contamination may occur when a sample containing low concentrations of analytes is analyzed immediately following a sample containing relatively high concentrations of analytes. As such, samples known to be clean should be analyzed first. To prevent carryover into subsequent samples, analysis of reagent blanks may be needed after the analysis of a sample containing high concentrations of analytes.

4.2.3 Isobaric elemental interferences in the ICP-MS are caused by isotopes of different elements forming atomic ions with the same nominal mass-to-charge ratio (m/z) as the target analyte. These can be managed by the selection of an alternate isotope or by the use of elemental interference equations. Most isobaric interferences that could affect the ICP-MS analysis for elements in this SOP have been identified. The basic elemental interference equations are based on natural isotopic abundances. The most precise coefficients for an instrument must be determined from the ratio of the net isotope signals that are observed for a known standard solution at a concentration sufficient to produce suitable counting statistics.

4.2.4 Physical interferences are effects associated with the sample nebulization and transport processes as well as ion-transmission efficiencies. Changes in viscosity can cause significant inaccuracies, especially in samples containing high concentrations of dissolved solids or high acid concentrations. These changes in matrix can cause significant signal suppression or enhancement. Dissolved solids can deposit on nebulizer tips and interface cones (reducing the orifice size and the instrument's performance). Internal standards can be used to correct for physical interferences if they are carefully matched to the analyte so that both elements react similarly to the matrix changes.

4.2.5 Memory interferences can occur when analytes from a previous sample contribute to signals measured from subsequent samples. The memory effects can result from analyte deposition of sample on the sample tubing, joints, nebulizer, spray chamber, torch, and/or interface cones. Routine maintenance on the sample introduction system is necessary in order to minimize the memory interferences. The memory effects must be taken into account when setting up a suitable rinse times. The evaluation of a minimum of three replicate integrations will help to determine memory problems.

5.0 Safety

Employees must abide by the policies and procedures in the Eurofins TestAmerica Environmental Health and Safety Manual (EHSM), the Eurofins TestAmerica Savannah Addendum to the EHSM, and this document.

This procedure may involve hazardous materials, operations, and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user to follow appropriate safety, waste disposal, and health practices under the assumption that all samples and reagents are potentially hazardous.

The analyst must protect himself/herself from exposure to the sample matrix. Many of the samples that are tested may contain hazardous chemical compounds or biological organisms. The analyst must, at a minimum, wear protective clothing (lab coat), eye protection (safety glasses or face shield), disposable gloves, and closed-toe, nonabsorbent shoes when handling samples.

5.1 Specific Safety Concerns or Requirements

Nitric and hydrochloric acids are extremely hazardous as oxidizers, corrosives, poisons, and are reactive. Inhalation of the vapors can cause coughing, choking, irritation of the nose, throat, and respiratory tract, breathing difficulties, and lead to pneumonia and pulmonary edema. Contact with the skin can cause severe burns, redness, and pain. Nitric acid can cause deep ulcers, and staining of the skin to a yellow or yellow-brown color. These acid vapors are irritating and can cause damage to the eyes. Contact with the eyes can cause permanent damage.

Samples that contain high concentrations of carbonates or organic matter, or samples that are at elevated pH can react violently when acids are added. Acids must be added to samples under a hood to avoid splash/splatter hazards and/or possibly toxic vapors that will be given off when the samples are acidified.

5.2 Primary Materials Used

The following is a list of the materials used in this procedure, which have a serious or significant hazard rating, and a summary of the primary hazards listed in their MSDS/SDS.

NOTE: This list does not include all materials used in the procedure. A complete list of materials used in this procedure can be found in the Reagents and Standards Section and the Equipment and Supplies Section of this SOP

Employees must review the information in the MSDS/SDS for each material before using it for the first time or when there are major changes to the MSDS/SDS. Electronic copies of MSDS/SDS can be found using the “MSDS” button on the Oasis homepage, on the EH&S webpage on Oasis, and via TALS File System Shares.

Material	Hazards	Exposure Limit[1]	Signs and Symptoms of Exposure
Hydrochloric Acid[2]	Corrosive Poison	5ppm Ceiling	Inhalation of vapors can cause coughing, choking, inflammation of the nose, throat, and upper respiratory tract, and in severe cases, pulmonary edema, circulatory failure, and death. Can cause redness, pain, and severe skin burns. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
Nitric Acid[2]	Corrosive Oxidizer Poison	2ppm TWA 4ppm STEL	Nitric acid is extremely hazardous; it is corrosive, reactive, an oxidizer, and a poison. Inhalation of vapors can cause breathing difficulties and lead to pneumonia and pulmonary edema, which may be fatal. Other symptoms may include coughing, choking, and irritation of the nose, throat, and respiratory tract. Can cause redness, pain, and severe skin burns. Concentrated solutions cause deep ulcers and stain skin a yellow or yellow-brown color. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
[1]Exposure limit refers to the OSHA regulatory exposure limit.			
[2]Always add acid to water to prevent violent reactions.			

6.0 Equipment and Supplies

6.1 Equipment and Instrumentation

Agilent 7700 / 7900 (or equivalent) equipped with an Octopole Reaction System (ORS). The ORS is a small enclosed chamber that can be pressurized with a collision/reaction gas and mounted on-axis to the quadrapole for high ion transmission. The reaction gases used are:

Helium (UHP grade) - reduces both matrix based interferences, such as chlorides, and simple plasma based interferences, such as argon oxide or the doubly charged argon argon+, attributed from the argon plasma.

Hydrogen (UHP grade) - reduces the intense plasma-based interferences which the helium mode may not be efficient enough to correct for, such as argon hydride, argon gas, or argon oxide.

The ORS corrects for most of the interferences associated with a non-collision cell ICP-MS, but there are a few analytes that are evaluated in a “no gas” or normal mode that still require an interference equation. They are typically:

Cd 111: (1 * 111) - (0.00124 * 95)
Sn 115: (1 * 115) - (118 * 0.016)
Pb 208: (1 * 206) + (1 * 207) + (1 * 208)

If analyzing drinking water samples in "normal" mode only (no collision or reaction gas), then the following interference equations are recommended by the EPA. These equations are guidelines and can differ from instrument to instrument.

As 75: (1 * 75) - [(3.127 * 77) - (0.815 * 82)]
Cd 111: (1 * 111) - [(1.073 * 108) - (0.712 * 106)]
Pb 208: (1 * 206) + (1 * 207) + (1 * 208)
Mo 98: (1 * 98) - (0.146 * 99)
V 51: (1 * 51) - [(3.127 * 53) - (0.113 * 52)]
In 115: (1 * 115) - (0.016 * 118)

Data System: Chemstation and TALS are used to acquire, store, reduce, and output ICP mass spectral data. This software has the capability of processing stored ICP-MS data. The software allows for the calculation of concentrations of analytes using the calibration curve.
A Master Equipment Listing is maintained by the Quality Assurance Department. This listing contains information regarding software and firmware for all equipment utilized in the laboratory.

6.2 Volumetric Containers and Dispensers

All volumetric labware must be verified in accordance with SOP SA-AN-100: *Laboratory Support Equipment (Verification and Use).* Refer to Attachment 10 for Labware Cleaning Procedures.

Volumetric Labware	Volume	Type (Quantitative / Qualitative)	Use	Verification Frequency	Laboratory Verification Criteria
Autosampler Vials	50mL	Qualitative	Holding sample on instrument	None	None
Eppendorf-Style Mechanical Pipettes	Various	QUANTITATIVE	Preparing Dilutions, Standard and Reagent Preparation and Spiking	Quarterly (Daily for DOD)	Accuracy = 2% Precision = 1%
Volumetric Flasks (Class A)	Various	QUANTITATIVE	Preparing Dilutions, Standard and Reagent Preparation	None (Class A)	None (Class A)

6.3 Lab Supplies

Argon gas supply and appropriate fittings

Cooling water supply

Hydrogen gas (UHP)

Helium gas (UHP)

Detergent – Citranox or comparable cleaner, used for washing non-disposable labware.

Filters – 0.45um syringe filters, used to filter samples for dissolved metals in the lab. Also used to remove particulates from sample digestions.

6.4 Sample Collection Containers

All sample collection containers are single-use disposable containers which limits the potential for contamination.

The routine sample collection containers supplied by the laboratory are:

Waters:
Total Metals:
250mL plastic with nitric acid – purchased with Certificate of Analysis attesting to purity.
Dissolved Metals:
250mL plastic – purchased with Certificate of Analysis attesting to purity.

Soils:
8oz plastic soil jar – purchased with Certificate of Analysis attesting to purity.

7.0 Reagents and Standards

7.1 Expiration Dates

Expiration dates (time from initial use or receipt to final use) for standard and reagent materials must be set according to the guidance in this SOP. Note: These are maximum expiration dates and are not to be considered an absolute guarantee of standard or reagent quality. Sound judgment must be used when deciding whether to use a standard or reagent. If there is doubt about the quality of a standard or reagent material, a new material must be obtained or the standard or reagent material verified. Data quality must not be compromised to extend a standard's life.

The expiration date of any standard or reagent must not exceed the expiration date of the standard or reagent that was used to prepare it.

7.2 Reagents

Reagents must be prepared and documented in accordance with SOP SA-AN-041*: Reagent and Standard Materials Procedures.*

Hydrochloric acid and nitric acid must be verified prior to use in accordance with the Eurofins TestAmerica Solvent Lot Testing Program.

Laboratory Reagent Water – ASTM Type II or better; water from the Modulab filtration system (i.e., ASTM Type I) should be used as the default.

Nitric Acid (HNO_3) - trace metal grade.

Storage: Store in a cool, dry, ventilated storage area with acid resistant floors and good drainage. Store away from sunlight, heat, water, and incompatible materials. Stable under ordinary conditions of use and storage.
Expiration: Manufacturer's expiration date

Hydrochloric Acid (HCl) - trace metal grade.
Storage: Store in a cool, dry, ventilated storage area with acid resistant floors and good drainage. Store away from sunlight, heat, water, and incompatible materials. Stable under ordinary conditions of use and storage.
Expiration: Manufacturer's expiration date

7.3 Standards

Standards must be prepared and documented in accordance with SOP SA-AN-041*: Reagent and Standard Materials Procedures.* Certificates of analysis or purity must be received with all purchased standards, and scanned and attached in TALS.

7.3.1 Calibration Stock Standards

The following individual analytes are purchased at the concentrations listed:
Aluminum – 10000mg/L
Antimony – 1000mg/L
Arsenic – 1000mg/L
Barium – 1000mg/L
Beryllium – 1000mg/L
Boron – 1000mg/L
Cadmium – 1000mg/L
Calcium – 10000mg/L
Chromium – 1000mg/L
Cobalt – 1000mg/L
Copper – 1000mg/L
Iron – 10000mg/L
Lead – 1000mg/L
Magnesium – 10000mg/L
Manganese – 1000mg/L
Mercury – 1000mg/L
Molybdenum – 1000mg/L
Nickel – 1000mg/L
Potassium – 10000mg/L
Selenium – 1000mg/L
Silver – 1000mg/L
Sodium – 10000mg/L
Strontium – 1000mg/L
Thallium – 1000mg/L
Tin – 1000mg/L
Titanium – 1000mg/L
Vanadium – 1000mg/L
Zinc – 1000mg/L
Uranium – 1000ug/L
Thorium – 1000ug/L

Storage: room temperature
Expiration: opened and unopened containers are given the manufacturer's expiration date

7.3.2 Initial Calibration Verification Stock Standards

The following individual analytes are routinely purchased from Absolute standards (this standard must be purchased from a vendor different than the vendor of the calibration standards) at the concentrations listed:

Aluminum – 10000mg/L
Antimony – 1000mg/L
Arsenic – 1000mg/L
Barium – 1000mg/L
Beryllium – 1000mg/L
Boron – 1000mg/L
Cadmium – 1000mg/L
Calcium – 10000mg/L
Chromium – 1000mg/L
Cobalt – 1000mg/L
Copper – 1000mg/L
Iron – 10000mg/L
Lead – 1000mg/L
Magnesium – 10000mg/L
Manganese – 1000mg/L
Mercury – 100mg/L
Molybdenum – 1000mg/L
Nickel – 1000mg/L
Potassium – 10000mg/L
Selenium – 1000mg/L
Silver – 1000mg/L
Sodium – 10000mg/L
Strontium – 1000mg/L
Thallium – 1000mg/L
Tin – 1000mg/L
Titanium – 1000mg/L
Vanadium – 1000mg/L
Zinc – 1000mg/L
Uranium – 1000ug/L
Thorium – 1000ug/L

Storage: room temperature
Expiration: opened and unopened containers are given the manufacturer's expiration date

7.3.3 Interference Check Standard

A/B Stock Standards:

Solution Component	Concentration Solution A (mg/L)	Concentration Solution B (mg/L)
Aluminum (Al)	1000	
Calcium (Ca)	1000	
Iron (Fe)	1000	
Magnesium (Mg)	1000	
Sodium (Na)	1000	
Phosphorus (P)	1000	
Potassium (K)	1000	
Sulfur (S)	1000	
Carbon (C)	2000	
Chloride (Cl)	10000	
Molybdenum (Mo)	20	
Titanium (Ti)	20	
Arsenic (As)		10
Cadmium (Cd)		10
Chromium (Cr)		10
Cobalt (Co)		10
Copper (Cu)		10
Manganese (Mn)		10
Nickel (Ni)		10
Silver (Ag)		10
Zinc (Zn)		10

Storage: room temperature
Expiration: opened and unopened containers are given the manufacturer's expiration date

7.3.4 Internal Standard Stock Standard

The following elements are used as the internal standards: Bi, In, Li6, Sc, Tb, Y, Ho, and Ge. A mixed standard containing all elements is purchased from a vendor at a concentration of 10mg/L.

Storage: room temperature
Expiration: opened and unopened containers are given the manufacturer's expiration date

7.3.5 Prepared Standards

Refer to Attachment 7 for information on prepared standards.

8.0 Sample Collection, Preservation, Shipment, and Storage

8.1 Water Samples

8.1.1 Total Metals

Water samples are routinely collected in 250mL plastic containers containing 3mL of a 1:3 nitric acid preservative. The preservative should be sufficient to achieve a sample pH of less than 2.

Although no temperature preservation is required, samples are routinely iced at the time of collection at 0-6°C (less than 6°C but not frozen). Samples are stored at room temperature until the time of digestion. Samples must be digested and analyzed within 6 months of collection, unless mercury is requested. Samples for mercury analysis must be digested and analyzed within 28 days of sample collection. Digestates are stored at room temperature until the time of analysis.

NCMs must be initiated for samples collected in improper containers and containing improper or insufficient preservatives.

8.1.2 Dissolved Metals

Water samples for dissolved metals are routinely filtered at the time of sampling and collected in 250mL plastic containers containing 3mL of a 1:3 nitric acid preservative. The preservative should be sufficient to achieve a sample pH of less than 2.

Note: If the sample is to be filtered in the laboratory, the sample must be collected in 250mL plastic container with no preservatives. Once filtered, the laboratory will add nitric acid to obtain a pH of less than 2. Unpreserved water samples that are to be filtered in the laboratory must be iced at the time of collection at 0-6°C (less than 6°C but not frozen). The samples must be kept refrigerated until the time of filtration and preservation.

> Note: The reference methods state that filtration must occur immediately; therefore, if the laboratory is requested to filter the samples - as opposed to a field filtration - appropriate narration must be included in the final report to denote this method deviation.

Although no temperature preservation is required when the samples are preserved with nitric acid, samples are routinely iced at the time of collection at 0-6°C (less than 6°C but not frozen). Samples are stored at room temperature until the time of digestion. Samples must be digested and analyzed within 6 months of collection, unless mercury is requested. Samples for mercury analysis must be digested and analyzed within 28 days of sample collection. Digestates are stored at room temperature until the time of analysis.

NCMs must be initiated for samples collected in improper containers and containing improper or insufficient preservatives.

8.2 Soil Samples

Soil samples are routinely collected in 8oz plastic soil containers.

Samples must be iced at the time of collection and maintained at 0-6°C (less than 6°C but not frozen) until the time of digestion and analysis. Samples must be digested and analyzed within 6 months of collection, unless mercury is requested. Samples for mercury analysis must be digested and analyzed within 28 days of sample collection. Digestates are stored at room temperature until the time of analysis.

9.0 Quality Control

SOP SA-QA-017: *Evaluation of Batch QC Data* and the SOP Summary in Attachment 3 provide requirements for evaluating QC data.

9.1 Batch QC

9.1.1 EPA 200.8 – Drinking Water

A digestion batch consists of up to 20 environmental samples and the associated QC items digested together within a 24 hour period.

The minimum QC items required for each digestion batch are: a method blank, a laboratory control sample (LCS), a low-level LCS (LLCS), a matrix spike (MS) to be performed on a minimum of 10% of samples or one per batch – whichever is greater, and a matrix spike duplicate (MSD).

This frequency equates to the following:

- For a batch of 10 or fewer samples, the minimum QC items are a method blank, an LCS, an LLCS, 1 matrix spike, and 1 matrix spike duplicate.
- For a batch of 11-20 samples, the minimum QC items are a method blank, an LCS, an LLCS, 1 matrix spike (from sample 1-10), another matrix spike (from sample 11-20), and a matrix spike duplicate.

The routine container supplied for this method is a 250mL container. 50mL is required for digestion. Reduced sample initial volumes may be necessary to achieve the required batch matrix spike frequency; however, the minimum digestion volume to be used for the matrix spike samples is 25mL. Note: Final volumes and spike amounts must be adjusted to compensate for these reduced initial volumes.

If there is insufficient sample volume to perform the required matrix spike(s), the LCS must be prepared in duplicate (i.e., LCSD). An NCM must be initiated on all affected samples to denote this situation. Insufficient sample volume is defined as receiving less than a total of 100mL.

Note: If an LCS and LCSD are performed, both QC items must be evaluated and reported. Acceptable recoveries (as well as %RPD) for both LCS and LCSD are required.

The sample preparation and digestion procedures are listed in the following SOPs:

Matrix	SOP
Aqueous samples	SA-ME-050

Batch QC must meet the criteria given in Attachment 3 of this SOP.

9.1.2 EPA 200.8 – Clean Water Act

An extraction batch consists of up to 20 environmental samples and the associated QC items extracted together within a 24 hour period.

The minimum QC items required for each extraction batch are: a method blank, a laboratory control sample (LCS), a matrix spike (MS) to be performed on a minimum of 10% of samples or one per batch – whichever is greater, and a matrix spike duplicate (MSD).

This frequency equates to the following:

- For a batch of 10 or fewer samples, the minimum QC items are a method blank, an LCS, 1 matrix spike, and 1 matrix spike duplicate.
- For a batch of 11-20 samples, the minimum QC items are a method blank, an LCS, 1 matrix spike (from sample 1-10), another matrix spike (from sample 11-20), and a matrix spike duplicate.

The routine container supplied for this method is a 250mL container. 50mL is required for extraction. Reduced sample initial volumes may be necessary to achieve the required batch matrix spike frequency; however, the minimum extraction volume to be used for the matrix spike samples is 25mL. Note: Final volumes and spike amounts must be adjusted to compensate for these reduced initial volumes.

If there is insufficient sample volume to perform the required matrix spike(s), the LCS must be prepared in duplicate (i.e., LCSD). An NCM must be initiated on all affected samples to denote this situation. Insufficient sample volume is defined as receiving less than a total of 100mL.

Note: If an LCS and LCSD are performed, both QC items must be evaluated and reported. Acceptable recoveries (as well as %RPD) for both LCS and LCSD are required.

The sample preparation and digestion procedures are listed in the following SOPs:

Matrix	SOP
Aqueous samples	SA-ME-050

Batch QC must meet the criteria given in Attachment 3 of the associated analytical SOP.

9.1.3 EPA 6020A and EPA 6020B

A digestion batch consists of up to 20 environmental samples and the associated QC items. The minimum QC items required for each digestion batch are: a method blank, a laboratory control sample (LCS), a matrix spike (MS), and a matrix spike duplicate (MSD) or a sample duplicate.

If there is insufficient sample to perform the MS/MSD or sample duplicate, an NCM must be initiated on all affected samples to denote this situation.

The routine container supplied for this method is a 250mL container. 50mL is required for digestion. Reduced sample initial volumes may be necessary to achieve the required batch matrix spike frequency; however, the minimum digestion volume to be used for the matrix spike samples is 25mL. Note: Final volumes and spike amounts must be adjusted to compensate for these reduced initial volumes.

If there is insufficient sample volume to perform the required matrix spike(s), the LCS must be prepared in duplicate (i.e., LCSD). An NCM must be initiated on all affected samples to denote this situation. Insufficient sample volume is defined as receiving less than a total of 100mL.

Note: If an LCS and LCSD are performed, both QC items must be evaluated and reported. Acceptable recoveries (as well as %RPD) for both LCS and LCSD are required.

The sample preparation and digestion procedures are listed in the following SOPs:

Matrix	SOP
Water samples	SA-ME-050
Soil samples	SA-ME-051

Batch QC must meet the criteria given in Attachment 3 of this SOP.

9.2 Instrument QC

9.2.1 Initial Calibration (ICAL)

The instrument must be calibrated daily in accordance with SOP SA-QA-016: *Evaluation of Calibration Curves.* This SOP provides requirements for establishing the calibration curve and gives the applicable formulas.

Instrument calibration is performed by analyzing a series of known standards. The methods require the calibration curve to consist of a minimum of a single standard and a blank; however, a multi-point calibration is used for all analytes.

Refer to Attachment 7 for the standard preparation instructions. Other standard concentrations may be used provided they support the reporting limit and are fully documented in accordance with SOP SA-AN-041.

Tabulate the concentrations and corresponding responses for each analyte. Establish a calibration curve by plotting the concentration along the x-axis and the corresponding response along the y-axis.

The regression coefficient (r^2) of the regression curve must be greater than 0.998 for the initial calibration curve to be acceptable.

9.2.2 Second Source Initial Calibration Verification (ICV)

Each calibration curve must be verified initially – prior to any sample analyses – in accordance with SOP SA-QA-016 with a standard obtained from a second source.

The ICV must be within +/-10% to be acceptable.

Refer to Attachment 7 for the standard preparation instructions. Another standard concentration may be used provided it is mid-level and fully documented in accordance with SOP SA-AN-041.

9.2.3 Initial Calibration Blank (ICB) / Continuing Calibration Blank (CCB)

The instrument must be shown to be free from contamination by the analysis of calibration blanks. Initial calibration blanks are analyzed at the beginning of each analytical run immediately following the ICV. Continuing calibration blanks are analyzed every 10 analyses immediately following each CCV.

Initial and continuing calibration blanks must be <1/2RL to be acceptable.

9.2.4 Continuing Calibration Verification

The initial calibration curve must be verified before and after every 10 analyses with a mid-level standard.

The CCV must be within +/-10% to be acceptable.

Refer to Attachment 7 for the standard preparation instructions. Another standard concentration may be used provided it is mid-level and fully documented in accordance with SOP SA-AN-041.

9.2.5 Interference Check Standard (ICSA and ICSAB)

The purpose of the Interference Check Standard is to prove that the instrument software is adequately correcting for common interferences through the use interelement correction factors. This check must be performed at the beginning of each analysis run. The concentrations of the target analytes must be within 20% of the true concentrations to be acceptable. The analyst must pay particular attention to false positives and false negatives for elements not present in the interference check solutions. Unspiked analytes should be less than two times the RL.

Note: The vendor has acknowledged trace impurities in the ICSA and ICSAB solution for the following analytes: Cadmium, Cobalt, Chromium, Strontium, Lead, and Manganese. As such, the presence of these analytes is not indicative of a matrix interference.

Note: WI DNR requires qualification of results if there is any detection in the ICSA for unspiked elements at or above the absolute value of the WI LOQ (i.e., 3.3 times the MDL).

9.2.6 Internal Standard (ISTD)

This procedure utilizes internals standards. See Attachment 7 for the concentrations and masses of the internal standard elements.

The internal standard solution is added to all standards, samples, and QC items by way of a peristaltic pump, a “T” connector, and a mixing coil at the instrument. The solution used is 1mg/L for each of the internal standard elements. Other concentrations may be used provided it is mid-level and fully documented in accordance with SOP SA-AN-041.

9.2.6.1 EPA 200.8

The response of the internal standard in all samples and QC items must be within 60-125% of the response of the internal standard in the calibration blank. If the response is outside this range, flush the instrument with rinse blank and re-analyze the calibration blank. If the responses are within limits, re-analyze the samples at a dilution of 2.

9.2.6.2 EPA 6020A and EPA 6020B

The response of the internal standard in all samples and QC items must not fall outside 30-150% of the response of the internal standard in the initial calibration standard. If the response is outside this range for instrument QC, terminate the analysis, correct the problem, and re-calibrate. If the response is outside this range for samples, re-analyze the sample at a dilution of 5.

9.2.7 Post Digestion Spike (PDS)

A post-digestion spike is performed on one sample per analytical batch to determine if matrix interferences are present. This post-digestion spike is evaluated if the serial dilution fails or if the analyte concentration is not at least 50 times the instrument detection limit. This should be the same sample selected for the serial dilution in Section 9.2.7.

9.2.7.1 Transfer 10mL of a digestate to a suitable vial.

9.2.7.2 Spike the sample with 20uL of the ICP-MS Calibration Stock Standard.

9.2.7.3 Analyze the spiked aliquot and an un-spiked aliquot (the un-spiked may have been analyzed previously and does not need to be reanalyzed).

9.2.7.4 Calculate the percent recovery of the post digestion spike as follows:

$$\%REC = \frac{C_{ps} - C_s}{C_2} \times 100$$

Where: C_{ps} = concentration of post digestion spike (ug/L)
C_s = concentration of un-spiked sample (ug/L)
C_2 = theoretical concentration of spike (ug/L)
(Refer to Attachment 7)

9.2.7.5 Evaluate the recovery using the following decision matrix. Limits for post digestion spikes are 80-120%.

Result of Post Digestion Spikes	Action
Within 80-120% limits	None
>120% recovery	Repeat analysis. Remake spiking solutions, re-spike, and reanalyze. Reanalyze un-spiked sample.
<80% recovery but >50% recovery	1) Dilute and re-spike. Elevate RL accordingly (for all associated samples). 2) Spike and evaluate all associated samples. 3) Spike and evaluate all associated samples by single point MSA. 4) Qualify all associated samples.
<50% recovery	Dilute digestate and repeat spike. ***Treat all samples associated with spike in the same manner as the spiked sample (i.e., spike or dilute samples).*** If recoveries are not 80-120%, analyze all associated samples by single point MSA. Note – high level of target analytes may inhibit spike recovery. Consult the supervisor in events where high levels of targets appear to be interfering.

Note: The >50% recovery of the post digestion spike is a benchmark below which samples may be biased high if corrected for spike recovery.

9.2.7.6 The post digestion spike and the method of standard additions must not be applied to samples analyzed at a dilution that produces a significant negative response. The analyst must use good judgment when evaluating data where the sample response is negative. Where a significant negative response is present, the digestate should be diluted and reanalyzed to determine the extent of the matrix interferences. If necessary, adjust the interference corrections and reanalyze the samples.

9.2.8 Single Point Method of Standard Additions (MSA)

The Method of Standard Additions (MSA) is performed for TCLP samples and when the post-digestion spike is less than 50%. Perform the MSA as follows:

Two identical aliquots of the sample digest, V_x, are taken. One aliquot is spiked with a solution of known concentration, C_s. The second aliquot is analyzed un-spiked (the small volume of standard added to the spiked sample should be disregarded). The concentration of both aliquots is measured and the sample concentration, C_x, is calculated as follows:

$$C_x = \frac{S_2 V_s C_s}{(S_1 - S_2) V_x}$$

Where: S_1 = absorbance or concentration of the spiked aliquot
S_2 = absorbance or concentration of the un-spiked aliquot
Vs = volume of spike solution

Example:		
	Sample concentration (S_2):	523ug/L.
	Spike solution concentration (C_s):	50,000ug/L
	Volume of spike solution (V_s):	0.10mL
	Volume of sample aliquots (V_x):	10mL
	Spiked sample concentration (S_1):	951ug/L

C_x = [(523)*(0.10)*(50,000)] / [(951-523)*10)] = [2,615,000]/[4280] = 611 ug/L

9.2.9 Dilution Test (Serial Dilution)

A 1/5 dilution is prepared and analyzed on one sample per batch to determine if matrix interferences are present.

9.2.9.1 Select a sample digestate that contains one or more target analytes at concentrations greater than 50 times the method detection limit.

9.2.9.2 Dilute the digestate by a factor of 5, and analyze the dilution using the same procedures used for the un-diluted aliquot.

9.2.9.3 Compare the results of the diluted and un-diluted aliquots of sample digestate.

9.2.9.4 If the results of the dilution are within +/-10% of the results of the undiluted sample, no matrix interference is present. If the results differ by greater than +/-10%, matrix interference should be suspected.

9.2.10 Determination of Linear Range of the ICP-MS

If the instrument is not calibrated over its entire linear range for a particular element, a linear range standard must be analyzed daily to validate the linear range.

9.2.10.1 The ICSA solution is utilized as a linear range standard for those elements included in the solution. For elements not contained in the ICSA solution, a linear range standard must be prepared and analyzed.

9.2.10.2 Prepare the standard at concentrations that are expected to define the linear range of the instrument. The calibration standards and the linear range standards must be matrix matched; that is, they have the same percentage of hydrochloric and nitric acids.

9.2.10.3 Analyze the standard(s) after the initial calibration is validated.

9.2.10.4 Compare the concentration of the linear range standard with its true concentration using the following equation:

$$PercentDifference = \left| \frac{Ccal - Ctrue}{Ctrue} \right| \otimes 100$$

Where:
C_{cal} = concentration determined from analysis
C_{true} = true concentration of the standard

If the percent difference is less than or equal to 10%, the linear range is confirmed at that concentration. If the percent difference is greater than 10%, repeat the analysis with a lower concentration. For elements validated in this manner, data may be reported up to 90% of that linear range before a dilution is required.

9.3 Corrective Action for Out-of-Control Data

When the quality control parameters do not meet the criteria set forth in this SOP, corrective action must be taken in accordance with SOP SA-QA-005: *Preventive and Corrective Action Procedures* the QC Summary Table in Attachment 3. SOP SA-QA-005 provides contingencies for out-of-control data and gives guidance for exceptionally permitting departures from approved policies and procedures. Nonconformance Memos must be initiated to document all instances where QC criteria are not met and all departures from approved policies and procedures.

10.0 Procedure

10.1 Sample Preparation

The sample preparation procedures are given in the following SOPs:

Matrix	SOP #
Aqueous Samples	SA-ME-050
Soil Samples	SA-ME-051

10.2 QC Sample Preparation

The QC sample preparation procedures are given in the following SOPs:

Matrix	SOP #
Aqueous Samples	SA-ME-050
Soil Samples	SA-ME-051

10.3 Analysis

10.3.1 Instrument Operating Conditions

The instrument conditions listed in this SOP are provided for guidance purposes. The actual conditions used by the laboratory may be slightly different from those listed here and must be documented in the instrument maintenance log, data system, and/or run log.

Instrument maintenance must be performed in accordance with Attachment 4 of this SOP. Turn the ICP-MS on and initiate the tune screen. Start the tune screen to allow the instrument to become thermally stable before analyzing the calibration standards. While the instrument is warming up, if the sample and skimmer cones are new or have been cleaned, aspirate the interference check solution (or similar solution) for about 15 minutes to pre-condition the cones.

Aspirate a 1ppb solution containing Lithium 7, Cesium, Yttrium, and Thallium.
An autotune using the “Start Up” function is performed before every calibration. This autotune process will optimize the following:

- Torch Axis
- EM
- Plasma Correction
- Resolution / Axis
- Performance Report
- P/A (Pulse to Analog)

The performance report should be reviewed and should meet the following conditions:

- Sensitivity: should yield counts greater than 1500 for Li7 and 2500 for Y and Tl
- Precision: should be less than 10% RSD of 200 replicates
- Oxides: must be less than 3% as CeO
- Doubly charged ions: must be less than 10%

10.3.1.2 Analyze the calibration standards and calibrate the ICP-MS in accordance with SOP SA-QA-016: *Evaluation of Calibration Curves.*

10.3.2 Internal Standard (ISTD)

Prior to analysis, internal standards must be added to all standards, samples, and QC items. The concentration of the internal standard must be the same in all calibration samples, field samples, and QC samples. The internal standard solution is added at the time of analysis by using a peristaltic pump, a "T" connection, and a mixing coil that is inserted in the sample uptake line before the instrument's nebulizer. The concentration of the internal standard solution is listed in Attachment 7 of this document.

10.3.3 Initial and Continuing Calibration Verifications

Calibrate the instrument using the standards and criteria described given in Section 9.2. Once the calibration has been established and verified with an ICV in accordance with Section 9.2, sample analysis may proceed.

Verify the calibration curve with a continuing calibration verification using the standards and criteria described given in Section 9.2.

10.3.4 Sample Analysis

The samples/digestates must be analyzed using the same procedures as those used for the calibration standards.

Note: It is the laboratory's default procedure to analyze soil samples and non-drinking water samples at a dilution factor of 5. The dilution is prepared immediately preceding analysis by diluting 2mL of digestate to a 10mL final volume. These dilution factors are captured by adjusting the default final volume in the Method Limit Groups. Reporting limits and MDLs have been elevated accordingly.

The default procedure is to include QC items (method blank, LCS, MS/MSD, and SD) in determining the maximum number of samples in the sequence.

10.3.5 Example Analytical Sequence

Analytical Sequence for samples immediately following an initial calibration:

Description	Comments
Blank	
Initial Calibration	Lowest standard is at or below the RL
ICV	Second Source
ICB	
Samples & Batch QC Items	RL Check Standard, ICSA, ICSAB, up to 7 additional analyses.
CCV	
CCB	
Samples & Batch QC Items	Up to 10 analyses, including QC.
CCV	
CCB	

Analytical Sequence for samples not immediately following an initial calibration:

Description	Comments
CCV	
CCB	
Samples & Batch QC Items	ICSA, ICSAB (minimum of every 12 hours) Up to 8 additional analyses.
CCV	
CCB	
Samples & Batch QC Items	Up to 10 analyses, including QC.
CCV	
CCB	

Note: For EPA 6020A and EPA 6020B, if the analysis run proceeds for more than 12 hours after the preceding ICSA/ICSAB pair, the analyst must repeat the analysis of the ICSA and ICSAB solutions.

The "up to 10 analyses" includes analysis of all analytical and batch QC items with the exception of the CCV and CCB analyses.

11.0 Calculations / Data Reduction

11.1 Data Reduction

Data must be evaluated in accordance with SOP SA-QA-002: *Data Generation and Review*.

11.1.1 Dilutions

If the concentration of a sample is above the calibration range (or linear range for single point curves) of the instrument the sample digestate must be diluted and reanalyzed.

11.1.2 Historical Data

Many of the laboratory's clients submit samples for repeat monitoring purposes. Prior to analysis, verify TALS Worksheet Notes to determine if historical data is available for review.

11.1.3 Chemical Relationships

When available, the following chemical relationships must be evaluated for each sample. If these relationships are not met the Supervisor must be contacted immediately.

- Total Results are ≥ Dissolved results (e.g. metals)

11.1.4 Drinking Water Compliance Evaluation

Public water suppliers (PWS) are governed by EPA-specified Maximum Contaminant Levels (MCL) above which indicates noncompliance. Many analytes also have a Maximum Contaminant Level Goal (MCLG), which is often lower than the MCL. The MCLs and MCLGs associated with this procedure are given in Attachment 8.

11.2 Calculations

11.2.1 The calculations associated with batch QC determinations are given in SOP SA-QA-017. Applicable calculations include accuracy (% recovery) and precision (%RPD).

11.2.2 The calculations associated with initial and continuing calibrations and are given in SOP SA-QA-016. Applicable calculations include determination for: calibration factor, standard deviation, relative standard deviation, relative response factor, and relative standard deviation.

11.2.3 The calculation to determine final concentration is given as follows:

Regression Curve:

$$FinalConcentration = CONC_{Sample} \otimes \frac{F}{I \times dw} \otimes D$$

Where:

$CONC_{Sample}$= Concentration of the sample
F = Final volume/weight
I = Initial volume/weight
D = Dilution factor
dw = % Solids decimal equivalent

Note: All dry weight corrections are performed automatically in TALS.

Note: This calculation assumes all applicable unit correction factors are applied.

12.0 Method Performance

12.1 Reporting Limit Verification (RLV)

At a minimum, RLVs must be performed initially upon method set-up in accordance with SOP SA-QA-007: *Determination and Verification of Detection and Reporting Limits*.

For analytes and methods certified by DOD ELAP, RLVs must also be performed quarterly thereafter. For analytes and methods certified by NELAC, RLVs must also be performed annually thereafter. Exceptions may be made for project-specific non-routine analytes.

12.1.1 Lower Limit of Quantitation Check (QCS)

EPA 6020A and EPA 6020B require a Lower Limit of Quantitation Check (LLQC) to be performed after establishing the reporting limit and on an as needed basis to demonstrate sensitivity. As such, the laboratory requires a Low-Level LCS (spiked at the reporting limit) to be performed annually, at a minimum. The recovery of the LLCS must be within 70-130% of the true value, or the reporting limit must be re-evaluated and elevated accordingly.

12.2 Method Detection Limit (MDL) Study

The MDL is the lowest concentration that can be detected for a given analytical method and sample matrix with 99% confidence that the analyte is present. MDLs reflect a calculated (statistical) value determined under ideal laboratory conditions in a clean matrix and may not be achievable in all environmental matrices. The current MDLs associated with this procedure are given in the Method Limit Group (MLG) in TALS.

At a minimum, MDL Studies must be performed initially upon method set-up in accordance with SOP SA-QA-007: *Determination and Verification of Detection and Reporting Limits*.

Note: MDL Studies are not required for non-routine analytes provided results are not reported below the RL (i.e., MDL equals RL in TALS).

12.3 Method Detection Limit Verification (MDLV)

At a minimum, MDLVs must be performed initially upon method set-up in accordance with SOP SA-QA-007: *Determination and Verification of Detection and Reporting Limits*.

For analytes and methods certified by DOD ELAP, MDLVs must also be performed quarterly thereafter. For analytes and methods certified by NELAC, MDLVs must also be performed annually thereafter.

Note: MDLVs are not required for non-routine analytes provided results are not reported below the RL (i.e., MDL equals RL in TALS).

12.4 Determination of the Instrument Detection Limit (IDL)

The instrument detection limit (IDL) is the concentration of analyte that can be statistically distinguished from the background noise of the instrument. The IDL limit must be determined quarterly, at a minimum, for each analyte in accordance with SOP SA-QA-007: *Determination and Verification of Detection and Reporting Limits.*

The IDL for EPA 6020A and EPA 6020B is defined as three times the standard deviation of seven replicate analyses of a blank solution analyzed over three non-consecutive days. The IDL for EPA 200.8 is defined as the concentration equivalent to the analyte signal which is equal to three times the standard deviation of a series of eight replicate measurements of the calibration blank signal at the selected mass (es). The IDL may be

elevated above the background noise (blank levels). The current IDL associated with this procedure is given in the Equipment Limit Group (ELG) in TALS.

12.5 QC Limit Generation, Control Charting, and Trend Analysis

The control limits for the batch QC items (LCS and MS/MSD) for this procedure are specified in the reference method and cannot be broadened; therefore, the laboratory defaults to the method-defined limits and does not utilize in-house or laboratory-derived limits for the evaluation of batch QC items.

Although the laboratory must default to the method-defined QC limits, control charting is a useful tool and is performed to assess analyte recoveries over time to evaluate trends. Control charting must be performed periodically (at a minimum annually) in accordance with SOP SA-QA-017: *Evaluation of Batch QC Data.*

12.6 Demonstrations of Capability

Initial and continuing demonstration of capability must be performed in accordance with SOP SA-QA-006*: Training Procedures.*

Prior to performing this procedure unsupervised, each new analyst who performs this analysis must demonstrate proficiency per method/analyte combination by successful completion of an initial demonstration of capability. The IDOC is performed by the analysis of 4 consecutive LCSs that meet the method criteria for accuracy and precision. The IDOC must be documented and routed to the QA Department for filing.

Annual continuing demonstrations of capability (CDOCs) are also required per analyst per method/analyte combination. The CDOC requirement may be met by the consecutive analysis of four LCS all in the same batch, by the analysis of four LCS analyzed in four consecutive batches (in different batches on different days), via acceptable results on a PT study, or analysis of client samples with statistically indistinguishable results when compared to another certified analyst. The CDOC must be documented and routed to the QA Department for filing.

12.7 Training Requirements

All training must be performed and documented in accordance with SOP SA-QA-006: *Training Procedures*.

Note: The SOPs listed in the Reference/Cross-Reference Section are applicable to this procedure. All employees performing this procedure must also be trained on these SOPs, and/or have a general understanding of these procedures, as applicable.

13.0 Pollution Control

It is Eurofins TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (e.g., examining recycling options, ordering chemicals based on quantity needed, preparing reagents based on anticipated usage and reagent stability,

etc.). Employees must abide by the policies in Section 13 of the Environmental Health and Safety Manual (EHSM) and the Savannah Addendum to the EHSM.

This procedure has been evaluated for opportunities to minimize the waste generated. Where reasonably feasible, pollution control procedures have been incorporated.

14.0 Waste Management

Waste management practices must be conducted consistent with all applicable federal, state, and local rules and regulations. All waste (i.e., excess reagents, samples, and method process wastes) must be disposed of in accordance with Section 9 of the Eurofins TestAmerica Savannah Addendum to the EHSM. Waste description rules and land disposal restrictions must be followed.

14.1 Waste Streams Produced by the Method

The following waste streams are produced when this method is carried out:

- Excess aqueous samples – Dispose according to characterization on the sample disposal sheets. Neutralize non-hazardous samples before disposal into drain/sewer. Transfer hazardous samples (identified on disposal sheets) to the waste department for disposal.

- Excess soil and solid samples – Dispose according to characterization on sample disposal sheets. Transfer non-hazardous samples to TCLP container for characterization in hazardous waste department. Transfer hazardous samples (identified on disposal sheets) to waste department for disposal.

- Acidic sample digestions – Neutralize before disposal into drain/sewer system.

Dispose of all metals standards with concentrations exceeding the TCLP threshold limits as a RCRA aqueous hazardous waste. The TCLP constituents and the respective threshold limits are:

Analyte	Disposal Limit (mg/L)
Arsenic	5.0
Barium	100
Cadmium	1.0
Chromium	5.0
Lead	5.0
Selenium	1.0
Silver	5.0
Mercury	0.20

Neutralize the standard by passing with sodium bicarbonate before transferring the waste to the RCRA aqueous hazardous waste drum.

All other metals standards may be disposed, using copious amounts of water, down a sink that flows to the neutralization pit.

15.0 References / Cross-References

- SOP SA-AN-100: *Laboratory Support Equipment (Verification and Use)*
- SOP SA-AN-041: *Reagent and Standard Materials Procedures*
- SOP SA-QA-002: *Data Generation and Review*
- SOP SA-QA-005: *Preventive and Corrective Action Procedures*
- SOP SA-QA-006*: Training Procedures*
- SOP SA-QA-007: *Determination and Verification of Detection and Reporting Limits*
- SOP SA-QA-015: *Homogenization, Compositing, and Segregation of Samples*
- SOP SA-QA-016: *Evaluation of Calibration Curves*
- SOP SA-QA-017: *Evaluation of Batch QC Data*
- Eurofins TestAmerica Savannah Quality Assurance Manual
- Eurofins TestAmerica Environmental Health and Safety Manual
- Eurofins TestAmerica Savannah Addendum to the Environmental Health and Safety Manual
- *Methods for Chemical Analysis of Water and Waste*; U.S EPA Office of Research and Development: Cincinnati, OHIO, March 1983.
- *Test Methods for Evaluating Solid Waste, Third Edition*; U.S. EPA Office of Solid Waste and Emergency Response: Washington, D.C., November 1986 (Revision III and IV).
  - SW-846 Chapter 3, Revision 3: Inorganic Analytes, December 1996
  - SW-846 Method 6020A, Revision 1: Inductively Coupled Plasma – Mass Spectrometry, February 2007
  - SW-846 Chapter 3, Revision 4: Inorganic Analytes, February 2007
  - SW-846 Method 6020B, Revision 2: Inductively Coupled Plasma – Mass Spectrometry, July, 2014
- *Methods for the Determination of Metals in Environmental Samples*; US EPA Office of Research and Development. Washington, DC, May 1994.
- EPA Method 200.8, Revision 5.4: Determination of Trace Elements in Waters and Wastes by Inductively Coupled Plasma – Mass Spectrometry, 1994

16.0 Method Modifications

16.1 Incorporation of Other Matrices

This procedure may be modified to analyze other matrices (e.g., wipe, waste, tissue, filter, and TCLP/SPLP leachate samples) based on the needs of the client. This will need to be arranged by the Project Manager at the initiation of the project.

Wipe, waste, filter, and tissue matrices are non-routine, and the laboratory is not currently NELAC certified for these matrices. The laboratory uses its routine soil RLs (converted for initial and final volumes, etc.) and soil QC limits to evaluate wipe, waste, filter, and tissue samples. Soil DOCs can be used to satisfy analyst demonstrations of capability for these types of non-routine matrices. The laboratory uses its routine soil RLs (converted for

initial and final volumes, etc.) and soil QC limits to evaluate TCLP/SPLP leachate samples. Water DOCs can be used to satisfy analyst demonstrations of capability for TCLP/SPLP matrices. Teflon chips, Ottawa sand, or equivalent is used as the blank matrix for wipes, wastes, filters, and tissues unless specifically requested otherwise by the project.

16.1.1 Collection and Handling Procedures for Non-Routine Matrices

Waste samples may be collected in 8oz plastic soil jars; however, it should be noted that an alternate container may be required as some organic wastes (oils) may not be conducive to plastic. Tissue samples may be collected in 8oz plastic soil jars. Wipes and filters may be collected in a variety of different containers.

Wipe, waste, and filter samples must be iced at the time of collection and maintained at 0-6°C (less than 6°C but not frozen) until the time of digestion/leaching. Samples for mercury must be digested/leached and analyzed within 28 days of collection. All other samples must be digested and analyzed within 6 months of collection. Digestates may be stored at room temperature until the time of analysis.

Tissue samples must be iced at the time of collection and maintained at -10°C to -20°C for up to 6 months. Samples for mercury must be digested/leached and analyzed within 28 days of thawing. All other samples must be digested and analyzed within 6 months of thawing. Digestates may be stored at room temperature until the time of analysis.

Wipe, waste, filter, and tissue matrices are prepared in the same manner as soil samples with the following exceptions:

- The initial amount for wipe or filter samples is 1 wipe or 1 filter.
- The initial amount for tissue and waste samples is 1.0-1.2g.

Once the TCLP/SPLP extraction procedure has been performed, the TCLP/SPLP leachate must be transferred to a 500mL plastic container and preserved with 1.0mL nitric acid to a pH <2. Preserved TCLP/SPLP leachates are stored at room temperature until the time of digestion. The leachate sample must be digested within 6 months of completion of the TCLP/SPLP extraction. Digestates are stored at room temperature until the time of analysis and must be analyzed within 6 months of completion of the TCLP/SPLP extraction.

16.1.2 Preparation and Analytical Procedures for Non-Routine Matrices

Wipe, waste, filter, and tissue samples are prepared in the same manner as routine soil samples as outlined in SOP SA-ME-051. TCLP/SPLP matrices are prepared in the same manner as routine water samples as outlined in SOP SA-ME-050. Refer to the applicable preparation SOPs for more information.

Wipe, waste, filter, tissue, and TCLP/SPLP matrices are analyzed in the same manner as routine samples as outlined in this SOP.

16.2 Other Considerations

16.2.1 EPA 6020A and 6020B note to use gold as a preservative for mercury; however, the laboratory has not implemented this preservative. The instrument manufacturer's recommendations do not include this reagent if HCl is used in standards and samples. Additionally, initial method validation did not indicate this preservative was necessary to prevent carryover as intended by the method.

16.2.3 EPA 200.8 requires the regression coefficient (r^2) of the regression curve to be greater than 0.995 for the initial calibration curve to be acceptable. EPA 6020A requires the r^2 of the regression curve to be greater than 0.998 for the initial calibration curve to be acceptable. The laboratory has adopted the 0.998 criteria for all three methods.

16.2.4 The EPA Manual for the Certification of Laboratories Analyzing Drinking Water requires a LFB at the MRL to be performed each day. The laboratory meets this requirement by preparing an LCS at the RL in each EPA 200.8 batch of drinking water samples. The EPA DW Manual does not specify criteria for the low-level LCS; therefore, the laboratory defaults to qualitative identification.

16.12 There is no method-defined batch precision requirement for EPA 200.8. The EPA does require precision for all samples analyzed under the Clean Water Act, and the laboratory routinely performs an MSD to support clients who require precision to be reported. If insufficient sample volume is provided to perform the MS/MSD, the laboratory performs the LCS in duplicate (i.e., LCS/LCSD).

16.13 It is the laboratory's default procedure to analyze samples at a dilution factor of 5. The dilution is prepared immediately preceding analysis by diluting 2mL of digestate to a 10mL final volume. These dilution factors are captured by adjusting the default final volume in the Method Limit Groups. Reporting limits and MDLs have been elevated accordingly.

16.2.5 The reference methods state that, for dissolved metals analysis, filtration must occur in the field. The laboratory may be requested to filter the samples upon receipt. If this occurs, then appropriate narration must be included in the final report to denote this method deviation.

16.2.6 EPA has acknowledged there is an error in the EPA 6020A reference method regarding the ISTD criteria (in one section the lower limit is listed as 30%, and in another section it is listed as 70%). The laboratory has adopted a lower limit of 30%, as is consistent with EPA Method 6020B. Additionally, there is no upper limit specified in the reference method. The laboratory has adopted an upper limit of 150% to ensure corrective action is taken when extremely high ISTD recovery is noted in samples.

17.0 Attachments

The following Tables, Diagrams, and/or Validation Data are included as Attachments:

Attachment 1: SOP Summary
Attachment 2: Sample Collection, Preservation, and Holding Time Table
Attachment 3: QC Summary
Attachment 4: Instrument Maintenance and Troubleshooting
Attachment 5: Analysis Masses, Internal Standards, and Tune Steps
Attachment 6: Element-specific Masses and Concentrations
Attachment 7: Standard Preparation
Attachment 8: Drinking Water MCLGs and MCLs
Attachment 9: Hardness Calculation Work Instruction
Attachment 10: Glassware Cleaning Procedures

Attachment 1:
SOP Summary

Sample Preparation Summary
Prior to analysis by ICP-MS, the sample must be solubilized or digested using the sample preparation method appropriate to the matrix.

Samples should be prepared according to the appropriate matrix-specific SOP.

Matrix	SOP
Aqueous Samples	SA-ME-050
Soil Samples	SA-ME-051

Sample Analysis Summary
Sample digestates are aspirated and nebulized into a spray chamber. A stream of argon gas carries the sample aerosol through the innermost of three concentric tubes and injects it into the middle of the donut-shaped plasma. The sample elements are dissociated, atomized, and excited to a higher energy level. The ions that are produced are entrained in the plasma gas and introduced, by means of an interface, into a mass spectrometer. The ions are sorted according to their mass to charge ratios and quantified with a channel mass spectrometer.

Analytical Sequence

Analytical Sequence for samples immediately following an initial calibration:

Description	Comments
Blank	
Initial Calibration	Lowest standard is at or below the RL
ICV	Second Source
ICB	
Samples & Batch QC Items	RL Check Standard, ICSA, ICSAB, up to 7 additional analyses.
CCV	
CCB	
Samples & Batch QC Items	Up to 10 analyses, including QC.
CCV	
CCB	

Analytical Sequence for samples not immediately following an initial calibration:

Description	Comments
CCV	
CCB	
Samples & Batch QC Items	ICSA, ICSAB Up to 8 additional analyses.
CCV	
CCB	
Samples & Batch QC Items	Up to 10 analyses, including QC.
CCV	
CCB	

Note: For EPA 6020A, if the analysis run proceeds for more than 12 hours after the ICSA/ICSAB pair, the analyst must repeat the analysis of the ICSA and ICSAB solutions.

The “up to 10 analyses” includes analysis of all analytical and batch QC items with the exception of the CCV and CCB analyses.

Attachment 2:
Sample Collection, Preservation, and Holding Time Table

Matrix	Routine Sample Container	Routine Sample Size	Minimum Sample Size	Dechlorination Agent	Chemical Preservation	Thermal Preservation	Holding Time[2]
Water	250mL plastic	50mL	25mL	Not Applicable	3mL 1:3 HNO3, to pH<2	Not Applicable	Mercury: 28 days Other Metals: 6 months
Soil	8oz plastic soil jar	1.0g	0.5g	Not Applicable	Not Applicable	0-6 °C[1]	Mercury: 28 days Other Metals: 6 months

[1]Samples are collected on ice and maintained at <6 °C with no frozen samples.
[2] Inclusive of digestion and analysis.

Note: If dissolved metals are requested, the sample must be filtered prior to the acid being added to the sample.

Attachment 3:
QC Summary

QC Item	Frequency	Criteria	Corrective Action
Initial Calibration: minimum 1 standard and 1 blank	Daily	Correlation ≥0.998 (for multipoint curves)	Recalibrate
Initial Calibration Verification Standard (ICV)	At the beginning of the analysis	Within ±10%	Recalibrate
Continuing Calibration Verification Standard (CCV)	At the beginning and end of the analysis, and every 10 samples	Within ±10% of the true value	Terminate the analysis, fix the problem and reanalyze the previous 10 samples.
Interference Check Standards (ICSA/ICSAB)	At the beginning of an analysis run	± 20% of the true values Unspiked analytes should be less than two times the RL. WI DNR requires qualification for analytes > LOQ (i.e., 3.3x MDL)	Terminate the analysis, correct the problem, recalibrate, and reanalyze all samples since the last ICS that was in control.
Calibration Blank (ICB/CCB)	After ICV and every CCV	<½ RL	Terminate the analysis, correct problem and reanalyze the previous 10 samples
Internal Standard	All samples and QC items	Refer to Section 9.2.5	Refer to Section 9.2.5
Batch Definition	Digested together w/in 24-hr timeframe; not to exceed 20 field samples	Not Applicable	Not Applicable
Method Blank	One per batch	|result|<RL DOD requires |result|<½ RL	Redigest and reanalyze batch

QC Item	Frequency	Criteria	Corrective Action
Laboratory Control Sample (LCS)	One per batch	TALS MLG	Redigest and reanalyze batch
Laboratory Control Sample Duplicate (LCSD)	One per batch, if insufficient sample provided for MS/MSD	TALS MLG	Redigest and reanalyze batch
Low-Level Laboratory Control Sample (LLCS) – spiked at the RL	EPA 200.8 DW: One per batch of 20 samples or less	Qualitatively identified	If the "regular" LCS meets criteria, initiate NCM and report data If the "regular" LCS does not meet criteria, redigest and reanalyze batch
Lower Limit of Quantitation Check (LLQC) – spiked at the RL	EPA 6020A / EPA 6020B: Annually, at a minimum, and as needed thereafter	EPA 6020A: 70-130% EPA 6020B: 80-120%	Re-evaluate RL and elevate accordingly
Matrix Spike (MS)	EPA 200.8: 10% of samples prepared; i.e., 2 separate matrix spikes per batch of twenty samples EPA 6020A: 5% of samples prepared; i.e., 1 matrix spike per batch of twenty samples	TALS MLG	Flag and report data
Matrix Spike Duplicate (MSD) or Sample Duplicate (SD)	EPA 200.8 (Clean Water Act), EPA 6020A: One MSD or sample duplicate per batch of twenty samples or less	TALS MLG	Flag and report data

QC Item	Frequency	Criteria	Corrective Action
Serial Dilution (1/5 Dilution)	One per batch of twenty samples or less	Refer to Section 9.2.7	Refer to Section 9.2.7
Post Digestion Spike (PDS)	One per batch of twenty samples or less	Refer to Section 9.2.6	Refer to Section 9.2.6
Reporting Limit Check Solution	EPA 200.8 and EPA 6020B: At the beginning of analysis run EPA 6020B: At beginning and end of analysis run.	EPA 200.8: Recovery +/-50% of the true concentration (if the instrument is not calibrated at or below the RL) EPA 6020A: 70-130% of the true concentration (if the instrument is not calibrated at or below the RL). EPA 6020B: 80-120% of the true concentration (if the instrument is not calibrated at or below the RL).	Stop the analysis, fix the problem and reanalyze the affected samples.
Initial Demonstration of Capability (IDOC)	Initially, per analyst, per analyte/method/matrix combination	Refer to SOP SA-QA-006	Refer to SOP SA-QA-006 Note: Unsupervised work must not begin until acceptable IDOC is obtained.
Continuing Demonstration of Capability	Annually, per analyst, per analyte/method combination	Refer to SOP SA-QA-006	Refer to SOP SA-QA-006

QC Item	Frequency	Criteria	Corrective Action
(CDOC)			
Reporting Limit Verification (RLV)	Upon method/instrument set-up, per analyte/method/matrix combination. Then quarterly thereafter (for DOD ELAP) or annually thereafter (for NELAC)	Refer to SOP SA-QA-007	Refer to SOP SA-QA-007
Method Detection Limit Study (MDL Study)	Upon method/instrument set-up, per analyte/method/matrix combination	Refer to SOP SA-QA-007	Refer to SOP SA-QA-007
MDL Verification (MDLV)	Upon method/instrument set-up, per analyte/method/matrix combination. Then quarterly thereafter (for DOD ELAP) or annually thereafter (for NELAC)	Refer to SOP SA-QA-007	Refer to SOP SA-QA-007

Attachment 4:
Instrument Maintenance and Troubleshooting

Instrument Labeling

Each instrument must be labeled with its name or ID (e.g., MSA, ICP-D, etc.). Additionally, non-operational instruments must be isolated from service or marked as being out of service. Each piece of equipment has an "Operational / Not Operational" sticker that is used for this purpose.

Maintenance Log

A maintenance log must be established for each piece of equipment used in the laboratory.

All maintenance that is performed on the instrument must be recorded in the log including:

- analyst or technician performing the maintenance
- date the maintenance was performed
- detailed explanation of the reason for the maintenance
- resolution of the problem and return to control
- all service calls from instrument representatives

Preventive Maintenance

Refer to the instrument manufacturer's guides for trouble-shooting items.

LABORATORY EQUIPMENT PREVENTIVE MAINTENANCE SCHEDULE

EQUIPMENT ITEM	Service Interval							SERVICE LEVEL
	D	W	M	Q	SA	A	AN	
Cones							X	Clean, as needed.
Intake							X	Wipe down as needed.
Lenses							X	Clean or replace as needed.
Nebulizer							X	Clean, as needed.
Pump Oil							X	Replace as needed.
Pump Tubing							X	Change as needed

D = daily; W = Weekly; M = monthly; Q = Quarterly; SA = semi-annually; A = annually; AN = as needed

Troubleshooting

Troubleshooting should be documented as outlined above. If possible, troubleshooting is best performed in a step-wise manner to systematically isolate instrument components. Refer to the instrument manufacturer's guides for specific information and strategies. Enlist assistance from technical and/or department management as needed.

Contingency Plan

Maintenance contracts are carried for most instrumentation and close contact is maintained with service personnel to ensure optimal instrument functioning. An extensive spare parts inventory is maintained for routine repairs, consisting of detectors, pump tubing, cones, lenses, torches, and other common instrumentation components. Since instrumentation is standardized throughout the laboratory network, spare parts and components can be readily exchanged among the network.

In general, the laboratory has at least one backup unit for each critical unit. In the event of instrument failure, portions of the sample load may be diverted to duplicate instrumentation, the analytical technique switched to an alternate approved technique (such as manual colorimetric determination as opposed to automated colorimetric determination), or samples shipped to another properly certified or approved Eurofins TestAmerica location.

Attachment 5:
Recommended Analysis Masses, Internal Standards, and Tune Steps

The following are the recommended analysis masses, internal standards, and tune steps:

Element	IS	Tune Step	Element	IS	Tune Step
Be 9	Li 6	1	Zn 66	Ge 74	3
B 11	Li 6	1	As 75	Ge 74	-3
Na 23	Sc 45	3	Se 78	Ge 74	2
Mg 24	Sc 45	3	Sr 88	Y 89	3
K 39	Ge 74	3	Mo 95	In 115	3
Ca 40	Sc 45	2	Ag 107	In 115	3
Ti 47	Sc 45	3	Cd 111	In 115	3
V 51	Ge 74	3	Sn 118	In 115	3
Cr 52	Ge 74	3	Sb 121	In 115	3
Mn 55	Ge 74	3	Ba 137	In 115	3
Fe 56	Sc 45	2	Hg 202	Bi 209	3
Co 59	Ge 74	3	Tl 205	Tb 159	3
Ni 60	Ge 74	3	Pb 208*	Tb 159	3
Cu 63	Ge 74	-3	U 238	Bi 209	1
Ca 44	Sc 45	3			

* Pb 208 = Pb 208 + Pb 207 + Pb 206

Note: Different masses and internal standards may be utilized, as matrix issues deem necessary.

Note: Agilent ICP-MS 7700 currently does not run H_2 reaction mode; only He collision mode and normal mode (no gas).

Tune Steps:
1. Normal mode: No gas
2. H_2 reaction mode: typical flows of 2.0 to 5.8 mL/min of Hydrogen gas
3. He collision mode: typical flows of 2.0 to 5.8 mL/min of Helium gas

Tune Check Criteria:
Instrument tunes must be performed before each calibration. A solution at ~1ppb of Be, Mg, Co, In, and Pb is analyzed, and the precision, mass calibration, and resolution are checked.

The following limits are used to evaluate the tune:
Mass calibration: +/- 0.1amu
Resolution check: <0.9amu at 10% peak height (baseline resolution)
Stability (5 reps): <5%

Attachment 6:
Recommended Element-specific Masses and Concentrations

Element	Mass/ Mode #	Calibration Conc. (ug/L)	ICV/CCV Conc. (ug/L)	RL Std. Conc. (ug/L)	Linear Range Std. Conc. (ug/L)*	Matrix Spike / Post Spike Conc.	
						Water (ug/L)	Soil (mg/kg)
Aluminum (Al)	27 / #1	10000, 5000, 2500, 1000,10	400/500	10	100000	5000	500
Antimony (Sb)	121 / #3	100,50,25,10,1.0	40/50	1.0	100	50	5
Arsenic (As)	75 / #2	100,50,25,10,0.5	40/50	0.50	2000	100	10
Barium (Ba)	137 / #3	100,50,25,10,1.0	40/50	1.0	2000	100	10
Beryllium (Be)	9 / #3	100,50,25,10,0.1	40/50	0.10	100	50	5.0
Boron (B)	11 / #3	200,100,50,20	80/100	20	2000	200	20
Cadmium (Cd)	111 / #3	100,50,25,10,0.5	40/50	0.50	100	50	5.0
Calcium (Ca)	40 / #1	10000,5000,100,50	4000/ 5000	50	100000	5000	500
Chromium (Cr)	52 / #2	100,50,25,10,1.0	40/50	1.0	2000	100	10
Cobalt (Co)	59 / #3	100,50,25,10,0.1	40/50	0.10	2000	50	5
Copper (Cu)	65 / #2	100,50,25,10,1.0	40/50	1.0	100	100	10
Iron (Fe)	56 / #1	10000,5000,100,50,20	4000/ 5000	20	100000	5000	500
Lead (Pb)	208 / #3	100,50,25,10,0.3	40/50	0.30	2000	50	5.0
Magnesium (Mg)	24 / #1	10000,5000,100,50	4000/ 5000	50	100000	5000	500
Manganese (Mn)	55 / #3	100, 50, 25, 10, 1.0	400/500	1.0	2000	500	50
Mercury (Hg)	202 / #3	5,2.5,1.25,0.5,0.16	2.0/2.5	0.16	100	5.0	0.50
Molybdenum (Mo)	95 / #3	100,50,25,10,1.0	40/50	1.0	2000	100	10
Nickel (Ni)	60 / #2	100,50,25,10,0.2	40/50	0.20	2000	100	10
Potassium (K)	39 / #2	10000,5000,100,50	4000/ 5000	50	100000	5000	500
Selenium (Se)	78 / #1	100,50,25,10,0.5	40/50	0.50	100	100	10
Silver (Ag)	107 / #3	100,50,25,10,0.2	40/50	0.20	100	50	5.0
Sodium (Na)	23 / #1	10000,5000,100,50	4000/ 5000	50	100000	5000	500

Element	Mass/ Mode #	Calibration Conc. (ug/L)	ICV/CCV Conc. (ug/L)	RL Std. Conc. (ug/L)	Linear Range Std. Conc. (ug/L)*	Matrix Spike / Post Spike Conc.	
						Water (ug/L)	Soil (mg/kg)
Strontium (Sr)	88 / #3	100,50,25,10,0.2	40/50	0.20	2000	100	10
Thallium (Tl)	205 / #3	100, 50, 25,10,0.2	8/10	0.20	20	40	4.0
Thorium (Th)	232 / #3	100,50,25,10,0.5	40/50	0.5	100	100	10
Tin (Sn)	118 / #3	100,50,25,10,1.0	40/50	1.0	2000	100	10
Titanium (Ti)	47 / #3	100,50,25,10,1.0	40/50	1.0	2000	100	10
Uranium (U)	238 / #3	100,50,25,10,0.5	40/50	0.5	100	100	10
Vanadium (V)	51 / #2	100,50,25,10,1.0	40/50	1.0	2000	100	10
Zinc (Zn)	66 / #3	100,50,25,10,4.0	40/50	4.0	2000	100	10

*For guidance only - instrument sensitivity will vary.

Attachment 7:
Standard Preparation

Note: All standards must be stored at room temperature. Standards containing mercury have a 28 day expiration date from the date prepared. All other standards have an expiration date of 6 months from date prepared.

All calibration standards and working standards are prepared using a rinse/blank solution containing 0.5% Hydrochloric Acid and 2% Nitric Acid by volume. The solution is prepared as follows:

Standard Final Volume (mL)	Hydrochloric acid (mL)	Nitric Acid (mL)
20000	100	400

Note: Adjustments to the final volume are acceptable provided that the acid by volume concentrations are adhered to.

CALIBRATION STANDARDS

Calibration Standard Stock Solution

Element	Parent Concentration (mg/L)	Volume Added (mL)	Final Volume (mL)	Final Volume (mg/L)
Aluminum (Al)	10000	1.0	100	100
Antimony (Sb)	1000	1.0		10
Arsenic (As)	1000	1.0		10
Barium (Ba)	1000	1.0		10
Beryllium (Be)	1000	1.0		10
Boron (B)	1000	2.0		20
Cadmium (Cd)	1000	1.0		10
Calcium (Ca)	10000	10		1000
Chromium (Cr)	1000	1.0		10
Cobalt (Co)	1000	1.0		10
Copper (Cu)	1000	1.0		10
Iron (Fe)	10000	10		1000
Lead (Pb)	1000	1.0		10
Magnesium (Mg)	10000	10		1000
Manganese (Mn)	1000	10		100
Mercury (Hg)	1000	0.050		0.50
Molybdenum (Mo)	1000	1.0		10
Nickel (Ni)	1000	1.0		10
Potassium (K)	10000	10		1000
Selenium (Se)	1000	1.0		10
Silver (Ag)	1000	1.0		10
Sodium (Na)	10000	10		1000
Strontium (Sr)	1000	1.0		10
Thallium (Tl)	1000	0.2		2.0
Thorium (Th)	1000	1.0		10
Tin (Sn)	1000	1.0		10
Titanium (Ti)	1000	1.0		10
Uranium (U)	1000	1.0		10
Vanadium (V)	1000	1.0		10
Zinc (Zn)	1000	1.0		10

The above standards may be grouped together into more than one stock standard to maximize the stability of the standards. For example, silver may need to be kept as a separate stock due to the stability of the silver in solution. Alternatively, a custom made vendor supplied Calibration Stock Standard may be purchased instead of making it in the laboratory.

WORKING CALIBRATION STANDARDS

Calibration Blank (ICB, CCB) – All standards are made up to the same acid concentrations as the calibration blank

Add 500mL to 600mL of reagent water to a clean 1L volumetric flask. Add 20mL of concentrated nitric acid (HNO_3) and 5.0mL of hydrochloric acid (HCl) to the volumetric flask. Dilute to a final volume of 1000mL with reagent water. Store the standard at room temperature. Other volumes may be prepared at the discretion of the lab. The nitric acid concentration must be 2% by volume, and the hydrochloric acid concentration must be 0.5% by volume.

Calibration Standards concentration (ug/L) – example for a final volume of 1000mL

Element	Standard 5 (10mL stock)	Standard 4 (5mL stock)	Standard 3 (2.5mL stock)	Standard 2 (1mL stock)
Aluminum (Al)	1000	500	250	100
Antimony (Sb)	100	50	25	10
Arsenic (As)	100	50	25	10
Boron (B)	200	100	50	20
Barium (Ba)	100	50	25	10
Beryllium (Be)	100	50	25	10
Cadmium (Cd)	100	50	25	10
Calcium (Ca)	10000	5000	2500	1000
Cobalt (Co)	100	50	25	10
Chromium (Cr)	100	50	25	10
Copper (Cu)	100	50	25	10
Iron (Fe)	10000	5000	2500	1000
Lead (Pb)	100	50	25	10
Magnesium (Mg)	10000	5000	2500	1000
Manganese (Mn)	1000	500	250	100
Mercury (Hg)	5.0	2.5	1.25	0.5
Molybdenum (Mo)	100	50	25	10
Nickel (Ni)	100	50	25	10
Potassium (K)	10000	5000	2500	1000
Selenium (Se)	100	50	25	10
Silver (Ag)	100	50	25	10
Sodium (Na)	10000	5000	2500	1000
Strontium (Sr)	100	50	25	10
Thallium (Tl)	20	10	5.0	2.0
Thorium (Th)	100	50	25	10
Tin (Sn)	100	50	25	10
Titanium (Ti)	100	50	25	10
Uranium (U)	100	50	25	10
Vanadium (V)	100	50	25	10
Zinc (Zn)	100	50	25	10

Note: The Standard 5 listed above is utilized when calibrating with a single standard and a blank.

Standard 1 is equivalent to the RL/PQL Working Standard described below.

CONTINUING CALIBRATION VERIFICATION (CCV) STANDARD

The calibration standard Level 4 is used as the continuing calibration verification standard.

INITIAL CALIBRATION VERIFICATION (ICV) SOLUTION

All ICV standards must be from a different source than those used for the calibration standards.
Note: ICV must be made fresh daily.

Preparation of the ICP-MS initial calibration verification solution

Element	Parent Concentration (mg/L)	Volume Added (mL)	Final Volume (mL)	Final Concentration (mg/L)
Aluminum (Al)	10000	0.40	100	40
Antimony (Sb)	1000	0.40		4.0
Arsenic (As)	1000	0.40		4.0
Boron (B)	1000	0.80		8.0
Barium (Ba)	1000	0.40		4.0
Beryllium (Be)	1000	0.40		4.0
Cadmium (Cd)	1000	0.40		4.0
Calcium (Ca)	10000	4.0		400.0
Cobalt (Co)	1000	0.40		4.0
Chromium (Cr)	1000	0.40		4.0
Copper (Cu)	1000	0.40		4.0
Iron (Fe)	10000	4.0		400.0
Lead (Pb)	1000	0.40		4.0
Magnesium (Mg)	10000	4.0		400.0
Manganese (Mn)	1000	1.0		40.0
Mercury (Hg)	100	0. 40		4.0
Molybdenum Mo)	1000	0.40		4.0
Nickel (Ni)	1000	0.40		4.0
Potassium (K)	10000	4.0		400.0
Selenium (Se)	1000	0.40		4.0
Silver (Ag)	1000	0.40		4.0
Sodium (Na)	10000	4.0		400.0
Strontium (Sr)	1000	0.40		4.0
Thallium (Tl)	1000	0.20		0.8
Tin (Sn)	1000	0.40		4.0
Thorium (Th)	1000	0.40		4.0
Titanium (Ti)	1000	0.40		4.0
Vanadium (V)	1000	0.40		4.0
Uranium (U)	1000	0.40		4.0
Zinc (Zn)	1000	0.40		4.0

Preparation of Initial Calibration Verification Working Standard

Dilute 1.0mL of the ICP-MS initial calibration verification solution to a final volume of 100mL using the same matrix solution as the calibration blank.

The final concentrations of the various elements are the same as listed in Table 1.

REPORTING LIMIT (RL) CHECK STANDARD

RL/PQL Stock A

Element	Parent Concentration (mg/L)	Volume Added (mL)	Final Volume (mL)	Final Concentration (mg/L)
Aluminum (Al)	10000	1.0	100	100
Antimony (Sb)	1000	1.0		10
Arsenic (As)	1000	0.5		5.0
Boron (B)	1000	20		200
Barium (Ba)	1000	1.0		10
Beryllium (Be)	1000	0.10		1.0
Cadmium (Cd)	1000	0.10		1.0
Calcium (Ca)	10000	5.0		500
Cobalt (Co)	1000	0.1		1.0
Chromium (Cr)	1000	1.0		10
Copper (Cu)	1000	1.0		10
Iron (Fe)	10000	2.0		200
Lead (Pb)	1000	0.30		3.0
Magnesium (Mg)	10000	5.0		500
Manganese (Mn)	1000	1.0		10
Molybdenum Mo)	1000	1.0		10
Nickel (Ni)	1000	1.0		10
Potassium (K)	10000	5.0		500
Selenium (Se)	1000	0.50		5.0
Sodium (Na)	10000	5.0		500
Strontium (Sr)	1000	0.20		2.0
Thallium (Tl)	1000	0.20		2.0
Thorium (Th)	1000	0.5		5.0
Tin (Sn)	1000	1.0		10
Titanium (Ti)	1000	1.0		10
Uranium (U)	1000	0.5		5.0
Vanadium (V)	1000	1.0		10
Zinc (Zn)	1000	4.0		40

***** Other standard concentrations may be used to verify higher or lower reporting limits.

RL/PQL Stock B

Element	Parent Standard (mg/L)	Volume Added (mL)	Final Volume (mL)	Final Concentration (mg/L)
Mercury (Hg)	1000	0.050	100	0.50

RL/PQL Stock C

Element	Parent Standard (mg/L)	Volume Added (mL)	Final Volume (mL)	Final Concentration (mg/L)
Silver (Ag)	1000	0.020	100	0.20

Preparation of RL/PQL Working Standard

Dilute 10uL of the RL/PQL Stock A, 32uL of the RL/PQL Stock B, and 100uL of the RL/PQL Stock C to a final volume of 100mL using the same matrix solution as the calibration blank.

ICP INTERFERENCE CHECK SOLUTIONS (for EPA 6020A and EPA 6020B)

Preparation of ICP Interference Check Solution A

Element	Parent Standard (mg/L)	Volume Stock A (mL)	Final Volume (mL)	Final Concentration (mg/L)
Aluminum (Al)	1000	50	500	100
Calcium (Ca)	1000			100
Iron (Fe)	1000			100
Magnesium (Mg)	1000			100
Sodium (Na)	1000			100
Phosphorus (P)	1000			100
Potassium (K)	1000			100
Sulfur (S)	1000			100
Carbon (C)	2000			200
Chloride (Cl)	10000			1000
Molybdenum (Mo)	20			2.0
Titanium (Ti)	20			2.0

Preparation of ICP Interference Check Solution AB

Element	Parent Concentration (mg/L)	Volume Stock A (mL)	Volume Stock B (mL)	Final Volume (mL)	Final Concentration (mg/L)
Aluminum (Al)	1000	50	1.0	500	100
Calcium (Ca)	1000				100
Iron (Fe)	1000				100
Magnesium (Mg)	1000				100
Sodium (Na)	1000				100
Phosphorus (P)	1000				100
Potassium (K)	1000				100
Sulfur (S)	1000				100
Carbon (C)	2000				200
Chloride (Cl)	10000				1000
Molybdenum (Mo)	20				2.0
Titanium (Ti)	20				2.0
Arsenic (As)	10				0.020
Cadmium (Cd)	10				0.020
Chromium (Cr)	10				0.020
Cobalt (Co)	10				0.020
Copper (Cu)	10				0.020
Manganese (Mn)	10				0.020
Nickel (Ni)	10				0.020
Silver (Ag)	10				0.020
Zinc (Zn)	10				0.020

ICP-MS LCS/MATRIX SPIKE SOLUTION

Preparation of the ICP-MS Matrix Spiking / Post Digestion Spiking Solution

Element	Parent Concentration (mg/L)	Volume Added (mL)	Final Volume (mL)	Final Concentration (mg/L)
Aluminum (Al)	10000	5.0	100	500
Antimony (Sb)	1000	0.5		5
Arsenic (As)	1000	1.0		10
Boron (B)	1000	2.0		20
Barium (Ba)	1000	1.0		10
Beryllium (Be)	1000	0.5		5.0
Cadmium (Cd)	1000	0.5		5.0
Calcium (Ca)	10000	5.0		500
Cobalt (Co)	1000	1.0		5.0
Chromium (Cr)	1000	1.0		10
Copper (Cu)	1000	1.0		10
Iron (Fe)	10000	5.0		500
Lead (Pb)	1000	0.50		5.0
Magnesium (Mg)	10000	5.0		500
Manganese (Mn)	1000	5.0		50
Mercury (Hg)	1000	0.050		0.50
Molybdenum Mo)	1000	1.0		10
Nickel (Ni)	1000	1.0		10
Potassium (K)	10000	5.0		500
Selenium (Se)	1000	1.0		10
Silver (Ag)	1000	0.50		5.0
Sodium (Na)	10000	5.0		500
Strontium (Sr)	1000	1.0		10
Thallium (Tl)	1000	0.4		4.0
Thorium (Th)	1000	1.0		10
Tin (Sn)	1000	1.0		10
Titanium (Ti)	1000	1.0		10
Uranium (U)	1000	1.0		10
Vanadium (V)	1000	1.0		10
Zinc (Zn)	1000	1.0		10

Separate mixtures can be utilized for stability considerations.

MDL SOLUTION

The MDL solution is used to determine the Method Detection Limit (MDL) of each target analyte (SOP SA-QA-007).

Preparation of Recommended MDL Intermediate Stock Solution

Element	Parent Concentration (mg/L)	Volume Added (mL)	Final Volume (mL)	Final Concentration (mg/L)
Antimony (Sb)	1000	1.25	100	12.5
Arsenic (As)	1000	1.0		10
Barium (Ba)	1000	1.0		10
Beryllium (Be)	1000	0.25		2.5
Cadmium (Cd)	1000	0.10		1.0
Cobalt (Co)	1000	0.20		2.0
Chromium (Cr)	1000	2.5		25.0
Copper (Cu)	1000	1.0		10
Lead (Pb)	1000	0.30		3.0
Magnesium (Mg)	10000	1.0		100
Manganese (Mn)	1000	1.0		10
Molybdenum Mo)	1000	10		10
Nickel (Ni)	1000	1.0		10
Selenium (Se)	1000	1.0		10
Silver (Ag)	1000	0.20		2.0
Strontium (Sr)	1000	0.50		5.0
Thallium (Tl)	1000	0.5		5.0
Tin (Sn)	1000	1.0		10
Titanium (Ti)	1000	1.25		12.5
Vanadium (V)	1000	5.0		50
Aluminum (Al)	10000	2.5		250
Boron (B)	1000	25.0		250.0
Calcium	10000	12.5		1250.0
Iron (Fe)	10000	2.5		250
Potassium (K)	10000	10.0		1000
Sodium (Na)	10000	10.0		1000
Zinc	1000	5.0		50.0

Preparation of MDL Working Solutions:

Preparation of the MDL Working solution is as follows dilute 2.0mL of the MDL Intermediate stock solution and 2.0mL of the Mercury Intermediate Calibration standard and bring to a final volume of 100mL. The standard is now ready for digestion.

Linearity Check Solutions:
The linearity check solutions are prepared individually according to the following equation:

$$Vs = \frac{Vlc \otimes Clc}{Cs}$$

Where: Vs = volume of stock standard (mL)
Cs = concentration of stock standard (mg/L)
Vlc = volume of linearity check standard to prepare (mL)
Clc = concentration of linearity check standard to prepare (mg/L)

The linearity check solutions are prepared at the concentrations specified in Attachment 6. Prepare sufficient volume to perform the linearity check, maintaining the hydrochloric acid concentration at 0.5% by volume and the nitric acid concentration at 1% by volume.

Internal Standards:
A solution at 1mg/L is prepared for all elements. That solution is added in-stream by the use of a T-fitting. The sample is pumped into the T-fitting with a white/white peristaltic pump tube and the internal standard is pumped in using a blue/orange pump tube.

Attachment 8:
Drinking Water MCLGs and MCLs

Element	Regulation	MCLG (mg/L)	MCL (mg/L)	Secondary Standard (mg/L)
Antimony	National Primary Drinking Water Regulations	0.006	0.0060	
Arsenic		0	0.010	
Barium		2.0	2.0	
Beryllium		0.0040	0.0040	
Cadmium		0.0050	0.0050	
Chromium		0.10	0.10	
Copper [1]		1.3	Action Level = 1.3	
Lead [1]		0	Action Level = 0.015	
Mercury		0.0020	0.0020	
Selenium		0.050	0.050	
Thallium		0.00050	0.0020	
Uranium		0	0.030	
Aluminum	National Secondary Drinking Water Regulations			0.05 – 0.2
Copper				1.0
Iron				0.30
Manganese				0.050
Silver				0.10
Zinc				5.0

[1] Lead and copper are regulated by a Treatment Technique that requires systems to control the corrosiveness of their water. If more than 10% of tap water samples exceed the action level, water systems must take additional steps. For copper, the action level is 1.3 mg/L, and for lead is 0.015 mg/L.

Attachment 9:
Hardness Calculation Work Instruction

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Work Instruction
FME157:05.23.07:0
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CALCULATION OF HARDNESS

(as Calcium Carbonate)

Method: SM2340B

Summary of Procedure:

An aqueous sample is analyzed for calcium and magnesium using ICP by methods EPA 200.7 or 6010B (SOP ME70: *Elements by ICP)*, or using ICP/MS by methods EPA 200.8 or 6020 (SOP ME74*: Elements by ICP/MS)*. The concentrations of these elements are converted to equivalents of calcium carbonate using the equation below and added together to give the final result.

Calculation:

Determine the concentration of calcium and magnesium as described in SOP ME70 or ME74.

Calculate the hardness as calcium carbonate using the following equation:

Hardness (as mg/L calcium carbonate) = (4.118 X [Mg]) + (2.497 X [Ca])

where:
[Mg] = Concentration of magnesium (mg/L)
[Ca] = Concentration of calcium (mg/L)

Quality Control:

Report the method blank as the default QC unless otherwise requested.

Formal detection limit studies, as described in 40CFR Part 136B and SOP QA07: *Determination of Detection Limits,* are not required for SM2340B.

This procedure is a calculation based on another analytical procedure; therefore, demonstration of capability (DOC) is accomplished by successful completion of analytical DOCs. (i.e. If the analyst has a successful DOC on file for Ca and Mg by EPA 200.7/6010B or EPA 200.8/6020, proficiency has been shown for SM2340B by default.)

Attachment 10:
Glassware Cleaning Procedures

GLASSWARE CLEANING PROCEDURES METALS DEPARTMENT
Graduated Cylinders 1. Scrub with hot, soapy H_2O and brush. 2. Rinse thoroughly with tap H2O. 3. Rinse with 10% HNO_3 4. Rinse thoroughly with DI H_2O.
Volumetric Flasks 1. Empty contents of flask 2. Squirt a small amount of cleaning detergent directly into volumetric flask 3. Fill flask 1/3 full with **HOT** H_2O 4. Replace top and shake flask. 5. Empty flask and rinse with **HOT** H_2O until no soap remains in flask. 6. Add approximately 10mL concentrated HNO_3 to 50mL, 100mL, and 250mL flasks ---- replace top and shake well. For 500mL or 1000mL flasks use 25mL and for 10mL flasks use 2 - 5mL of concentrated HNO_3. 7. Rinse 3 times with DI H_2O, filling flask 1/3 full and replacing top. Store until needed. *** NEVER PLACE VOLUMETRIC FLASKS OR TOPS IN SINK OR DISHPAN WITH OTHER DIRTY DISHES.**
***Dispose of all acid waste in accordance with the Eurofins TestAmerica Savannah Addendum to the Corporate Safety Manual.**

18.0 Revision History

Summary of Changes:

Added reference to the Master Equipment Listing maintained by QA to Section 6.1. ANAB Audit 2020.

Summary of Changes from Previous Revision 13:

- Minor editorial, grammatical, and/or formatting changes made.
- Updated SOP signatories to reflect current responsibilities and titles.
- Updated company name and logo where applicable.
- Updated Attachment 7, Attachment 5 and Attachment 6 with current information.
- Updated Attachment 9 with current logo.
- Updated section 10.3.1 with current instrument instructions.

ORGANOCHLORINE PESTICIDES AND POLYCHLORINATED BIPHENYLS (PCBs) BY GC/ECD

(Methods: EPA 608, EPA 608.3, EPA 8081B, and EPA 8082A)

Approvals (Signature/Date):

Kimberly Chamberlain 11/23/2021

Kim Chamberlain Date
Quality Assurance Manager /Technical Director
Environmental Health and Safety Coordinator

Brad Mullis 11/23/2021

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Department Manager

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1.0 Scope and Application

This SOP gives the procedures for the determination of organochlorine pesticides (including single component pesticides, toxaphene, and technical chlordane) and polychlorinated biphenyls (PCBs) as Aroclors via gas chromatography/electron capture detection (GC/ECD).

> Note: This SOP outlines two options for toxaphene quantitation. The first option is the laboratory's default option and includes single peak quantitation. The second option is utilized to meet project-specific objectives and includes quantitation via total area under the curve (TAUC).

The routine matrices performed by this procedure are waters and soils. Other matrices which may be performed include wipes, leachates, tissues, and wastes.

A complete target analyte list, the reporting limits (RL), the method detection limits (MDL), and the accuracy and precision criteria associated with this procedure are provided in the LIMS Method Limit Groups (MLGs).

This SOP was written by and for TestAmerica's Savannah laboratory.

2.0 Summary of Method

Liquid samples are extracted using continuous liquid-liquid or separatory funnel extraction with methylene chloride. The extract is concentrated, the solvent is exchanged to hexane, and the final volume of the extract is adjusted.

1. If the High Volume preparation procedure is requested, 1000mL of sample is extracted and concentrated to a final volume of 5mL.
2. If the Low Volume Initiative (LVI) preparation procedure is requested, 250mL of sample is extracted and concentrated to a final volume of 1mL

Soil samples are extracted using the microwave extraction procedure. Approximately 15g of the homogenized sample is dried with anhydrous sodium sulfate and extracted with 1:1 acetone/hexane. The extract is concentrated to a final volume of 5mL.

The preparation procedures may also incorporate copper (sulfur) clean-up or acid clean-up (used for PCBs and TUAC only), as outlined in the applicable preparation SOP.

> Note: Sulfuric acid clean-up is required for the Toxaphene TAUC analysis/projects.

The extract is analyzed by gas chromatography using dual capillary columns (different phases) and dual electron capture (EC) detectors. This configuration allows for simultaneous detection and confirmation of the target compounds. Identification of the target compounds in samples is performed by comparing the retention times of the peaks with standards analyzed under the same GC conditions. Quantitation is performed using the internal standard calibration technique.

This SOP is based on the following methods: EPA Methods 608, 608.3, 8081B, 8082A, the TAUC Method, and the Task Force Method 1.

3.0 Definitions

Refer to the Glossary Section of the *Quality Assurance Manual* (QAM) for a complete listing of applicable definitions and acronyms.

4.0 Interferences

4.1 Procedural Interferences

4.1.1 Interferences may be caused by contaminants in solvents, reagents, glassware, and other sample processing apparatus and can make identification and/or quantification of the target analytes difficult.

4.1.2 All sample collection containers are single-use disposable containers which limits the potential for contamination. All non-disposable labware must be scrupulously cleaned in accordance with the posted Labware Cleaning Instructions to ensure it is free from contaminants and does not contribute artifacts.

4.1.3 High purity reagents and solvents are used to help minimize interference problems. Acetone, hexane and methylene chloride must be verified prior to use in accordance with the TestAmerica Solvent Lot Testing Program.

4.1.4 Instrument and/or method blanks are routinely used to demonstrate all reagents and apparatus are free from interferences under the conditions of the analysis.

4.2 Matrix Interferences

4.2.1 Matrix interferences may be caused by contaminants that are co-extracted from the sample matrix. The sample may require cleanup or dilution prior to analysis to reduce or eliminate the interferences. All possible cleanup procedures are listed below. If matrix interferences continue after a cleanup has been performed, the sample is diluted as needed for data analysis. If a cleanup is used, the method blank and laboratory control standard must also be subjected to the cleanup.

Clean-up Procedure	Application	Effectiveness
Sulfuric acid	PCBs Toxaphene TAUC*	Eliminates polar and some unsaturated hydrocarbon interferences
Copper	Pest/PCBs	Eliminates elemental sulfur

*Sulfuric acid clean-up is required for the Toxaphene TAUC analysis/projects.

Copper is effective at removing elemental sulfur, which elutes as a very large peak in the middle of the chromatogram. Treatment with sulfuric acid is a very effective means of removing polar compounds and unsaturated compounds from the sample matrix. These two cleanups require fewer sample manipulations, and should have minimal negative impact on the recoveries of the target compounds and surrogates.

4.2.2 Interfering contamination may occur when a sample containing low concentrations of analytes is analyzed immediately following a sample containing relatively high concentrations of analytes. As such, samples known to be clean should be analyzed first. To prevent carryover into subsequent samples, analysis of reagent blanks may be needed after the analysis of a sample containing high concentrations of analytes.

5.0 Safety

Employees must abide by the policies and procedures in the TestAmerica Environmental Health and Safety Manual (EHSM), the TestAmerica Savannah Addendum to the EHSM, and this document.

This procedure may involve hazardous materials, operations, and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user to follow appropriate safety, waste disposal, and health practices under the assumption that all samples and reagents are potentially hazardous.

The analyst must protect himself/herself from exposure to the sample matrix. Many of the samples that are tested may contain hazardous chemical compounds or biological organisms. The analyst must, at a minimum, wear protective clothing (lab coat), eye protection (safety glasses or face shield), disposable nitrile gloves, and closed-toe, nonabsorbent shoes when handling samples.

5.1 Specific Safety Concerns or Requirements

Acetone and hexane are flammable solvents. They can cause irritation to the respiratory tract. Overexposure can cause fatigue, confusion, headache, dizziness, and drowsiness.

The gas chromatograph contains zones that have elevated temperatures. The analyst needs to be aware of the locations of those zones, and must cool them to room temperature prior to working on them.

There are areas of high voltage in the gas chromatograph. Depending on the type of work involved, either turn the power to the instrument off, or disconnect it from its source of power.

5.2 Primary Materials Used

The following is a list of the materials used in this procedure, which have a serious or significant hazard rating, and a summary of the primary hazards listed in their MSDS/SDS.

Note: This list does not include all materials used in the procedure. A complete list of materials used in this procedure can be found in the Reagents and Standards Section and the Equipment and Supplies Section of this SOP.

Employees must review the information in the MSDS/SDS for each material before using it for the first time or when there are major changes to the MSDS/SDS. Electronic copies of MSDS/SDS can be found using the "MSDS Online" button on the Oasis homepage, on the EH&S webpage on Oasis, and through LIMS File System Shares.

Material	Hazards	Exposure Limit[1]	Signs and Symptoms of Exposure
Acetone	Flammable	1000ppm TWA	Inhalation of vapors irritates the respiratory tract. May cause coughing, dizziness, dullness, and headache.
Hexane	Flammable Irritant	500ppm TWA	Inhalation of vapors irritates the respiratory tract. Overexposure may cause lightheadedness, nausea, headache, and blurred vision. Vapors may cause irritation to the skin and eyes.
[1]Exposure limit refers to the OSHA regulatory exposure limit.			

6.0 Equipment and Supplies

6.1 Equipment and Instrumentation

Analytical Balance – Verify in accordance with SOP SA-AN-100 *Laboratory Support Equipment (Verification and Use)*

Top-loading Balance – Verify in accordance with SOP SA-AN-100 *Laboratory Support Equipment (Verification and Use)*

Data System – CHROM and Chemstation software are used to acquire, store, reduce, and output gas chromatographic/electron capture data. The software have the capability of processing stored GC data by recognizing a GC peak within any given retention time window, comparing the retention time of the GC peak with retention time window of a known standard. The software also allows integration of the peak, calculation of response factors and/or construction of a linear regression calibration curve, calculation of response factor statistics (mean and standard deviation), and calculation of concentrations of analytes using either the calibration curve or the response factors.

Agilent Gas Chromatograph (6890 or 6890N) – equipped with dual detectors (an ECD pair) and an autosampler.

Recommended column pairs:

CLP I fused silica capillary column 30m x 0.25mm ID x 0.25um film
CLP II fused silica capillary column 30m x 0.25mm ID x 0.20um film

The recommended configuration is to connect two capillary columns to a single injection port via a y-splitter and a length of guard column. The ends of each column are connected to separate detectors. A single injection is split between the two columns to provide simultaneous detection and confirmation of the target compounds.

6.2 Volumetric Containers and Dispensers

All volumetric labware must be verified in accordance with SOP SA-AN-100: *Laboratory Support Equipment (Verification and Use).* Refer to Attachment 16 for Labware Cleaning Procedures.

Volumetric Labware	Volume	Type (Quantitative / Qualitative)	Use	Verification Frequency	Laboratory Verification Criteria
Glass Transfer Pipettes	Various	Qualitative	Extract Aliquotting	None	None
Glass Disposable Pipettes	Various	QUANTITATIVE	Extract Aliquotting; Dilution Preparation	Per Lot	Accuracy = 2% Precision = 1%
Mechanical Eppendorf-Style Pipettes	Various	QUANTITATIVE	Standard and Dilution Preparation	Initially, prior to first use, then quarterly thereafter (Daily per DOD)	Accuracy = 2% Precision = 1%
Extract Vials	12mL	Qualitative	Extract Storage	None	None
Autosampler Vials (w/ Septa & Cap)	2mL	Qualitative	Instrument Analysis	None	None
Gas Tight Syringes	Various	QUANTITATIVE	Standard Preparation	None, if received w/ COA	None, if received w/ COA
Volumetric Containers	Various	QUANTITATIVE	Standard Preparation	None, Class A	None, if received w/ COA

6.3 Lab Supplies

Detergent – FL70 or equivalent, used for washing non-disposable labware.

6.4 Sample Collection Containers

All sample collection containers are pre-cleaned, single-use disposable containers, which limits the potential for contamination. The routine sample collection containers supplied by the laboratory are:

High Volume preparation procedure = 1L amber without preservative.
Low Volume preparation procedure = 250mL amber without preservative

7.0 Reagents and Standards

7.1 Expiration Dates

Expiration dates (time from initial use or receipt to final use) for standard and reagent materials must be set according to the guidance in this SOP. Note: These are maximum expiration dates and are not to be considered an absolute guarantee of standard or reagent quality. Sound judgment must be used when deciding whether to use a standard or reagent. If there is doubt about the quality of a standard or reagent material, a new material must be obtained or the standard or reagent material verified. Data quality must not be compromised to extend a standard's life – i.e., when in doubt, throw it out.

The expiration date of any standard or reagent must not exceed the expiration date of the standard or reagent that was used to prepare it; that is, the "children may not outlive the parents".

Unless listed elsewhere in this SOP, the expiration dates given below apply.

7.2 Reagents

Reagents must be prepared and documented in accordance with SOP SA-AN-041: *Reagent and Standard Materials Procedures.*

Acetone, Hexane and methylene chloride must be verified prior to use in accordance with the TestAmerica Solvent Lot Testing Program.

Hexane - residue grade or better; J.T. Baker 9262-03 (4L)
Storage: Room temperature, Flammable Solvent cabinet
Expiration: Unopened: Manufacturer's expiration date
Opened: Manufacturer's expiration date

7.3 Standards

Standards must be prepared and documented in accordance with SOP SA-AN-041: *Reagent and Standard Materials Procedures.* Certificates of analysis (COAs) or purity must be received with all purchased standards, and scanned and attached to the standard in LIMS.

Attachment 9 contains the recipes for the calibration standards for the routine target compounds.

Note: Stock standards must be stored per the manufacturer's recommendations (i.e., room temperature, refrigerator, or freezer). Secondary dilutions and working standards must be stored at 0-6°C (less than 6°C but not frozen).

8.0 Sample Collection, Preservation, Shipment, and Storage

8.1 Aqueous Samples

Aqueous samples for the High Volume preparation procedure are routinely collected in unpreserved 1L amber glass containers. Aqueous samples for the Low Volume preparation procedure are routinely collected in unpreserved 250mL amber glass containers.

Samples must be iced at the time of collection and maintained at 0-6°C (less than 6°C but not frozen) until the time of preparation. Samples must be prepared within 7 days of collection. Extracts must be stored separately at 0-6°C (less than 6°C but not frozen) until the time of analysis and analyzed within 40 days of extraction.

Samples must be checked for pH and for the presence of residual chlorine prior to preparation. NCMs must be initiated for samples collected in improper containers, for samples with extreme pH, or for sample containing residual chlorine. Refer to the applicable preparation SOP for sample preservation check information.

Note: EPA SW-846 Update IV Chapter 4 re-defined the holding times for PCBs as "NONE". The EPA Methods Update Rule re-defined holding times for PCBs as one year from collection to extraction and one year from extraction to analysis. The laboratory has incorporated the extended preparation holding times for PCBs on a project-specific basis, as allowed by that particular state agency and client.

8.2 Soil Samples

Soil samples are routinely collected in 8oz glass soil jars.

Samples must be iced at the time of collection and maintained at 0-6°C (less than 6°C but not frozen) until the time of preparation. Samples must be prepared within 14 days of collection. Extracts must be stored separately at 0-6°C (less than 6°C but not frozen) until the time of analysis and analyzed within 40 days of extraction.

9.0 Quality Control

SOP SA-QA-017: *Evaluation of Batch QC Data* and the SOP Summary in Attachment 3 provide requirements for evaluating QC data.

9.1 Batch QC

An extraction batch consists of up to 20 environmental samples and the associated QC items extracted together within a 24 hour period.

The minimum QC items required for each extraction batch are as follows:

EPA 608:
a method blank and a laboratory control sample (LCS), a matrix spike (MS) per 10% of samples analyzed, and a matrix spike duplicate (MSD).

This frequency equates to the following:

- For a batch of 10 or fewer samples, the minimum QC items are a method blank, an LCS, 1 matrix spike, and 1 matrix spike duplicate.
- For a batch of 11-20 samples, the minimum QC items are a method blank, an LCS, 1 matrix spike (from sample 1-10), another matrix spike (from sample 11-20), and a matrix spike duplicate.

8000-series and EPA 608.3:
A method blank (MB) and a laboratory control sample (LCS), a matrix spike (MS), and a matrix spike duplicate (MSD).

The routine container supplied for the High Volume preparation procedure is a 1L container. The routine container supplied for the Low Volume preparation procedure is a 250mL container. The minimum sample volume required for the High Volume preparation procedure is 500mL. Reduced volumes are not applicable to the Low Volume preparation procedure.

Note: Final volumes and spike amounts must be adjusted to compensate for these reduced initial volumes.

If there is insufficient sample volume to perform the required matrix spike(s), an NCM must be initiated on all affected samples to denote this situation. Insufficient sample volume is defined as receiving less than a total of 2L for the High Volume preparation procedure or less than 3 full 250mL containers for the Low Volume (LVI) preparation procedure.

If there is insufficient sample volume available to perform the batch MS/MSD, the LCS must be prepared in duplicate (i.e., LCSD). If an LCS and LCSD are performed, both QC items must be evaluated and reported. Acceptable recoveries (as well as %RPD) for both LCS and LCSD are required. An NCM must be initiated on all samples within the batch to denote this situation.

Note: Unless the client has specified which sample to use as the matrix spike, the matrix spike must be selected at random from the samples in the batch. Field QC (e.g., equipment blanks, trip blanks, and field blanks) must not be used for MS/MSD unless specifically requested to do so by the client or unless there is insufficient sample available for performing MS/MSD using an actual field sample.

Batch QC must meet the criteria given in Attachment 3 of this SOP.

Note: The laboratory has defined the analyte listed in Attachment 11 as a poor or erratic performer and allows for exceptions to the LCS/LCSD, MS/MSD, and Sporadic Marginal Exceedance criteria for this analyte. An NCM must be initiated to denote this situation.

Note: The use of Sporadic Marginal Exceedances, as defined in SOP SA-QA-017, is not permitted for samples originating from South Carolina.

9.2 Instrument QC

9.2.1 PEVAL Breakdown Standard

The column(s) must be evaluated prior to the analysis of the calibration standards and once every 12-hour clock for EPA 8081B and once every 24-hour clock for EPA 608 and EPA 608.3. The column evaluation is performed by injecting a PEVAL standard that contains Endrin and p,p'-DDT and calculating the percent breakdown of these compounds. The standard used for determining the percent breakdown must not contain any compounds that coelute with Endrin, DDT, or any of the corresponding breakdown products.

Note: This column evaluation does not have to be performed if PCBs are the only target compounds required. PCBs are stable and not subject to breakdown in the injection port.

Inject the Endrin/DDT breakdown standard. Check the peak integrations and calculate the breakdown as follows:

$$\%\text{Breakdown Endrin} = \frac{\text{Response(Endrin Aldehyde + Endrin Ketone)}}{\text{Response(Endrin + Endrin aldehyde + Endrin Ketone)}} \otimes 100$$

$$\%\text{Breakdown DDT} = \frac{\text{Response(DDE + DDD)}}{\text{Response(DDT + DDE + DDD)}} \otimes 100$$

The response (area) must be used to evaluate the breakdown. Do not use concentrations and do not "undetect" peaks that are below the RL or MDL. All peaks detected by the data system must be included in the percent breakdown calculation.

Breakdown Criterion

EPA 8081B and EPA 608: The breakdown for each compound must be less than 15% to be acceptable.

EPA 608.3: The breakdown for each compound must be less than 20% to be acceptable.

If the breakdown exceeds criteria the instrument requires column and/or injector port maintenance. The maintenance may include, but is not limited to, replacing the septum, clipping the front of the guard column, replacing the glass injector sleeve, and scrubbing (cleaning) the injector port.

Note: If 2,4'-DDT and its degradates, 2,4'-DDD and 2,4'-DDE, are target compounds, the system must be tested for breakdown of 2,4'-DDT. The same % breakdown criterion is used to evaluate the column. Calculate the breakdown as follows:

$$\%\text{Breakdown}\,2,4'\text{-DDT} = \frac{\text{Response}(2,4'\text{-DDE} + 2,4'\text{-DDD})}{\text{Response}(2,4'\text{-DDT} + 2,4'\text{-DDE} + 2,4'\text{-DDD})} \otimes 100$$

9.2.2 Initial Calibration (ICAL)

The instrument must be calibrated in accordance with SOP SA-QA-016: *Evaluation of Calibration Curves.* This SOP provides requirements for establishing the calibration curve and gives the applicable formulas.

Instrument calibration is performed by analyzing a series of known standards. The calibration curve must consist of a minimum of 5 standards for the EPA 8000-series methods and a minimum of 3 standards for EPA 608 and EPA 608.3. The lowest level calibration standard must be at or below the reporting limit, and the remaining standards will define the working range of the analytical system.

Note: A minimum of 6 points is required for a quadratic curve. Higher order curves are not permitted. Some programs and agencies (e.g., SC DHEC) do not allow the use of quadratic curves. Refer to the Project Requirement Summary and/or Project Plan to determine if this curve type is prohibited.

The initial calibration standard concentrations currently in use in the laboratory are listed in Attachment 9. Refer to Attachment 9 for the standard preparation instructions. Other standard concentrations may be used provided they support the reporting limit and are fully documented in accordance with SOP SA-AN-041.

9.2.2.1 ICAL Linearity Criteria

The preferred method of quantitation is the average response factor. The relative standard deviation (%RSD) of the calibration standards must be <20% for the EPA 8000-series methods, <15% for EPA 608.3 and <10% for EPA 608 for the initial calibration curve to be acceptable.

Another initial calibration option is to evaluate a regression curve. If the regression curve option is chosen, the regression coefficient (r^2) must be greater than or equal to 0.990 to be acceptable.

If these criteria are not met, then re-calibration is required before sample analysis can proceed.

Note: There are several additional requirements that must be met for PCB analyses. These requirements include the following:

1. Selection of peaks of appropriate height.
   When selecting peaks for the quantitation of Aroclors all peaks selected must be 25% of the height of the largest peaks in the Aroclor pattern, except for Aroclor 1268 where the requirement is 10%. Additionally, the peaks selected should have relatively good peak shapes and have as little co-elution with other peaks in the pattern.

2. Resolution requirements.
   The mid-level AR1660 ICAL standard must meet a resolution criteria of <75% for the triplet near the end of the AR1260 pattern on at least one column. The preferred column will be the CLP-I column. The resolution is defined as:

   (Height of Valley / Average of the Two Peak Heights)*100%

   All pesticide analytes must be at least 80% baseline resolution on at least one column.

3. Non-AR1660 Standard Requirements
   A single point calibration is typically used to quantitate the non-AR1660 Aroclors. If this option is used, then the average response factor quantitation must be employed for these analytes.
   - When single point calibration is used for the non-AR1660 analytes, a quadratic curve fit may only be used for AR1660 **if** the %RSD criteria outlined above are met.
   - Additionally, if an Aroclor that is typically calibrated as a single-point is detected in a sample analyzed by EPA 608, then the sample must be reanalyzed after a multi-point curve has been established.
   - Alternatively, a multi-point calibration may be used for the non-AR1660 Aroclors. If this option is used, a quadratic curve may be employed when %RSD criteria exceeds that outlined above, provided the curve type chosen produces accurate quantitation over the entire working range of the instrument.
   - **Note:** DOD QSM requires a multi-point calibration to be used for quantitation of all detected Aroclors.

4. AR1016 Pattern Recognition Standard
   A single standard containing only AR1016 must be analyzed within the ICAL sequence, to establish pattern recognition of AR1016 versus AR1242.

Note: Refer to Attachment 11 for information on acceptance criteria for poor and/or erratic performing compounds.

9.2.2.2 ICAL Readback / % Error Criteria

Each calibration curve must be evaluated to ensure that the quantitation over the entire working range is accurate. This is accomplished via CHROM by reviewing the % Error for each analyte in each calibration level.

The re-calculated concentrations of each analyte should be within 50% for the low point and within 30% for the remaining points.

If these criteria are not met, an alternate curve type that meets criteria should be utilized to ensure a better fit, or another initial calibration curve should be performed.

9.2.3 Second Source Initial Calibration Verification (ICV)

The calibration curve must be verified after the initial calibration is established, prior to any sample analyses, in accordance with SOP SA-QA-016 with a standard obtained from a second source.

The initial calibration verification standard concentration currently in use in the laboratory is equivalent to Level 4 of the ICAL. Another standard concentration may be used provided it is mid-level and fully documented in accordance with SOP SA-AN-041.

EPA 608:
The ICV is acceptable if the %RSDs for all of the analytes in the ICV is less than 15%.

EPA 8081B and EPA 8082A and DODQSM V5:
The ICV is acceptable if the %RSDs for all of the analytes in the ICV is less than 20%.

EPA 608.3:
The ICV is acceptable if the %RSDs for all of the analytes in the ICV are within the method defined limits. These limits can be viewed in the LIMS MLG.

Note: Refer to Attachment 11 for information on acceptance criteria for poor and/or erratic performing compounds.

Note: ICVs are not required for the non-AR1660 PCBs. The AR1660 ICV is used to satisfy this evaluation.

9.2.4 Initial Calibration Blank (ICB) / Continuing Calibration Blank (CCB)

The instrument must be shown to be free from contamination by the analysis of calibration blanks. Initial calibration blanks are analyzed at the beginning of each sequence. Continuing calibration blanks are analyzed in each clock.

Initial and continuing calibration blanks must be <1/2RL to be acceptable.

9.2.5 Continuing Calibration Verification

An analytical clock is defined as 24 hours (for EPA 608 and EPA 608.3) or 12 hours (for EPA 8081B and EPA 8082A).

EPA 8081B and EPA 8082A require that no more than 20 field samples and/or batch QC items (total) be analyzed within an analytical clock. The default procedure is to include field and batch QC items (e.g., method blank, LCS, MS/MSD, and SD) in determining the maximum number of samples in an analytical clock. Instrument QC (e.g., CCVs, PEVAL, and instrument blanks) are not included in this count of 20.

For EPA 608, the initial calibration curve must be verified at the beginning of each 24-hour clock with a mid-level standard. As this is an internal standard method, a capping CCV is not required. The CCV must be within +/-15% to be acceptable.

For EPA 608.3, the initial calibration curve must be verified at the beginning and end of each 24-hour clock with a mid-level standard. The CCV must be within the method defined limits to be acceptable. An additional, reporting limit CCV (RLCCV) is incorporated into the procedure for EPA 608.3. This CCV can be analyzed at the end of each 24-hour after the mid-level standard for analytes outside method criteria. This can be used to demonstrate sensitivity at the reporting limit can be obtained. Samples requiring target analytes that are outside the acceptance criteria in the closing CCV may be reported if those analytes are qualitatively identified in the RLCCV and are non-detect in the samples. An NCM must be initiated to denote this situation. Refer to the applicable CCV section of Attachment 3 for more information on how the RLCCV is utilized.

For EPA 8081B and EPA 8082A, the initial calibration curve must be verified at the beginning of each 12-hour clock with a mid-level standard. As this is an internal standard method, a capping CCV is not required. The CCV must be within +/-20% to be acceptable.

For EPA 8082A, the opening CCV set must include the non-AR1660 single point mid-level standards for retention time updates and pattern recognition. If a sample has a positive detection for any non-AR1660 Aroclor, a CCV for that Aroclor must be analyzed and meet criteria prior to sample analysis. Additionally, the mid-level CCV standard must meet the resolution requirements outlined in Section 9.2.2.1.

For EPA 8081B and 8082A DODQSM V5: The CCV must be analyzed before sample analysis, after every 10 field samples and at the end of the sequence. Multi-component analytes (ie, Toxaphene, Chlordane and Arochlors, other than 1016/1260) are required before sample analysis. All must be within +/- 20% D.

- Corrective Action: Immediately analyze two consecutive CCVs. If both pass, samples may be reported. If either fails or two consecutive CCVS cannot be run, repeat analysis of CCV(s) and all associated samples. Recalibrate if necessary and reanalyze all associated samples since last acceptable CCV(s).

Note: Refer to Attachment 11 for information on acceptance criteria for poor and/or erratic performing compounds.

Note: If the CCV is high biased and samples are non-detect for the affected analyte, data may be reported with qualification. Additionally, if one column is marginally outside criteria for an analyte, this column may be used for qualitative identification, only, with quantitative results for that compound reported from the other, passing column. An NCM must be initiated to address this situation.

Refer to Attachment 8 for the standard preparation instructions. Another standard concentration may be used provided it is mid-level and fully documented in accordance with SOP SA-AN-041.

- Note: The routine CCV standards are the Pest single-peak mix, AR1660 standard, technical chlordane, and toxaphene.

- Note: For DOD samples which required a multi-point curve for all Aroclor detections, then an additional CCV is required for the Aroclor of concern.

- Note: For the Toxaphene TAUC analysis/projects, the initial calibration curve must be verified at the beginning of the 12-hour clock with a mid-level Toxaphene standard. The CCV must be within +/-20% to be acceptable.

9.2.6 Internal Standard (ISTD)

This procedure is an internal standard (ISTD) procedure. 1-Bromo-2-nitrobenzene (BNB) is the internal standard.

Prior to analysis, this internal standard must be added to all standards, samples, and QC items. The concentration of the internal standard must be the same in all calibration samples, field samples, and QC samples. A concentration of 0.08ug/mL is used.

The response of the ISTD in the ICV/CCV must be within 50-150% of the response of the ISTD in the CCV-level standard in the initial calibration sequence. If the response is outside of this range, the analysis of the CCV must be repeated and any samples associated with the CCV must also be re-analyzed. Repeated failure of the ISTD response will require re-calibration.

Some projects require the ISTD of the daily CCV to be within 30 seconds of the RT of the ICAL. Due to instrument maintenance, it is possible for the retention time of the daily CCV to be outside of this window even though all other criteria are met. As such, the laboratory updates the RT of the ISTD and all analytes daily with the first CCV in the sequence.

The response of the ISTD in the samples and batch QC items must be within 50-200% of the response and within 30 seconds of the previous CCV.

DODQSM V5: Retention time within 0.06RRT units from the retention time of the midpoint standard in the ICAL. Internal standard signal within -50% to + 100% of the ICAL midpoint. On days when ICAL is not performed, initial CCV is used.

If the response or retention time is outside of the ranges listed above, corrective action must be taken which can include reanalysis of the extract, re-spiking extract with ISTD and re-analysis, or re-calibration of the analytical system. Obvious matrix interferences are qualified and noted in an NCM.

9.2.7 Surrogate

This procedure uses 2 surrogates to evaluate the extraction process. Decachlorobiphenyl (DCB) and Tetrachloro-m-xylene (TCMX) are the surrogates. Prior to preparation, these surrogates are added to all samples and QC items. The concentration of the surrogate is the same in all field samples and QC samples. A concentration of 0.50ug/L for water samples, and a concentration of 16.7ug/kg is used for soil samples.

The percent recovery of the surrogate in all field samples and QC samples must be within the limits listed in the Method Limit Groups (MLGs) in LIMS. If the percent recovery is outside of this range, the analysis of the sample must be repeated. Barring obvious matrix interferences, repeated failure of the surrogate percent recovery may indicate re-extraction is necessary.

Note: If one of the surrogates is within the acceptance limits and the other surrogate recovery is >10%, a field sample is deemed acceptable and re-extraction and re-analysis is not automatically performed.

9.2.7.1 Surrogate Dilution Factor Threshold

Due to the level of dilution required for samples, surrogates may be diluted out. As such, recoveries will be reported as “0D” in dilutions greater than DF5. Control limits will not apply to samples analyzed at dilutions of greater than DF5.

An NCM must be initiated to denote this situation.

9.3 Corrective Action for Out-of-Control Data

When the quality control parameters do not meet the criteria set forth in this SOP, corrective action must be taken in accordance with SOP SA-QA-005: *Preventive and Corrective Action Procedures* the QC Summary Table in Attachment 3. SOP SA-QA-005 provides contingencies for out-of-control data and gives guidance for exceptionally permitting departures from approved policies and procedures. Nonconformance Memos must be initiated to document all instances where QC criteria are not met and all departures from approved policies and procedures.

10.0 Procedure

10.1 Sample Preparation

The sample preparation procedures are given in the following SOPs:

Matrix	SOP
Aqueous Samples	SA-EX-030
Soil Samples	SA-EX-040

10.2 Analysis

10.2.1 Instrument Operating Conditions

The instrument conditions listed in this SOP are provided for guidance purposes. The actual conditions used by the laboratory may be slightly different from those listed here and must be documented in the instrument maintenance log, data system, and/or run log.

Instrument maintenance must be performed in accordance with Attachment 4 of this SOP.

The goal is to have maximum separation between the target compounds in the shortest run time while maintaining sufficient sensitivity to detect the target compounds at the reporting limit and MDL (if required).

Two columns are connected to the injection port using a press-tight glass y-splitter and a guard column, a two-hole ferrule, or a glass tee to provide simultaneous detection and confirmation of the target analytes.

Example GC Parameters
Injector: 220-240°C
Detector: 300-320°C
Carrier Gas Flow: Helium at 2-5mL/min (per column)
Make-up Gas Flow: Nitrogen at 50-100mL/min (per detector) – see manufacturer's recommended flows

Initial pressure: 10.00psi for 2.5 minutes
Pressure program 1: 20psi/min to 35psi, hold 0 minutes
Pressure program 2: 25psi/min to 45psi, hold 0 minutes
Pressure rate: 13.00psi/minute
Final pressure: 55psi for 0 minutes

Total flow: 60.1mL/min (helium)

Temperature program
Initial Temp: 110°C
Initial Hold: 0.5 minutes
Program Rate1: 45°C per minute to 200°C, hold 0 minutes
Program Rate2: 12.5°C per minute to 230°C, hold 0 minutes
Program Rate3: 30°C per minute to 295°C, hold 0 minutes
Program Rate4: 25°C per minute to 330°C, hold 2.03 minutes
Maximum Temp: 340°C

Run Time: Approximately 9 minutes

Detector: Dual electron capture
Detector temperature: 325°C
Makeup flow: 60-100mL/min (nitrogen)

Signal data rate: 20hz

Auto-injector
Sample washes: 0
Sample pumps: 4
Injection volume: 1.0uL
Syringe size: 10uL
Post Injection Solvent A washes: 4
Post Injection Solvent B washes: 4 (use hexane as wash solvent)
Viscosity delay: 0 seconds
Plunger speed: fast
Pre-injection dwell: 0.00minutes
Post injection dwell: 0.00minutes

Note: These conditions and parameters are given for guidance. The columns/phases, GC conditions, and instrument parameters may be modified to optimize each analytical system.

10.2.1.1 Determination of Retention Time Windows

The procedure for the determination of retention time windows is given in SOP SA-QA-008: *Evaluation of Chromatographic Data*. Retention time windows (RTW), i.e., the length of time the instrument will scan for the analyte, must be established initially upon instrument set-up and verified annually.

Retention times (RT), i.e., the elution time of the analyte, are verified daily with the analysis of the ICAL or CCV. The retention time for the CCV must fall within the daily retention time window as defined in SOP SA-QA-008.

10.2.2 Initial and Continuing Calibration

Calibrate the instrument using the standards and criteria described in Section 9.2.2. Once the calibration has been established and verified with an ICV in accordance with Section 9.2.3, sample analysis may proceed.

Verify the calibration curve with a continuing calibration verification using the standards and criteria described given in Section 9.2.5.

10.2.3 Sample Analysis

The term “clock time” defines the continuing calibration verification frequency. The clock time starts at the injection of the PEVAL. The analysis of samples and batch QC items may continue until the clock time expires. A new PEVAL and CCV (i.e., a new clock) is required to proceed with the analysis of more samples and/or batch QC items. An analytical clock is defined as 24 hours (for EPA 608 and EPA 608.3) or 12 hours (for EPA 8081B and EPA 8082A).

EPA 8081B and EPA 8082A require that no more than 20 field samples and/or batch QC items (total) be analyzed within an analytical clock. The default procedure is to include field and batch QC items (e.g., method blank, LCS, MS/MSD, and SD) in determining the maximum number of samples in an analytical clock. Instrument QC (e.g., CCVs, PEVAL, and instrument blanks) are not included in this count of 20.

10.2.3.1 Remove the extracts from the refrigerator and allow them to come to room temperature.

Transfer 1.0mL of the extract to an autosampler vial and add 0.10mL of the internal standard solution to give an internal standard concentration of 0.08ug/mL. The concentration of the internal standard must be the same in all calibration samples, field samples, and QC samples.

The sample extract must be injected using the same injection volume used for the calibration standards. Samples that are known to be relatively clean should be analyzed first. Samples suspected of containing high concentrations should be analyzed last. Instrument blanks may be analyzed after suspected high concentration samples to allow the detector response to stabilize.

10.2.4 Example Analytical Sequence

Refer to Attachment 1 for an example analytical sequence.

Guidance for the evaluation of the calibration standards, field samples, and QC items are summarized in Section 9 and in Attachment 3.

11.0 Calculations / Data Reduction

11.1 Data Reduction

Data evaluation must be performed in accordance with SA-QA-008: *Evaluation of Chromatographic Data.* This SOP includes specific information regarding the evaluation of chromatographic data, including the requirements for performing manual integrations and the evaluation of retention times.

Data must be evaluated in accordance with SOP SA-QA-002: *Data Generation and Review*.

11.1.1 Target Analyte Identification

The judgment and experience of the analyst and his/her colleagues are important factors in the evaluation of chromatographic data. Inspect each chromatogram to ensure that the peaks are properly identified and that the correct areas have been associated with the corresponding standard peak RT in the data system tabulation.

The evaluation of chromatograms for target compounds must take into account the calibration of the analytical system (initial and continuing calibration response and retention times); the recovery and retention time shift of the surrogate compounds, whether the peak response falls within the working range of the calibration; and the integration of the peaks. The analyst must also take into account the results from the method blank and lab control sample before reporting quantitative data. SOP SA-QA-008: *Evaluation of Chromatographic Data* provides additional guidance for the evaluation of chromatographic data. This guidance is summarized in the following sections.

Refer to Attachment 6 and 7 for specific instructions on toxaphene identification and quantitation including the single peak option and the TAUC Method, respectively.

11.1.2 Manual Integrations

Manual integrations must be documented in accordance with SOP SA-QA-008. Data systems should be adjusted to minimize operator intervention. All chromatographic peaks must be evaluated for overall peak shape and “reasonableness” of integration. Under no circumstances should manual integrations be used to change reasonable data system integrations in order to meet calibration or QC criteria.

11.1.3 Dual Column Reporting

Refer to SOP SA-QA-008: *Evaluation of Chromatographic Data* for information on assessing and reporting data from dual columns.

11.1.4 Surrogate Evaluation

Two surrogates, TCMX and DCB, are spiked into each sample and QC item prior to preparation. Given the complicated nature of GC-ECD chromatograms, assessing surrogate recovery is frequently complicated by co-eluting positive and negative interferences. Evaluate the surrogates in the same manner as the target compounds using the guidance above.

Two surrogates are added to all samples and field samples. The laboratory's policy is:

- both surrogates must be within the recovery limits for method blanks and LCS
- one surrogate must fall within the recovery limits for all samples and the other surrogate must have a recovery of at least 10%.
- dilution cannot be used as a justification for not re-analyzing or re-extracting a field sample if the sample can be analyzed at a dilution factor of five or less.

TCX	DCB	Action
Within limits	Within limits	Evaluate and report data
Within limit	Below LCL but >10% recovery	Evaluate and report data
Below LCL but >10% recovery	Within limits	
Below LCL	Below LCL	-Reanalyze -Re-extract unacceptable samples
Above UCL	Above UCL	Evaluate and report data if not target compounds are detected and recovery is <50% above the UCL

LCL = lower control limit UCL = upper control limit

Note: For samples that contain Aroclor 1268, TCX is used to determine if the surrogate recovery is acceptable. DCB is a component of AR1268 and will bias the recovery high.

Refer to Section 11.1.5.1 for information on the surrogate dilution threshold factor.

11.1.5 Dilutions

If the response for an analyte exceeds the working range of the system, a dilution is required. Unless otherwise specified by a client QAPP, results from a single analysis are reported as long as the largest target analyte (when multiple analytes are present) is in the upper half if the calibration range. When reporting results from dilutions, appropriate data flags must be used or qualification in a case narrative provided to the client.

For clients who require the laboratory to provide lower detection limits, a general guide would be to report the dilution detailed above and one additional run at a dilution factor 1/10 of the dilution with the highest target in the upper half of the calibration curve. For example, if samples analyzed at a 1/50 dilution resulted in a target in the upper half of the calibration curve, the sample would be analyzed at a dilution factor of 5 to provide lower reporting limits.

Prepare dilutions for sample extracts where the target compounds exceed the calibration range as follows:

Dilution Factor (DF)	Volume of Sample Extract	Final Volume* (mL)
2	0.50	1.0
5	0.20	1.0
10	0.10	1.0
20	0.50	10
50	0.20	10
100	0.10	10
200	Perform DF10, then DF20	
500	Perform DF10, then DF50	
1000	Perform DF10, then DF100	
2000	Perform DF20, then DF100	
5000	Perform DF50, then DF100	
10000	Perform DF100, then DF100	
20000	Perform DF20, then DF1000	
50000	Perform DF50, then DF1000	
100000	Perform DF100, then DF1000	

* Final Solvent = Hexane

Transfer 1.0mL of the dilution to an autosampler vial and add 100uL of the internal standard.

11.1.5.1 Surrogate Dilution Threshold Factor

Surrogates may be diluted out if the concentration of target compounds is high or the presence of non-target compounds interferes with the quantification of the target compounds. Undetect surrogates in the sample when the dilution factor is greater than 5. As such, recoveries must be reported as “0D”, and control limits will not apply.

An NCM must be initiated to denote this situation.

11.1.5.2 Dilutions and MS/MSD Recoveries

Matrix spike recoveries are not reported for dilutions of greater than 5. An NCM is generated for instances where the dilution prohibits evaluation of the MS/MSD recoveries. In instances where the unspiked sample concentration is more than four times the concentration of the target compound spiked into the MS and MSD, the results are qualified with “4” or other suitable flag.

An NCM must be initiated to denote this situation.

11.1.6 Chemical Relationships

The analyst must be aware of the following chemical relationships:

Alpha-BHC, beta-BHC, delta-BHC, and gamma-BHC are isomers and will generally be present together in a sample. Gamma-BHC is usually the predominant isomer present and the only BHC isomer on the regulated drinking water list.

When 4,4'-DDT (p,p;-DDT) is present in a sample, its breakdown products, 4,4'-DDD and 4,4'-DDE will usually be present, too.

11.1.7 Historical Data

Many of the laboratory's clients submit samples for repeat monitoring purposes. Prior to analysis, verify LIMS Worksheet Notes and/or use the LIMS Historical Data Tracker feature to determine if historical data is available for review.

11.2 Calculations

11.2.1 The calculations associated with batch QC determinations are given in SOP SA-QA-017. Applicable calculations include accuracy (% recovery) and precision (%RPD).

11.2.2 The calculations associated with initial and continuing calibrations and are given in SOP SA-QA-016. Applicable calculations include determination for: calibration factor, standard deviation, relative standard deviation, relative response factor, and relative standard deviation.

11.2.3 The calculation to determine final concentration is given as follows:

$$FinalConcentration = CONC_{Sample} \otimes \frac{F}{I \times dw} \otimes D$$

Where:

$CONC_{Sample}$= Concentration of the sample
F = Final volume/weight
I = Initial volume/weight
D = Dilution factor
dw = % Solids decimal equivalent

Note: All dry weight corrections are performed automatically in LIMS.

Note: This calculation assumes all applicable unit correction factors are applied.

12.0 Method Performance

12.1 Reporting Limit Verification (RLV)

At a minimum, RLVs must be performed initially upon method set-up in accordance with SOP SA-QA-007: *Determination and Verification of Detection and Reporting Limits*.

For analytes and methods certified by DOD ELAP and TNI, RLVs must also be performed quarterly. Exceptions may be made for project-specific non-routine analytes.

12.2 Method Detection Limit (MDL) Study

The current MDLs associated with this procedure are given in the Method Limit Group (MLG) in LIMS.

At a minimum, MDL Studies must be performed initially upon method set-up in accordance with SOP SA-QA-007: *Determination and Verification of Detection and Reporting Limits*.

Replicates must be analyzed quarterly in accordance with SOP SA-QA-007.

Note: MDL Studies are not required for non-routine analytes provided results are not reported below the RL (i.e., MDL equals RL in LIMS).

12.3 Method Detection Limit Verification (MDLV)

At a minimum, MDLVs must be performed initially upon method set-up in accordance with SOP SA-QA-007: *Determination and Verification of Detection and Reporting Limits*.

For analytes and methods certified by DOD ELAP and Texas TRPP, MDLVs must also be performed quarterly.

Note: MDLVs are not required for non-routine analytes provided results are not reported below the RL (i.e., MDL equals RL in LIMS).

12.2 QC Limit Generation, Control Charting, and Trend Analysis

12.2.1 EPA 608 and EPA 608.3
The control limits for the batch QC items (LCS/LCSD and MS/MSD) for this procedure are specified in the reference method and cannot be broadened; therefore, the laboratory defaults to the method-defined limits and does not utilize in-house or laboratory-derived limits for the evaluation of batch QC items.

12.2.2 EPA 8081B and EPA 8082A
The control limits for the batch QC items (LCS/LCSD and MS/MSD) for this procedure are not specified by the reference method; therefore, the laboratory defaults to in-house and/or laboratory-derived limits for the evaluation of batch QC items.

12.2.3 Even when the laboratory must default to the method-defined QC limits, control charting is a useful tool and is performed to assess analyte recoveries over time to evaluate trends. Control charting must be performed periodically (at a minimum annually) in accordance with SOP SA-QA-017: *Evaluation of Batch QC Data.*

12.1 Demonstrations of Capability

Initial and continuing demonstration of capability must be performed in accordance with SOP SA-QA-006*: Training Procedures.*

Prior to performing this procedure unsupervised, each new analyst who performs this analysis must demonstrate proficiency per method/analyte combination by successful completion of an initial demonstration of capability. The IDOC is performed by the analysis of 4 consecutive LCSs that meet the method criteria for accuracy and precision. The IDOC must be documented and routed to the QA Department for filing.

Annual continuing demonstrations of capability (CDOCs) are also required per analyst per method/analyte combination. The CDOC requirement may be met by the consecutive analysis of four LCS all in the same batch, by the analysis of four LCS analyzed in four consecutive batches (in different batches on different days), via acceptable results on a PT study, or analysis of client samples with statistically indistinguishable results when compared to another certified analyst. The CDOC must be documented and routed to the QA Department for filing.

12.2 Training Requirements

All training must be performed and documented in accordance with SOP SA-QA-006: *Training Procedures*.

Note: The SOPs listed in the Reference/Cross-Reference Section are applicable to this procedure. All employees performing this procedure must also be trained on these SOPs, and/or have a general understanding of these procedures, as applicable.

13.0 Pollution Control

It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (e.g., examining recycling options, ordering chemicals based on quantity needed, preparing reagents based on anticipated usage and reagent stability, etc.). Employees must abide by the policies in Section 13 of the Environmental Health and Safety Manual and the Savannah Addendum to the EHSM.

This procedure has been evaluated for opportunities to minimize the waste generated. Where reasonably feasible, pollution control procedures have been incorporated.

14.0 Waste Management

Waste management practices must be conducted consistent with all applicable federal, state, and local rules and regulations. All waste (i.e., excess reagents, samples, and method process wastes) must be disposed of in accordance with Section 9 of the TestAmerica Savannah Addendum to the EHSM. Waste description rules and land disposal restrictions must be followed.

14.1 Waste Streams Produced by the Method

The following waste streams are produced when this method is carried out:

- Excess aqueous samples – Dispose according to characterization on the sample disposal sheets. Neutralize non-hazardous samples before disposal into drain/sewer. Transfer hazardous samples (identified on disposal sheets) to the waste department for disposal.

- Excess soil and solid samples –All soil samples require incineration for disposal. Transfer RCRA hazardous and non-hazardous samples to the yard box for disposal. Samples classified as PCB hazardous are transferred to the waste coordinator for drumming. All samples classified as foreign soils are transferred to the waste coordinator for disposal. .

- Flammable waste (hexane and methanol from extracts, rinsings, and standards) - Transfer to a satellite container designated for flammable waste and transfer to waste disposal department when the container is full. Leave 10% of headspace in the satellite container.

- Methylene chloride extracts - Dispose according to characterization on sample disposal sheets. Transfer extract to chlorinated waste container.

- Samples treated with sulfuric acid- Transfer vials to Hexane Vial Waste container.

15.0 References / Cross-References

- SOP SA-AN-100: *Laboratory Support Equipment (Verification and Use)*
- SOP SA-AN-041: *Reagent and Standard Materials Procedures*
- SOP SA-EX-015: *Toxicity Characteristic Leaching Procedure (TCLP) and Synthetic Precipitation Leaching Procedure (SPLP)*
- SOP SA-EX-030: *Liquid Extraction Procedures: Continuous Liquid-Liquid and Separatory Funnel*
- SOP SA-EX-040: *Sonication Procedures*
- SOP SA-EX-042: *Waste Dilution Extraction*
- SOP SA-QA-002: *Data Generation and Review*
- SOP SA-QA-005: *Preventive and Corrective Action Procedures*
- SOP SA-QA-006*: Training Procedures*
- SOP SA-QA-007: *Determination and Verification of Detection and Reporting Limits*
- SOP SA-QA-008: *Evaluation of Chromatographic Data*
- SOP SA-QA-015: *Homogenization, Compositing, and Segregation of Samples*
- SOP SA-QA-016: *Evaluation of Calibration Curves*
- SOP SA-QA-017: *Evaluation of Batch QC Data*
- TestAmerica Savannah Quality Assurance Manual
- TestAmerica Environmental Health and Safety Manual (CW-E-M-001)
- TestAmerica Savannah Addendum to the Environmental Health and Safety Manual
- Test Methods for Evaluating Solid Waste, Third Edition with Revisions and Updates, SW-846; U.S. EPA Office of Solid Waste and Emergency Response: Washington, DC, December 1996 and February 2007.
  - Method 8000B: Determinative Chromatographic Separations, Revision 2; December 1996.
  - Method 8000C: Determinative Chromatographic Separations, Revision 3; March 2003.
  - Method 8081B: Organochlorine Pesticides by Gas Chromatography, Revision 2; February 2007
  - Method 8082A: Polychlorinated Biphenyls (PCBs) by Gas Chromatography, Revision 1; February 2007
- *Code of Federal Regulations, Title 40, Part 136*; U.S. Government Printing Office: Washington, DC, July 1, 1988.
  - Method 608: Organochlorine Pesticides and PCBs
  - Method 608.3: Organochlorine Pesticides and PCBs by GC/HSD
- Project-specific Toxaphene TAUC Method (Task Force 1)
  - Application of Standard Methods for the Determination of Toxaphene in Environmental Media" (Carlin, Revells, and Reed; Chemosphere 41 (2000), 481-486)
  - Procedure for the Determination of Toxaphene; US EPA Region IV; August 14, 1997.

16.0 Method Modifications

16.1 Incorporation of Other Matrices

This procedure may be modified to analyze other matrices (e.g., wipe, waste, tissue, and TCLP/SPLP leachate samples) based on the needs of the client. This will need to be arranged by the Project Manager at the initiation of the project.

Wipe, waste, and tissue matrices are non-routine, and the laboratory is not currently NELAC certified for these matrices. The laboratory uses its routine soil RLs (converted for initial and final volumes, etc.) and default QC limits to evaluate wipe, waste, filter, and tissue samples. Soil DOCs can be used to satisfy analyst demonstrations of capability for these types of non-routine matrices. The laboratory uses its routine aqueous RLs (converted for initial and final volumes, etc.) and default QC limits to evaluate TCLP/SPLP leachate samples. Water DOCs can be used to satisfy analyst demonstrations of capability for TCLP/SPLP matrices. Teflon chips, Ottawa sand, or equivalent is used as the blank matrix for wipes, wastes, and tissues unless specifically requested otherwise by the project.

16.1.1 Collection and Handling Procedures

Waste (Oil) Samples:
Waste (oil) samples are routinely collected in 8oz soil containers with PTFE-lined lids. Waste (oil) samples must be iced at the time of collection and maintained at 4°C (less than 6°C but not frozen) until the time of preparation. Samples must be prepared within 14 days of collection. Extracts must be stored at 0-6°C (less than 6°C but not frozen) until the time of analysis and analyzed within 40 days of extraction.

Wipe Samples:
Wipe samples are routinely collected in 40mL VOA vials. Wipe samples must be iced at the time of collection and maintained at 4°C (less than 6°C but not frozen) until time of preparation. Samples must be prepared within 14 days of collection. Extracts must be stored at 0-6°C (less than 6°C but not frozen) until the time of analysis and analyzed within 40 days of extraction.

Tissue Samples:
Tissue samples are routinely collected in plastic containers with the size dependent upon the type of tissue being collected. Plastic jars or plastic baggies can be used. Upon receipt, samples must be placed in the freezer at -10° to -20°C if extraction/digestion cannot be completed that day and must be kept frozen until the time of preparation. A holding time of six months from the date of collection for frozen fish fillets is recommended by the Alabama Department of Environmental Management (ADEM) and will be used for all biological tissues. Once the tissue has been thawed it must be stored at 0-6°C (less than 6°C but not frozen) and preparation must take place within 14 days. Extracts must be stored at 0-6°C (less than 6°C but not frozen) until the time of analysis and analyzed within 40 days of extraction.

TCLP/SPLP Leachate Samples

Once the TCLP/SPLP extraction procedure has been performed, the leachate is transferred to a 1L glass container. TCLP/SPLP leachates must be stored at 0-6°C (less than 6°C with no frozen samples) until the time of preparation. The leachate sample must be prepared within 7 days of completion of the TCLP/SPLP extraction. Extracts must be stored at 0-6°C (less than 6°C but not frozen) until the time of analysis and analyzed within 40 days of extraction.

16.1.2 Preparation and Analytical Procedures

Wipe, waste, and tissue samples are prepared in the same manner as routine soil samples as outlined in SOP SA-EX-040 and SA-EX-042. TCLP/SPLP matrices are prepared in the same manner as routine water samples as outlined in SOP SA-EX-030. Refer to the applicable preparation SOPs for more information.

Waste oils are prepared by using 0.5g of sample and brought up to a final volume of 10mL.

Wipe, waste, filter, tissue, and TCLP/SPLP matrices are analyzed in the same manner as routine samples as outlined in this SOP.

16.2 Other Considerations

16.2.1 EPA Methods 608 and 608.3 were written specifically for industrial and municipal wastewater samples; however, the laboratory may perform other types of water samples using this procedure.

16.2.2 The procedures for chlorinated pesticides (EPA 8081B) and PCBs (EPA 8082A) are given as separate methods in SW-846. The extraction and the analysis are combined in this SOP to reduce the time of extraction and analysis, and to reduce the amount of solvent used in the procedures (one extraction instead of two). If interferences or high levels of non-PCB compounds are present, a portion of the extract may be subjected to the acid cleanup and reanalyzed.

16.2.3 The reference methods do not require the analysis of an ICV. NELAC requires an ICV; however, it does not list specific criteria. The laboratory has adopted the default criteria listed in Section 9 for ICVs for these methods.

16.2.4 The laboratory allows one surrogate compound to be outside acceptance limits, in field samples and MS/MSD, provided their recovery is greater than 10%.

16.2.5 The reference methods do not require the analysis of an instrument blank; however, the laboratory routinely analyzes instrument blanks items and has adopted in-house criteria as outlined in this SOP.

16.2.6 The laboratory has incorporated the minimum batch QC items as outlined in Section 9.1. Some additional QC items are routinely performed to satisfy common regulatory and/or client requests for precision data and/or to facilitate scheduling and data evaluation.

16.2.7 The laboratory has incorporated the evaluation of the breakdown check standard (PEVAL) for EPA 608, EPA 608.3 and the EPA 8000-series methods. This standard is required by EPA 608.3 and the EPA 8000-series methods, but is only recommended for EPA 608.

16.2.8 EPA SW-846 Update IV Chapter 4 re-defined the holding times for PCBs as "NONE". The EPA Methods Update Rule re-defined holding times for PCBs as one year from collection to extraction and one year from extraction to analysis. The laboratory has incorporated the extended preparation holding times for PCBs on a project-specific basis, as allowed by that particular state agency and client.

16.2.9 The laboratory's procedure has been modified from the project-specific Toxaphene TAUC reference documents as follows:

- Quantification of Toxaphene TAUC does not use "valley-to-valley" integration; instead it instructs the analyst to assign the baseline based on the instrument blank. "Valley-to-valley" integration excludes the unresolved components of toxaphene, commonly referred to as the "humpogram". The procedure in this SOP includes the humpogram as an integral part of the total area under the curve, since these unresolved peaks are part of the response for toxaphene.

- Due to advances in technology since the time the Toxaphene TAUC reference documents were written, the laboratory has incorporated the use of alternate GC instrument configuration and columns, and the microwave extraction technique. Additionally, the toxaphene reference standard is procured from a commercial vendor which may not be an exact match to the Hercules X16189-49 standard. For this reason any vendor standard purchased must be compared to the Hercules standard prior to use for the analysis of TAUC.

- Further procedure-specific information on integration, identification, and quantitation procedures for Toxaphene TAUC is given in Attachment 7.

16.2.10 The laboratory has defined the analytes listed in Attachment 11 as poor or erratic performers and allows for exceptions to the ICAL, ICV, CCV, LCS, MS/MSD, and Sporadic Marginal Exceedance criteria for this analyte as outlined in this SOP. An NCM must be initiated to denote this situation.

Note: The use of Sporadic Marginal Exceedances, as defined in SOP SA-QA-017, is not permitted for samples originating from South Carolina.

16.2.11 The laboratory allows for the following exceptions to the continuing calibration verification criteria. If the CCV is high biased and samples are non-detect for the affected analyte, data may be reported with qualification. Additionally, if one column is marginally outside criteria for an analyte, this column may be used for qualitative identification, only, with quantitative results for that compound reported from the other, passing column. An NCM must be initiated to address this situation.

16.2.12 Due to the complex nature of PCB analyses, the laboratory has incorporated some additional QC requirements for this procedure. These requirements include resolution checks, AR1016 pattern recognition standard, peak height criteria for peak assignment, etc., and will allow for better evaluation of these compounds.

16.2.13 EPA 608.3 requires a mid level standard to be analyzed at the end of each analytical clock. Due to the nature of the samples analyzed by this method it is highly likely that one or more analytes will not meet method criteria in the closing standard. Therefore, the laboratory allows for an additional reporting limit standard (RLCCV) to be analyzed after the mid level standard at the end of an analytical clock. This standard can be used to demonstrate sensitivity at the reporting limit can be obtained. Samples requiring target analytes that are outside the acceptance criteria in the closing CCV may be reported if those analytes are qualitatively identified in the RLCCV and are non-detect in the samples. An NCM must be initiated to denote this situation.

17.0 Attachments

The following Tables, Diagrams, and/or Validation Data are included as Attachments:

Attachment 1: SOP Summary
Attachment 2: Sample Collection, Preservation, and Holding Time Table
Attachment 3: QC Summary
Attachment 4: Instrument Maintenance and Troubleshooting
Attachment 5: Analysis of Multiple Peak Compounds - PCB as Aroclors
Attachment 6: Analysis of Multiple Peak Compounds - Toxaphene (Single Peak Method)
Attachment 7: Analysis of Multiple Peak Compounds - Toxaphene (TAUC Method)
Attachment 8: Analysis of Multiple Peak Compounds - Technical Chlordane
Attachment 9: Standard Preparation Recipes
Attachment 10: Example Retention Times and Relative Retention Times
Attachment 11: Poor and Erratic Performer Information
Attachment 12: Glassware Cleaning Procedures

Attachment 1: SOP Summary

Sample Preparation Summary
Liquid samples are extracted using continuous liquid-liquid extraction with methylene chloride. The extract is concentrated, the solvent is exchanged to hexane, and the final volume of the extract is adjusted.

5. If the High Volume preparation procedure is requested, 1000mL of sample is extracted and concentrated to a final volume of 5mL.
6. If the Low Volume Initiative (LVI) preparation procedure is requested, 250mL of sample is extracted and concentrated to a final volume of 1.0mL.

Soil samples are extracted using the sonication or microwave extraction procedure. Approximately 15g of the homogenized sample is dried with anhydrous sodium sulfate and extracted with 1:1 acetone/hexane. The extract is concentrated to a final volume of 5mL.

The preparation procedures may also incorporate copper (sulfur) clean-up and acid clean-up (used for PCBs and TAUC only).

Samples should be prepared according to the appropriate matrix-specific SOP.

Matrix	SOP
Aqueous Samples	SA-EX-030
Soil Samples	SA-EX-040

Sample Analysis Summary
The extract is analyzed by gas chromatography using dual capillary columns (different phases) and dual electron capture (EC) detectors. This configuration allows for simultaneous detection and confirmation of the target compounds. Identification of the target compounds in samples is performed by comparing the retention times of the peaks with standards analyzed under the same GC conditions. Quantitation is performed using the internal standards calibration technique.

Example Analytical Sequence

Analysis Item	Comments
Initial Calibration Sequence	PEVAL - Endrin/DDT Breakdown Check AR1016 AR1660 ICAL (6-pt) AR1660 ICV Remaining Aroclors (1-pt) Pesticide ICAL (6-pt) Pesticide ICV Toxaphene ICAL (6-pt) Toxaphene ICV

	Technical Chlordane ICAL (6-pt) Technical Chlordane ICV AP9 ICAL (6-pt) AP9 ICV
Instrument Blank	
Field Samples and Batch QC Items	Analyze until clock time expires. EPA 608 and EPA 608.3 = 24 hours EPA 8000-series = 12 hours (or 20 samples/batch QC, whichever comes first)
PEVAL	Endrin/DDT Breakdown Check
CCV	Pesticide CCV AR1660 CCV Toxaphene CCV Technical Chlordane CCV AP9 CCV (if requested) AR1221/1254 CCV (if needed) AR1232/1262 CCV (if needed) AR1242/1268 CCV (if needed) AR1248 CCV (if needed)
Instrument Blank	
Field Samples and Batch QC Items	Analyze until clock time expires. EPA 608 and EPA 608.3 = 24 hours EPA 8000-series = 12 hours (or 20 samples/batch QC, whichever comes first)
CCV	Pesticide CCV AR1660 CCV Toxaphene CCV Technical Chlordane CCV AP9 CCV (if requested) AR1221/1254 CCV (if needed) AR1232/1262 CCV (if needed) AR1242/1268 CCV (if needed) AR1248 CCV (if needed)

*Note 1 – PEVAL Breakdown Check is not required for PCB-only analyses
*Note 2 – For the TAUC Method, a Toxaphene CCV must be analyzed at the beginning every 12-hour clock. The Parlar 11/Parlar 69 RTW Standard must be analyzed at the start of every day for retention time determination
*Note 3 – For DOD, only 10 samples are allowed per clock.

Attachment 2:
Sample Collection, Preservation, and Holding Time Table

Matrix	Routine Sample Container	Routine Sample Size	Minimum Sample Size	Chemical Preservation	Thermal Preservation	Dechlorination Agent	Holding Time
Water (High Volume Prep)	1L amber glass	1L	500mL	None	0-6°C[1]	None	7 days to extract[2] 40 days to analyze[3] PCBs Only[4]: 1 year to extract[2] 40 days to analyze[3]
Water (Low Volume Prep)	250mL amber glass	250mL	250mL	None	0-6°C[1]	None	7 days to extract[2] 40 days to analyze[3] PCBs Only[4]: 1 year to extract[2] 40 days to analyze[3]
Soil	8 oz glass soil jar	15g	15g*	None	0-6°C[1]	None	14 days to extract[2] 40 days to analyze[3] PCBs Only[4]: 1 year to extract[2] 40 days to analyze[3]

[1]Samples must be maintained at 0-6°C, with no frozen samples.
[2]From sample collection.
[3]From sample preparation.
[4]Project-specific criteria, as allowed by the client and/or agency
***Reduced sample prep amounts may be used per specific project requirements or with approval from client or project manager.

Attachment 3:
QC Summary

QC Item	Frequency	Criteria	Corrective Action
Clock Time	EPA 608 and EPA 608.3: 24 hours EPA 8000-series: 12 hours or 20 field samples/batch QC, whichever comes first	Clock time starts with the injection of the PEVAL. Analysis of samples and QC items must conclude within expiration of clock time. Subsequent analysis requires new opening PEVAL and CCV.	Not Applicable
Breakdown Check (PEVAL)	At beginning of each clock	Breakdown <15% for both DDT and Endrin Breakdown <20% for 608.3	-Re-analyze check solution -Perform injector port and/or column maintenance and re-analyze
Initial Calibration (ICAL)	Upon instrument set-up, and after unsuccessful CCV	EPA 608: 3-point minimum; RSD <10%; r^2>0.990 %RSE<50% for low point; <30% for other points EPA 608.3: 3-point minimum; RSD <15%; r^2>0.990 %RSE<50% for low point; <30% for other points EPA 8000-series: 5-point point minimum; RSD <20%; r^2>0.990 Readback: $\leq$50% for low point; $\leq$30% for other points	Refer to SOP SA-QA-016

QC Item	Frequency	Criteria	Corrective Action
		Note: When using single point ICAL for non-AR1660 analytes, then AR1660 must meet %RSD criteria if using quadratic curve. *Refer to Attachment 11 for Poor Performer Information	
Initial Calibration Verification (ICV) - Second Source	After each ICAL	EPA 608: %D<15% EPA 608.3: %D within LIMS MLG limits EPA 8081B & EPA 8082A: %D<20% *Refer to Attachment 11 for Poor Performer Information	Refer to SOP SA-QA-016
Continuing Calibration Verification (CCV)	EPA 608: Initially, and every 24 hours thereafter EPA 608.3: Initially and at the end of every analytical clock 8000-series: Initially, after every 12 hours (or 20 field samples/batch QC), whichever comes first	EPA 608: %D<15% EPA 608.3: %D within LIMS MLG limits EPA 8081B & EPA 8082A: %D<20% *Refer to Attachment 11 for Poor Performer Information	Refer to SOP SA-QA-016

QC Item	Frequency	Criteria	Corrective Action
	Toxaphene TAUC: Initially, after every 12 hours		
Reporting Limit Continuing Calibration Verification (RLCCV)	EPA 608.3 Only: At the end of an analytical clock when the closing CCV has analytes outside criteria.	Affected analytes must be qualitatively identified.	- Perform instrument maintenance - Reanalyze affected samples. - Recalibrate
Calibration Blank (CCB/ICB)	After ICV and every CCV	<1/2 RL	- Terminate the analysis; correct problem; reanalyze affected samples.
Internal Standard (ISTD)	All field, batch QC, & instrument QC samples	CCV: - Response of the internal standard must be within a range of 50-150% of the ICAL mid-level std Samples: - Response within 50-200% of previous CCV - RT within window defined by	-Evaluate chromatogram and integrations. -Reanalyze or dilute and reanalyze -Flag data

QC Item	Frequency	Criteria	Corrective Action
		previous CCV	
Extraction Batch Definition	Extracted together w/in 24-hr period; Not to exceed 20 field samples	Not Applicable	Not Applicable
Method Blank (MB)	One per batch	<½ RL	Refer to SOP SA-QA-017
Laboratory Control Sample (LCS)	One per batch	Within LIMS MLG limits	Refer to SOP SA-QA-017
Laboratory Control Sample Duplicate (LCSD)	One per batch, if insufficient sample for MS/MSD	Within LIMS MLG limits	Refer to SOP SA-QA-017
Matrix Spike (MS)	EPA 608: 10% of samples 8000-series and EPA 608.3: Per batch	Within LIMS MLG limits	Refer to SOP SA-QA-017
Matrix Spike Duplicate (MSD)	Per batch	Within LIMS MLG limits	Refer to SOP SA-QA-017
Surrogates	All field, batch QC, & instrument QC samples	Within LIMS MLG limits Surrogate DTF = 5	Refer to SOP SA-QA-017
Retention Time Window (RTW) Determination	Annually, after major instrument maintenance, and with each new column	Refer to SOP SA-QA-008	Refer to SOP SA-QA-008
Initial Demonstration of Capability	Initially, per analyst, per analyte/method/matrix combination	Refer to SOP SA-QA-006	Refer to SOP SA-QA-006

QC Item	Frequency	Criteria	Corrective Action
(IDOC)			Note: Unsupervised work must not begin until acceptable IDOC is obtained.
Continuing Demonstration of Capability (CDOC)	Annually, per analyst, per analyte/method/matrix combination	Refer to SOP SA-QA-006	Refer to SOP SA-QA-006
Reporting Limit Verification (RLV)	Upon method/instrument set-up, per analyte/method/matrix combination. Then quarterly thereafter (for DOD ELAP) or annually thereafter (for non-DOD ELAP)	Refer to SOP SA-QA-007	Refer to SOP SA-QA-007
Method Detection Limit Study (MDL)	Upon method/instrument set-up, per analyte/method/matrix combination	Refer to SOP SA-QA-007	Refer to SOP SA-QA-007
MDL Verification (MDLV)	Upon method/instrument set-up, per analyte/method/matrix combination. Then quarterly thereafter (for DOD ELAP) or annually thereafter (for non-DOD ELAP)	Refer to SOP SA-QA-007	Refer to SOP SA-QA-007

Attachment 4:
Instrument Maintenance and Troubleshooting

LABORATORY EQUIPMENT PREVENTIVE MAINTENANCE SCHEDULE								
	Service Interval							
EQUIPMENT ITEM	D	W	M	Q	SA	A	AN	SERVICE LEVEL
Guard Column/Injector							X	Change sleeve and cut front of guard column, recommended daily
Septum							X	Replace , recommended daily
Splitless Disc							X	Replace , recommended daily
Autosampler							X	Syringe cleaned or replaced as needed
Column							X	Change column

D = daily; W = Weekly; M = monthly; Q = Quarterly; SA = semi-annually; A = annually; AN = as needed

Troubleshooting

Troubleshooting should be documented as outlined above. If possible, troubleshooting is best performed in a step-wise manner to systematically isolate instrument components. Refer to the instrument manufacturer's guides for specific information and strategies. Enlist assistance from technical and/or department management as needed.

Contingency Plan

Maintenance contracts are carried for most instrumentation and close contact is maintained with service personnel to ensure optimal instrument functioning. An extensive spare parts inventory is maintained for routine repairs. Since instrumentation is standardized throughout the laboratory network, spare parts and components can be readily exchanged among the network.

In general, the laboratory has at least one backup unit for each critical unit. In the event of instrument failure, portions of the sample load may be diverted to duplicate instrumentation, the analytical technique switched to an alternate approved technique (such as manual colorimetric determination as opposed to automated colorimetric determination), or samples shipped to another properly certified or approved TestAmerica location.

Maintenance Log

A maintenance log must be established for each piece of equipment used in the laboratory. All maintenance that is performed on the instrument must be recorded in the log including:

- Type of maintenance performed (Note: This includes preventative/routine maintenance; non-routine maintenance; maintenance performed by an external vendor; updates to software versions; etc.)
- analyst or technician performing the maintenance
- date the maintenance was performed
- detailed explanation of the reason for the maintenance
- resolution of the problem and return to control
- all service calls from instrument representatives
- Resolution of the problem (i.e., the Return to Control)

Instrument Labeling

Each instrument must be labeled with its name or ID (e.g., MSA, ICP-D, etc.). Additionally, non-operational instruments must be isolated from service or marked as being out of service. Each piece of equipment has an “Operational / Not Operational” sticker that is used for this purpose.

Attachment 5:
Analysis of Multiple Peak Compounds

Evaluation of PCBs as Aroclors

PCBs are reported as Aroclors, which are mixtures of PCB congeners. The Aroclors are named by percentage of chlorine in each mixture with the prefix 12- to represent the biphenyl molecule.

Aroclor	% Chlorine by Weight
1221	21
1232	32
1016	41.5
1242	42
1248	48
1254	54
1260	60
1262	62
1268	68

The exception to this naming convention is AR1016, which is about 42% chlorine by weight. AR1016 and AR1242 have very similar chromatograms. Both Aroclors have almost the same weight of chlorine by weight and nearly the same PCB congeners.

Aroclors are identified by matching the pattern of the sample with standards analyzed under the same analytical conditions. Interference may occur due to the presence of non-target analytes or due to "weathering" or degradation of the Aroclor in the environment. The presence of multiple Aroclors will also complicate identification and quantitation. Many matrix interferences may be reduced or eliminated by treating the sample extract with copper and sulfuric acid prior to analysis. The applicable preparation SOPs detail this procedure.

Note: Do not use the acid cleanup **on the entire extract** if pesticides are also to be reported as many of the pesticides are not stable in acid or strong oxidizer. Use a separate aliquot of sample to subject to acid clean-up if pesticides and PCBs are required.

When a pattern matching an Aroclor is encountered, it is quantitated using 3-5 characteristic peaks. AR1016 and AR1260 are quantitated using the average RF/CF determined during initial calibration. The other Aroclors are quantitated against the RF/CF determined from their single-point analysis during initial calibration. Samples should be diluted when the amount of PCB in a sample extract exceeds the calibration range defined in initial calibration. Note that the AR1660 standard defines the working range for all the Aroclors (i.e., if AR1660 was calibrated from 0.05ug/mL to 1.5ug/mL, and a sample extract was analyzed containing 10ug/mL of AR1232, that extract would require dilution to get the amount of AR1232 to be less than 1.5ug/mL). If a sample contains any of the single point Aroclors (that is, Aroclors other than AR1016 and AR1260), then that associated standard must be run within 72 hours of the sample to determine retention time shifts and pattern recognition.

In the 3-5 peak approach, use each peak in the standard to calculate a calibration factor for that peak, using the total mass of PCB in the standard. These calibration factors are then used to calculate the concentration of each corresponding peak in the sample chromatogram and the 3-5 resulting concentrations are averaged to provide the final result for the sample.

"Weathering" is the loss of part of the Aroclor pattern due to biological or chemical degradation of individual PCBs. When weathering is suspected, try to match the later eluting peaks first. Flag the results for a weathered Aroclor pattern as tentatively identified or make a note in the case narrative.

The presence of multiple Aroclors can be a problem to identify since most Aroclors have at least a few peaks in common. The easiest case would be to have early and late eluting Aroclors present. The most difficult cases will involve the presence of Aroclors with the same relative chlorine level.

Note: When choosing individual peaks for quantitation, compare their responses in the sample and standard. If the peaks chosen do not correlate well (i.e. ratios to other peaks are close) between the sample and standard, review the chromatograms for other possible peaks for quantitation.

Calibration/Quantitation Peaks

The Aroclors are routinely calibrated and quantified in samples using five peaks on each column over the retention time range. The default peaks are listed the tables below. Note: The peaks used for quantitation are subject to change due to age of column, resolution with pesticide compounds, etc. The peaks selected should have a peak height at least 25% of the largest peak within the Aroclor.

AR1221

Peak	Column 1 Restek CLPesticides			Column 2 Restek CLPesticides II		
	RT	RRT	Response Ratio	RT	RRT	Response Ratio
ISTD	2.840	1.000		2.603	1.000	
1	4.453	1.568	1.0	4.633	1.780	1.0
2	4.713	1.660	0.46	4.877	1.873	0.70
3	4.787	1.685	2.4	4.980	1.913	2.6

AR1232

Peak	Column 1 Restek CLPesticides			Column 2 Restek CLPesticides II		
	RT	RRT	Response Ratio	RT	RRT	Response Ratio
ISTD	2.840	1.000		2.607	1.000	
1	5.400	1.901	1.0	5.680	2.182	1.0
2	6.187	2.178	2.0	6.017	2.311	0.19
3	6.417	2.259	0.77	6.073	2.333	0.42
4	6.527	2.298	0.54	6.440	2.474	1.7
5	7.820	2.754	0.38	6.673	2.563	0.76

AR1016

Peak	Column 1 Restek CLPesticides			Column 2 Restek CLPesticides II		
	RT	RRT	Response Ratio	RT	RRT	Response Ratio
ISTD	2.842	1.000		2.607	1.000	
1	5.400	1.899	1.0	5.667	2.174	1.0
2	6.187	2.176	2.2	6.017	2.308	0.25
3	6.417	2.257	0.85	6.073	2.330	0.50
4	6.527	2.295	0.61	6.437	2.469	1.9
5	7.820	2.750	0.47	6.673	2.560	0.89

AR1242

Peak	Column 1 Restek CLPesticides			Column 2 Restek CLPesticides II		
	RT	RRT	Response Ratio	RT	RRT	Response Ratio
ISTD	2.843	1.000		2.607	1.000	
1	5.400	1.899	1.0	5.680	2.179	1.0
2	6.187	2.176	2.1	6.020	2.309	0.20
3	6.417	2.257	0.83	6.073	2.330	0.46
4	5.527	2.295	0.58	6.440	2.471	1.9
5	7.820	2.750	0.15	6.673	2.560	0.84

AR1248

Peak	Column 1 Restek CLPesticides			Column 2 Restek CLPesticides II		
	RT	RRT	Response Ratio	RT	RRT	Response Ratio
ISTD	2.843	1.000		2.607	1.000	
1	5.397	1.898	1.0	5.667	2.174	1.0
2	5.743	2.020	0.39	6.437	2.469	2.6
3	6.187	2.176	2.4	7.727	2.964	2.1
4	6.420	2.258	1.0	8.007	3.072	0.51
5	6.527	2.295	0.67	8.083	3.101	1.2

AR1254

Peak	Column 1 Restek CLPesticides			Column 2 Restek CLPesticides II		
	RT	RRT	Response Ratio	RT	RRT	Response Ratio
ISTD	2.840	1.000		2.603	1.000	
1	7.820	2.754	1.0	8.280	3.181	1.0
2	8.300	2.923	1.7	8.637	3.318	0.96
3	9.073	3.195	1.9	9.523	3.658	1.5
4	9.593	3.378	1.7	9.937	3.817	1.2
5	9.923	3.494	1.5	10.560	4.056	1.2

AR1260

Peak	Column 1 Restek CLPesticides			Column 2 Restek CLPesticides II		
	RT	RRT	Response Ratio	RT	RRT	Response Ratio
ISTD	2.842	1.000	NA	2.607	1.000	NA
1	10.610	3.732	1.0	11.040	4.235	1.0
2	10.693	3.761	0.54	11.133	4.271	0.60
3	11.113	3.909	0.50	11.553	4.432	1.0
4	11.573	4.070	2.1	11.677	4.480	0.55
5	12.063	4.243	0.80	11.983	4.597	2.3

AR1262

Peak	Column 1 Restek CLPesticides			Column 2 Restek CLPesticides II		
	RT	RRT	Response Ratio	RT	RRT	Response Ratio
ISTD	2.840	1.000		2.607	1.000	
1	10.610	3.736	1.0	11.040	4.241	1.0
2	11.040	3.887	0.76	11.553	4.438	0.87
3	11.573	4.075	1.8	11.983	4.603	1.8
4	13.127	4.622	0.60	13.623	5.233	0.71

AR1268

Peak	Column 1 Restek CLPesticides			Column 2 Restek CLPesticides II		
	RT	RRT	Response Ratio	RT	RRT	Response Ratio
ISTD	2.843	1.000		2.607	1.000	
1	12.117	4.261	1.0	12.603	4.835	1.0
2	12.183	4.285	1.1	12.687	4.867	1.0
3	12.513	4.401	0.79	13.127	5.036	0.85
4	13.127	4.617	0.34	13.623	5.226	0.36
5	13.617	4.789	1.9	14.197	5.446	2.1

Calibration Standards
Apply this guidance to both columns/detectors.

Adjust the baseline, if needed, to ensure a consistent integration between samples, standards, and QC items.

Inspect the peaks used for quantitation to make sure that the peaks are properly distinguishable from closely eluting peaks. Note: This step is important because as the column degrades resolution between the peaks may decrease and the integration parameters adjusted. Be sure to apply the same integration parameters to the calibration standards and samples.

ICAL: Update the method with the retention times and responses of the standards.

CCV: Update the method with the retention times of the standards.

Qualitative Identification
The Aroclors are subject to degradation in the environment; that is, exposure to the environment will cause loss of some of the peaks in the sample pattern that are present in the standard. In general, the peaks at the higher retention times are degraded by loss of chlorine from the PCB congeners, resulting in the loss of the peak and/or the formation of compounds with lower levels of chlorination, which elute at earlier retention times. Degradation may also occur as the lighter PCB congeners volatilize. Degradation of the Aroclor patterns makes qualitative identification difficult and biases the quantitation significantly if the peaks used for quantitation are if not present in the pattern.

Apply this guidance to both columns/detectors.

Adjust the baseline, if needed, to ensure a consistent integration between samples, standards, and QC items.

Compare the overall pattern of the sample to the most recently analyzed Aroclor standard. Since the AR1016/AR1260 mixture contains all of the PCB congeners in all of the other Aroclors and because it is analyzed in every clock, overlaying the sample with the AR1660 standard can help in the evaluation for the presence of PCB in the sample. If a substantial number of peaks line up with the AR1660 standard, locate the clock where the most recent Aroclor single points were analyzed to compare the sample with the patterns in the other Aroclors.

Evaluate the ratios of the 3-5 quantitation peaks identified by the data system and compare to the standard(s).

Inspect the peaks used for quantitation to make sure that the peaks are properly distinguishable from closely eluting peaks.

Manually integrate peaks where the data system did not integrate peaks in the same manner as the calibration standards. The most common types of manual integration involve “splitting

peaks" that are not resolved by the current integration parameters or identifying peaks as targets because of loss of resolution of peak symmetry.
Identify the Aroclor that most resembles the pattern of the sample and un-detect (mark as "not detected") in the data system quant file.

Inspect the chromatogram for the presence of interfering peaks from other target compounds or from non-target matrix interferences. The most common interference peaks will be from other Aroclors present in the sample matrix. Refer to Attachment 9 for guidance on retention times and relative retention times of potential interfering peaks.

"Un-assign" the quantitation peaks that are subject to interference.

Compare the average concentration (of the five quantitation peaks) from column 1 with the average concentration from column 2 and evaluate and qualify according to the guidance in the table for evaluating dual column results.

Attachment 6:
Analysis of Multiple Peak Compounds

Toxaphene
(Single Peak Method)

Toxaphene is a mixture of chlorinated camphenes and has a complex and characteristic pattern when analyzed by GC/ECD. This pattern will degrade in the environment with the most of the degradation coming from "reductive de-chlorination" where the higher molecular weight components lose chlorine and form components with lower molecular weights. This degradation makes identification and quantification of toxaphene residues very problematic as the pattern shifts "forward" as the lower molecular weight components become more prominent.

Calibration/Quantitation Peaks
Toxaphene is routinely calibrated and quantified in samples using five peaks on each column over the retention time range. The default peaks are provided in the table below:

Peak	Restek CLPesticides			Restek CLPesticides II		
	RT	RRT	Response Ratio	RT	RRT	Response Ratio
ISTD	2.843	1.000		2.607	1.000	
1	10.460	3.679	1.0	10.633	4.079	1.0
2	10.883	3.810	1.1	11.093	4.256	1.1
3	11.560	4.066	1.6	11.970	4.592	0.92
4	12.170	4.280	1.1	12.647	4.852	0.51
5	12.433	4.373	0.65	12.977	4.978	0.38

Calibration Standards:
Apply the following guidance to both columns/detectors:

Inspect the peaks used for quantitation to make sure that the peaks are properly distinguishable from closely eluting peaks.

Note: This is important because as the column degrades resolution between the peaks may decrease and the integration parameters adjusted. Be sure to apply the same integration parameters to the calibration standards and samples.

ICAL: Update the method with the retention times and responses of the standards.
CCV: Update the method with the retention times of the standards.

Qualitative Identification

Toxaphene is subject to degradation in the environment; that is, exposure to the environment will cause loss of some of the peaks in the sample pattern that are present in the standard. A procedure where the sample pattern matches the standard and a procedure where the sample pattern does not match the pattern but a "toxaphene-like" pattern is present are provided.

a. Samples where pattern matches standard pattern

- Apply the following guidance to both columns/detectors:
- Compare the overall pattern of the sample to the most recently analyzed toxaphene standard.
- If the sample and standard chromatograms agree well, quantify toxaphene using the five characteristic peaks used for calibration as follows:

    - Inspect the peaks used for quantitation to make sure that the peaks are properly distinguishable from closely eluting peaks.
    - Inspect the chromatogram for the presence of interfering peaks from other target compounds or from non-target matrix interferences. The most common interference peak is 4,4'-DDT on column 1 (RTX CLPesticides column).
    - Manually integrate peaks where the data system did not integrate peaks in the same manner as the calibration standards. The most common types of manual integration involve "splitting peaks" that are not resolved by the current integration parameters or identifying peaks as targets because of loss of resolution of peak symmetry.
    - "Undetect" the toxaphene quantitation peaks that are subject to interference. Again, the most common interference peak is 4,4'-DDT on column 1 (RTX CLPesticides column). If 4,4-DDT is present, "undetect" toxaphene peak 1. In this case, the quantitation will be based on the average of the remaining quantitation peaks.
    - Compare the average concentration (of the five quantitation peaks) from column 1 with the average concentration from column 2 and evaluate and qualify according to the guidance in the table for evaluating dual column results.
    - Provide an NCM describing the interference peaks, if present, and how the quantitation of toxaphene was affected.

Example:

Sample X contained toxaphene and 4,4-DDT. Since 4,4-DDT coelutes with the first peak used to quantify toxaphene on column 1, that toxaphene peak was "undetected" and not included in the quantification of toxaphene on column 1. The concentration of 4,4-DDT was most likely biased high on column 1 because it co-elutes with a peak from toxaphene and was reported from Column 2.

Samples where sample pattern does not matches standard pattern closely

- Apply the following guidance to both columns/detectors:
- Compare the overall pattern of the sample to the most recently analyzed toxaphene standard.
- If the sample and standard chromatograms do not agree well, that is, the pattern and ratios of the quantitation peaks are not similar, as is the case of degraded toxaphene, evaluate the data as follows:

  Note: Contact the supervisor, department manager, or Technical Director as soon as toxaphene is tentatively identified. The evaluation of samples for degraded toxaphene is difficult and will require the consultation of the most experienced analysts.

- Inspect the chromatogram for the presence of the peaks used for quantitation. The presence of three or more quant peaks on both columns, especially the first three, indicates that toxaphene may be present.
- Try to match all the peaks (not just peaks used for quantitation) in the sample to all the early peaks in the toxaphene standard. The ratios will not be the same but the presence of peaks in the sample eluting at the same RT as the peaks in the early part of the toxaphene chromatogram is additional evidence that degraded toxaphene is present.
- Inspect the chromatogram for the presence of interfering peaks from other target compounds or from non-target matrix interferences. The most common interference peak will be from 4,4-DDT, which co-elutes with toxaphene peak 1 on Column 1 and is often associated with the presence of toxaphene in the environment. Refer to Attachment 9 for guidance on retention times and relative retention times of potential interfering peaks.
- Identify the sample as containing degraded toxaphene if the overall pattern of the peaks is similar to the toxaphene standard but the peaks elute at earlier RT AND the peaks in the first part of the sample chromatogram correspond reasonably to the early eluting peaks in the toxaphene standard.
- Quantify degraded toxaphene using the total response of all peaks eluting within the pattern in the sample against the total response of all peaks between the first and last eluting peaks in the toxaphene standard.
- Compare the concentration from column 1 with the concentration from column 2 and evaluate and qualify according to the guidance in the table for evaluating dual column results.
- Additionally, qualify the data in an NCM, noting that toxaphene was identified even though the patterns of peaks did not match the standard exactly and that toxaphene quantified against a single standard using total response.

Example:
A pattern similar to toxaphene was present on both columns. The sample's chromatograms contained peaks that match the peaks eluting in the first part of the toxaphene standard pattern. The sample was quantified by comparing the total area of the peaks in the sample with the total area of the peaks in the toxaphene standard. The results were qualified with "J" to indicate that the concentration was estimated because the sample pattern does not match the standard pattern but there was sufficient evidence to identify the presence of toxaphene in the sample.

Attachment 7:
Analysis of Multiple Peak Compounds

Toxaphene
(TAUC Method)

This Attachment outlines the "total area under the curve" (TAUC) procedure for integrating calibration, quality control, and field sample chromatograms. This approach gives a conservative concentration; that is, the result is usually biased high when compared to the "five peak quantitation" described in Attachment 6. This approach quantitates all peaks within the retention time range from Parlar 11 to Parlar 69, regardless of qualitative identification.

Retention Time Window Determination

Establish the retention time window (RTW) for toxaphene using a time range from 0.10 minutes before Parlar 11 to 0.10 minutes after Parlar 69. This window will include all of the major and most of the minor peaks in toxaphene. This window was chosen based on client instruction and ensures that each analyst would use the same RT range for toxaphene. Use this window on both columns.

This RTW must be re-established at the beginning of every sequence via re-analysis of the RTW Standard (i.e., Parlar 11/Parlar 69).

Note: The surrogates and internal standard must fall outside of the retention time range of peaks in the toxaphene standard and, as such, will be excluded from the quantitation.

Attachment 8
Analysis of Multiple Peak Compounds

Technical Chlordane

Technical Chlordane is a mixture of at least 11 major components and 30 or more minor components that is used to prepare specific pesticide formulations. The following components are significant: α-chlordane and γ-chlordane, trans-nonachlor, and heptachlor. The α-chlordane and γ-chlordane isomers are the most prevalent and their detection as single components is a good indicator that technical chlordane may be present.

When the GC pattern of the sample resembles that of the Technical Chlordane standard, quantitate chlordane by comparing the area of 3 to 5 major peaks. Heptachlor and heptachlor epoxide should not be included in this quantitation but rather should be quantitated and reported separately.

Calibration/Quantitation Peaks
Technical chlordane is routinely calibrated and quantified in samples using five peaks on each column over the retention time range. The default peaks are provided in the tables below:

Peak	Restek CLPesticides			Restek CLPesticides II		
	RT	RRT	Response Ratio	RT	RRT	Response Ratio
ISTD	2.843	1.000		2.607	1.000	
1	6.387	2.246	1.0	6.353	2.437	1.0
2	7.323	2.576	1.4	7.483	2.871	1.4
3	8.443	2.970	3.7	8.637	3.313	3.5
4	8.693	3.057	5.4	8.927	3.425	3.0
5	10.130	3.563	0.92	10.570	4.055	0.94

Calibration Standards
Apply the following guidance to both columns/detectors:

- Inspect the peaks used for quantitation to make sure that the peaks are properly distinguishable from closely eluting peaks.

  Note: This is important because as the column degrades resolution between the peaks may decrease and the integration parameters adjusted. Be sure to apply the same integration parameters to the calibration standards and samples.

ICAL: Update the method with the retention times and responses of the standards.
CCV: Update the method with the retention times of the standards.

Qualitative Identification

Technical chlordane is subject to degradation in the environment; that is, exposure to the environment will cause loss of some of the peaks in the sample pattern that are present in the standard. In general, if alpha chlordane and gamma chlordane are detected in the sample and technical chlordane is a target compound, report the sample as containing technical chlordane.

b. Samples where pattern matches standard pattern

- Apply the following guidance to both columns/detectors:
- Compare the overall pattern of the sample to the most recently analyzed technical chlordane standard.
- If the sample and standard chromatograms agree well, quantify technical chlordane using the five characteristic peaks used for calibration as follows:
- Inspect the peaks used for quantitation to make sure that the peaks are properly distinguishable from closely eluting peaks.
- Inspect the chromatogram for the presence of interfering peaks from other target compounds or from non-target matrix interferences.
- Manually integrate peaks where the data system did not integrate peaks in the same manner as the calibration standards. The most common types of manual integration involve “splitting peaks” that are not resolved by the current integration parameters or identifying peaks as targets because of loss of resolution of peak symmetry.
- “Undetect“ the technical chlordane quantitation peaks that are subject to interference.
- Compare the average concentration (of the five quantitation peaks) from column 1 with the average concentration from column 2 and evaluate and qualify according to the guidance in the table for evaluating dual column results.

c. Samples where sample pattern does not matches standard pattern closely

- Apply this guidance to both columns/detectors.
- Compare the overall pattern of the sample to the most recently analyzed technical chlordane standard.
- If the sample and standard chromatograms do not agree well, that is, the pattern and ratios of the quantitation peaks are not similar, as is the case of degraded technical chlordane, evaluate the data as follows:

  Note: Contact the supervisor, department manager, or Technical Director as soon as technical chlordane is tentatively identified. The evaluation of samples for degraded technical chlordane is difficult and will require the consultation of the most experienced analysts.

  - Inspect the chromatogram for the presence of the peaks used for quantitation. The presence of three or more quant peaks on both columns, especially peaks TCHLOR-3 (gamma chlordane) and TCHLOR-4 (alpha chlordane), indicates that technical chlordane is present.
  - Try to match all the peaks (not just peaks used for quantitation) in the sample to the peaks in the standard. The ratios will most likely not agree but the presence of peaks in the sample eluting at the same RT along with the

presence of heptachlor and alpha and gamma chlordane is additional evidence that degraded technical chlordane is present.

- Inspect the chromatogram for the presence of interfering peaks from other target compounds or from non-target matrix interferences. Refer to Attachment 9 for guidance on retention times and relative retention times of potential interfering peaks.
- Identify the sample as containing technical chlordane if the overall pattern of the peaks is similar to the technical chlordane standard and peaks are present for alpha chlordane and gamma chlordane.

- Quantify technical chlordane as the average of the quantitation peaks present in the sample matrix. Alternatively, use the total response of all peaks eluting within the pattern in the sample against the total response of all peaks between the first and last eluting peaks in the technical chlordane standard.

- Compare the concentration from column 1 with the concentration from column 2 and evaluate and qualify according to the guidance in the table for evaluating dual column results.

- Additionally, qualify the data in an NCM, noting that technical chlordane was identified even though the patterns of peaks did not match the standard exactly and note how the quantitation was done: either by the average of the quantitation peaks present or that technical chlordane was quantified against a single standard using total response.

Attachment 9
Standard Preparation Instructions

608/8081 PEST Standard Preparation Recipe
Initial Calibration, Continuing Calibration Verification (CCV), and Initial Calibration Verification (ICV)

PEST A/B Stock Standard Mixes

Stock/Mix	LIMS ID	Vendor/ Part Number	Concentration (ug/mL)
Pest Routine Pesticides	SGABCAL	Restek 32415	2000
Pest Routine Pesticides 2nd Source	SGABICV	Restek 32415.sec	2000
Pest Surrogate Stock	SGPESTSURR	Restek 32000	200
Pest Internal Standard (bromonitrobenzene (BNB))	BNBISTD	Chemservice F2319S	1000

Expiration date: Unopened ampuls: manufacturer's date of expiration Opened ampules: 1 year

Pest ISTD Working Standard (LIMS ID = SGBNB_wk)

Stock/Mix	Aliquot Volume (uL)	Final Volume (mL)	Final Concentration (ug/mL)
Pesticide Internal Standard	800	1000	0.8

Solvent: hexane Expiration: 6 months

Pest Intermediate Working Standard (LIMS ID = INTPESTAB)

Stock/Mix	Aliquot Volume (uL)	Final Volume (mL)	Final Concentration (ug/mL)
Routine Pesticide Stock	50 (SGABCAL)* 800 (SGPESTSURR)	10	10 16

Solvent: hexane Expiration: 6 months *If using Supelco solution at 1000 ug/mL then use 100 uL.

Pest Calibration Standards

Cal Level	LIMS ID	Intermediate Aliquot	Final Volume (mL)	Pest ISTD Working Std Volume (mL)
1	SGPEST1	12.5uL	50mL	5.0
2	SGPEST2	25uL	50mL	5.0
3	SGPEST3	50uL	50mL	5.0
4	SGPEST4	500uL	250mL	25
5	SGPEST5	200uL	50mL	5.0
6	SGPEST6	400uL	50mL	5.0

Solvent: hexane Expiration: 6 months

Pest ICV Intermediate Working Standard (LIMS ID = INTPESTABICV)

Stock/Mix	Aliquot Volume (uL)	Final Volume (mL)	Final Concentration (ug/mL)
Routine Pesticide 2ND Source Stock	50	10	10

Solvent: hexane Expiration: 6 months *If using Supelco solution at 1000 ug/mL then use 100 uL

Pest ICV Standard (LIMS ID = SGABICV)

Cal Level	ICV Stock	Surrogate Stock	Final Volume	Pest ISTD Working Std Volume (mL)
4 ICV	100uL	8uL	50mL	5mL
Dilute to the final volume and then add the Pesticide ISTD working Standard				

Solvent: hexane Expiration: 6 months

Pest On-Column Concentrations (ug/mL)

Compound	CAL 1	CAL 2	CAL 3	CAL 4	CAL 5	CAL 6
Bromonitrobenzene (ISTD)	0.08	0.08	0.08	0.08	0.08	0.08
Tetrachloro-m-xylene Decachlorobiphenyl	0.0040	0.0080	0.016	0.032	0.064	0.128
Alpha-BHC, Gamma-BHC, Beta-BHC, Delta-BHC, Heptachlor, Aldrin, Heptachlor epoxide, Gamma-Chlordane, Alpha-Chlordane Endosulfan I, 4,4'-DDE, Dieldrin, Endrin 4,4'-DDD, Endosulfan II, 4,4'-DDT, Endrin aldehyde. Methoxychlor, Endosulfan sulfate, Endrin ketone	0.0025	0.0050	0.010	0.020	0.040	0.08

Storage: glass container with Teflon-lined cap; in standards' refrigerator at 0-6°C.

508/608/8081 AR1660 Standard Preparation Recipe
Initial Calibration, Continuing Calibration Verification (CCV), and
Initial Calibration Verification (ICV)

Aroclor Stock Standard Mixes

Stock/Mix	LIMS ID	Vendor/ Part Number	Final Concentration (ug/mL)
AR1660 Stock	SG1660CAL (or AR1660)	Restek 32039	1000
AR1660 Stock-2nd Source	SG1660ICV	Restek 32039.sec	1000
Pest Surrogate Stock	SGPESTSURR	Restek 32000	200
Pesticide Internal Standard (bromonitrobenzene (BNB))	BNBISTD	Chemservice F2319S	1000

Expiration date: Unopened ampules: manufacturer's date of expiration Opened ampules: 1 year

Pest ISTD Working Standard (LIMS ID = SGBNB_wk)

Stock/Mix	Aliquot Volume (uL)	Final Volume (mL)	Conc. (ug/mL)
Pesticide Internal Standard	800	1000 (1.0L)	0.08

Solvent: hexane Expiration: 6 months

AR1660 Initial Calibration Standards

Cal Level	LIMS ID	AR1660 Stock	Surrogate Stock	Final Volume	Pest ISTD Working Standard
1	SG1660-1	2.5uL	1.0uL	50mL	5.0
2	SG1660-2	5uL	2uL	50mL	5.0
3	SG1660-3	12.5uL	4uL	50mL	5.0
4	SG1660-4	125uL	40uL	250mL	25
5	SG1660-5	50uL	16ul	50mL	5.0
6	SG1660-6	75uL	32uL	50mL	5.0
Dilute to the final volume and then add the Pesticide ISTD working Standard					

Solvent: hexane Expiration: 6 months

AR1660 ICV Standard (LIMS ID = SGPCBICV_)

Cal Level	AR1660 ICV Stock	Surrogate Stock	Final Volume	Pest ISTD Working Standard
4 ICV	25uL	8uL	50mL	5mL
Dilute to the final volume and then add the Pesticide ISTD working Standard				

Solvent: hexane Expiration: 6 months

AR1660 On-Column Concentrations (ug/mL)

Compound	CAL 1	CAL 2	CAL 3	CAL 4	CAL 5	CAL 6
Bromonitrobenzene (ISTD)	0.08	0.08	0.08	0.08	0.08	0.08
Tetrachloro-m-xylene, DCB	0.0040	0.008	0.016	0.032	0.064	0.128
Aroclor 1016/AR1260	0.05	0.1	0.25	.50	1.0	1.5

Storage: glass container with Teflon-lined cap; in standards' refrigerator at 0-6°C.

508/608/8081 Technical Chlordane Standard Preparation Recipe
Initial Calibration, Continuing Calibration Verification (CCV), and
Initial Calibration Verification (ICV)

Stock Standard Mixes

Stock/Mix	LIMS ID	Vendor/ Part Number	Concentration (ug/mL)
Technical chlordane	SGCHLORCAL_	Restek 32072	5000
Technical chlordane ICV (2nd Source)	SGCHLORICV_	Restek 32072.sec	5000
Pesticide Internal Standard (bromonitrobenzene (BNB))	BNBISTD	Chemservice F2319S	1000

Expiration: Unopened ampuls: manufacturer's date of expiration Opened ampules: 1 year

Pest ISTD Working Standard (LIMS ID = SGBNB_wk)

Stock/Mix	Aliquot Volume (uL)	Final Volume (mL)	Conc. (ug/mL)
Pesticide Internal Standard	800	1000 (1.0L)	0.08

Solvent: hexane Expiration: 6 months

Technical Chlordane Intermediate Working Standard (LIMS ID = INTCHLOR)

Stock/Mix	Aliquot Volume (uL)	Final Volume (mL)	Final Concentraton (ug/mL)
Technical Chlordane Stock	1000	10	500

Solvent: hexane Expiration: 6 months

Technical Chlordane Initial Calibration Standards

Cal Level	LIMS ID	TCHLOR Stock	Final Volume	Pest ISTD Working Standard
1	SGCHLOR1	2.5uL	50mL	5mL
2	SGCHLOR2	5uL	50mL	5mL
3	SGCHLOR3	12.5uL	50mL	5mL
4	SGCHLOR4	125uL	250mL	50mL
5	SGCHLOR5	50uL	50mL	5mL
6	SGCHLOR6	125uL	50mL	5mL
Dilute to the final volume and then add the Pesticide ISTD working Standard				

Solvent: hexane Expiration: 6 months

Technical Chlordane ICV Intermediate Working Standard (LIMS ID = INTCHLORICV)

Stock/Mix	Aliquot Volume (uL)	Final Volume (mL)	Final Concentraton (ug/mL)
Technical Chlordane Source Stock	100	10	50

Solvent: hexane Expiration: 6 months

Technical Chlordane ICV Standard (LIMS ID = SGCHLRICV_)

Cal Level	TCHLOR ICV Stock	Final Volume	Pest ISTD Working Standard
4 ICV	250uL	50mL	5mL
Dilute to the final volume and then add the Pesticide ISTD working Standard			

Solvent: hexane Expiration: 6 months

Technical Chlordane On-Column Concentrations (ug/mL)

Compound	CAL 1	CAL 2	CAL 3	CAL 4	CAL 5	CAL 6
Bromonitrobenzene (ISTD)	0.08	0.08	0.08	0.08	0.08	0.08
Technical chlordane	0.025	0.050	0.125	0.25	0.50	1

Storage: **Glass container with Teflon-lined cap; in standards' refrigerator at 0-6°C.**

508/608/8081 Toxaphene Standard Preparation Recipe
Initial Calibration, Continuing Calibration Verification (CCV), and
Initial Calibration Verification (ICV)

Stock Standard Mixes

Stock/Mix	LIMS ID	Vendor/ Part Number	Concentration (ug/mL)
Toxaphene Calibration Stock	SGTOXCAL_	Restek 32071	5000
Toxaphene ICV (2nd Source)	SGTOXICV_	Restek 32071.sec	5000
Pesticide Internal Standard (bromonitrobenzene (BNB))	BNBISTD_	Chemservice F2319S	1000

Expiration: Unopened ampuls: manufacturer's date of expiration Opened ampuls: 1 year

Pest ISTD Working Standard (LIMS ID = SGBNB_wk)

Stock/Mix	Aliquot Volume (uL)	Final Volume (mL)	Conc. (ug/mL)
Pesticide Internal Standard	800	1000 (1.0L)	0.08

Solvent: hexane Expiration: 6 months

Toxaphene Initial Calibration Standards

Cal Level	LIMS ID	TOX Stock (uL)	Final Volume (mL)	Pest ISTD Working Standard (mL)
1	SGTOX1	2.5	50	5.0
2	SGTOX2	5	50	5.0
3	SGTOX3	12.5	50	5.0
4	SGTOX4	125	250	25.0
5	SGTOX5	50	50	5.0
6	SGTOX6	100	50	5.0
Dilute to the final volume and then add the Pesticide ISTD working Standard				

Solvent: hexane Expiration: 6 months

Toxaphene ICV Intermediate Working Standard (LIMS ID = INTTOXICV)

Stock/Mix	Aliquot Volume (uL)	Final Volume (mL)	Final Concentration (ug/mL)
Toxaphene Source Stock	100	10	50

Solvent: hexane Expiration: 6 months

Toxaphene ICV Standard (LIMS ID = SGICVTOX_)

Cal Level	TOX ICV Intermediate	Final Volume	Pest ISTD Working Standard
4 ICV	125uL	50mL	5.0mL
Dilute to the final volume and then add the Pesticide ISTD working Standard			

Solvent: hexane Expiration: 6 months

Toxaphene On-Column Concentrations (ug/mL)

Compound	CAL 1	CAL 2	CAL 3	CAL 4	CAL 5	CAL 6
Bromonitrobenzene (ISTD)	0.08	0.08	0.08	0.08	0.08	0.08
Toxaphene	0.25	0.50	1.25	2.5	5.0	10

Storage: Glass container with Teflon-lined cap; in standards' refrigerator at 0-6°C.

508/608/8081 Single Point PCB Standard Preparation Recipe
Initial Calibration, Continuing Calibration Verification (CCV), and
Initial Calibration Verification (ICV)

Aroclor Stock Standard Mixes

Stock/Mix	LIMS ID	Vendor/ Part Number	Concentration (ug/mL)
AR1221	SG1221CAL	Restek 32007	1000
AR1254	SG1254CAL	Restek 32011	1000
AR1232	SG1232CAL	Restek 32008	1000
AR1262	SG1262CAL	Restek 32409	1000
AR1242	SG1242CAL	Restek 32009	1000
AR1268	SG1268CAL	Restek 32410	1000
AR1248	SG1248CAL	Restek 32010	1000
AR1221 ICV	SG1221ICV	Restek 32007.sec	1000
AR1254 ICV	SG1254ICV	Restek 32011.sec	1000
AR1232 ICV	SG1232ICV	Restek 32008.sec	1000
AR1262 ICV	SG1262ICV	Restek 32409.sec	1000
AR1242 ICV	SG1242ICV	Restek 32009.sec	1000
AR1268 ICV	SG1268ICV	Restek 32410.sec	1000
AR1248 ICV	SG1248ICV	Restek 32010.sec	1000
Pesticide Internal Standard (bromonitrobenzene (BNB))	BNBISTD	Chemservice F2319S	1000

Expiration date: Unopened ampules: manufacturer's date of expiration Opened ampules: 1 year

Pest ISTD Working Standard (LIMS ID = SGBNB_wk)

Stock/Mix	Aliquot Volume (uL)	Final Volume (mL)	Conc. (ug/mL)
Pesticide Internal Standard	800	1000	0.08

Solvent: hexane Expiration: 6 months

AR2154 Calibration Standard (LIMS ID = SG21/54-4)

Cal Level	AR1221 Stock	AR1254 Stock	Final Volume	Pest ISTD Working Standard	AR2154 Conc (ug/mL)	ISTD Conc (ug/mL)
4	25uL	25uL	50mL	5mL	0.50	0.08
Dilute to the final volume and then add the Pesticide ISTD working Standard						

Solvent: hexane Expiration: 6 months

AR3262 Calibration Standard (LIMS ID = SG32/62-4)

Cal Level	AR1232 Stock	AR1262 Stock	Final Volume	Pest ISTD Working Standard	AR3262 Conc (ug/mL)	ISTD Conc (ug/mL)
4	25uL	25uL	50mL	5mL	0.50	0.08
Dilute to the final volume and then add the Pesticide ISTD working Standard						

Solvent: hexane Expiration: 6 months

AR4268 Calibration Standard (LIMS ID = SG42/68-4)

Cal Level	AR1242 Stock	AR1268 Stock	Final Volume	Pest ISTD Working Standard	AR4268 Conc (ug/mL)	ISTD Conc (ug/mL)
4	25uL	25uL	50mL	5mL	0.50	0.08
Dilute to the final volume and then add the Pesticide ISTD working Standard						

Solvent: hexane Expiration: 6 months

AR1248 Calibration Standard (LIMS ID = SG 1248-4)

Cal Level	AR1248 Stock	Final Volume	Pest ISTD Working Standard	AR1248 Conc (ug/mL)	ISTD Conc (ug/mL)
4	25uL	50mL	5mL	0.50	0.08
Dilute to the final volume and then add the Pesticide ISTD working Standard					

Solvent: hexane Expiration: 6 months

Storage: glass container with Teflon-lined cap; in standards' refrigerator at 0-6°C.

Note: If a multi-point ICAL is required for any of the single-point PCBs then follow the instructions for 1016/1260 multi-point curve, excluding the surrogates.

508/608/8081 Column/Instrument Checks
DDT/Endrin Breakdown

Stock Standard Mixes

Stock/Mix	LIMS ID	Vendor/ Part Number	Concentration (ug/mL)
PEVAL Stock	SGPEM_	Restek 32417	Endrin 100 p,p'-DDT 200
2,4'-DDT PEVAL Stock	SG24PEM_	Restek 32200	1000
Pesticides Internal Standard (bromonitrobenzene (BNB))	BNBISTD_	Chemservice F2319S	1000

Expiration date: Unopened ampules: manufacturer's date of expiration Opened ampules: -1 year

Pest ISTD Working Standard (LIMS ID = SGBNB_wk)

Stock/Mix	Aliquot Volume (uL)	Final Volume (mL)	Conc. (ug/mL)
Pesticide Internal Standard	800	1000 (1.0L)	0.08

Solvent: hexane Expiration: 6 months

PEVAL Run Standard (LIMS ID = SGPEVAL_)

Stock/Mix	Aliquot Volume (uL)	Final Volume (mL)	Conc. (ug/mL)
PEVAL Stock	80	250	Endrin 0.032 p,p'-DDT 0.064
Dilute to final volume and then add 25mL of the Pesticide ISTD Working Standard			

Solvent: hexane Expiration: 6 months

2,4-DDT PEVAL Run Standard (LIMS ID = SG24PEVAL_)

Stock/Mix	Aliquot Volume (uL)	Final Volume (mL)	Conc. (ug/mL)
2,4'-DDT PEVAL Stock	20 (0.020mL)	50.0	0.40
Dilute to final volume and then add 5.0mL of the Pesticide ISTD Working Standard			

Solvent: hexane Expiration: 6 months

508/608/8081 Surrogate and Spiking Solution Preparation Recipes

Stock Standard Mixes

Stock/Mix	LIMS ID	Vendor/ Part Number	Concentration (ug/mL)
Pesticides Intermediate Mix	INTPESTABICV_		10
AR 1016 / AR 1260 Mix	SG1660CAL_	Restek 32039	1000
Pesticides Surrogate Stock (TCX and DCB)	SGPESTSURR_	Restek 32000	200

Expiration date: Unopened ampules: manufacturer's date of expiration Opened ampules:1 year

608 Working Surrogate Standard (LIMS ID = PESTwkSURR_)

Stock/Mix	Aliquot Volume (uL)	Final Volume (mL)	Conc. (ug/mL)
Pesticides Surrogate Stock (TCX and DCB)	1250 (1.25mL)	500	0.50 each

Solvent: methanol Expiration: 6 months

Pesticide Working Spiking Standard (LIMS ID = 608wkSPIKE__)

Stock/Mix	Aliquot Volume (uL)	Final Volume (mL)	Conc. (ug/mL)
Pesticides Intermediate Mix	1000 (1.0mL)	100	0.10

Solvent: methanol Expiration: 6 months

1660 Working PCB Spiking Standard (LIMS ID = 1660wkSPIK_)

Stock/Mix	Aliquot Volume (uL)	Final Volume (mL)	Conc. (ug/mL)
AR 1016 / AR 1260 Mix	1000	100	10 each

Solvent: methanol Expiration: 6 months

Storage: glass container with Teflon-lined cap; in standards' refrigerator at 0-6°C.

APIX and Additional Standards

APIX/Additions Calibration Mix (LIMSID = SGAP9CAL; Vendor/Part Number = Restek CS-16683)

Compound	Concentration (ug/mL)
Chlorobenzilate	500
Kepone	500
Hexachlorobenzene	100
Isodrin	25
Mirex	100
2,4-DDT, 2,4-DDD, 2,4-DDE	50 each

Expiration: Unopened ampules: manufacturer's expiration date; Opened ampules: 1 year from open date

APIX/Additions ICV Mix (LIMSID = SGAP9ICV; Vendor/Part Number = Restek CS-16683.SS)

Stock/Mix	Concentration (ug/mL)
Chlorobenzilate	500
Kepone	500
Hexachlorobenzene	100
Isodrin	25
Mirex	100
2,4-DDT,2,4-DDD,2,4-DDE	50 each

Expiration: Unopened ampules: manufacturer's expiration date; Opened ampules: 1 year from open date

Pest ISTD (LIMSID = BNBISTD; Vendor/Part Number = Chemservice F2319S)

Parent Standard	Aliquot Volume (uL)	Final Volume (mL)	Concentration (ug/mL)
Pesticide Internal Standard (bromonitrobenzene (BNB))	800uL	1000 (1.0L)	1000

Expiration: Unopened ampules: manufacturer's expiration date; Opened ampules: 1 year from open date

Pest ISTD Working Standard (LIMSID = SGBNB_wk)

Parent Standard	Aliquot Volume (uL)	Final Volume (mL)	Final Concentration (ug/mL)
Pesticide Internal Standard	800uL	1000 (1.0L)	0.8

Solvent: Hexane Expiration: 6 months from prep date

APIX/Additions Working Standard (LIMSID = INT8081ADD_)

Parent Standard	Aliquot Volume (uL)	Final Volume (mL)	Final Concentration (ug/mL)
SGAP9CAL	400	10	Various – see below

Compound	Final Concentration (ug/mL)
Chlorobenzilate	20
Kepone	20
Hexachlorobenzene	4
Isodrin	1
Mirex	4
2,4-DDT, 2,4-DDD, 2,4-DDE	2

Solvent: Hexane Expiration: 6 months from prep date

APIX/Additions Calibration Standards

Cal Level	LIMS ID	SGADDNINT Aliquot (uL)	Final Volume (mL)	Pest ISTD Working Std Volume (mL)
1	SGADDNS-1	30	25	2.5
2	SGADDNS-2	62.5	25	2.5
3	SGADDNS-3	125	25	2.5
4	SGADDNS-4	1000	100	10
5	SGADDNS-5	500	25	2.5
6	SGADDNS-6	1000	25	2.5

Solvent: Hexane Expiration: 6 months from prep date

APIX/Additions On-Column Concentrations (ug/mL)

Compound	CAL 1	CAL 2	CAL 3	CAL 4	CAL 5	CAL 6
Bromonitrobenzene (ISTD)	0.08	0.08	0.08	0.08	0.08	0.08
Chlorobenzilate	0.024	0.05	0.10	0.2	0.4	0.8
Kepone	0.024	0.050	0.10	0.20	0.40	0.8
Hexachlorobenzene	0.0048	0.01	0.02	0.04	0.08	0.16
Isodrin	0.0012	0.0025	0.005	0.01	0.02	0.04
Mirex	0.0048	0.01	0.02	0.04	0.08	0.16
2,4-DDT, 2,4-DDD, 2,4-DDE	0.0024	0.005	0.01	0.02	0.04	0.08

Storage: Glass container with Teflon-lined cap; in standards' refrigerator at 0-6°C.

APIX/Additions ICV (LIMSID = INT8081ADDICV_)

Parent Standard	Aliquot Volume (uL)	Final Volume (mL)	Final Concentration (ug/mL)
SGAP9ICV	400	10	Various – see below

Compound	Final Concentration (ug/mL)
Chlorobenzilate	20
Kepone	20
Hexachlorobenzene	4
Isodrin	1
Mirex	4
2,4-DDT, 2,4-DDD, 2,4-DDE	2

Solvent: Hexane Expiration: 6 months from prep date

APIX ICV Standard (LIMS ID = SGADDNSICV_)

Cal Level	LIMS ID	APIX ICV Intermediate Aliquot (uL)	Final Volume (mL)	Pest ISTD Working Std Volume (mL)
4 ICV	SGADDNSICV	250	25	2.5

Solvent: Hexane Expiration: 6 months from prep date

Attachment 10
Example Retention Times and Relative Retention Times
(Potential Interference Compounds)

Compound	Column 1 Restek CLPesticides	
	Retention Time*	Relative Retention Time
Bromonitrobenzene (ISTD)	2.843	1.000
Tetrachloro-m-xylene (SURR)	4.153	1.461
AR1221-1	4.453	1.568
AR1221-2	4.713	1.660
AR1221-3	4.787	1.685
Hexachlorobenzene	4.843	1.705
Alpha-BHC	5.147	1.811
AR1248-1	5.397	1.898
AR1016-1	5.400	1.899
AR1232-1	5.400	1.901
AR1242-1	5.400	1.899
Gamma-BHC (Lindane)	5.700	2.006
AR1248-2	5.743	2.020
Beta-BHC	5.857	2.061
Delta-BHC	6.170	2.170
AR1016-2	6.187	2.176
AR1232-2	6.187	2.178
AR1242-2	6.187	2.176
AR1248-3	6.187	2.176
TCHLOR-1	6.387	2.246
AR1016-3	6.417	2.257
AR1232-3	6.417	2.259
AR1242-3	6.417	2.257
AR1248-4	6.420	2.258
AR1242-4	6.527	2.295
Heptachlor	6.527	2.295
AR1016-4	6.527	2.295
AR1232-4	6.527	2.298
AR1248-5	6.527	2.295
Aldrin	7.070	2.487
TCHLOR-2	7.323	2.576
Isodrin	7.687	2.707
AR1016-5	7.820	2.750
AR1254-1	7.820	2.754
AR1232-5	7.820	2.754
AR1242-5	7.820	2.750
2,4-DDE	8.183	2.881
Compound	**Column 1 Restek CLPesticides**	

	Retention Time*	Relative Retention Time
Heptachlor epoxide	8.210	2.887
AR1254-2	8.300	2.923
Gamma-Chlordane	8.443	2.970
TCHLOR-3	8.443	2.970
TCHLOR-4	8.693	3.057
Alpha-Chlordane	8.700	3.060
4,4'-DDE	8.853	3.114
Endosulfan I	8.973	3.156
AR1254-3	9.073	3.195
2,4-DDD	9.187	3.235
Dieldrin	9.430	3.317
AR1254-4	9.593	3.378
2,4-DDT	9.657	3.400
Endrin	9.857	3.467
Kepone	9.923	3.494
AR1254-5	9.923	3.494
4,4'-DDD	9.980	3.510
Chlorobenzilate	10.007	3.523
TCHLOR-5	10.130	3.563
Endosulfan II	10.260	3.608
TOX-1	10.460	3.679
4,4'-DDT	10.470	3.682
AR1260-1	10.610	3.732
AR1262-1	10.610	3.736
AR1260-2	10.693	3.761
TOX-2	10.833	3.810
Endrin aldehyde	11.000	3.869
AR1262-2	11.040	3.887
AR1260-3	11.113	3.909
Methoxychlor	11.317	3.980
Mirex	11.533	4.061
TOX-3	11.560	4.066
AR1260-4	11.573	4.070
AR1262-3	11.573	4.075
Endosulfan sulfate	11.750	4.132
AR1260-4	12.063	4.243
AR1268-1	12.117	4.261
TOX-4	12.170	4.280
AR1268-2	12.183	4.285
Endrin ketone	12.220	4.298
Compound	**Column 1 Restek CLPesticides**	
	Retention Time*	**Relative Retention**

		Time
TOX-5	12.433	4.373
AR1268-3	12.513	4.401
AR1262-4	13.127	4.622
AR1268-4	13.127	4.617
AR1268-5	13.617	4.789
Decachlorobiphenyl (SURR)	13.943	4.904

Compound	Column 2 Restek CLPesticides II	
	Retention Time	Relative Retention Time
Bromonitrobenzene (ISTD)	2.607	1.000
Tetrachloro-m-xylene (SURR)	4.100	1.584
AR1221-1	4.633	1.780
AR1221-2	4.877	1.873
Hexachlorobenzene	4.967	1.908
AR1221-3	4.980	1.913
Alpha-BHC	5.220	2.003
AR1016-1	5.667	2.174
AR1248-1	5.667	2.174
AR1232-1	5.680	2.182
AR1242-1	5.680	2.179
Gamma-BHC (Lindane)	5.843	2.242
Beta-BHC	5.997	2.300
AR1016-2	6.017	2.308
AR1232-2	6.017	2.311
AR1242-2	6.020	2.309
AR1016-3	6.073	2.330
AR1232-3	6.073	2.333
AR1242-3	6.073	2.330
TCHLOR-1	6.353	2.437
AR1016-4	6.437	2.469
AR1248-2	6.437	2.469
AR1232-4	6.440	2.474
AR1242-4	6.440	2.471
Delta-BHC	6.513	2.499
Heptachlor	6.613	2.537
AR1016-5	6.673	2.560
AR1232-5	6.673	2.563
AR1242-5	6.673	2.560
Aldrin	7.200	2.762
TCHLOR-2	7.483	2.871
Compound	Column 2 Restek CLPesticides II	
	Retention Time	Relative Retention Time

AR1248-3	7.727	2.964
Isodrin	7.930	3.046
AR1248-4	8.007	3.072
AR1248-5	8.083	3.101
Heptachlor epoxide	8.277	3.175
AR1254-1	8.280	3.181
AR1254-2	8.637	3.318
TCHLOR-3	8.637	3.313
Gamma-Chlordane	8.640	3.315
2,4-DDE	8.657	3.325
Alpha-Chlordane	8.890	3.426
TCHLOR-4	8.927	3.425
4,4'-DDE	9.037	3.467
Endosulfan I	9.270	3.556
AR1254-3	9.523	3.658
Dieldrin	9.557	3.666
2,4-DDD	9.643	3.704
AR1254-4	9.937	3.817
Chlorobenzilate	10.107	3.882
Endrin	10.110	3.878
2,4-DDT	10.197	3.917
Kepone	10.307	3.959
4,4'-DDD	10.330	3.964
Endosulfan II	10.490	4.024
AR1254-5	10.560	4.056
TCHLOR-5	10.570	4.055
TOX-1	10.633	4.079
4,4'-DDT	10.883	4.175
AR1260-1	11.040	4.235
AR1262-1	11.040	4.241
Endrin aldehyde	11.083	4.252
TOX-2	11.093	4.256
AR1260-2	11.133	4.271
AR1260-3	11.553	4.432
AR1262-2	11.553	4.438
Endosulfan sulfate	11.560	4.435
AR1260-4	11.677	4.480
TOX-3	11.970	4.592
AR1260-4	11.983	4.597
AR1262-3	11.983	4.603
Methoxychlor	12.097	4.641
Mirex	12.393	4.761
Endrin ketone	12.473	4.785
Compound	**Column 2 Restek CLPesticides II**	
	Retention Time	**Relative Retention Time**
AR1268-1	12.603	4.835

TOX-4	12.647	4.852
AR1268-2	12.687	4.867
TOX-5	12.977	4.978
AR1268-3	13.127	5.036
AR1262-4	13.623	5.233
AR1268-4	13.623	5.226
AR1268-5	14.197	5.446
Decachlorobiphenyl (SURR)	14.657	5.623

***Note**: These retention times are listed as guidance and reflect the retention times of the target compounds at the time this SOP was released. Changes in instrument configuration, length of column, etc. will affect these values.
***Note:** It has been found that in some situations the elution order of 4,4-DDe and endosulfan I can switch on CLP I column.

Attachment 11:
Poor Responder Information

The following analytes have been identified, in the reference method and/or via historical data, to be a poor and/or erratic performers:

Kepone
Chlorobenzilate (This analyte is impacted by the presence of Kepone due to its close elution.)

These analytes are exempt from the LCS, MS, MSD, Sporadic Marginal Exceedance, ICAL, ICV, and CCV criteria as listed in this SOP.

Note: The use of Sporadic Marginal Exceedances, as defined in SOP SA-QA-017, is not permitted for samples originating from South Carolina.

Attachment 12: Glassware Cleaning Procedures

GLASSWARE CLEANING PROCEDURES

SEMIVOLATILE GC LAB

1. Scrub with tap water and Liquinox.
2. Rinse 3 times thoroughly with tap water.
3. Rinse 3 times thoroughly with Acetone.
4. Place volumetric top-down within storage rack and allow to air dry.
5. Store in closed drawer.

FSG046:12.04.13:0

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18.0 Revision History

Summary of Changes for Revision 24:
- Added reference to preparation via separatory funnel to section 2.0.
- Added containers to section 8.1 and 8.2. LVI prep is performed using 250mL amber unpreserved.
- Added 608.3 breakdown criteria to section 9.2 of 20%.
- Added DODQSM V5 criteria to section 9.2.3.
- Removed requirement for capping Toxaphene CCV when using TAUC reporting in section 9.2.5.
- Updated section 12.0 to reflect TNI2016, DOD and TRPP requirements.

Summary of changes for Revision 23:
- Updated expiration dates of purchased and intermediate standards. Standards purchased in flame-sealed ampules will expire 1 year from opening and intermediates or working standards will expire 6 months after preparation.
- Updated CCV analysis requirement for TAUC to be in line with SW-846 revision 4. Updates are in section 9.2.5 and Attachment 3.

Summary of Changes:
- Minor editorial, grammatical, and/or formatting changes made.
- Updated SOP signatories to reflect current responsibilities and titles.
- Added note that SC DHEC prohibits the use of sporadic marginal exceedances. This change stems from SC DHEC Technical Deficiency Letter dated 05/14/19. Section 9.1, Section 16.2.10, and Attachment 11
- Added %RSE requirement. (This change stems from SC DHEC Technical Deficiency Letter dated 05/14/19.) Section 9.2.2.2 and Attachment 3

SEMI-VOLATILE COMPOUNDS BY GC/MS

(Methods: EPA 625, EPA 625.1, EPA 8270D, EPA 8270E, EPA 8270D_LL, EPA 8270E_LL, EPA 8270D_LL_PAH, and EPA 8270E_LL_PAH)

Approvals (Signature/Date):

Brad Mullis — 10/27/2021

Brad Mullis — Date
Department Manager

Kimberly Chamberlain — 10/27/2021

Kimberly Chamberlain — Date
Quality Assurance Manager / Technical Director
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1.0 SCOPE AND APPLICATION

This SOP contains the procedures for the determination of extractable semi-volatile organic compounds (SVOC) by gas chromatography/mass spectrometry (GC/MS).

The routine matrices performed by this procedure are waters and soils. Other matrices which may be performed include wipes, leachates, tissues, and wastes.

A complete target analyte list, the reporting limits (RL), the method detection limits (MDL), and the accuracy and precision criteria associated with this procedure are provided in the TALS Method Limit Groups (MLGs).

2.0 SUMMARY OF METHOD

A measured volume or weight of sample is extracted using continuous liquid-liquid or microwave extraction procedures. The extract is then analyzed by GC/MS. Qualitative identification of the target compounds in the extract is based on the retention time and the relative abundance of the characteristic masses determined from standards analyzed on the same GC/MS under the same conditions. Quantitative analysis is performed using the internal standard technique with a single characteristic ion.

This SOP is based on the following methods: EPA 625, EPA 625.1, EPA 8270E, and EPA 8270D.

Note: This SOP contains the procedures for several variations of the SW-846 methods. These variations include:

- EPA 8270D/EPA 8270E = Routine EPA 8270D/EPA 8270E method.
- EPA 8270D_LL/EPA 8270E_LL = Low-level EPA 8270D/EPA 8270E method, providing lower RLs and MDLs than the routine method. This low-level method can incorporate a polynuclear aromatic hydrocarbon (PAH) sublist option to accommodate low-level PAH reporting.
- EPA 8270D_LL_PAH/EPA 8270E_LL_PAH = Low-level EPA 8270D/ EPA 8270E PAH-only method, providing lower RLs and MDLs than the routine method. This method is used for low-level PAH analytes only and ,is used for DOD QSM V5 samples, and incorporates ortho-terphenyl (OTP) as the surrogate compound. Refer to the DOD QSM document for analytical specifications required in addition to those listed in this SOP.

These three sets of procedures incorporate slightly different standard concentrations, surrogate compounds, instrument configuration, and/or QC evaluation criteria, which are outlined separately in the applicable sections of this SOP.

This SOP also gives the procedures for analyzing and reporting samples using the MS in the Selective Ion Monitoring (SIM) mode.

3.0 DEFINITIONS

Refer to the Glossary Section of the *Quality Assurance Manual* (QAM) for a complete listing of applicable definitions and acronyms.

4.0 INTERFERENCES

4.1 Procedural Interferences

Interferences may be caused by contaminants in solvents, reagents, glassware, and other sample processing apparatus and can make identification and/or quantification of the target analytes difficult.

All sample collection containers are single-use disposable containers which limits the potential for contamination. All non-disposable labware must be scrupulously cleaned in accordance with the posted Labware Cleaning Instructions to ensure it is free from contaminants and does not contribute artifacts.

High purity reagents and solvents are used to help minimize interference problems. Acetone and methylene chloride must be verified prior to use in accordance with the TestAmerica Solvent Lot Testing Program.

Instrument and/or method blanks are routinely used to demonstrate all reagents and apparatus are free from interferences under the conditions of the analysis.

4.2 Matrix Interferences

Matrix interferences may be caused by contaminants that are co-extracted from the sample matrix. The extract may require dilution prior to analysis to reduce or eliminate the interferences.

Interfering contamination may occur when a sample containing low concentrations of analytes is analyzed immediately following a sample containing relatively high concentrations of analytes. As such, samples known to be clean should be analyzed first. To prevent carryover into subsequent samples, analysis of reagent blanks may be needed after the analysis of a sample containing high concentrations of analytes.

If there is interference with the primary ion, then secondary ions may be used for quantification. If a secondary ion is used for quantification, the linearity of the secondary ion must be established by meeting the criteria in Section 11.

The basic conditions of the initial extraction may cause hydrolysis and degradation of some target compounds. The degradation may be pronounced in phthalate esters.

Refer to Section 11.1.5 for more information on the chemical relationships of these compounds.

5.0 SAFETY

Employees must abide by the policies and procedures in the TestAmerica Environmental Health and Safety Manual (EHSM), the TestAmerica Savannah Addendum to the EHSM, and this document.

This procedure may involve hazardous materials, operations, and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user to follow appropriate safety, waste disposal, and health practices under the assumption that all samples and reagents are potentially hazardous.

The analyst must protect himself/herself from exposure to the sample matrix. Many of the samples that are tested may contain hazardous chemical compounds or biological organisms. The analyst must, at a minimum, wear protective clothing (lab coat), eye protection (safety glasses or face shield), disposable gloves, and closed-toe, nonabsorbent shoes when handling samples.

5.1 Specific Safety Concerns or Requirements

The toxicity or carcinogenicity of chemicals used in this method has not been precisely defined. Each chemical should be treated as a potential health hazard, and exposure to these chemicals should be minimized.

Methylene chloride is a carcinogen and an irritant. It causes irritation to the respiratory tract and has a strong narcotic effect with symptoms of mental confusion, light-headedness, fatigue, nausea, vomiting, and headache. It may be absorbed through the skin and can cause irritation and pain to the skin and eyes.

The gas chromatograph and mass spectrometer contain zones that have elevated temperatures. The analyst must be aware of the locations of those zones, and must cool them to room temperature prior to working on them.

The mass spectrometer is under deep vacuum. The mass spectrometer must be brought to atmospheric pressure prior to working on the source.

There are areas of high voltage in both the gas chromatograph and the mass spectrometer. Depending on the type of work involved, either turn the power to the instrument off, or disconnect it from its source of power.

5.2 Primary Materials Used

The following is a list of the materials used in this procedure, which have a serious or significant hazard rating, and a summary of the primary hazards listed in their MSDS/SDS.

NOTE: This list does not include all materials used in the procedure. A complete list of materials used in this procedure can be found in the Reagents and Standards Section and the Equipment and Supplies Section of this SOP

Employees must review the information in the MSDS/SDS for each material before using it for the first time or when there are major changes to the MSDS/SDS. Electronic copies of MSDS/SDS can be found using the "MSDS" link on the Oasis homepage and on the EH&S webpage on Oasis.

Material	Hazards	Exposure Limit [1]	Signs and Symptoms of Exposure
Acetone	Flammable	1000ppm TWA	Inhalation of vapors irritates the respiratory tract. May cause coughing, dizziness, dullness, and headache.
Methylene Chloride	Carcinogen Irritant	25ppm TWA 125ppm STEL	Causes irritation to respiratory tract. Has a strong narcotic effect with symptoms of mental confusion, light-headedness, fatigue, nausea, vomiting and headache. Causes irritation, redness and pain to the skin and eyes. Prolonged contact can cause burns. Liquid degreases the skin. May be absorbed through skin.
[1]Exposure limit refers to the OSHA regulatory exposure limit.			

6.0 EQUIPMENT AND SUPPLIES

6.1 Equipment and Instrumentation

Gas Chromatograph - Agilent 6890, 7890, or equivalent with compatible autosampler, split/splitless injector, and direct capillary interface.

Mass Spectrometer- Agilent 5973, 5975, or equivalent

Top-loading Balance – Verify in accordance with SOP SA-AN-100: *Laboratory Support Equipment (Verification and Use)*

6.2 Analytical Data System / Software / Hardware

Chemstation software is used on a Windows-based PC to schedule and acquire data. CHROM software is used on a Windows-based PC to store, reduce/evaluate, and output the data to the laboratory's LIMS system (i.e., TALS). CHROM software has the capability of processing stored GC/MS data by recognizing a GC peak within any given retention time window, comparing the mass spectrum from the GC peak with spectral data in a user-created data base, and generating a list of tentatively identified compounds with their retention times and scan numbers. The software also allows integration of the ion abundance of any specific ion between specified time or scan number limits, calculation of response factor , construction of a linear regression calibration curve, calculation of response factor statistics (mean and standard deviation), and calculation of concentrations of analytes using either the calibration curve or the response factors.

6.3 Volumetric Labware

Volumetric Containers must be verified in accordance with SOP SA-AN-100: *Laboratory Support Equipment (Verification and Use)*. Refer to Attachment 12 for Glassware Cleaning Procedures.

Volumetric Labware	Volume	Type (Quantitative / Qualitative)	Use	Verification Frequency	Laboratory Verification Criteria
Gas-Tight Microliter Syringes	Various	QUANTITATIVE	Dilution, Reagent, and Standard Preparation	None (if received with COA)	None (if received with COA)
Volumetric Flasks (Class A)	Various	QUANTITATIVE	Dilution, Reagent, and Standard Preparation	None (Class A)	None (Class A)
Disposable Transfer Pipettes	Various	Qualitative	Sample Transfer	None	None
Autosampler Vials (w/Crimp Cap)	1mL	Qualitative	Instrument Analysis	None	None
Standard Storage Vials (Amber, Glass, w/ Screw Cap)	2mL	Qualitative	Standard Storage	None	None

6.4 Lab Supplies

Column – Rxi-5 SilMS 30ms; 0.25mmx0.25um or equivalent

Injector Liner – 4mm ID quartz or 4mm glass, deactivated

7.0 REAGENTS AND STANDARDS

7.1 Expiration Dates

Expiration dates (time from initial use or receipt to final use) for standard and reagent materials must be set according to the guidance in this SOP. Note: These are maximum expiration dates and are not to be considered an absolute guarantee of standard or reagent quality. Sound judgment must be used when deciding whether to use a standard or reagent. If there is doubt about the quality of a standard or reagent material, a new material must be obtained or the standard or reagent material verified. Data quality must not be compromised to extend a standard's life.

The expiration date of any standard must not exceed the expiration date of the standard that was used to prepare it.

7.2 Reagents

Reagents must be prepared and documented in accordance with SOP SA-AN-041*: Reagent and Standard Materials Traceability.*

Acetone and methylene chloride must be verified prior to use in accordance with the TestAmerica Solvent Lot Testing Program.

Methylene chloride – pesticide grade, for preparation of analytical standards
Storage: Room temperature
Expiration: Unopened and Opened - Manufacturer's expiration date

Acetone – pesticide grade, for cleaning glassware.
Storage: Room temperature
Expiration: Unopened and Opened - Manufacturer's expiration date

7.3 Standards

Standards must be prepared and documented in accordance with SOP SA-AN-041*: Reagent and Standard Materials Traceability.* Certificates of analysis or purity must be received with all purchased standards, and attached to the standard in TALS.

Refer to Attachment 8 for the laboratory's current standards and recipes.

8.0 Sample Collection, Preservation, Shipment, and Storage

8.1 Aqueous Samples

Aqueous samples are routinely collected in 1L or 500mL amber glass containers with PTFE-lined lids.

Samples must be iced at the time of collection and maintained at 0-6°C (less than 6°C but not frozen) until the time of preparation. Samples must be prepared within 7 days of collection. Extracts must be stored in the refrigerator at 0-6°C until the time of analysis and analyzed within 40 days of extraction. Sample extracts must be allowed to come to room temperature prior to analysis.

Note: In the presence of samples containing residual chlorine, phenol-d5 has been known to react to form chlorinated phenolic compounds that are not detected as the original spiked surrogate. Therefore, aqueous samples must be evaluated for the presence of residual chlorine prior to extraction. The presence or absence of residual chlorine is documented on the preparation sheet completed at extraction.

8.2 Soil Samples

Soil samples are routinely collected in 8oz soil containers with PTFE-lined lids.

Samples must be iced at the time of collection and maintained at 0-6°C (less than 6°C but not frozen) until the time of preparation. Samples must be prepared within 14 days of collection. Extracts must be stored in the refrigerator at 0-6°C until the time of analysis and analyzed within 40 days of extraction. Sample extracts must be allowed to come to room temperature prior to analysis.

9.0 Quality Control

SOP SA-QA-017: *Evaluation of Batch QC Data* and the SOP Summary in Attachment 3 provide requirements for evaluating QC data.

9.1 Batch QC

An extraction batch consists of up to 20 environmental samples and the associated QC items.

For EPA 625, the laboratory's default QC items performed for each extraction batch are: a method blank, a laboratory control sample (LCS), a matrix spike (MS) per 10% of samples extracted, and a matrix spike duplicate (MSD).

For EPA 8270D, EPA 8270D_LL, EPA 8270D_LL_PAH, EPA 8270E, EPA 8270E_LL, EPA 8270E_LL_PAH, and EPA 625.1 the laboratory's default QC items performed for each extraction batch are: a method blank, a laboratory control sample (LCS), a matrix spike (MS), and a matrix spike duplicate (MSD).

Note: Unless the client has specified which sample to use as the matrix spike, the matrix spike must be selected at random from the samples in the batch. Field QC (e.g., equipment blanks, trip blanks, and field blanks) must not be used for MS/MSD unless specifically requested to do so by the client or unless there is insufficient sample available for performing MS/MSD using an actual field sample.

Note: LCS and LCSD are performed if insufficient sample is provided for MS/MSD. If an LCS and LCSD are performed, both QC items must be evaluated and reported. Acceptable recoveries (as well as %RPD) for both LCS and LCSD are required.

Refer to applicable preparation SOP listed in Section 10.1 for further information on batch QC.

Batch QC must meet the criteria given in Attachment 3 of this SOP.

9.1.1 Poor Performers

As indicated in SW-846 and/or via assessment of laboratory control sample (LCS) recoveries and control charts, the compounds listed in Attachment 10 are poor performers and/or behave erratically. These compounds will not be included in the LCS/LCSD/MS/MSD marginal exceedance count for EPA 8270D or EPA 8270E, as outlined in SOP SA-QA-017: *Evaluation of Batch QC Data,* provided they are qualitatively detected.

Note: An NCM must be initiated to denote this situation.

Note: The use of Sporadic Marginal Exceedances, as defined in SOP SA-QA-017, is not permitted for samples originating from South Carolina.

9.2 Instrument QC

9.2.1 DFTPP Tune

A solution containing DFTPP (decafluorotriphenyl phosphine) must be analyzed at the beginning of each analytical clock as a check on the tune of the instrument. The analytical clock begins with the injection of the DFTPP standard. The analytical clock is defined as 12 hours for EPA 8270D, EPA 8270D_LL, EPA 8270D_LL_PAH, EPA 8270E, EPA 8270E_LL, EPA 8270E_LL_PAH and EPA 625.1. The analytical clock is defined as 24 hours for EPA 625.

Meeting the DFTPP tuning criteria demonstrates that the instrument is measuring the proper masses in the proper ratios. The DFTPP analysis takes place under the same instrument conditions as the calibration standards and samples except that a different temperature program can be used to allow for the timely elution of DFTPP. All other instrument conditions including the mass range, scan rate, and multiplier voltage must be identical.

A 1uL aliquot of the 50ng/uL DFTPP/Column Evaluation solution is utilized for EPA 625, EPA 625.1, EPA 8270E and EPA 8270D. A 1uL aliquot of the 5ng/uL DFTPP/Column Evaluation solution is utilized for EPA 8270D_LL, EPA 8270D_LL_PAH, EPA 8270E_LL, and EPA 8270E_LL_PAH.

The DFTPP solution must also contain benzidine, pentachlorophenol, and p,p'-DDT at the following concentrations:

EPA 625, EPA 625.1, EPA 8270E and EPA 8270D – 50ug/mL
EPA 8270E_LL, EPA 8270E_LL_PAH, EPA 8270D_LL and 8270D_LL_PAH – 5ug/mL

Refer to Section 10 for the DFTPP evaluation criteria.

9.2.2 Initial Calibration (ICAL)

The instrument must be calibrated in accordance with SOP SA-QA-016: *Evaluation of Calibration Curves.* This SOP provides requirements for establishing the calibration curve and gives the applicable formulas.

Instrument calibration is performed by analyzing a series of known standards. The calibration curve must consist of a minimum of 3 standards for EPA 625 and a minimum of 5 standards for EPA 625.1, EPA 8270D, EPA 8270E, EPA 8270D_LL, EPA 8270E_LL, EPA 8270E_LL_PAH and EPA 8270D_LL_PAH. The lowest level calibration standard must be at or below the reporting limit, and the remaining standards will define the working range of the analytical system.

Note: A minimum of 6 points is required for a quadratic curve. Higher order curves are not permitted. Some programs and agencies (e.g., SC DHEC) do not allow the use of quadratic curves. Refer to the Project Requirement Summary to determine if this curve type is prohibited.

The initial calibration standard concentrations currently in use in the laboratory are as follows:

EPA 8270D / EPA 8270E/ EPA 625 / EPA 625.1

Standard Level	Concentration (ug/mL)
1	5
2	10
3	20
4	40
6	80
7	100
8	200

EPA 8270D_LL/ EPA8270D_LL_PAH/ EPA 8270E_LL/EPA_8270E_LL_PAH

Standard Level	Concentration (ug/mL)
1	0.10
2	0.20
3	1.0
4	2.0
5	5.0
6	10
7	15
8	20

Refer to Attachment 8 for the standard preparation instructions. Other standard concentrations may be used provided they support the reporting limit and are fully documented in accordance with SOP SA-AN-041.

ICAL evaluation steps and acceptance criteria vary between the methods. Refer to Attachment 1 for a summary of the method-specific requirements.

9.2.2.1 ICAL Readback / % Error Criteria

Each calibration curve must be evaluated to ensure that the quantitation over the entire working range is accurate. This is accomplished via CHROM by reviewing the % Error for each analyte in each calibration level.

The re-calculated concentrations of each analyte must be within 50%D for the low point and within 30%D for the remaining points.

If these criteria are not met, an alternate curve type that meets criteria should be utilized to ensure a better fit, or another initial calibration curve should be performed.

9.2.3 Instrument Blanks

The instrument must be shown to be free from contamination by the analysis of instrument blanks or method blanks. Instrument blanks are analyzed at the beginning of each clock before analysis of any samples. The instrument blanks should be analyzed following the ICV or CCV. Analysis of a batch method blank may substitute for the analysis of an instrument blank provided analysis is acceptable.

Instrument blanks must be <1/2RL to be acceptable.

9.2.4 Second Source Initial Calibration Verification (ICV)

The calibration curve must be verified initially – prior to any sample analyses – in accordance with SOP SA-QA-016 with a standard obtained from a second source.

Refer to Attachment 8 for the standard preparation instructions. Another standard concentration may be used provided it is mid-level and fully documented in accordance with SOP SA-AN-041.

ICV evaluation steps and acceptance criteria vary between the methods. Refer to Attachment 1 for a summary of the method-specific requirements.

9.2.5 Continuing Calibration Verification (CCV)

The initial calibration curve must be verified at the beginning of each clock with a mid-level standard. The analytical clock is defined as 12 hours for EPA 625.1, EPA 8270D, EPA 8270E, EPA 8270E_LL, EPA 8270E_LL_PAH, EPA 8270D_LL, and EPA 8270D_LL_PAH and is defined as 24 hours for EPA 625. The initiation of the clock begins with the injection of the DFTPP.

Refer to Attachment 8 for the standard preparation instructions. Another standard concentration may be used provided it is mid-level and fully documented in accordance with SOP SA-AN-041.

DODQSMV5.0 requires a closing calibration verification (CCVC) to be analyzed at the end of each 12hour clock. Analysis should occur within the 12 hour clock window.

RLCCV (reporting level calibration verification) is incorporated into the procedure for EPA 8270D, EPA 8270E, EPA 8270E_LL, EPA 8270E_LL_PAH, EPA 8270D_LL, and EPA 8270D_LL_PAH. This CCV is used to demonstrate sensitivity at the reporting limit can be obtained. Refer to the applicable CCV section of Attachment 1 for more information on how the RLCCV is utilized.

Note: CCV evaluation steps and acceptance criteria vary between the methods. Refer to Attachment 1 for a summary of the method-specific requirements.

9.2.6 Isomer Resolution Criteria

The GC resolution of structural isomers must be monitored in the ICAL and CCV. In the ICAL, use the calibration level that will be utilized as the CCV.

Structural isomers that produce very similar mass spectra should be identified as individual isomers if they have sufficiently different GC retention times. Sufficient GC resolution is achieved if the height of the valley between two isomer peaks is less than 50% of the average of the two peak heights. Otherwise, structural isomers are identified as isomeric pairs. These criteria are important for the identification of benzo(b)fluoranthene and benzo(k)fluoranthene.

Note: The resolution should be monitored utilizing the signal resolution of the extracted ion profiles for the quantitation ion for each analyte. Sufficient GC resolution is needed to identify the individual isomeric peaks by retention time. If the software is able to integrate and define separate peaks for the isomeric pairs, then sufficient resolution has been achieved. If there is sufficient evidence to support the identification of the individual component, then the component is identified, quantified, and reported.

9.2.7 Internal Standard (ISTD)

This procedure is an internal standard (ISTD) procedure. The internal standards used are 1,4-dichlorobenzene-d4, naphthalene-d8, acenaphthene-d10, phenanthrene-d10, chrysene-d12, and perylene-d12.

Prior to analysis, the internal standards must be added to all standards, samples, and QC items. The concentration of the internal standards must be the same in all calibration samples, field samples, and QC samples. A concentration of 40ug/mL is used for EPA 8270D, EPA 8270E, EPA 625 and EPA 625.1. A concentration of 2.0ug/mL is used for EPA 8270E_LL, EPA 8270E_LL_PAH, EPA 8270D_LL and EPA 8270D_LL_PAH.

9.2.7.1 ICV/CCV ISTD Criteria

The response of the ISTD in the ICV/CCVIS must be within a factor of 2 of the response of the ISTD in the CCV-level standard in the initial calibration sequence, identified as the ICIS. Due to the number of analytes reported in this method, multiple CCVs can be analyzed. The primary CCV is defined as the CCVIS and is used to monitor ISTD response in samples. If the response is outside of the required range, the analysis of the CCVIS must be repeated and any samples associated with the CCVIS must also be re-analyzed. Repeated failure of the ISTD response may require instrument maintenance, re-preparation of standards, and/or re-calibration.

9.2.7.2 Sample and Batch QC ISTD Criteria

The response of the ISTD in samples and batch QC items must be within a factor of 2 of the response of the CCVIS in the associated analytical clock. If the response is outside of this range, corrective action must be taken. Corrective actions include re-analysis (for field samples) and/or re-extraction (for batch QC items).

9.2.8 Surrogates

This procedure uses surrogates to evaluate the extraction process.

The surrogates for EPA 625, EPA 625.1, EPA 8270D, EPA 8270E, EPA 8270E_LL and EPA 8270D_LL are phenol-d5(acid), 2-fluorophenol (acid), 2,4,6-tribromophenol (acid), nitrobenzene-d5 (base), 2-fluorobiphenyl (base), and p-terphenyl-d14 (base),

The surrogate for EPA 8270D_LL_PAH and EPA 8270E_LL_PAH is ortho-terphenyl (OTP).

Prior to preparation, the surrogate analytes are added to all samples and QC items. The concentration of the surrogate is the same in all field samples and QC samples. A concentration of 100ug/mL is used for EPA 625, EPA 625.1, EPA 8270E and EPA 8270D. A concentration of 10ug/mL is used for EPA 8270D_LL and EPA 8270E_LL. A concentration of 2.0ug/mL is used EPA 8270D_LL_PAH and EPA 8270E_LL_PAH.

The percent recovery of the surrogate in all field samples and QC samples must be within the control limits listed in the Method Limit Groups (MLGs) in TALS.

9.2.8.1 Surrogate Dilution Factor Threshold

Due to the level of dilution required for samples, surrogates may be diluted out. As such, recoveries will be reported as “0D” in dilutions at 1:10 or greater. Control limits will not apply to samples analyzed at dilutions of 1:10 or greater.

An NCM must be initiated to denote this situation.

9.2.8.2 One Acid / One Base Surrogate Exception

The laboratory allows one acid and one base surrogate compound to be outside acceptance limits, in field samples and MS/MSD, provided their recovery is greater than 10%. All surrogate compounds must pass in method blanks and LCS/LCSD.

An NCM must be initiated to denote this situation.

Note: DODQSM does not permit this allowance. All surrogates must recover within limits established in the DODQSM.

Note: SC DHEC does not permit this allowance. All surrogates must meet criteria for samples originating from South Carolina.

9.3 Corrective Action for Out-of-Control Data

When the quality control parameters do not meet the criteria set forth in this SOP, corrective action must be taken in accordance with SOP SA-QA-005: *Preventive and Corrective Action Procedures* and the QC Summary Table in Attachment 3. SOP SA-QA-005 provides contingencies for out-of-control data and gives guidance for exceptionally permitting departures from approved policies and procedures. Nonconformance Memos must be initiated to document all instances where QC criteria are not met and all departures from approved policies and procedures.

10.0 Procedure

10.1 Preparation

10.1.1 Aqueous Sample Preparation

For continuous liquid-liquid extraction, the sample is transferred to a continuous liquid-liquid extractor or separatory funnel, adjusted to a specific pH, as required by the analyte list, and extracted using methylene chloride. The extract is concentrated to a predetermined final volume (typically 1mL) using the Zymark nitrogen blow-down concentrator procedure.

Refer to SOP SA-EX-030: *Liquid Extraction Procedures: Continuous Liquid-Liquid and Separatory Funnel* for specifics on the aqueous and leachate sample preparation process.

10.1.2 Soil Sample Preparation

For microwave extraction, the sample is transferred to a Teflon extraction vessel. A 1:1 acetone/methylene chloride mixture is added to the sample in the vessel. The vessel is placed on the instrument and the sample is extracted at an elevated temperature and pressure. The vessel is cooled to room temperature; the extract is passed through sodium sulfate to remove the water from the sample; and the extract is collected in a concentration tube. The extract is concentrated to a predetermined final volume (typically 1mL) in methylene chloride using the Zymark nitrogen blow-down concentrator procedure.

Refer to SOP SA-EX-040: *Soil Extraction Procedures (Microwave and Sonication)* for specifics on the soil sample preparation process.

10.2 Analysis

10.2.1 Instrument Operating Conditions

Note: The instrument conditions listed in this SOP are provided for guidance purposes. The actual conditions used by the laboratory may be slightly different from those listed here and must be documented in the instrument maintenance log, data system, and/or run log.

Instrument maintenance must be performed in accordance with Attachment 4 of this SOP.

The goal is to have maximum separation between the target compounds in the shortest run time while maintaining sufficient sensitivity to detect the target compounds at the MDL.

Example GC Parameters
Injector: 250-280°C
Injector Mode:

- Split – EPA 8270D, EPA 8270E, EPA 625, and EPA 625.1
- Splitless – EPA 8270E_LL, EPA 8270E_LL_PAH, EPA 8270D_LL and EPA 8270D_LL_PAH

Column – Rx-5SilMS 30M x 0.25mm x 0.25um; or equivalent

Carrier Gas Flow: Helium at 0.5-1.0mL/min (column)
Mass spectrometer interface: 280-300°C
Mass spectrometer source temperature: 230°C
Mass spectrometer quad temp: 150C
Mass range: 35-500amu, with a minimum scan time of 1.0 scan per second

Temperature Program:

Initial Temp:	55°C
Initial Hold:	1.5 min
Program Rate:	30°C/min to 190°C, 32°C/min to 320°C
Final Temp:	320°C (hold until elution time of Benzo(ghi)perylene or last eluting analyte)
Injected Volume:	1-2uL per column
Inlet Purge Time:	0.8 minutes

The injection volume must be the same for all standards and sample extracts.

10.2.2 DFTPP Analysis and Evaluation

The DFTPP standard must be analyzed at the beginning of each clock. The analytical clock is defined as 12 hours for EPA 625.1, EPA 8270D, EPA 8270E, EPA 8270E_LL, EPA 8270E_LL_PAH, EPA 8270D_LL, and EPA 8270D_LL_PAH, and is defined as 24 hours for EPA 625. The initiation of the clock begins with the injection of the DFTPP.

10.2.2.1 DFTPP Spectrum Criteria

The spectrum of the DFTPP must meet the criteria for each method listed in Attachment 5. Background subtraction is required, and must be accomplished using a single scan no more than 20 scans prior to the elution of DFTPP. CHROM software is programmed to perform this subtraction during processing of the DFTPP. The time utilized for the background subtraction is displayed on the tune report generated in CHROM.

Background subtraction is designed to eliminate column bleed and instrument background ions.

Note: The DFTPP analysis should be evaluated as to the relative size of the DFTPP peak under the m/z 198 profile. A benchmark area window should be established for each instrument and data system. Area outside of this window suggests instrumental problems such as a bad injection, clogged autosampler syringe, leaking injector, reduced or elevated detector sensitivity, improper electron multiplier voltage selection, wrong tune method or tune file selected for this analysis, PFTBA valve left open, etc.

If the DFTPP fails to meet the criteria, the instrument may require tuning (manually or automatically with PFTBA). Depending on the nature of the results from the DFTPP analysis, other corrective measures may include remaking the DFTPP standard, cleaning the mass spectrometer source, etc.

10.2.2.2 Peak Tailing Factor Criteria

The analysis of benzidine and pentachlorophenol serves as a check on the system performance. The tailing factor for pentachlorophenol and benzidine is calculated as outlined in Attachment 6 and must meet the following criteria:

EPA 625:
pentachlorophenol <5
benzidine <3

EPA 625.1, EPA 8270D, EPA 8270E, EPA 8270E_LL, EPA 8270E_LL_PAH, EPA 8270D_LL, and EPA 8270D_LL_PAH:
pentachlorophenol <2
benzidine <2

If the criteria above are not met, perform injector port and column maintenance and reanalyze the DFTPP standard.

10.2.2.3 DDT Breakdown Criteria

Percent breakdown is calculated using the areas from the total ion chromatogram using following equation:

$$\%DDTBreakdown = \frac{(areaDDE + areaDDD)}{(areaDDT + areaDDE + areaDDD)} x\,100$$

The percent breakdown of p,p'-DDT must not exceed 20% for the methods cited above.

Note: There is no DDT percent breakdown requirement for EPA 625.

10.2.3 Initial and Continuing Calibration

Calibrate the instrument using the standards and criteria described given in Section 9.2.2. Once the calibration has been established and verified with an ICV in accordance with Section 9.2.4, sample analysis may proceed.

Verify the calibration curve with a continuing calibration verification using the standards and criteria described given in Section 9.2.5 and Attachment 1.

10.2.4 Sample Analysis

The sample extract must be injected using the same injection volume used for the calibration standards. Samples that are known to be relatively clean should be analyzed first. Samples suspected of containing high concentrations should be analyzed last. Instrument blanks may be analyzed after suspected high concentration samples to allow the detector response to stabilize.

The clock time starts with the injection of the DFTPP. The analysis standards, QC and samples may continue until the clock time expires. Sample analysis must initiate within the clock time defined by the applicable method. For EPA 8270D, EPA 8270E, EPA 8270E_LL, EPA 8270E_LL_PAH, EPA 8270D_LL, and EPA 8270D_LL_PAH, the clock time is 12 hours. For EPA 625, the clock time is 24 hours.

Example Analytical Sequence

Description	Comments
DFTPP	Clock time begins.
Initial Calibrations	
ICVs	
Instrument Blank	Instrument Blank or Method Blank may be analyzed
Samples & Batch QC Items	EPA 625: Clock time ends at 24 hours from DFTPP EPA 625.1 EPA 8270D, EPA 8270E, EPA 8270E_LL, EPA8270E_LL_PAH, EPA 8270D_LL, and EPA 8270D_LL_PAH: Clock time ends at 12 hours from DFTPP
CCVC	Capping CCV for DODQSM
DFTPP	Clock time begins.
CCVs	
RL CCV	EPA 8270D, EPA 8270E, EPA 8270E_LL, EPA 8270E_LL_PAH, EPA 8270D_LL, and EPA 8270D_LL_PAH only
Instrument Blank	Instrument Blank or Method Blank may be analyzed
Samples & Batch QC Items	EPA 625: Clock time ends at 24 hours from DFTPP EPA 625.1 EPA 8270D, EPA 8270E, EPA 8270E_LL, EPA 8270E_LL_PAH, EPA 8270D_LL, and EPA 8270D_LL_PAH: Clock time ends at 12 hours from DFTPP
CCVC	Capping CCV for DODQSM

11.0 Calculations / Data Reduction

11.1 Data Reduction

Data evaluation must be performed in accordance with SA-QA-008: *Evaluation of Chromatographic Data.* This SOP includes specific information regarding the evaluation of chromatographic data, including the requirements for performing manual integrations and the evaluation of retention times.

Data review and reporting must be performed in accordance with SA-QA-002: *Data Generation and Review.*

11.1.1 Target Analyte Identification

A target compound is identified by the visual comparison of the sample mass spectrum with the mass spectrum of the target compound from the standard spectral library. CHROM utilizes the NIST/NBS library for reference spectra.

The following criteria must be met in order to positively identify a compound:

1) Elution of the sample component within +/-0.06 RRT (relative retention time) units of the daily standard containing that compound. RRT is calculated as follows:

$$RRT = \frac{retention time of the target compound}{retention time of the associated internal standard}$$

2) Correspondence of the target compound spectrum and the standard component mass spectrum.

   All ions present in the standard component mass spectrum at a relative intensity greater than 10% (most abundant ion = 100%) should be present in the sample component mass spectrum. Other ions may be present in the sample component. Coelution of a non-target compound with a target compound will make the identification of the target compound more difficult. Ions due to the non-target compound should be subtracted from the sample component spectrum as part of the background to account for the discrepancy between the sample spectrum and the standard spectrum.

   The relative intensities of the ions present in the sample component spectrum should agree within ±30% of the relative intensities of the ions in the standard reference spectrum. For example, an ion with an abundance of 50% in the reference spectrum should have a corresponding abundance between 20% and 80% in the sample component spectrum. The intensities of the ions in the standard reference spectrum are updated utilizing the continuing calibration standard. If the relative intensities are outside 30% in sample, the data will be flagged with an R by CHROM.

3) The intensities of the characteristic ions of a compound must maximize in the same scan or within one scan of each other. Selection of a peak by a data system target compound search routine where the search is based on the presence of a target chromatographic peak containing ions specific for the target compound at a compound-specific retention time will be accepted as meeting this criterion.

4) Structural isomers that produce very similar mass spectra should be identified as individual isomers if they have sufficiently different GC retention times. Sufficient GC resolution is achieved if the height of the valley between two isomer peaks is less than 50% of the average of the two peak heights. Otherwise, structural isomers are identified as isomeric pairs.

   If the above criteria are not met, the analyst should seek help from a senior analyst or Supervisor. If there is sufficient evidence to support the identification of the component, then the component is identified, quantified, and reported. If there is not sufficient evidence to support identification of the component, then an NCM must be generated to note that the isomers should be identified as the isomeric pair.

5) Ions present in the sample spectrum, but not in the reference spectrum, should be reviewed for possible subtraction from the sample spectrum because of over-lapping or co-eluting peaks.

   Ions present in the reference spectrum, but not in the sample spectrum, should be reviewed for possible subtraction from the sample spectrum because of co-eluting peaks.

11.1.2 Evaluation of Tentatively Identified Compounds (TICs)

For samples containing components not associated with the calibration standards, a library search on a reference library, such as the NIST/NBS, may be conducted in order to identify the non-target compounds. Note: TICs can not be provided from a SIM analysis.

Refer to SOP SA-QA-008: *Evaluation of Chromatographic Data* for the laboratory's TIC procedures.

11.1.3 Dilutions

It is acceptable to report results from a single analysis as long as the largest target analyte (when multiple analytes are present) is in the upper half of the calibration range. If the laboratory does not have historical data and acquires data for a sample with passing internal standards and surrogates but one or more analytes exceed the calibration range, the laboratory will report the over-range (i.e., E-values) with an "Acceptable" TALS status and re-analyze the extract only for the compounds that exceeded the calibration range. The secondary analysis will be reported as a "DL" in TALS, and surrogates will not be reported from this analysis. This is known as the "Condensed Dilution Format". When reporting results from dilutions, appropriate data flags must be used or qualification in a case narrative provided to the client.

For clients who require the laboratory to provide lower detection limits, a general guide is to report the dilution detailed above and one additional run at a dilution factor 1/10 of the dilution with the highest target in the upper half of the calibration curve. For example, if samples analyzed at a 1/50 dilution resulted in a target in the upper half of the calibration curve, the sample is re-analyzed at a dilution factor of 1/5 to provide lower reporting limits for the remaining compounds.

Note: If samples are analyzed at a dilution factor of 1:10 or greater, report the surrogate result as 0%D, to denote a dilution above the Dilution Threshold. Control limits will not apply to samples analyzed at dilutions of 1:10 or greater.
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Dilute samples according to the following table:

Dilution Factor	Extract Aliquot (uL)	ISTD Aliquot (uL)		Final Volume* (mL)
		Routine	Low Level	
2	500	10	5	1.0
5	200	16	8	1.0
10	100	18	9	1.0
20	50	19	9.5	1.0
50	20	20	10	1.0
100	10	20	10	1.0

*Final solvent = methylene chloride

11.1.4 Historical Data

Many of the laboratory's clients submit samples for repeat monitoring purposes. Prior to analysis, verify TALS Notes and/or use the TALS Historical Data Tracker feature to determine if historical data is available for review.

11.1.5 Chemical Relationships

The analyst must be aware of the following chemical relationships:

- Several analytes reported as "total" are summed from the individual components. For example, Total Cresols are summed as follows:

  Total cresols = m/p-cresols + o-cresol

  Note that o-cresol and m/p-cresol are also reported as individual analytes.

- Several analytes are identified as summary analytes. These analytes break into multiple peaks upon analysis. Each component's response must be utilized to calculate a response for the summary analyte. The following are examples of summary analytes and their associated components.

  Aramite, Total = aramite peak 1 + aramite peak 2
  Diallate, Total = diallate peak 1 + diallate peak 2
  Isosafrole, Total = Isosafrole peak 1 and Isosafrole peak 2

- Chromatographically unresolved isomers are reported together since isomers cannot be resolved by differences in mass. For example, m-cresol and p-cresol are reported as m&p-cresols.

- It is important that closely eluting isomers be resolved chromatographically so that the analyte can be properly identified. The most critical separation is benzo(b)fluoranthene and benzo(k)fluoranthene.

- Acid/Base Compounds

  Basic Compounds: In aqueous samples, several target compounds are soluble in methylene chloride only at basic pH (>12). These compounds form methylene chloride insoluble salts at acidic pH and remain in the aqueous phase. When the pH is adjusted to basic, the ionic compound reverts to its original form and can be extracted out of aqueous solution. Examples of these compounds include pyridine (TCLP, AP9), benzidine (625PP), and a,a-dimethylphenethylamine (AP9). If acid-only extraction is performed, basic compounds will not be extracted and detected.

  Neutral Compounds: In aqueous samples, neutral compounds can extract into methylene chloride at either acidic pH (<2) or at basic pH (>12). That is, these compounds do not convert to salts or ionic forms at either acidic pH. If the acid pH is performed first, the compounds partition into the methylene chloride and would be detected in the acid fraction; if the basic pH extraction is performed first, these compounds partition into the methylene chloride and would be detected in the base fraction. Exceptions include the phthalate esters (e.g., dimethyl phthalate, diethyl phthalate) and other esters which may be irreversibly converted (hydrolyzed) to salts if subjected to the basic pH extraction first.

  Acid Compounds: In aqueous samples, the acidic compounds can be extracted into methylene chloride at acid pH (pH<2). The acidic compounds are the phenols and benzoic acid. At basic pH, the phenols forms water soluble salts which are not soluble in methylene chloride. When the pH is adjusted to <2 the salt is converted back to the phenol or acid form. Some phenols (2,4-dimethyl phenol and the cresols) do not completely ionize at basic pH and may be present in both the acid and base fractions of a dual pH extraction.

- Single pH (<2) Extractions

  Single pH extractions are performed at pH<2 and include the compounds that are soluble in methylene chloride at acidic pH. The primary application of the single pH extraction is for the routine compound list, which includes most of the compounds that are monitored for and are detected in field samples. The advantages of the single pH extraction are a shorter extraction time and efficient extraction of all phenolic compounds and compounds subject to hydrolysis under basic pH conditions. The drawback is that basic compounds are not extracted under single pH conditions.

- Dual pH Extractions

  Dual pH extractions may be performed with the basic pH extraction first followed by the acidic pH or the acidic pH extraction may be performed first followed by the basic pH extraction. The table below summarizes some of the positive and negative aspects of dual pH extractions.

Extraction	Pros	Cons
Acidic pH first followed by basic pH Examples: TCLP, Appendix IX	No hydrolysis of phthalate esters	Acid and base/neutral surrogates are both extracted into solvent at acidic pH. There is no surrogate to determine whether the sample pH was adjusted to basic pH, to determine the extraction efficiency of the basic pH extraction
Basic pH first followed by acidic pH Example: EPA 625 PP	Acid compounds (phenols) and base/neutral compounds can be separated into two extracts. The partitioning of the target analytes into separate extracts can sometimes help to minimize the effect of the sample matrix on the target compounds.	Some compounds may be partitioned into both the acid and base/neutral extracts. Examples include 2,4-dimethylphenol and the cresol compounds.
		Phthalate esters are converted (hydrolyzed) to salts under basic conditions which causes irreversible loss of the compounds. Dimethyl phthalate and diethyl phthalate are the most effected compounds.
		Some phenolic compounds may have reduced recoveries

- Compounds with similar structures and properties are often found together in a sample or in the samples from the same project or site. That is, when one of that type of compound is detected, the analyst should be looking for other compounds of that type. For example, when one PAH compound (e.g., naphthalene, phenanthrene, benzo(a)pyrene, etc.) is detected, the analyst should expect other PAH compounds to be present. When chlorinated benzenes (e.g., 1,2-DCB, 1,3-DCB, 1,4-DCB) are present, the analyst should be aware that other chlorinated benzenes may be present. When pentachlorophenol is detected, the analyst should also look for tetrachlorophenols and trichlorophenols.

11.1.6 Confirmation of GC Analyses

If required, GC/MS can be used to confirm some GC-only analytes. Based on the response of the standard, concentrate the SVOC GC extract to an appropriate final volume.

Note for SVOC: The pesticide extracts cannot be used to confirm the presence of herbicides since the extract has not been properly prepared.

- Add an appropriate volume of internal standard to the extract or sample to give the same concentration as in the calibration standard. Analyze the extract under the same conditions as the standard.

- Compare the retention time of the sample to the retention time of the standard.

If a peak is detected at the retention time of the target compound containing the selected masses in the same ratio as the standard, the peak is confirmed as the target

compound and the concentration is calculated. The relative intensities of the ions in the sample should agree within ±20% of the intensities of the ions in the standard.

If a peak is not present at the appropriate retention time or if the ratios of the ions are not the same as the standard, the analyte is not confirmed.

11.2 Calculations

11.2.1 The calculations associated with batch QC determinations are given in SOP SA-QA-017. Applicable calculations include accuracy (% recovery) and precision (%RPD).

11.2.2 The calculations associated with initial and continuing calibrations and are given in SOP SA-QA-016. Applicable calculations include determination for: calibration factor, standard deviation, relative standard deviation, and relative response factor.

11.2.3 The calculation to determine final concentration is given as follows:

$$FinalConcentration = CONC_{Sample} \otimes \frac{F}{I \times dw} \otimes D$$

Where:

$CONC_{Sample}$= Concentration of the sample
F = Final volume/weight
I = Initial volume/weight
D = Dilution factor
dw = % Solids decimal equivalent (applicable to soil samples, only)

- **Note: This calculation assumes all applicable unit correction factors are applied.**
- The calculation used for water samples is the same as above with the exception that there is no % Solids factor applied.
- All dry weight corrections are performed automatically in TALS.

12.0 Method Performance

12.1 Reporting Limit Verification (RLV)

At a minimum, RLVs must be performed initially upon method set-up in accordance with SOP SA-QA-007: *Determination and Verification of Detection and Reporting Limits*.

For analytes and methods certified by DOD ELAP, RLVs must also be performed quarterly thereafter. For all other analytes and methods, RLVs must also be performed annually thereafter. Exceptions may be made for project-specific non-routine analytes.

12.2 Method Detection Limit (MDL) Study

The MDL is the lowest concentration that can be detected for a given analytical method and sample matrix with 99% confidence that the analyte is present. MDLs reflect a calculated (statistical) value determined under ideal laboratory conditions in a clean matrix and may not be achievable in all environmental matrices. The current MDLs associated with this procedure are given in the Method Limit Group (MLG) in TALS.

At a minimum, MDL Studies must be performed initially upon method set-up in accordance with SOP SA-QA-007: *Determination and Verification of Detection and Reporting Limits*.

Note: MDL Studies are not required for non-routine analytes provided results are not reported below the RL (i.e., MDL equals RL in TALS).

12.3 Method Detection Limit Verification (MDLV)

At a minimum, MDLVs must be performed initially upon method set-up in accordance with SOP SA-QA-007: *Determination and Verification of Detection and Reporting Limits*.

For analytes and methods certified by DOD ELAP, MDLVs must also be performed quarterly thereafter

12.4 QC Limit Generation, Control Charting, and Trend Analysis

12.4.1 EPA 625 and EPA 625.1

The control limits for the batch QC items (LCS, MS/MSD, SD) for this procedure are specified in the reference method and cannot be broadened; therefore, the laboratory defaults to the method-defined limits and does not utilize in-house or laboratory-derived limits for the evaluation of batch QC items.

Although the laboratory must default to the method-defined QC limits, control charting is a useful tool and is performed to assess analyte recoveries over time to evaluate trends. Control charting must be performed periodically (at a minimum annually) in accordance with SOP SA-QA-017: *Evaluation of Batch QC Data.*

12.4.2 EPA 8270D, EPA 8270E, EPA 8270E_LL, EPA 8270E_LL_PAH, EPA 8270D_LL, and EPA 8270D_LL_PAH

The control limits for the batch QC items (LCS, MS/MSD, SD) for this procedure are not specified by the reference method; therefore, the laboratory defaults to in-house and/or laboratory-derived limits for the evaluation of batch QC items.

Control charting is a useful tool and is performed to assess analyte recoveries over time to evaluate trends. Control charting must be performed periodically (at a minimum annually) in accordance with SOP SA-QA-017: *Evaluation of Batch QC Data.*

12.5 Demonstrations of Capability

Initial and continuing demonstration of capability must be performed in accordance with SOP SA-QA-006*: Training Procedures.*

Prior to performing this procedure unsupervised, each new analyst who performs this analysis must demonstrate proficiency per method/analyte combination by successful completion of an initial demonstration of capability. The IDOC is performed by the analysis of 4 consecutive LCSs that meet the SOP criteria for accuracy and precision. The IDOC must be documented and routed to the QA Department for filing.

Annual continuing demonstrations of capability (CDOCs) are also required per analyst per method/analyte combination. The CDOC requirement may be met by the consecutive analysis of four LCS all in the same batch, by the analysis of four LCS analyzed in four consecutive batches (in different batches on different days), via acceptable results on a PT study, or analysis of client samples with statistically indistinguishable results when compared to another certified analyst. The CDOC must be documented and routed to the QA Department for filing.

Note: EPA 625.1 and EPA 625 has method specific DOC limits that must be met.
The DODQSM limits must be utilized if evaluating DOC for EPA 8270D or EPA 8270E

12.6 Training Requirements

All training must be performed and documented in accordance with SOP SA-QA-006: *Training Procedures*.

Note: The SOPs listed in the Reference/Cross-Reference Section are applicable to this procedure. All employees performing this procedure must also be trained on these SOPs, and/or have a general understanding of these procedures, as applicable.

13.0 Pollution Control

It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (e.g., examining recycling options, ordering chemicals based on quantity needed, preparing reagents based on anticipated usage and reagent stability, etc.). Employees must abide by the policies in Section 13 of the Environmental Health and Safety Manual and the Savannah Addendum to the EHSM.

This procedure has been evaluated for opportunities to minimize the waste generated. Where reasonably feasible, pollution control procedures have been incorporated.

14.0 Waste Management

Waste management practices must be conducted consistent with all applicable federal, state, and local rules and regulations. All waste (i.e., excess reagents, samples, and method process wastes) must be disposed of in accordance with Section 9 of the TestAmerica Savannah Addendum to the EHSM. Waste description rules and land disposal restrictions must be followed.

14.1 Waste Streams Produced by the Method

The following waste streams are produced when this method is carried out:

- Excess samples, reagents, and standards must be disposed in accordance with the TestAmerica Savannah Addendum to the EHSM.
- Flammable waste (acetone from extracts, rinsings, and standards) – Transfer to a satellite container designated for flammable waste and transfer to waste disposal department when the container is full with 10% headspace.
- Methylene chloride extracts – Dispose in drums using Hexane Vial waste stream which has been coded for Methylene Chloride.
- Methylene chloride used to rinse glassware, etc. – Transfer to chlorinated waste container.
- Excess aqueous samples – Dispose according to characterization from sample disposal program. If non-hazardous, dispose down drain/sewer. If hazardous, transfer to hazardous waste department for drumming. PCB Hazardous require disposal via drumming. Do dispose of any samples containing oil or strong smells via the sewer system. Route these to the waste coordinator.
- Excess soil and solid samples – Dispose according to characterization from disposal program. Non-RCRA Hazardous and RCRA Hazardous may be disposed via yardbox. PCB and Foreign Soil Hazardous are routed to the waste coordinator.
- Excess oil samples – Transfer to waste department for storage/disposal

14.2 Waste Disposal

Material	Treatment	Destination
Samples (Non-Hazardous Water)	Neutralization if applicable	Sink
Samples (Hazardous Water)	Neutralization if applicable	RCRA Hazardous Water
Samples (Unconfirmed Soils)	None	Soil Yardbox
Samples (Hazardous Soils)	None	RCRA Soils Yardbox
Autosampler Vials	None	Hexane Vials
Excess Standards (Non-PCB)	None	Hexane Vials
Excess Standards (PCB)	None	PCB Waste

15.0 References / Cross-References

- SOP SA-AN-100: *Laboratory Support Equipment (Verification and Use)*
- SOP SA-AN-041: *Reagent and Standard Materials Traceability*
- SOP SA-EX-015: *Toxicity Characteristic Leaching Procedure (TCLP) and Synthetic Precipitation Leaching Procedure (SPLP)*
- SOP SA-EX-030: *Liquid Extraction Procedures: Continuous Liquid-Liquid and Separatory Funnel*
- SOP SA-EX-040*:Soil Extraction Procedures (Microwave and Sonication)*
- SOP SA-EX-042: *Waste Dilution Extraction*
- SOP SA-QA-002: *Data Generation and Review*
- SOP SA-QA-005: *Preventive and Corrective Action Procedures*
- SOP SA-QA-006*: Training Procedures*
- SOP SA-QA-007: *Determination and Verification of Detection and Reporting Limits*
- SOP SA-QA-008: *Evaluation of Chromatographic Data*
- SOP SA-QA-015: *Homogenization, Compositing, and Segregation of Samples*
- SOP SA-QA-016: *Evaluation of Calibration Curves*
- SOP SA-QA-017: *Evaluation of Batch QC Data*
- TestAmerica Savannah Quality Assurance Manual
- TestAmerica Environmental Health and Safety Manual
- TestAmerica Savannah Addendum to the Environmental Health and Safety Manual
- Method 8000B: *Test Methods for Evaluating Solid Wastes, Third Edition, SW-846;* U.S. EPA Office of Solid Waste and Emergency Response: Washington, DC.
- Method 8000C: *Test Methods for Evaluating Solid Wastes, Revision 3;* U.S. EPA Office of Solid Waste and Emergency Response: Washington, DC.
- Method 8270D: *Test Methods for Evaluating Solid Wastes, Third Edition, SW-846;* U.S. EPA Office of Solid Waste and Emergency Response: Washington, DC.
- Method 8270E: *Test Methods for Evaluating Solid Wastes, Third Edition, SW-846;* U.S. EPA Office of Solid Waste and Emergency Response: Washington, DC.
- EPA Method 625*: Base/Neutrals and Acids.* 40 CFR Part 136, Appendix A, July 1, 1995.
- EPA Method 625.1*: Base/Neutrals and Acids by GC/MS.* 40 CFR Part 136, Appendix A, December 2016.

16.0 Method Modifications

16.1 Incorporation of Other Matrices

This procedure may be modified to analyze other matrices (e.g., wipe, waste, tissue, and TCLP/SPLP leachate samples) based on the needs of the client. This will need to be arranged by the Project Manager at the initiation of the project.

Wipe, waste, and tissue matrices are non-routine, and the laboratory is not currently NELAC certified for these matrices. The laboratory uses its routine soil RLs (converted for initial and final volumes, etc.) and default QC limits to evaluate wipe, waste, filter, and tissue samples. Soil DOCs can be used to satisfy analyst demonstrations of capability for these types of non-routine matrices. The laboratory uses its routine aqueous RLs (converted for initial and final volumes, etc.) and default QC limits to evaluate TCLP/SPLP leachate samples. Water DOCs can be used to satisfy analyst demonstrations of

capability for TCLP/SPLP matrices. Teflon chips, Ottawa sand, or equivalent is used as the blank matrix for wipes, wastes, and tissues unless specifically requested otherwise by the project.

16.1.1 Collection and Handling Procedures

Waste (Oil) Samples:
Waste (oil) samples are collected in 8oz soil containers with PTFE-lined lids. Waste (oil) samples must be iced at the time of collection and maintained at 0-6°C until the time of preparation. Samples must be prepared within 14 days of collection. Extracts must be stored in the refrigerator at 0-6°C until the time of analysis and analyzed within 40 days of extraction.

Wipe Samples:
Wipe samples are routinely collected in 40mL VOA vials. Wipe samples must be iced at the time of collection and maintained at 0-6°C until time of preparation. Samples must be prepared within 14 days of collection. Extracts must be stored in the refrigerator at 0-6°C until the time of analysis and analyzed within 40 days of extraction.

Tissue Samples:
Tissue samples are routinely collected in plastic containers with the size dependent upon the type of tissue being collected. Plastic jars or plastic baggies can be used. Upon receipt, samples must be placed in the freezer at <-10°C if extraction/digestion cannot be completed that day. Samples must be prepared within 14 days of defrosting. Extracts must be stored in the refrigerator at 0-6°C until the time of analysis and analyzed within 40 days of extraction.

TCLP/SPLP Leachate Samples
Once the TCLP/SPLP extraction procedure has been performed, the leachate is transferred to a 1L glass container. TCLP/SPLP leachates must be stored at 0-6°C until the time of preparation. The leachate sample must be prepared within 7 days of completion of the TCLP/SPLP extraction. Extracts must be stored in the refrigerator at 0-6°C until the time of analysis and analyzed within 40 days of extraction.

16.1.2 Preparation and Analytical Procedures

Wipe, waste, and tissue samples are prepared in the same manner as routine soil samples as outlined in SOP SA-EX-040. TCLP/SPLP matrices are prepared in the same manner as routine water samples as outlined in SOP SA-EX-030. Refer to the applicable preparation SOPs for more information.

Wipe, waste, filter, tissue, and TCLP/SPLP matrices are analyzed in the same manner as routine samples as outlined in this SOP.

16.2 Other Considerations

16.2.1 SW-846 allows alternate criteria to be used for DFTPP evaluation. As such the laboratory has incorporated criteria from the following method:

- EPA 8270D, EPA 8270E, EPA 8270E_LL, EPA 8270E_LL_PAH, EPA 8270D_LL, and EPA 8270D_LL_PAH tune criteria are taken from EPA 625.

16.2.2 The laboratory allows one acid and one base surrogate compound to be outside acceptance limits, in field samples and MS/MSD, provided their recovery is greater than 10%. All surrogate compounds must pass in method blanks and LCS/LCSD.

16.2.3 EPA Method 625 does not require the analysis of an ICV. NELAC requires an ICV; however, it does not list specific criteria. The laboratory has adopted the default criteria listed in Section 9.2.3 for ICVs for these methods.

Additionally, EPA Method 8270D lists recommended criteria of 30%D for the ICV and acknowledges that alternative acceptance limits may be appropriate based on project-specific data quality objectives. The laboratory defaults to the criteria outlined in this SOP; however, more stringent, project-specific requirements can be accommodated upon client request.

16.2.4 The laboratory has defined the analytes listed in Attachment 10 as poor or erratic performers and allows for exceptions to the ICV, CCV, LCS, MS/MSD, and Sporadic Marginal Exceedance criteria for these analytes by EPA method 8270D and EPA 8270E as outlined in this SOP.

Note: The use of Sporadic Marginal Exceedances, as defined in SOP SA-QA-017, is not permitted for samples originating from South Carolina.

16.2.5 EPA Method 8270D or EPA 8270E does not place a cap on an individual analyte's %D or %RSD when evaluating the CCV. The laboratory has adopted more stringent in-house requirements as outlined in this SOP.

16.2.6 Due to maintenance procedures, the laboratory allows internal standard retention times in continuing calibration checks (CCVs) for EPA 8270D, EPA 8270E, EPA 8270E_LL, EPA 8270E_LL_PAH, EPA 8270D_LL, and EPA 8270D_LL_PAH to vary by more than 30 seconds when compared to the retention time of the internal standards in the most recent initial calibration. A component of column maintenance is to remove a portion of the front of the column to eliminate reactive spots caused by injection of field samples. As the column is shortened, the retention times of the internal standards are also shortened. If peak resolution and sensitivity are maintained by meeting the CCV criteria, calibration of the analytical system is not required.

Note: This modification is used for CCVs only. The laboratory has made no similar modification to sample ISTD evaluation.

16.2.8 EPA Method 8270D and 8270E indicate indeno(1,2,3)pyrene and di-n-octylphthalate will be quantitated using ISTD Perylene-12. The laboratory uses Chrysene-d12 as this compound is more stable.

16.2.9 EPA Method 8270D states the method blank must be less than the MDL, 5% of the regulatory limit, or 5% of the sample result, whichever is greater. The laboratory's criteria for the method blank is <1/2RL. More stringent, project-specific requirements can be accommodated upon client request.

16.2.10 EPA Method 8270D specifies a reporting limit standard quantitation criteria of 30% when utilizing a linear fit for the ICAL. The laboratory defaults to EPA 8270E criteria which allows 50%D at the RL level and 30%D of all other levels.

16.2.11 EPA 8270D_LL_PAH and EPA 8270E_LL_PAH is performed for DOD samples only. This procedure incorporates the ortho-terphenyl surrogate included in the EPA 8310B reference method (i.e., PAHs via HPLC analysis).

16.2.12 Method 8270D states in section 11.6.1 The reference mass spectrum must be generated by the laboratory using the conditions of this method. The reference spectra utilized by the laboratory in CHROM for peak identification is generated by the NIST library. The mass ratios for the ions are updated by the continuing calibration analysis.

17.0 Attachments

The following Tables, Diagrams, and/or Validation Data are included as Attachments:

Attachment 1: SOP Summary
Attachment 2: Sample Collection, Preservation, and Holding Time Table
Attachment 3: QC Summary
Attachment 4: Preventative Maintenance and Troubleshooting
Attachment 5: DFTPP Criteria
Attachment 6: Example Tailing Factor Calculation
Attachment 7: Target Compound Information: Quant ions and ISTDs
Attachment 8: Standard Preparation Postings
Attachment 9: Procedures for SIM Analyses
Attachment 10: Poor Responder Information
Attachment 11: EPA 8270D Minimum RRF Table
Attachment 12: EPA 625.1 QC Recovery Criteria
Attachment 13: Glassware Cleaning Procedures

Attachment 1:
SOP Summary

Sample Preparation Summary
Samples should be prepared according to the appropriate matrix-specific SOP.

Matrix	SOP
Water	SA-EX-030
Soil	SA-EX-040

Aqueous Sample Preparation:
For continuous liquid-liquid extraction, the sample is adjusted to a specific pH, as required by the analyte list, transferred to a continuous liquid-liquid extractor or separatory funnel, and extracted using methylene chloride. The extract is concentrated to a predetermined final volume (typically 1mL) using the Zymark nitrogen blow-down concentrator procedure.

Soil Sample Preparation:
For microwave extraction, the sample is transferred to a Teflon extraction vessel. A 1:1 acetone/methylene chloride mixture is added to the sample in the vessel. The vessel is placed on the instrument and the sample extracted at an elevated temperature and pressure. The vessel is cooled to room temperature, the extract is passed through sodium sulfate to remove the water from the sample, and the extract is collected in a concentration tube. The extract is concentrated to a specified final volume (typically 1mL) in methylene chloride using the Zymark nitrogen blow-down concentrator procedure.

Sample Analysis Summary
The extract is analyzed by GC/MS. Qualitative identification of the target compounds in the extract is based on the retention time and the relative abundance of the characteristic masses determined from NIST library. Quantitative analysis is performed using the internal standard technique with a single characteristic ion.

An example of the Analytical Sequence is given in Section XXX.

Initial and Continuing Calibration Requirements Summary

1.0 EPA 625

1.1 Initial Calibration (ICAL)

1.1.1 Linearity Criteria

All compounds must be evaluated for linearity. The following steps outline the evaluation:

1. The relative standard deviation (%RSD) of the calibration standards must be ≤35% for the initial calibration curve to be acceptable.
2. A minimum of 3 points is required. The lowest calibration point must be at or lower than the reporting limit (RL).
3. If the calibration is utilizing enough points to establish a linear or quadratic fit, then a regression curve may be used. Utilize a regression curve in accordance with SOP SA-QA-016. The criteria for the regression coefficient is r^2 ≥0.990. If the %RSD is less than 35%, a regression curve may still be used if it provides a better calibration model over the calibration range than the average RF.

1.2 Second Source Initial Calibration Verification (ICV)

The initial calibration verification (ICV) is acceptable if the %D is ≤35% for each analyte. If the %D criteria are not met, re-calibration is required.

1.3 Continuing Calibration (CCV)

All analytes in the CCV must be ≤20% of the true value to be acceptable.

2.0 EPA 8270D, EPA 8270E, EPA 8270E_LL, EPA 8270E_LL_PAH, EPA 8270D_LL, and EPA 8270D_LL_PAH

2.1 Initial Calibration (ICAL)

2.1.1 Minimum Relative Response Factor (RRF) Criteria

EPA 8270D: The minimum RRF criteria and control analytes are listed in Attachment 11. If the minimum RRF criteria for each compound in each level of the ICAL are not met, analysis of an RLCCV is required in each clock. An NCM is required to denote this situation.
EPA8270E: The minimum RRF criteria listed in Attachment 11 is advisory.

2.1.2 Linearity Criteria

All compounds must be evaluated for linearity. The following steps outline the evaluation:

1. Determine the %RSD for each compound. The %RSD of each compound must be ≤20% to be acceptable.

2. If the %RSD criteria are not met, the next option is to utilize a linear regression curve for that compound, in accordance with SOP SA-QA-016.

   For a linear regression curve to be acceptable, the regression coefficient (r^2) must be ≥0.990.

   For a quadratic curve to be acceptable, the regression coefficient (r^2) must be ≥0.990.

3. If the r^2 criteria are not met, the only remaining option is to utilize the average response factor and report that compound as estimated. In this situation, an NCM must be initiated to describe the issue.

   Note: SW-846 does not put a cap on an individual analyte's %RSD; however, the laboratory has adopted the requirement that no individual analyte can exceed 50% RSD. Therefore, if any analyte's %RSD is >50 %, then re-calibration is required.

4. If more than 10% of the analytes do not meet both the %RSD criteria or meet the r^2 criteria, then recalibration is required.

   Note: Several standard mixes are utilized to perform an initial calibration for the full list of target analytes (e.g., TCL, Appendix IX, etc). Each of these mixes constitutes its own initial calibration. Therefore, when evaluating the numbers of acceptable analytes, each mix will be evaluated separately. Re-calibration need only involve the affected mixes.

5. For any analyte associated with a calibration that does not meet RSD or 0.990 or minimum response factor, an RLCCV must be analyzed with each subsequent clock.

   The RLCCV is a CCV analyzed at the reporting limit for the affected analyte. The criterion for the RLCCV is detection only; however, the standard qualitative identification criteria in the method must be met.
   NOTE: An NCM must be initiated to denote this situation. Any positive results should be noted as estimated. Non-detect results for analyte may be reported without flagging if there has been an analysis of the RLCCV.

2.2 Second Source Initial Calibration Verification (ICV)

The initial calibration verification (ICV) is acceptable if the %D of each analyte in the ICV is less than or equal 30%.

DODQSMV5: ICV must be ≤ 20%D for all analytes. If criteria is not met, re-calibration is required.

2.2.1 ICV Poor Performers

Refer to Attachment 10 for the identification of poor and/or erratic performing analytes. These analytes are allowed a %D >30% but must be <50%D to be acceptable. If there are poor performers that exceed 50%D, the data may be reported provided results are noted as estimated. An NCM must be initiated to denote this situation.

DODQSMV5 does not address poor performers. ICV must be ≤ 20%D for all analytes

2.3 Continuing Calibration Verification (CCV)

2.3.1 Minimum RRF Criteria

The minimum RRF for each compound is listed in Attachment 11.

EPA 8270D: If the minimum RRF criteria are not met, analysis may proceed with the analysis of the RLCCV to demonstrate sensitivity. An NCM must be initiated for any detections associated with a minimum RRF failure.
EPA 8270E: RRF criteria is advisory.

Note: The RRF criteria for certain analytes have been modified for the EPA 8270D_LL method and are documented in Attachment 11.

2.3.2 %D Criteria

EPA 8270D and EPA 8270E : The CCV must be ≤ 20%D.

The reference method makes the exception, however, such that due to the large numbers of analytes in each CCV, some analytes may not meet these criteria.

- If more than 20% of the analytes in a CCV exceed the %D criteria, the CCV is unacceptable, and re-analysis is required.
- If less than 20% of the analytes in a CCV exceed the %D criteria, analyze an RLCCV.
- Analytes that do not meet CCV %D criteria may be reported if the affected analytes are qualitatively identified in the RL CCV.
- If the affected analyte is not detected in the associated client sample, the result is reported without qualification.
- If the affected analyte is detected in the associated client sample, the result must be reported as estimated.

Note: An NCM must be initiated to denote this situation
Note: SW-846 does not put a cap on an individual analyte's %D; however, the laboratory has the requirement that no individual analyte can exceed

50%D. Therefore, if any analyte's %D is >50%, then corrective action is required. Corrective actions include instrument maintenance, re-injection, and/or re-calibration.

DODQSMV5:

- All analytes in the CCV must be ≤ 20 %D. A closing CCV(CCVC) must be analyzed within the 12 hour clock and be ≤ 50% D.
- If the CCV does not meet acceptance criteria and corrective action is not taken, the analysis of 2 passing CCVs is required prior to the analysis of samples.

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CCV Poor Performers

Refer to Attachment 10 for the identification of poor and/or erratic performing analytes. These analytes are allowed a %D >20% but must be <50 %D to be acceptable. If there are poor performers that exceed 50%D, the data may be reported provided results are noted as estimated. An NCM must be initiated to denote this situation. The poor performers must be counted in the total number of allowances within a CCV analysis.

DODQSMV5 does not address poor performers for EPA 8270D or EPA 8270E. Address via NCM if analyte is not considered a compound of concern.

3.0 EPA 625.1

3.1 Initial Calibration (ICAL)

All compounds must be evaluated for linearity. The following steps outline the evaluation:

1. The relative standard deviation (%RSD) of the calibration standards must be ≤35% for the initial calibration curve to be acceptable.
2. A minimum of 5 points are required. The lowest calibration point must be at or lower than the reporting limit (RL).
3. If the calibration is utilizing enough points to establish a linear or quadratic fit, then a regression curve may be used. Utilize a regression curve in accordance with SOP SA-QA-016. The criteria for the regression coefficient is r^2 ≥0.990. If the %RSD is less than 35%, a regression curve may still be used if it provides a better calibration model over the calibration range than the average RF.

3.2 Second Source Initial Calibration Verification (ICV)

The initial calibration verification (ICV) is acceptable if the recovery meets the method defined limits established in EPA 625.1. If the %D criteria are not met, re-calibration is required.

3.3 Continuing Calibration Verification (CCV)

All analytes in the CCV must meet the method defined recovery limits established in EPA 625.1. See Attachment 12

If the %D criteria are not met, then corrective action is required. Corrective actions include instrument maintenance, re-injection, and/or re-calibration.

Attachment 2:
Sample Collection, Preservation, and Holding Time Table

Matrix	Routine Sample Container	Routine Sample Size	Minimum Sample Size	Chemical Preservation	Thermal Preservation	Dechlorination Agent	Holding Time
Water	1L or 500mL amber glass	1L	500mL	None	0-6 °C[1]	None	7 days to extract 40 days to analyze
Soil	8oz glass soil jar	15g	15g	None	0-6°C[1]	None	14 days to extract 40 days to analyze

[1]Samples must be maintained at 0-6 °C, with no frozen samples.

Attachment 3:
QC Summary

QC Item	Method	Frequency	Criteria	Corrective Action
Clock Time	EPA 625	24 hours	Clock time starts with the injection of the DFTPP. Analysis of samples and QC items must conclude within expiration of clock time. Subsequent analysis requires new DFTPP.	Not applicable
	All Other Methods	12 Hours		
Tune/Column Evaluation Standard (DFTPP) - Spectrum Criteria	All Methods	At beginning of each clock	Spectrum Criteria: Refer to Attachment 5.	- Perform instrument maintenance - Re-tune.
Tune/Column Evaluation Standard (DFTPP) - Tailing Factor Criteria	EPA 625	At beginning of each clock	Pentachlorophenol <5 Benzidine <3	- Perform instrument maintenance - Re-tune.
	All Other Methods		Pentachlorophenol <2 Benzidine <2	
Tune/Column Evaluation Standard (DFTPP) - Breakdown Criteria	All Other Methods	At beginning of each clock	<20%	- Perform instrument maintenance - Re-tune.
	EPA 625		None	

QC Item	Method	Frequency	Criteria	Corrective Action
Initial Calibration (ICAL)	EPA 625	Upon instrument set-up, and after unsuccessful CCV	3-point Minimum %RSD <35%	-Re-analyze standard(s) -Prepare new standard(s) and reanalyze -Perform injector port maintenance and reanalyze standards -Replace column and/or clean source, and reanalyze standards
	EPA 625.1		5-point Minimum %RSD <35%	
	EPA 8270D EPA 8270E EPA 8270E_LL EPA 8270E_LL_PAH EPA 8270D_LL EPA 8270D_LL_PAH		5-point Minimum RRF per Attachment 11. %RSD ≤20%. If %RSD > 20%, use curve fit w/ r^2 ≥0.990; If linear fit, RL Level: 30% of true; If r^2 <0.990, use %RSD (allowed for <10% of total # analytes; no analyte >50% RSD). %RSE <50% for low point; <30% for all other points.	
	SIM		3-point Minimum r^2 ≥0.990	

Initial Calibration Verification (ICV) - Second Source	EPA 625	After each ICAL	%D <35%	-Reanalyze standard -Prepare new standard and reanalyze -Recalibrate
	EPA 625.1		Within method-defined limits (in TALS MLG)	
	EPA 8270D EPA 8270E EPA 8270E_LL EPA 8270E_LL_PAH EPA 8270D_LL EPA 8270D_LL_PAH		%D <30% Poor performers %D <50% as per Attachment 10. An NCM is required to denote analytes outside criteria. DODQSM: All analytes ≤ 20%D. No allowance for poor performers	

QC Item	Method	Frequency	Criteria	Corrective Action
Continuing Calibration Verification (CCV)	EPA 625	Per clock (Analyze after DFTPP)	%D ≤20%	-Reanalyze standard -Prepare new standard and reanalyze -Recalibrate Note 1: If <20% total # analytes >20%D, evaluate RL CCV. RL CCV must be qualitatively identified. - If client sample is ND for affected analyte, report unqualified result. - If client sample has detection for affected analyte, report result as estimated. DODQSMV5: If CCV is outside criteria, then analysis of two passing CCVs is required if no corrective action performed (ie reprep or maintenance)
	EPA 625.1		Within method-defined limits (in TALS MLG)	
	EPA 8270D EPA 8270E EPA 8270E_LL EPA 8270E_LL_PAH EPA 8270D_LL EPA 8270D_LL_PAH		RRF per Attachment 11. %D ≤20%. Poor performers per Attachment 10. See Note 1. DODQSM: All analytes ≤ 20%D.	
Reporting Limit Continuing Calibration Verification (RL CCV)	EPA 8270D EPA 8270E EPA 8270E_LL EPA 8270E_LL_PAH EPA 8270D_LL EPA 8270D_LL_PAH	Per clock, if needed. (Analyze after CCV) When RRF criteria is not met in ICAL, or CCV RRF or CCV %D criteria not met.	Affected analytes must be qualitatively identified.	- Perform instrument maintenance - Reanalyze affected samples. - Recalibrate

QC Item	Method	Frequency	Criteria	Corrective Action
Extraction Batch Definition	All Methods	Extracted together w/in 24-hr timeframe; not to exceed 20 field samples	Not Applicable	Not Applicable
Method Blank (MB)	All Methods	One per extraction batch	<1/2RL	Refer to SOP SA-QA-017
Laboratory Control Sample (LCS)	All Methods	One per extraction batch	Within TALS MLG Limits	Refer to SOP SA-QA-017
Laboratory Control Sample Duplicate (LCSD)	All Methods	One per extraction batch, when insufficient sample is provided for MS/MSD/SD	Within TALS MLG Limits	Refer to SOP SA-QA-017
Matrix Spike (MS)	All Methods	One per extraction batch	Within TALS MLG Limits	Refer to SOP SA-QA-017
Matrix Spike Duplicate (MSD)	All Methods	One per extraction batch	Within TALS MLG Limits	Refer to SOP SA-QA-017
Initial Demonstration of Capability (IDOC)	All Methods	Initially, per analyst, per analyte/method/matrix combination	Refer to SOP SA-QA-006	Refer to SOP SA-QA-006 Note: Unsupervised work must not begin until acceptable IDOC is obtained.

QC Item	Method	Frequency	Criteria	Corrective Action
Continuing Demonstration of Capability (CDOC)	All Methods	Annually, per analyst, per analyte/method/matrix combination	Refer to SOP SA-QA-006	Refer to SOP SA-QA-006
Reporting Limit Verification (RLV)	All Methods	Upon method/instrument set-up, per analyte/method/matrix combination. Then quarterly thereafter (for DOD ELAP) or annually thereafter (for non-DOD ELAP)	Refer to SOP SA-QA-007	Refer to SOP SA-QA-007
Method Detection Limit Study (MDL)	All Methods	Upon method/instrument set-up, per analyte/method/matrix combination	Refer to SOP SA-QA-007	Refer to SOP SA-QA-007
MDL Verification (MDLV)	All Methods	Upon method/instrument set-up, per analyte/method/matrix combination. Then quarterly thereafter (for DOD ELAP) or annually thereafter (for non-DOD ELAP)	Refer to SOP SA-QA-007	Refer to SOP SA-QA-007

Attachment 4:
Preventative Maintenance and Troubleshooting

LABORATORY EQUIPMENT PREVENTIVE MAINTENANCE SCHEDULE								
EQUIPMENT ITEM	**Service Interval**							**SERVICE LEVEL**
	D	**W**	**M**	**Q**	**SA**	**A**	**AN**	
Septum							**X**	Replace, recommended daily
Splitless Disc							**X**	Replace, recommended daily
Column/Injector							**X**	Change sleeve and cut front of column, recommended daily
Autosampler							**X**	Clean syringe as needed; replace syringe as needed
Injector Port							**X**	Replace injector port as needed
Lines							**X**	Flush lines as needed; replace lines as needed
Column							**X**	Change column as needed
Mass Spectrometer							**X**	Clean as needed
Rough Pump							**X**	Change oil as needed

D = daily; W = Weekly; M = monthly; Q = Quarterly; SA = semi-annually; A = annually; AN = as needed

Troubleshooting

Troubleshooting should be documented as outlined above. If possible, troubleshooting is best performed in a step-wise manner to systematically isolate instrument components. Refer to the instrument manufacturer's guides for specific information and strategies. Enlist assistance from technical and/or department management as needed.

Contingency Plan

An extensive spare parts inventory is maintained for routine repairs. Since instrumentation is standardized throughout the laboratory network, spare parts and components can be readily exchanged among the network.

In general, the laboratory has at least one backup unit for each critical unit. In the event of instrument failure, portions of the sample load may be diverted to duplicate instrumentation, the analytical technique switched to an alternate approved technique (such as manual colorimetric determination as opposed to automated colorimetric determination), or samples shipped to another properly certified or approved TestAmerica location.

Maintenance Log

A maintenance log must be established for each piece of equipment used in the laboratory. All maintenance that is performed on the instrument must be recorded in the log including:

- Type of maintenance performed (Note: This includes preventative/routine maintenance; non-routine maintenance; maintenance performed by an external vendor; updates to software versions; etc.)
- analyst or technician performing the maintenance
- date the maintenance was performed
- detailed explanation of the reason for the maintenance
- resolution of the problem and return to control
- all service calls from instrument representatives
- Resolution of the problem (i.e., the Return to Control)

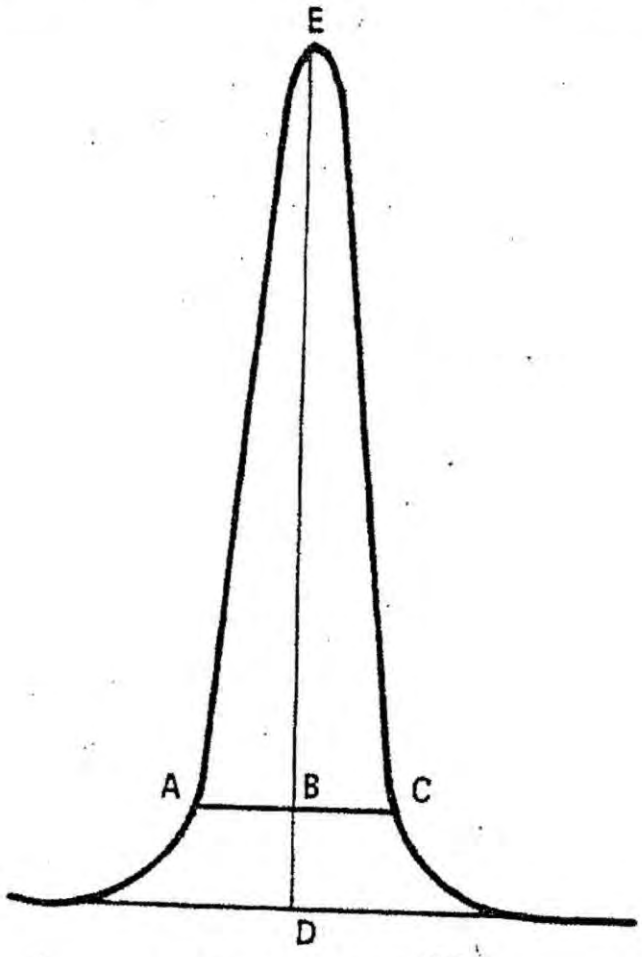
Instrument Labeling

Each instrument must be labeled with its name or identification. Additionally, non-operational instruments must be isolated from service or marked as being out of service. Each piece of equipment has an “Operational / Not Operational” sticker that is used for this purpose.

Attachment 5:
DFTPP Criteria

m/z	Ion Abundance Criteria
51	30-60% of mass 198
68	<2% of mass 69
70	<2% of mass 69
127	40-60% of mass 198
197	<1.0% of mass 198
198	Base peak, 100% relative abundance
199	5-9% of mass 198
275	10-30% of mass 198
365	>1% of mass 198
441	Present but less than mass 443
442	>40% of mass 198
443	17-23% of mass 442

Attachment 6:
Example Tailing Factor Calculation



$$\text{TAILING FACTOR} = \frac{BC}{AB}$$

Example calculation: Peak Height = DE = 100 mm
10% Peak Height = BD = 10 mm
Peak Width at 10% Peak Height = AC = 23 mm
AB = 11 mm
BC = 12 mm

$$\text{Therefore: Tailing Factor} = \frac{12}{11} = 1.1$$

Attachment 7:
Target Compound Information: Quant Ions and ISTDs*

PARAMETER	Quant Ion	Secondary Ions		ISTD	pH 2 (Only)	pH 2 *and* pH 11 (i.e., Dual pH Required)
1,1'-Biphenyl	154	76		3	X	
1-Chloronaphthalene	162	127	164	3	X	
1,2,3-Trichlorobenzene	216	214		1		X
1,2,4,5-Tetrachlorobenzene	216	214	179	3		X
1,2,4-Trichlorobenzene	180	182	145	2	X	
1,2-Dichlorobenzene	146	148		1	X	
1,2-Diphenylhydrazine	77	105	182	4	X	
1,3,5-Trichlorobenzene	180	145		1		X
1,3,5-Trinitrobenzene	213	74	120	4		X
1,3-Dichlorobenzene	146	148	111	1	X	
1,3-Dinitrobenzene	168	76	50	3		X
1,4-Dichlorobenzene	146	148	111	1	X	
1,4-Dioxane	88	58	45	1	X	
1,4-Naphthoquinone	158	104	76	3		X
1,4-Dinitrobenzene	168	76	50	3		
1-Diallate	86	43	234	4		X
1-Methylnaphthalene	142	141		2	X	
1-Naphthylamine	143	115	116	3		X
2,2'-oxybis[1-chloropropane]	45	121		1	X	
2,3,4,5-Tetrachlorophenol	232	230	131	3		
2,3,4,6-Tetrachlorophenol	232	230	131	3	X	
2,3,5,6-Tetrachlorophenol	232	96	131	3	X	
2,3,6-Trichlorophenol	196	198		2	X	
2,3-Dimethylphenol	107	122	121	1	X	
2,4 & 2,5-Dimethylphenol	107	122	121	1	X	
2,4,5-Trichlorophenol	196	198	200	3	X	
2,4,6-Trichlorophenol	196	198	200	3	X	
2,4-Dinitrochlorobenzene	202	110	75	2		X
2,4-Dichlorophenol	162	164	98	2	X	
2,4-Dimethylphenol	122	107	121	2	X	
2,4-Dinitrophenol	184	63	154	3	X	
2,4-Dinitrotoluene	165	89	63	3	X	
2,5-Dimethylphenol	107	122	121	1	X	
2,5-Dinitrophenol	184	63		1	X	
2,6-Dichlorophenol	162	164	98	2	X	
2,6-Dimethylphenol	107	122	121	1	X	
2,6-Dinitrotoluene	165	89	63	3	X	
2-Acetylaminofluorene	181	180	223	5		X
2-Chloronaphthalene	162	164	127	3	X	

PARAMETER	Quant Ion	Secondary Ions		ISTD	pH 2 (Only)	pH 2 *and* pH 11 (i.e., Dual pH Required)
2-chloronitrobenzene/4-chloronitrobenzene	157	111	75	2		X
2-Chlorophenol	128	130	64	1	X	
2-Diallate	86	43	234	4		X
2-Mercaptobenzothiazole	167	108		5		X
2-Methylnaphthalene	142	141		2	X	
2-Methylphenol	107	108	77	1	X	
2-Naphthylamine	143	115	116	3		X
2-Nitroaniline	65	92	138	3	X	
2-Nitrobiphenyl	152	115		3		X
2-Nitrophenol	139	109	65	2	X	
2-Picoline	93	66		1		X
2-Toluidine	106	107	79	2		X
3 & 4 Methylphenol	107	108		1	X	
3,3'-Dichlorobenzidine	252	254	126	5	X	
3,3'-Dimethylbenzidine	212	196	106	5		X
3,4-Dichloronitrobenzene	109	133	191	2		X
3,4-Dimethylphenol	107	122	121	1	X	
3-Methylcholanthrene	268	252	253	5		X
3-Nitroaniline	138	108	92	3	X	
3-Nitrobiphenyl	152	199		4		X
3-Nitrochlorobenzene	157	111	75	2		X
3-Nitrophenol	139	65		1	X	
4,6-Dinitro-2-methylphenol	198	105	121	4	X	
4-Aminobiphenyl	169	168	170	4		X
4-Bromophenyl phenyl ether	248	250	141	4	X	
4-Chloro-3-methylphenol	107	144	142	2	X	
4-Chloroaniline	127	129	65	2	X	
4-Chloronitrobenzene	157	111	75	2		X
4-Chlorophenol	65	128		2	X	
4-Chlorophenyl phenyl ether	204	141	206	3	X	
4,4'-Methylene bis(2-chloroaniline)	231	266	268	5		X
4-Nitroaniline	138	108	92	3	X	
4-Nitrobiphenyl	152	199		4		X
4-Nitrophenol	65	109	139	3	X	
4-Nitroquinoline-1-oxide	174	101	128	4		X
5-Nitro-o-toluidine	152	77	106	3		
6-Methylchrysene	242	241	239	5		X
7,12-Dimethylbenz(a)anthracene	256	239	241	6		X
Acenaphthene	154	153	152	3	X	
Acenaphthylene	152	151	153	3	X	
Acetophenone	105	77	51	2	X	
Acrylamide	71	44	55	1		X

PARAMETER	Quant Ion	Secondary Ions		ISTD	pH 2 (Only)	pH 2 *and* pH 11 (i.e., Dual pH Required)
alpha,alpha-Dimethyl phenethylamine	58	91	42	2		X
alpha-Pinene	93	121		1		X
Aniline	93	66		1		X
Anthracene	178	176	179	4	X	
Aramite, Total	185	191	319	5		X
Aramite-1	185	191	319	5		X
Aramite-2	185	191	319	5		X
Atrazine	200	173	215	4	X	
Benzaldehyde	77	105		1	X	
Benzidine	184	92	185	4		X
Benzo[a]anthracene	22	229	226	5	X	
Benzo[a]pyrene	252	125	253	6	X	
Benzo[b]fluoranthene	252	253	125	6	X	
Benzo[g,h,i]perylene	276	277	138	6	X	
Benzo[k]fluoranthene	252	253	125	6	X	
Benzoic acid	105	122		2	X	
Benzyl alcohol	108	79	77	1	X	
Bis(2-chloroethoxy)methane	93	123	95	2	X	
Bis(2-chloroethyl)ether	63	93	95	1	X	
Bis(2-ethylhexyl) phthalate	149	167	279	5	X	
Butyl benzyl phthalate	149	91	206	5	X	
Caprolactam	113	55		2	X	
Carbazole	167			4	X	
Catechol	110	64		1		X
Chlorobenzilate	139	251	111	5	X	
Chrysene	228	226	229	5	X	
Di(2-ethylhexyl)adipate	129	57		3	X	
Diallate	86	43		4		X
Dibenz(ah) acridine	279	280	278	6	X	
Dibenz(aj)acridine	279	280	278	6	X	
Dibenz(a,h)anthracene	287	139	279	6	X	
Dibenzofuran	168	139		3	X	
Diethyl phthalate	149	177	150	3	X	
Dimethoate	87	93	125	4		X
Dimethyl phthalate	163	194	164	3	X	
Dimethyl terephthalate	194	135		2		X
Di-n-butyl phthalate	149	150	104	4	X	
Di-n-octyl phthalate	149	43		5	X	
Dinoseb	211	163	147	4	X	
Disulfoton	88	60		4		X
Ethyl methanesulfonate	79	109	97	1		X
Ethyl Parathion	109	97		4		X

PARAMETER	Quant Ion	Secondary Ions		ISTD	pH 2 (Only)	pH 2 *and* pH 11 (i.e., Dual pH Required)
Famphur	218	93	125	4		X
Fluoranthene	202	203	101	4	X	
Fluorene	166	165	167	3	X	
Hexachlorobenzene	284	142	249	4	X	
Hexachlorobutadiene	225	223	227	2	X	
Hexachlorocyclopentadiene	237	235	272	3	X	
Hexachloroethane	117	201	199	1	X	
Hexachlorophene	196	198		6		X
Hexachloropropene	213	211	215	2		X
Hexadecane	57	43	71	1	X	
Indene	57	43	71	1	X	
Indeno[1,2,3-cd]pyrene	276	138		5	X	
Isodrin	193	195	66	4	X	
Isophorone	82	95	138	2	X	
Isosafrole	162	104	131	2		X
Kepone	272	237	357	5		X
Methapyrilene	97	58	191	4		X
Methyl Benzoate	105	77	51	1		X
Methyl methanesulfonate	80	79	65	1		X
Methyl parathion	109	125		4		X
Methyl Phenols, Total	107	108		1	X	
Monomethyl Terephthalate	149	121		3		X
Naphthalene	128	129		2	X	
n-Decane	57	43	41	1	X	
Nitrobenzene	77	123	65	2	X	
N-Nitro-o-toluidine	152	77	106	3		X
N-Nitrosodiethylamine	102	42	44	1	X	
N-Nitrosodimethylamine	42	74		1		X
N-Nitrosodi-n-butylamine	84	57	41	2		X
N-Nitrosodi-n-propylamine	70	42		1	X	
N-Nitrosodiphenylamine	169	168	167	4	X	
N-Nitrosomethylethylamine	88	42	43	1		X
N-Nitrosomorpholine	56	86		1		X
N-Nitrosopiperidine	114	42	55	2		X
N-Nitrosopyrrolidine	100	41	42	2		X
N-Octadecane	57	43	41	**1**	X	
o,o',o''-Triethylphosphorothioate	65	97	93	2		X
p-Dimethylamino azobenzene	120	225	77	5		X
Pentachlorobenzene	250	248	252	3		X
Pentachloroethane	167	165	169	1		X
Pentachloronitrobenzene	237	295	142	4		X
Pentachlorophenol	266	264	268	4	X	

PARAMETER	Quant Ion	Secondary Ions		ISTD	pH 2 (Only)	pH 2 *and* pH 11 (i.e., Dual pH Required)
Phenacetin	108	109	179	3		X
Phenanthrene	178	176	179	4	X	
Phenol	94	66	65	1	X	
Phenyl ether	170	141		3		X
Phenylmercapatan	110	66	109	1		X
Phorate	75	121		4		X
p-Phenylene diamine	108	80	107	2		X
Pronamide	173	175	145	4		X
Pyrene	202	200	203	5	X	
Pyridine	79	52	51	1		X
Quinoline	129	102		1		X
Safrole, Total	162	104	135	2		X
Sulfotepp	97	65		4		X
Thionazin	107	96	97	4		X
Toluic acid	91	119	136	2		X
SURROGATES						
Nitrobenzene-d5[1]	82	128	54	2		
2-Fluorobiphenyl[1]	172	171		3		
Phenol-d5 [2]	99	71		1		
2-Fluorophenol [2]	112	64		1		
2,4,6-Tribromophenol [2]	330	332	144	3		
Terphenyl-d141	244	122	212	5		
OTP[2] (8270D_LL_PAH Only)	244	122	212	5		

[1]Base Surrogate
[2]Acid Surrogate

INTERNAL STANDARDS

1,4-Dichlorobenzene-d4	152	150	115	1		
Naphthalene-d8	136	68		2		
Acenaphthene-d10	164	162	160	3		
Phenanthrene-d10	188	94	80	4		
Chrysene-d12	240	236	120	5		
Perylene-d12	264	265	260	6		

For a complete list of target analytes for each method refer to the TALS Method Limit Groups (MLGs).

Attachment 8:
Standard Preparation Information

Purchased Standards

Vendor Name	Standard Description	Part Number	Concentration (ug/mL)
Supelco	8270 Tuning Mix	47548-U	1000
Restek	8270 L1/S1	570666	1000
Restek	8270 L1/S1 SS	570666.sec	1000
Restek	8270 L1/S9	569730	2000
Restek	8270 L1/S9 SS	569730.SEC	2000
Restek	8270 L1/S10	569731	2000
Restek	8270 L1/S10 SS	569731.SEC	2000
Restek	8270 L1/S11	569732	2000
Restek	8270 L1/S11	569732.SEC	2000
Restek	8270 L2/S1	567678	1000
Restek	8270 L2/S1 SS	567678.sec	1000
Restek	8270 L2/S2	569733	1000
Restek	8270 L2/S2 SS	569733.SEC	1000
Restek	8270 L2/S3	567680	2000
Restek	8270 L2/S3 SS	567680.sec	2000
Restek	8270 L2/S4	567681	1000
Restek	8270 L2/S4 SS	567681.sec	1000
Restek	8270 L2/S5	567682	2000
Restek	8270 L2/S5 SS	567682.sec	2000
Restek	8270 L2/S7	568726	2000
Restek	8270 L2/S7 SS	568726.sec	2000
Restek	8270 L2/S8	568727	2000
Restek	8270 L2/S8 SS	568727.sec	2000
Restek	8270 L2/ADD	568396	2000
Restek	8270 L2/ADD SS	568396.sec	2000
Restek	8270 L3/S1	567683	2000
Restek	8270 L3/S1 SS	567683.sec	2000
Restek	Hexachlorophene	568395	5000
Restek	Hexachlorophene SS	568395.sec	5000
Restek	8270 SURR	567685	5000
Restek	o-Terphenyl	31066	2000
Restek	8270 Internal Standard	567684	2000

Expiration:
Un-opened: Manufacturer's expiration date
Opened: 6 months from date opened or manufacturer's expiration date, whichever is sooner

Storage:
Un-opened: Manufacturer's suggested
Opened: Refrigerated at 0-6°C.

Prepared Standards

Note:
The following conditions apply for all prepared standards:
Final Solvent = Methylene Chloride
Storage = 0-6°C
Expiration = 3 months from preparation date

EPA 625 / EPA 8270D / EPA 625.1 / EPA 8270E DFTPP
TALS Name = SMDFTPP050

Parent Standard Name	Parent Standard Concentration (ug/mL)	Aliquot (uL)	Final Volume (mL)	Final Concentration (ug/mL)
8270 Tuning Mix	1000	500	10	50

SM LIST 1A INT
TALS Name = SM-List1AInt

Parent Standard Name	Parent Standard Concentration (ug/mL)	Aliquot (uL)	Final Volume (mL)	Final Concentration (ug/mL)
8270 L1/S1	1000	4000	10	400
8270 L1/S9	2000	2000		
8270 SURR	5000	1000		

SM LIST 1B INT
TALS Name = SM-List1BInt

Parent Standard Name	Parent Standard Concentration (ug/mL)	Aliquot (uL)	Final Volume (mL)	Final Concentration (ug/mL)
8270 L1/S11	2000	2000	10	400
8270 L1/S10	2000	2000		

SM LIST 2-AP9 INT
TALS Name = SM-L2-AP9INT

Parent Standard Name	Parent Standard Concentration (ug/mL)	Aliquot (uL)	Final Volume (mL)	Final Concentration (ug/mL)
8270 L2/ADD	2000	1000	10	200
8270 L2/S1	1000	2000		
8270 L2/S2	1000	2000		
8270 L2/S3	2000	1000		
8270 L2/S5	2000	1000		

SM-OP-INT
TALS Name = SM-8270OPINTC

Parent Standard Name	Parent Standard Concentration (ug/mL)	Aliquot (uL)	Final Volume (mL)	Final Concentration (ug/mL)
8270 L2/S4	1000	2000	5	400
8270 L2/S7	2000	1000		
8270 L2/S8	2000	1000		

LIST 1A Second Source ICV
TALS Name = SM-LIST1A-ICV

Parent Standard Name	Parent Standard Concentration (ug/mL)	Aliquot (uL)	Final Volume (mL)	Final Concentration (ug/mL)
8270 L1/S1-SS	1000	160	2	80
8270 L1/S9-SS	2000	80		
8270 SURR	5000	32		
8270 ISTD	2000	40		

LIST 1B Second Source ICV
TALS Name = SM-LIST1B-ICV

Parent Standard Name	Parent Standard Concentration (ug/mL)	Aliquot (uL)	Final Volume (mL)	Final Concentration (ug/mL)
8270 L1/S10-SS	2000	80	2	80
8270 L1/S11-SS	2000	80		
8270 ISTD	2000	40		

LIST 2 Second Source ICV
TALS Name = SM-LIST2-ICV

Parent Standard Name	Parent Standard Concentration (ug/mL)	Aliquot (uL)	Final Volume (mL)	Final Concentration (ug/mL)
8270 L2/S1-SS	1000	160	2	80
8270 L2/S2-SS	1000	160		
8270 L2/S3-SS	2000	80		
8270 L2/S5-SS	2000	80		
8270 L2/ADD-SS	2000	80		

Hexachlorophene/Organophos Second Source ICV
TALS Name = SM-HEXAOPHICV

Parent Standard Name	Parent Standard Concentration (ug/mL)	Aliquot (uL)	Final Volume (mL)	Final Concentration (ug/mL)
R-HEXA-SS	5000	320	2	80
8270 L2/S4-SS	1000	160		
8270 ISTD	2000	40		
8270L2/S8-SS	2000	80		
8270L2/S7-SS	2000	80		

Low-Level Internal Standard Solution
TALS Name = SM-LLISTD

Parent Standard Name	Parent Standard Concentration (ug/mL)	Aliquot (uL)	Final Volume (mL)	Final Concentration (ug/mL)
8270 Internal Standard Mix	2000	1000	10	200

8270LL Full Standard
TALS Name = SM-LL BNA INT

Parent Standard Name	Parent Standard Concentration (ug/mL)	Aliquot (uL)	Final Volume (mL)	Final Concentration (ug/mL)
8270 L1/S1	1000	500	10	50
8270 L2/ S10	2000	250		
8270 L1/ S11	2000	250		
8270 L1/ S9	2000	250		
8270 SURR	5000	100		
OTP	2500	200		

8270LL AP-9 Intermediate Mix
TALS Name = SMLLA9INT(CR)

Parent Standard Name	Parent Standard Concentration (ug/mL)	Aliquot (uL)	Final Volume (mL)	Final Concentration (ug/mL)
8270 L2/S1	1000	500	10	50
8270L2/S2	1000	500		
8270 L2/S3	2000	250		
8270 L2/S5	2000	250		
8270 L2/ADD	2000	250		

8270LL Hexachlorophene/Organophos Intermediate Mix
TALS Name = SM-OP-INT

Parent Standard Name	Parent Standard Concentration (ug/mL)	Aliquot (uL)	Final Volume (mL)	Final Concentration (ug/mL)
8270 L2/S4	1000	500	10	50
8270L2/S7	2000	250		
8270 L2/S8	2000	250		

Low-Level LIST 1 Second Source ICV
TALS Name = SMLLBNAICV

Parent Standard Name	Parent Standard Concentration (ug/mL)	Aliquot (uL)	Final Volume (mL)	Final Concentration (ug/mL)
8270 L1/S1-SS	1000	20	2	10
8270 L1/S10-SS	2000	10		
8270 L1/ S11-SS	2000	10		
8270 L1/ S9-SS	2000	10		
8270 SURR	5000	4		
OTP	2000	8		

Low-Level Appendix 9 Second Source ICV
TALS Name = SMLLAP9ICV

Parent Standard Name	Parent Standard Concentration (ug/mL)	Aliquot (uL)	Final Volume (mL)	Final Concentration (ug/mL)
8270 L2/S1	1000	10	2	10
8270 L2/S2	2000	20		
8270 L2/S3	2000	20		
8270 L2/S5	2000	10		
8270 L2/ADD	5000	10		
LLISTD Solution	2000	20		

Low-Level Hexachlorophene/Organophos Second Source ICV
TALS Name = LLOPHhexICV

Parent Standard Name	Parent Standard Concentration (ug/mL)	Aliquot (uL)	Final Volume (mL)	Final Concentration (ug/mL)
R-HEXA-SS	5000	200	2	10
8270 L2/S4-SS	1000	20		
LLISTD Solution	200	20		
8270 L2/S7-SS	2000	10		
8270 L2/S8-SS	2000	10		

SM List 1A Calibration Curve

ICAL Level ID	Parent Standard	Parent Standard Concentration (ug/mL)	Parent Standard Aliquot (uL)	ISTD Aliquot (uL)	Final Volume (mL)	Final Concentration (ug/mL)
LIST 1A-005	SM LIST 1A INT	200	25	40	2	5
LIST 1A-010	SM LIST 1A INT	200	50	40	2	10
LIST 1A-020	SM LIST 1A INT	200	100	40	2	20
LIST 1A-040	SM LIST 1A INT	200	200	40	2	40
LIST 1A-060	SM LIST 1A INT	200	300	40	2	60
LIST 1A-080	SM LIST 1A INT	200	400	40	2	80
LIST 1A-100	SM LIST 1A INT	200	500	40	2	100
LIST 1A-160	SM LIST 1A INT	200	800	40	2	160
LIST 1A-200	SM LIST 1A INT	200	1000	40	2	200

SM List 1B Calibration Curve

ICAL Level ID	Parent Standard	Parent Standard Concentration (ug/mL)	Parent Standard Aliquot (uL)	ISTD Aliquot (uL)	Final Volume (mL)	Final Concentration (ug/mL)
LIST 1B-005	SM LIST 1B INT	200	25	40	2	5
LIST 1B-010	SM LIST 1B INT	200	50	40	2	10
LIST 1B-020	SM LIST 1B INT	200	100	40	2	20
LIST 1B-040	SM LIST 1B INT	200	200	40	2	40
LIST 1B-060	SM LIST 1B INT	200	300	40	2	60
LIST 1B-080	SM LIST 1B INT	200	400	40	2	80
LIST 1B-100	SM LIST 1B INT	200	500	40	2	100
LIST 1B-160	SM LIST 1B INT	200	800	40	2	160
LIST 1B-200	SM LIST 1B INT	200	1000	40	2	200

SM List 2 Calibration Curve

ICAL Level ID	Parent Standard	Parent Standard Concentration (ug/mL)	Parent Standard Aliquot (uL)	ISTD Aliquot (uL)	Final Volume (mL)	Final Concentration (ug/mL)
LIST 2-005	SM-LIST 2-AP9 INT	200	50	40	2	5
LIST 2-010	SM-LIST 2-AP9 INT	200	100	40	2	10
LIST 2-020	SM-LIST 2-AP9 INT	200	200	40	2	20
LIST 2-050	SM-LIST 2-AP9 INT	200	500	40	2	50
LIST 2-080	SM-LIST 2-AP9 INT	200	800	40	2	80
LIST 2-100	SM-LIST 2-AP9 INT	200	1000	40	2	100
LIST 2-200	SM-LIST 2-AP9 INT	200	2000	40	2	200

Hexachlorophene/Ophos Calibration Curve

ICAL Level ID	Parent Standard	Parent Standard Concentration (ug/mL)	Parent Standard Aliquot (uL)	ISTD Aliquot (uL)	Final Volume (mL)	Final Concentration (ug/mL)
HEXAOP-005	SMOPINT	1000	10	40	2	5
	R-HEXA	5000	20			50
HEXAOP-010	SMOPINT	1000	20	40	2	10
	R-HEXA	5000	40			100
HEXAOP-020	SMOPINT	1000	40	40	2	20
	R-HEXA	5000	80			200
HEXAOP-050	SMOPINT	1000	100	40	2	50
	R-HEXA	5000	200			500
HEXAOP-080	SMOPINT	1000	160	40	2	80
	R-HEXA	5000	320			800
HEXAOP-100	SMOPINT	1000	200	40	2	100
	R-HEXA	5000	400			1000
HEXAOP-200	SMOPINT	1000	400	40	2	200
	R-HEXA	5000	800			2000

SM 8270LL BNA Working Standards

ICAL Level ID	Parent Standard	Parent Standard Concentration (ug/mL)	Parent Standard Aliquot (uL)	LL ISTD Aliquot (uL)	Final Volume (mL)	Final Concentration (ug/mL)
LLBNA0.20	BNA 50	50	8	40	2	0.20
LLBNA 1.0	BNA 50	50	40	40	2	1.0
LLBNA 2.0	BNA 50	50	80	40	2	2.0
LLBNA 5.0	BNA 50	50	200	40	2	5.0
LLBNA 10	BNA 50	50	400	40	2	10
LLBNA 15	BNA 50	50	600	40	2	15
LLBNA 20	BNA 50	50	800	40	2	20

SM 8270LL BNA Working Standards

ICAL Level ID	Parent Standard	Parent Standard Concentration (ug/mL)	Parent Standard Aliquot (uL)	LL ISTD Aliquot (uL)	Final Volume (mL)	Final Concentration (ug/mL)
LLAP91.0	LLAP9 Intermediate	50	40	20	2	1.0
LLAP92.0	LLAP9 Intermediate	50	80	20	2	2.0
LLAP95.0	LLAP9 Intermediate	50	200	20	2	5.0
LLAP910.0	LLAP9 Intermediate	50	400	20	2	10
LLAP915.0	LLAP9 Intermediate	50	600	20	2	15
LLAP920.0	LLAP9 Intermediate	50	800	20	2	20

Low-Level Hexachlorophene/Ophos Calibration Curve

ICAL Level ID	Parent Standard	Parent Standard Concentration (ug/mL)	Parent Standard Aliquot (uL)	ISTD Aliquot (uL)	Final Volume (mL)	Final Concentration (ug/mL)
LLOphHex1.0	SM-OP-INT	50	40	20	2	1.0
	R-HEXA	5000	20			50
LLOphHex2.0	SM-OP-INT	50	80	20	2	2.0
	R-HEXA	5000	40			100
LLOphHex5.0	SM-OP-INT	50	200	20	2	5.0
	R-HEXA	5000	100			250
LLOphHex10.0	SM-OP-INT	50	400	20	2	10
	R-HEXA	5000	200			500
LLOphHex15.0	SM-OP-INT	50	600	20	2	15
	R-HEXA	5000	300			750
LLOphHex20.0	SM-OP-INT	50	800	20	2	20
	R-HEXA	5000	400			1000

Attachment 9:
Procedures for SIM Analyses

Analytical Procedures for SIM Analysis

The mass spectrometer (MS) may be used in the selected ion monitoring mode to increase the sensitivity of the GC/MS analysis. In SIM mode, the MS is set to monitor for only a few selected ions; therefore, more ions of the selected mass(es) can be filtered and counted, resulting in an increase of sensitivity of 10 to 100 fold over scan monitoring, depending on the compound. SIM GC/MS analysis sacrifices selectivity for sensitivity since only a few characteristic ions are monitored. A spectral match cannot be made against a reference library and tentatively identified compounds (TICs) cannot be determined from a SIM run. In general, the SIM analysis should be used only when a few compounds are required to be monitored. SIM can also be used to confirm the presence of a target compound determined by GC when the routine GC/MS scan analysis cannot provide confirmation.

The following procedures are based on the guidance in SW-846 Method 8270D/8270E. The analytical sequence is the same as is given in the associated analytical SOPs, with mass tune criteria and calibration verification every 12 or 24 hours.

- Analyze the 50 ug/mL DFTPP in the scan mode. Evaluate the DFTPP against the acceptance criteria given in previous sections in the SOP.

- Determine the approximate retention time of the target by analyzing the target analyte(s) by GC/MS scan.

- Set the MS to monitor for the characteristic ions (minimum of two ions) for the target analyte(s) and internal standard(s). The ion dwell time should be set to give at least six integrations across the peak. A dwell time of 50-100ms is common.

- Prepare and analyze a minimum of three calibration standards for the target compounds. The lowest standard should be at the required quantitation limit and the other two standards should define the working range of the GC/MS. The internal standard(s) should be at a concentration of approximately 4ug/mL for SVOC.

- Evaluate the resulting calibration curve according to the initial calibration procedures given in SOP SA-QA-016. If r^2 is >0.990, the calibration curve is acceptable.

  If the initial calibration criteria are not met, action must be taken to bring the analytical system into compliance with the criteria. This action may include injection port maintenance, source cleaning, changing the column, or replacement of injection port lines and assembly. In any case, if the criteria are not met, the initial calibration must be repeated. The analyst must be aware of the 24-hour clock for the DFTPP analysis in 625 SIM and the 12-hour clock for the DFTPP analysis in 8270 SIM.

SIM Analysis of Target Compounds

- Add an appropriate volume of internal standard to the extract or sample to give the same concentration as in the calibration standards. Analyze the extract or sample under the same conditions as the standard.

Note: It is advised that the extract be split before addition of ISTD solution. One split can be utilized for scan analysis and second split reserved for SIM analysis.

- Compare the retention time of the sample to the retention time of the standard.

  If a peak is detected at the retention time of the target compound containing the selected masses in the same ratio as the standard, the peak is confirmed as the target compound and the concentration is calculated. The relative intensities of the ions in the sample should agree within ±20% of the intensities of the ions in the standard.

  If a peak is not present at the appropriate retention time or if the ratios of the ions are not the same as the standard, the analyte is not confirmed

- If the concentration of the target compound exceeds the highest calibration standard, analyze a more dilute aliquot of the sample or extract, maintaining the internal standard concentration at the same level as the calibration standards.

Attachment 10:
Poor Responder Information

The following analytes have been identified, in the reference method and/or via historical data, to be poor and/or erratic performers:

1,2,4,5-Tetrachlorobenzene
1,3,5-Trinitrobenzene
1,3-Dinitrobenzene
1,4-Dinitrobenzene
1,4-dioxane
1,4-Naphthoquinone
1-Naphthylamine
2,3,4,6-Tetrachlorophenol
2,3,5,6-Tetrachlorophenol
2,4-dinitrophenol
2,6-Dichlorophenol
2-Acetylaminofluorene
2-Naphthylamine
2-Picoline
2-secbutyl-4,6-dinitrophenol (Dinoseb)
3&4-methylphenol
3,3'-Dimethylbenzidine
3'3-Dichlorobenzidine
3-Methylcholanthrene
4,4"-Methylenebis(2-chloroaniline)
4,4-methylenebis (2-chloroaniline)
4,6-Dinitro-2-methyl- phenol,
4-Aminobiphenyl
4-Chloroaniline,
4-nitrophenol
4-Nitroquinoline-1-oxide
5-Nitro-o-toluidine
6-Methylchrysene
7,12-Dimethylbenz(a)anthracene
a,a-Dimethyl-phenethylamine
Acrylamide
Aniline
Aramite (#1)
Aramite (#2)
Atrazine
Benzaldehyde
Benzidine
Benzoic Acid
Benzyl Alcohol
Biphenyl
Caprolactam
Chlorobenzilate
Diallate (#1)
Diallate (#2)
Dibenz(a,h)acridine
Dibenz(a,j)acridine
Dimethoate
Diphenylamine
Disulfoton
Dodecanol
Ethyl methanesulfonate
Famphur
Hexachlorocyclopentadiene
Hexachloroethane
Hexachlorophene
Hexachloropropene
Hydroquinone
Isodrin
Isosafrole (#1)
Isosafrole (#2)
Kepone
Methapyrilene
Methyl methanesulfonate
Methyl Parathion
N-Nitrosodiethylamine
n-Nitrosodimethylamine
n-Nitrosodi-n-butylamine
N-Nitrosomethylethylamine
N-Nitrosomorpholine
N-Nitrosopiperidine
N-Nitrosopyrrolidine
o,o,o-Triethylphosphorothioate
o-Toluidine
p-(Dimethylamino)azobenzene
Parathion
Pentachlorobenzene
Pentachloroethane
Pentachloronitrobenzene
Pentachlorophenol
Phenacetin
Phenol
Phorate
p-Phenylenediamine
Pronamide
Pyridine
Quinoline
Safrole
Sulfotepp
Thionazin

These analytes are exempt from the LCS, MS, MSD, Sporadic Marginal Exceedance, ICV, and CCV criteria for EPA method 8270D and EPA 8270E as listed in this SOP.

Attachment 11:
EPA 8270D Minimum RRF Criteria

Analyte	Minimum RRF Criteria
1,2,4,5-Tetrachlorobenzene	0.010
1,1'-Biphenyl	0.010
2,3,4,6-Tetrachlorophenol	0.010
2,2'-Oxibis(1-chloroporopane)	0.010
2-Chloronaphthalene	0.800
2-Chlorophenol	0.800
2-Methylphenol	0.700
2-Methylnaphthalene	0.400
2-Nitroaniline	0.010
2-Nitrophenol	0.100
2,4-Dichlorophenol	0.200
2,4-Dimethylphenol	0.200
2,4-Dintirophenol	0.010
2,4-Dinitrotoluene	0.200
2,4,5-Trichlorophenol	0.200
2,4,6-Trichlorophenol	0.200
2,6-Dinitrotoluene	0.200
3-Nitroaniline	0.010
3,3'-Dichlorobenzidine	0.010
4-Bromophenyl phenyl ether	0.100
4-Chloro-3-methylphenol	0.200
4-Chloroaniline	0.010
4-Chlorophenyl phenyl ether	0.400
4-Methylphenol	0.600
4-Nitroaniline	0.010
4-Nitrophenol	0.010
4,6-Dinitro-2-methylphenol	0.010
Acenaphthene	0.900
Acenaphthylene	0.900
Acetophenone	0.010
Atrazine	0.010
Anthracene	0.700
Benzaldehyde	0.010
Benzo(a)anthracene	0.800
Benzo(a)pyrene	0.700
Benzo(b)fluoranthene	0.700
Benzo(g,h,i)perylene	0.500
Benzo(k)fluoranthene	0.700
Bis(2-choroethyl)ether	0.700
Bis (2-chloroethoxy)methane	0.300
Bis (2-ethylhexyl)phthalate	0.010
Butyl benzyl phthalate	0.010

Caprolactam	0.010
Carbazole	0.010
Analyte	**Minimum RRF Criteria**
Chrysene	0.700
Dibenz(a,h)anthracene	0.400
Dibenzofuran	0.800
Diethyl phthalate	0.010
Dimethyl phthalate	0.010
Di-n-butyl phthalate	0.010
Di-n-octyl phthalate	0.010
Fluoranthene	0.600
Fluorene	0.900
Hexachlorobenzene	0.100
Hexachlorobutadiene	0.010
Hexachlorocyclopentadiene	0.050
Hexachloroethane	0.300
Indeno(1,2,3-cd)pyrene	0.500
Isophorone	0.400
Naphthalene	0.700
Nitrobenzene	0.200
N-Nitroso-di-n-propylamine	0.500
N-Nitroso-di-phenylamine	0.010
Pentachlorophenol	0.050
Phenanthrene	0.700
Phenol	0.800
Pyrene	0.600

Attachment 12:
625.1 QC Acceptance Criteria

Table 6 – QC Acceptance Criteria – Method 625[1]					
Analyte	Range for Q (%)[2]	Limit for s (%)[3]	Range for $\bar{X}$ (%)[3]	Range for P_1, P_2(%)[3]	Limit for RPD (%)
Acenaphthene	70-130	29	60-132	47-145	48
Acenaphthylene	60-130	45	54-126	33-145	74
Aldrin	7-152	39	7-152	D-166	81
Anthracene	58-130	40	43-120	27-133	66
Benzo(a)anthracene	42-133	32	42-133	33-143	53
Benzo(b)fluoranthene	42-140	43	42-140	24-159	71
Benzo(k)fluoranthene	25-146	38	25-146	11-162	63
Benzo(a)pyrene	32-148	43	32-148	17-163	72
Benzo(ghi)perylene	13-195	61	D-195	D-219	97
Benzyl butyl phthalate	43-140	36	D-140	D-152	60
beta-BHC	42-131	37	42-131	24-149	61
delta-BHC	D-130	77	D-120	D-120	129
bis(2-Chloroethyl)ether	52-130	65	43-126	12-158	108
bis(2-Chloroethoxy)methane	52-164	32	49-165	33-184	54
bis(2-Chloroisopropyl) ether	63-139	46	63-139	36-166	76
bis(2-Ethylhexyl) phthalate	43-137	50	29-137	8-158	82
4-Bromophenyl phenyl ether	70-130	26	65-120	53-127	43
2-Chloronaphthalene	70-130	15	65-120	60-120	24
4-Chlorophenyl phenyl ether	57-145	36	38-145	25-158	61
Chrysene	44-140	53	44-140	17-168	87
4,4'-DDD	D-135	56	D-135	D-145	93
4,4'-DDE	19-130	46	19-120	4-136	77

[2] Test concentration = 100 µg/mL
[3] Test concentration = 100 µg/L
Q = Calibration verification (Sections 7.3.1 and 13.4)
s = Standard deviation for four recovery measurements in the DOC test (Section 8.2.4).
$\bar{X}$ = Average recovery for four recovery measurements in the DOC test (Section 8.2.4).
P_1, P_2 = MS/MSD recovery (Section 8.3.2, Section 8.4.2).
RPD = MS/MSD relative percent difference (RPD; Section 8.3.3).
D = Detected; result must be greater than zero.

Table 6 – QC Acceptance Criteria – Method 625[1]					
Analyte	Range for Q (%)[2]	Limit for s (%)[3]	Range for $\bar{X}$ (%)[3]	Range for P_1, P_2(%)[3]	Limit for RPD (%)
4,4'-DDT	D-171	81	D-171	D-203	135
Dibenz(a,h)anthracene	13-200	75	D-200	D-227	126
Di-*n*-butyl phthalate	52-130	28	8-120	1-120	47
3,3'-Dichlorobenzidine	18-213	65	8-213	D-262	108
Dieldrin	70-130	38	44-119	29-136	62
Diethyl phthalate	47-130	60	D-120	D-120	100
Dimethyl phthalate	50-130	110	D-120	D-120	183
2,4-Dinitrotoluene	53-130	25	48-127	39-139	42
2,6-Dinitrotoluene	68-137	29	68-137	50-158	48
Di-*n*-octyl phthalate	21-132	42	19-132	4-146	69
Endosulfan sulfate	D-130	42	D-120	D-120	70
Endrin aldehyde	D-189	45	D-189	D-209	75
Fluoranthene	47-130	40	43-121	26-137	66
Fluorene	70-130	23	70-120	59-121	38
Heptachlor	D-172	44	D-172	D-192	74
Heptachlor epoxide	70-130	61	71-120	26-155	101
Hexachlorobenzene	38-142	33	8-142	D-152	55
Hexachlorobutadiene	68-130	38	38-120	24-120	62
Hexachloroethane	55-130	32	55-120	40-120	52
Indeno(1,2,3-cd)pyrene	13-151	60	D-151	D-171	99
Isophorone	52-180	56	47-180	21-196	93
Naphthalene	70-130	39	36-120	21-133	65
Nitrobenzene	54-158	37	54-158	35-180	62
N-Nitrosodi-*n*-propylamine	59-170	52	14-198	D-230	87
PCB-1260	19-130	77	19-130	D-164	128
Phenanthrene	67-130	24	65-120	54-120	39
Pyrene	70-130	30	70-120	52-120	49
1,2,4-Trichlorobenzene	61-130	30	57-130	44-142	50
4-Chloro-3-methylphenol	68-130	44	41-128	22-147	73
2-Chlorophenol	55-130	37	36-120	23-134	61
2,4-Dichlorophenol	64-130	30	53-122	39-135	50
2,4-Dimethylphenol	58-130	35	42-120	32-120	58
2,4-Dinitrophenol	39-173	79	D-173	D-191	132
2-Methyl-4,6-dinitrophenol	56-130	122	53-130	D-181	203
2-Nitrophenol	61-163	33	45-167	29-182	55
4-Nitrophenol	35-130	79	13-129	D-132	131
Pentachlorophenol	42-152	52	38-152	14-176	86
Phenol	48-130	39	17-120	5-120	64
2,4,6-Trichlorophenol	69-130	35	52-129	37-144	58

[1] Acceptance criteria are based upon method performance data in Table 7 and from EPA Method 1625. Where necessary, limits for recovery have been broadened to assure applicability to concentrations below those used to develop Table 7.

[2] Test concentration = 100 µg/mL
[3] Test concentration = 100 µg/L
Q = Calibration verification (Sections 7.3.1 and 13.4)
s = Standard deviation for four recovery measurements in the DOC test (Section 8.2.4).
$\bar{X}$ = Average recovery for four recovery measurements in the DOC test (Section 8.2.4).
P_1, P_2 = MS/MSD recovery (Section 8.3.2, Section 8.4.2).
RPD = MS/MSD relative percent difference (RPD; Section 8.3.3).
D = Detected; result must be greater than zero.

Attachment 13:
Glassware Cleaning Procedures

GLASSWARE CLEANING PROCEDURES

SEMIVOLATILE GC/MS DEPARTMENT

1. Rinse glassware 3 times with methylene chloride
2. Place glassware, top-down, within storage rack and allow to air dry.
3. Store glassware in closed drawer.

FSM108:05.14.14:0

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18.0 Revision History

Summary of Changes for Revision 11:
- Revised water and soil container volumes to Section 8.0 and Attachment 2.
- Revised section 9.2.2 for current calibration levels.
- Added ICAL readback criteria to section 9.2.2.1.
- Added DODQSM5 criteria to section 9.2.5 and 9.2.8.2.
- Added 625.1,625 and DODQSM requirements to section 12.5 for DOC.
- Revised section 14.1 for current disposal procedures.
- Added reference to 8000C in Section 15.
- Added DODQSM criteria to Attachment 1 and Attachment 3.
- Added Attachment 12 for 625.1 Recovery Criteria and revised Glassware Cleaning Procedures to Attachment 13.

Summary of Changes for Revision 10:
- Addition of standard level in section 9.2.2 to be consistent with current calibration levels used in the laboratory.

Summary of Changes from Previous Revision:
-Updated SOP signatories to reflect current responsibilities and titles.
-Incorporated references to EPA 8270E.
-Incorporated the following changes as per SC DHEC Technical Deficiency Letter dated 05/18/19:
-Added note the SC DHEC prohibits the use of sporadic marginal exceedance. Section 9.1.1 and Section 16.2.4
-Corrected spelling of decafluorotriphenyl phosphine. Section 9.2.1
-Clarified calculation to reference waters. Section 11.2.3.
-Added requirement to evaluate %RSE. Section 9.2.2.1 and Attachment 3

VOLATILE COMPOUNDS BY GC/MS

(Methods: EPA 624, EPA 624.1, EPA 8260B, EPA 8260C, EPA 8260D and SM6200B)

Approvals (Signature/Date):

10/05/2021

Steven Proctor
Department Manager
Date

10/05/2021

Kimberly Chamberlain
Quality Assurance Manager / Environmental Health and Safety Coordinator
Date

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1.0 SCOPE and APPLICATION

This SOP contains the procedures for the determination of volatile organic compounds (VOC) by purge and trap gas chromatography/mass spectrometry (GC/MS). This procedure is applicable to a wide variety of low molecular weight compounds that have low aqueous solubility and boiling points less than 200°C.

The routine matrices for this procedure are waters (EPA 624, EPA 624.1, EPA 8260B, EPA 8260C, EPA 8260D and SM6200B) and soils (for EPA 8260B, EPA 8260C, and EPA 8260D); however, this procedure may be adapted to accommodate other matrices as outlined in Section 16.1. This procedure may also be used to perform Custer Rule analyses for chloroform only (via a modified EPA 624 analysis).

Note: This SOP incorporates state-specific requirements for EPA 8260B and EPA 8260C soils collected in Alaska. The term "EPA 8260_AK" is used in this SOP to designate those requirements that differ from the standard procedure, which include a medium-level sample collection procedure, an additional surrogate fortified into the methanol in the out-going sample container, state-specific reporting limits, and initial calibration concentration levels.

A complete target analyte list, the reporting limits (RL), the method detection limits (MDL), and the accuracy and precision criteria associated with this procedure are provided in the LIMS Method Limit Groups (MLGs). Attachment 8 outlines the routine analytes and the associated ions.

2.0 SUMMARY OF METHOD

Volatile organic compounds (VOCs) are purged from the sample matrix with nitrogen. The VOCs are transferred from the sample matrix to the vapor phase. The vapor is swept through a sorbent tube where the VOCs are trapped. After the purging is completed, the trap is heated and backflushed with helium to desorb the VOCs onto a GC column. The GC is temperature-programmed to separate the VOCs, which are then detected by a mass spectrometer. Qualitative identification of the target compounds in the sample is based on the relative retention time and the mass spectra of the characteristic masses (ions) determined from standards analyzed on the same GC/MS under the same conditions. Quantitative analysis is performed using the internal standard technique with a single characteristic ion.

Water samples are routinely purged at ambient conditions; however, a heated purge may be used if required by the project. A 5mL purge volume is used as the default for EPA8260, EPA 624 and EPA 624.1. The calibration standards and the associated QC must be analyzed under the same conditions and volume. This sample introduction procedure is based on EPA 5030B, EPA 5030C, and EPA 624. For SM6200B a 20mL purge volume is utilized as the default.

Low-level (nominally<1mg/kg) soil samples are purged at 40°C in a purge and trap instrument designed to add water and internal standards to the vial containing the sample

without breaking the seal. The sample is stirred during purging to thoroughly mix the soil and water. The calibration standards and associated QC are purged under the same conditions. This sample introduction procedure is based on EPA 5030B or 5030C (for soil samples collected in bulk) and EPA 5035 or EPA 5035A (for soil samples collected via Encore/Terracore devices).

Medium level soils (nominally>1mg/kg), waste samples, and soil samples for EPA 8260_AK are extracted with methanol. An aliquot of the methanol extract is injected into reagent water. The methanol extract/reagent water is purged at a heated temperature (i.e., 40°C) for routine soils and at ambient temperature for wastes and EPA 8260_AK.

This SOP is based on EPA 624, EPA 624.1, EPA 8260B, EPA 8260C, EPA 8260D and SM6200B.

3.0 DEFINITIONS

Refer to the Glossary Section of the *Quality Assurance Manual* (QAM) for a complete listing of applicable definitions and acronyms.

4.0 INTERFERENCES

4.1 Procedural Interferences

4.1.1 Interferences may be caused by contaminants in solvents, reagents, glassware, and other sample processing apparatus and can make identification and/or quantification of the target analytes difficult.

4.1.2 All sample collection containers are single-use disposable containers which limits the potential for contamination. All non-disposable labware must be scrupulously cleaned in accordance with the posted Labware Cleaning Instructions (found in Attachment 14) to ensure it is free from contaminants and does not contribute artifacts.

4.1.3 High purity reagents and solvents are used to help minimize interference problems. Methanol and Hydrochloric Acid must be verified prior to use in accordance with the Eurofins TestAmerica Solvent Lot Testing Program.

4.1.4 Instrument and/or method blanks are routinely used to demonstrate all reagents and apparatus are free from interferences under the conditions of the analysis.

4.1.5 VOCs commonly used in the laboratory are potential sources of contamination. Methylene chloride, acetone, Freon-113, MEK, hexane, toluene, and isopropanol are used in the laboratory and tend to present the most problems.

4.1.5 The Teflon seals of the purge and trap device can absorb and outgas many of the compounds that are included in this method. These Teflon fittings should be periodically checked for integrity. If the contamination is suspected, the fittings may be heated at 105°C for one hour or replaced.

4.1.6 The addition of acid to the sample during collection will cause the degradation of several target compounds. Acrolein and acrylonitrile recovery may be reduced and 2-chloro-ethyl vinyl ether will be completely degraded. The recovery of 2-chloroethyl vinyl ether will also be reduced as the purge and trap lines become acidic. For this reason, unpreserved vials must be utilized if these analytes are requested.

4.2 Matrix Interferences

4.2.1 Matrix interferences may be caused by contaminants that are purged from the sample matrix.

Contamination by carryover can occur whenever high concentration samples and low concentration samples are analyzed sequentially. As such, samples known to be clean should be analyzed first. When practical, high concentration samples should be followed by a blank to check for cross-contamination. If the targets found in the highly concentrated sample are found in subsequent samples, the analyst must verify that the P/T systems are not contributing contamination to the subsequent samples. If the target compound(s) are not present in subsequent samples, the analysis of a blank is not required but may be a prudent preventative measure. Frequent trap bakeout and purging of the entire purging system may be necessary when carry-over is suspected. Reagent blanks must be analyzed when contamination is suspected to ensure that the system is free from contamination.

A common type of contamination is from samples containing high concentrations of hydrocarbons such as gasoline or mineral spirits. At high concentrations, these compounds may cause elevated baselines that can obscure the mass ion signals of target compounds with masses similar to the hydrocarbons. For example, a common mass in hydrocarbons is mass 43, which is also present in many ketones. Mineral spirits at high concentrations can be very problematic as it contains hydrocarbons beyond C12, which can linger in the purge and trap unit and carryover for quite a few samples. High concentrations of hydrocarbons can also degrade the trap much more quickly than would be expected.

4.2.3 The volatiles laboratory must be kept as free from contamination as possible. Highly contaminated samples must be segregated from routine samples. Contact with sections of the laboratory where solvents are used should be minimized. Refrigerator and freezer blanks must be prepared, stored, and analyzed to evaluate the sample storage areas for possible contamination. Guidance is provided in SOP SA-QA-015: *Homogenization, Compositing, and Segregation of Samples.*

4.2.4 Matrix interferences may be overcome by the use of the secondary ions for quantitation. An example of this is the use of mass 82 for quantitation with chlorobenzene-d5 internal standard when a potential co-eluter, 1,1,1,2-tetrachloroethane, is a target compound. One of the mass fragments of 1,1,1,2-tetrachloroethane is mass 117, which is the recommended quantitation ion for chlorobenzene-d5. The use of the secondary ions should be used for quantitation in such cases when the laboratory can clearly demonstrate matrix problems. Mass 58 is recommended for quantitation of acetone due to the elution of a hydrocarbon at the same retention time.

5.0 SAFETY

Employees must abide by the policies and procedures in the Eurofins TestAmerica Environmental Health and Safety Manual (EHSM), the Eurofins TestAmerica Savannah Addendum to the EHSM, and this document.

This procedure may involve hazardous materials, operations, and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user to follow appropriate safety, waste disposal, and health practices under the assumption that all samples and reagents are potentially hazardous.

The analyst must protect himself/herself from exposure to the sample matrix. Many of the samples that are tested may contain hazardous chemical compounds or biological organisms. The analyst must, at a minimum, wear protective clothing (lab coat), eye protection (safety glasses or face shield), disposable gloves, and closed-toe, nonabsorbent shoes when handling samples. Note: Cut-resistant gloves must be worn, or other hand protection material used, when opening and closing VOA vials.

5.1 Specific Safety Concerns or Requirements

The gas chromatograph and mass spectrometer contain zones that have elevated temperatures. The analyst must be aware of the locations of those zones, and must cool them to room temperature prior to working on them.

The mass spectrometer is under deep vacuum. The mass spectrometer must be brought to atmospheric pressure prior to working on the source.

There are areas of high voltage in both the gas chromatograph and the mass spectrometer. Depending on the type of work involved, either turn the power to the instrument off, or disconnect it from its source of power.

The exit vent of the split injector must have a carbon trap in-line to collect the volatile compounds that are vented during the injection of the sample. The traps should be changed as needed and must be disposed of in accordance with Section 9 of the Eurofins TestAmerica Savannah Addendum to the EHSM.

Methanol is a flammable solvent. It can cause irritation to the respiratory tract. Overexposure can cause fatigue, confusion, headache, dizziness, and drowsiness.

Hydrochloric acid is extremely hazardous as an oxidizer, a corrosive, a poison, and is reactive. Inhalation of the vapors can cause coughing, choking, irritation of the nose, throat, and respiratory tract, breathing difficulties, and lead to pneumonia and pulmonary edema. Contact with the skin can cause severe burns, redness, and pain. Acid vapors are irritating and can cause damage to the eyes. Contact with the eyes can cause permanent damage. Concentrated acids should be used in a fully functional fume hood.

5.2 Primary Materials Used

The following is a list of the materials used in this procedure, which have a serious or significant hazard rating, and a summary of the primary hazards listed in their SDS/MSDS.

Note: This list does not include all materials used in the procedure. A complete list of materials used in this procedure can be found in the Reagents and Standards Section and the Equipment and Supplies Section of this SOP

Employees must review the information in the SDS/MSDS for each material before using it for the first time or when there are major changes to the SDS/MSDS. Electronic copies of SDS/MSDS can be found using the "MSDS" link on the Oasis homepage and on the EH&S webpage on Oasis.

Material	Hazards	Exposure Limit[1]	Signs and Symptoms of Exposure
Methanol	Flammable Poison Irritant	200ppm TWA	A slight irritant to the mucous membranes. Toxic effects exerted upon nervous system, particularly the optic nerve. Symptoms of overexposure may include headache, drowsiness and dizziness. Methyl alcohol is a defatting agent and may cause skin to become dry and cracked. Skin absorption can occur; symptoms may parallel inhalation exposure. Irritant to the eyes.
Hydrochloric Acid	Corrosive Poison	5ppm Ceiling	Inhalation of vapors can cause coughing, choking, inflammation of the nose, throat, and upper respiratory tract, and in severe cases, pulmonary edema, circulatory failure, and death. Can cause redness, pain, and severe skin burns. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
[1]Exposure limit refers to the OSHA regulatory exposure limit.			
Note: Always add acid to water to prevent violent reactions.			

6.0 EQUIPMENT AND SUPPLIES

6.1 Equipment and Instrumentation

A list of the instruments, with their basic configuration, is provided in Attachment 9. These instruments were in use at the time of SOP revision. Other instruments and configurations may be used provided they are fully documented and validated in accordance with laboratory procedures.

Top-loading Balance – Verify in accordance with SOP SA-AN-100: *Laboratory Support Equipment (Verification and Use)*

6.2 Analytical Data System / Software / Hardware

Chemstation software is used on a Windows-based PC to schedule and acquire data. CHROM software is used on a Windows-based PC to store, reduce/evaluate, and output the data to the laboratory's LIMS system. CHROM software has the capability of processing stored GC/MS data by recognizing a GC peak within any given retention time window, comparing the mass spectrum from the GC peak with spectral data in a user-created data base, and generating a list of tentatively identified compounds with their retention times and scan numbers. The software also allows integration of the ion abundance of any specific ion between specified time or scan number limits, calculation of response factors as or construction of a linear regression calibration curve, calculation of response factor statistics (mean and standard deviation), and calculation of concentrations of analytes using either the calibration curve or the response factors.

The QA department maintains a Master Equipment Listing for all equipment utilized by the laboratory. This listing provides information such as the model, serial number and the software or firmware utilized.

6.3 Lab Supplies

Supelco Vocarb 3000 trap or equivalent – Other traps may be used as long as the target compounds can be detected at the required quantitation limit.

Recommended Column: J&W DB-624: 20m x 0.18mm ID, 1.8um film

6.4 Volumetric Containers and Dispensers

All volumetric labware must be verified in accordance with SOP SA-AN-100: *Laboratory Support Equipment (Verification and Use).* Refer to Attachment 6 for Labware Cleaning Procedures.

Volumetric Labware	Volume	Type (Quantitative / Qualitative)	Use	Verification Frequency	Laboratory Verification Criteria
Volumetric Flasks (Class A)	Various	QUANTITATIVE	Standard Preparation	None (Purchased Class A)	None (Purchased Class A)
Gas-Tight Syringes	Various	QUANTITATIVE	Standard Preparation Spike Addition	None, if received with COA	None, if received with COA
Pump-Style Mechanical Pipettes	5mL	QUANTITATIVE	Reagent/Water Dispensing	Initially, prior to first use, then quarterly thereafter (Daily for DOD)	Accuracy = 2% Precision = 1%
VOA Vials*	40mL	QUANTITATIVE	Sample Collection Instrument Analysis	Per Lot	Accuracy = 2% Precision = 1%

*Note: The VOA vials used to collect samples have been verified to actually contain closer to 43mL. As such, spiking amounts for ISSU have been adjusted accordingly for instruments that are unable to automatically add ISSU during the sample transfer step of the autosampler. Standards are prepared in 50mL volumetric containers, and poured into the VOA vial for analysis on the instrument.

6.5 Sample Collection Containers

All sample collection containers are single-use disposable containers which limits the potential for contamination. Containers are received from vendors with a statement that the containers are suitable for the intended use and have been tested and certified to be free of contamination.

Refer to SOP SA-VO-001: *Preparation, Screening, and Storage of Volatiles Samples* for the containers routinely used to collect field samples.

7.0 REAGENTS AND STANDARDS

7.1 Expiration Dates

Expiration dates (time from initial use or receipt to final use) for standard and reagent materials must be set according to the guidance in this SOP. Note: These are maximum expiration dates and are not to be considered an absolute guarantee of standard or reagent quality. Sound judgment must be used when deciding whether to use a standard or reagent. If there is doubt about the quality of a standard or reagent material, a new material must be obtained or the standard or reagent material verified. Data quality must not be compromised to extend a standard's life.

The expiration date of any standard must not exceed the expiration date of the standard that was used to prepare it.

7.2 Reagents

Reagents must be prepared and documented in accordance with SOP SA-AN-041: *Reagent and Standard Materials Traceability.*

Methanol and Hydrochloric Acid must be verified prior to use in accordance with the Eurofins TestAmerica Solvent Lot Testing Program addressed in SOP SA-AN-041.

7.2.1 Reagent water – ASTM Type II; lab generated water

7.2.2 Blank Sand – Macro Chemicals; used as blank matrix for soil samples

7.2.3 Methanol – for purge and trap analysis; J.T. Baker 9077-02 (1L)

7.3 Standards

Standards must be prepared and documented in accordance with SOP SA-AN-041*: Reagent and Standard Materials Traceability.* Certificates of analysis or purity must be received with all purchased standards, and scanned and attached in LIMS.

The recipes for the preparation of standards are provided in Attachment 10. The recipes contain the stock standards, preparation steps, storage, and expiration dates for the routine target compounds.

8.0 SAMPLE COLLECTION, PRESERVATION AND HOLDING

Refer to Attachment 2 for a summary of the routine containers, holding times, preservation requirements, etc.

Refer to SOP SA-VO-001: *Preparation, Screening, and Storage of Volatiles Samples* for information on the preservation and dechlorination checks required for these methods.

9.0 QUALITY CONTROL

SOP SA-QA-017: *Evaluation of Batch QC Data* and the QC Summary in Attachment 3 provide requirements for evaluating QC data.

9.1 Batch QC

All batch QC must meet the criteria given in Attachment 3 of this SOP.

9.1.1 Soil Samples

A batch consists of up to 20 environmental samples and the associated QC items. The laboratory defaults to the following as the minimum QC items required for each batch: a method blank (MB), a laboratory control sample (LCS), a matrix spike (MS), and a matrix spike duplicate (MSD).

If there is insufficient sample to perform the MS/MSD, the LCS must be prepared in duplicate (i.e., LCS/LCSD). An NCM must be initiated on all affected samples to denote this situation. Insufficient sample is defined as receiving less than 30g for bulk samples or less than 4 Encores/Terracores.

Note: If an LCS and LCSD are performed, both QC items must be evaluated and reported. Acceptable recoveries (as well as %RPD) for both LCS and LCSD are required.
Note: Unless the client has specified which sample to use as the matrix spike, the matrix spike must be selected at random from the samples in the batch. Field QC (e.g., equipment blanks, trip blanks, and field blanks) must not be used for MS/MSD unless specifically requested to do so by the client or unless there is insufficient sample available for performing MS/MSD using an actual field sample.

9.1.2 Aqueous Samples

A batch consists of up to 20 environmental samples and the associated QC items. The minimum QC items required for each batch are as follows:

EPA 8260B, EPA 8260C, EPA 8260D and SM6200B:
Method blank (MB), a laboratory control sample (LCS), a matrix spike (MS), and a matrix spike duplicate (MSD).

EPA 624 and EPA 624.1:
Method blank (MB), a laboratory control sample (LCS), a matrix spike (MS) performed per 10% of samples analyzed, and a matrix spike duplicate (MSD). This equates to 1 MS and 1MSD for a batch of 10 or less samples or equates to 1 MS (from sample 1-10), 1 MS (from sample 11-20), and 1 MSD for a batch of 11-20 samples.

If there is insufficient sample to perform the required MS and/or MSD, the LCS must be prepared in duplicate (LCSD). An NCM must be initiated on all affected samples to denote when insufficient sample is provided to perform a MS/MSD. Insufficient sample is defined as receiving less than less than 4 vials.

Note: It is not uncommon to for the laboratory to be supplied with insufficient containers to perform MS/MSD; therefore, the laboratory routinely performs LCS/LCSD as a default. If an LCS and LCSD are performed, both QC items must be evaluated and reported. Acceptable recoveries (as well as %RPD) for both LCS and LCSD are required.

Note: Unless the client has specified which sample to use as the matrix spike, the matrix spike must be selected at random from the samples in the batch. Field QC (e.g., equipment blanks, trip blanks, and field blanks) must not be used for MS/MSD unless specifically requested to do so by the client or unless there is insufficient sample available for performing MS/MSD using an actual field sample.

9.1.3 Poor Performers / Erratic Compounds

As indicated in EPA 8260B, EPA 8260C and EPA 8260D and/or via assessment of laboratory control sample recoveries and control charts, the compounds listed in Attachment 11 are Poor Performers and/or behave erratically. T
hese compounds will not be included in the LCS/LCSD/MS/MSD marginal exceedance count, provided their %R is >10%.
Refer to SOP SA-QA-017 for information on marginal exceedance.

An NCM must be initiated to denote this situation.

Note: DOD QSM 5.3 does not identify poor performing analytes for EPA 8260. The laboratory will allow the use of the poor performer exception in LCS/LCSD/MS/MSD if the analytes are not identified as a compound of concern by the client.

Note: The state of South Carolina does not allow the use of marginal exceedances. Poor performers must meet a limit of 60-140%. South Carolina UST work must meet a limit of 70-130%.

9.2 Instrument QC

The term “clock time” or “analytical clock” refers to the amount of time that can pass before additional instrument QC items must be performed. The analytical clock begins with the injection of the BFB, and all subsequent injections must be completed before the clock time expires - at which point new instrument QC is performed and a new clock is initiated.

The clock times are defined as follows:

- EPA 8260B, EPA 8260C, EPA 8260D, and SM6200B = 12 hours
- EPA 624 = 24 hours
- EPA 624.1= 12 hours (clock time starts after the analysis of the MB but may not exceed 14 hours total)
- EPA 624 Cluster Rule (chloroform only) = 8 hours

Note: Due to instrument configurations employing dual concentrators, most of the laboratory instruments can analyze more than 20 injections within the designated clock times. An analytical batch is still defined as 20 field samples; therefore, if more than 20 field samples are analyzed within a clock, additional batch QC is required (i.e., another method blank, LCS, and LCSD or MS/MSD must be performed).

9.2.1 BFB Tune Check

9.2.1.1 For EPA 8260B, EPA 8260C, EPA 8260D, EPA 624, and EPA 624.1, Fifty nanograms of 4-BFB must be analyzed at the beginning of each clock as a check on the "tune" of the mass spectrometer. Meeting the tuning criteria ensures that the instrument is measuring the proper masses in the proper ratios. The 4-BFB analysis takes place under the same instrument conditions as the calibration standards and samples except that a different temperature program can be used to allow for the timely elution of 4-BFB. All other

instrument conditions must be identical - the mass range, scan rate, and multiplier voltage.

If the instrument is configured for direct injection, 50ng of 4-BFB may be injected directly on to the column. If the purge and trap is used to analyze the 4-BFB, the purge and trap conditions must be the same as for the calibration standards and samples.

For SM6200B, twenty-five nanograms of 4-BFB must be analyzed at the beginning of each clock as a check on the "tune" of the mass spectrometer. Meeting the tuning criteria ensures that the instrument is measuring the proper masses in the proper ratios. The 4-BFB analysis takes place under the same instrument conditions as the calibration standards and samples except that a different temperature program can be used to allow for the timely elution of 4-BFB. All other instrument conditions must be identical - the mass range, scan rate, and multiplier voltage.
If the instrument is configured for direct injection, 25ng of 4-BFB may be injected directly on to the column. If the purge and trap is used to analyze the 4-BFB, the purge and trap conditions must be the same as for the calibration standards and samples.

9.2.1.2 Evaluation of the 4-BFB peak

9.2.1.2.1 The chromatogram must exhibit acceptable baseline behavior and the 4-BFB peak must be symmetrical (i.e., Gaussian). A spectrum of the baseline that shows high abundances of mass 40 (Argon) and mass 44 (carbon dioxide) may indicate a leak or contaminated carrier gas.

9.2.1.2.2 The spectrum of the 4-BFB must meet the criteria listed in Attachment 7. Background subtraction must be straightforward and designed only to eliminate column bleed or instrumental background. Scans ±1 scan from the apex can be evaluated for the 4-BFB criteria. Consecutive scans within this range can be averaged to meet the criteria.

9.2.1.2.3 The 4-BFB analysis must be evaluated as to the relative size of the 4-BFB peak under the m/z 95 profile. A benchmark area window must be established for each instrument. Response outside of this window suggests instrumental problems such as a poor purge, clogged jet separator, leak in the Tekmar purging device, reduced or elevated detector sensitivity, improper electron multiplier voltage selection, wrong tune method or tune file selected for this analysis, PFTBA valve left open, or other anomalies.

9.2.1.2.4 If the 4-BFB fails to meet the acceptance criteria, the instrument may require tuning (manually or automatically with PFTBA). Depending on the nature of the results from the 4-BFB analysis, other corrective measures may include remaking the 4-BFB standard and/or cleaning the mass spectrometer source.

9.2.2 Trap Check Standard (Optional)

The trap check standard is used to evaluate the condition of the trap by monitoring the formation of chloromethane and bromomethane. Chloromethane and bromomethane may be formed on a degraded trap by thermal decomposition of halogenated compounds.

9.2.2.1 Prepare the trap check standard by injecting 2uL of a 50ug/mL bromoform standard into 5mL of reagent water. Other sample volumes may be used but the sample must transfer 100ng of bromoform to the column. Add the internal standards and surrogates. Analyze the sample using the same analytical system conditions used for samples and standards.

9.2.2.2 Evaluate the chromatogram for the presence of chloromethane and bromomethane. Compare the response to the 5.0 ug/L standard. The response must be less than or equal to the reporting limit.

Note: Ensure that the spectra match the reference spectra and that the most abundant ions are present for both compounds - chloromethane (m/z 50, 52) and bromomethane (94, 96).

9.2.2.3 If the trap check standard does not meet the acceptance criteria, the trap must be replaced and conditioned.

9.2.3 Initial Calibration (ICAL)

The instrument must be calibrated in accordance with SOP SA-QA-016: *Evaluation of Calibration Curves.* This SOP provides requirements for establishing the calibration curve and gives the applicable formulas.

Instrument calibration is performed by analyzing a series of known standards. The calibration curve must consist of a minimum of 5 standards. The lowest level calibration standard must be at or below the reporting limit, and the remaining standards will define the working range of the analytical system. A minimum of 6 standard is required for a non-linear or quadratic regression.

The initial calibration standard concentrations currently in use in the laboratory are as follows:

EPA 624, 624.1, 8260B, EPA 8260C and EPA 8260D:

Standard Level	Concentration (ng)	Final Concentration – Waters (ug/L)	Final Concentration – Soils (ug/kg)
1	5	1.0	5.0
2	10	2.0	10
3	25	5.0	25
4	50	10	50*
5	100	20	75
6	250	50*	100
7	500	100	150
8	1000	200	200

*CCV Level

SM6200B:

Standard Level	Concentration (ng)	Final Concentration – Waters (ug/L)
1	2.5	0.5
2	5	1.0
3	12.5	2.5
4	25	5
5	50	10
6	100	20
7	125	25
8	250	50

*CCV- Alternate throughout the batch

EPA 8260_AK Soils
(Incorporates 1:40 dilution associated with the methanol extraction)

Standard Level	Concentration (ng)	Final Concentration – Soils (ug/kg)
1	0.50	20
2	1.0	40
3	2.0	80
4	5.0	200
5	10	400*
6	25	1000
7	50	2000

*CCV Level

Refer to Attachment 10 for the standard preparation instructions. Other standard concentrations may be used provided they support the reporting limit and are fully documented in accordance with SOP SA-AN-041.

9.2.3.2 Initial Calibration (ICAL) Criteria

The instrument must be calibrated in accordance with SOP SA-QA-016: *Evaluation of Calibration Curves.* This SOP provides requirements for establishing the calibration curve and evaluation.

9.2.3.2.1 EPA 8260B ICAL:

The initial calibration is evaluated specifically for the calibration check compounds (CCC) and the system performance check compounds (SPCC). The CCC and SPCC criteria are given in Attachment 5 of this SOP. The %RSD criteria for CCC and minimum RRF for SPCC must be met before the analysis of samples can begin.

After the CCC and SPCC initial calibration criteria have been met, each target must be evaluated for linearity. The relative standard deviation of the calibration standards must be <15% for the initial calibration curve to be acceptable.

If one or more compounds do not meet the %RSD criterion, the next option is to evaluate a regression curve. The regression coefficient (r^2) of the regression curve must be greater than 0.990 for the initial calibration curve to be acceptable.

A minimum of 6 points is required for a quadratic curve. Higher order curves are not permitted.

Readback Criteria:
EPA 8260B does not specify read-back criteria. The laboratory requires that the low-level standard recover within 50% of true value and that all other calibration levels be within +/- 30% of the true concentration.

Note: Some programs and agencies (e.g., SC DHEC) do not allow the use of quadratic curves. Refer to the Project Requirement Summary and/or Project Plan to determine if this curve type is prohibited.

9.2.3.2.2 EPA 8260C and EPA 8260D ICAL:

RSD and regression Criteria: The relative standard deviation (RSD) must be less than 20% difference. If a compound exceeds 20% RSD, a regression curve must be employed. The regression coefficient (r^2) of the regression curve must be greater than 0.990 for the initial calibration curve to be acceptable.

Note: A minimum of 6 points is required for a quadratic curve. Higher order curves are not permitted. Some programs and agencies (e.g., SC DHEC) do not allow the use of quadratic curves. Refer to the Project Requirement Summary and/or Project Plan to determine if this curve type is prohibited.

RRF Criteria: A list of compounds with minimum relative response factor criteria is listed in Attachment 6. RRF criteria must be met for EPA 8260C and is advisory for EPA 8260D.

Note: 10% of the analytes in the calibration may be outside criteria. If analytes do meet criteria, they are considered estimated. An NCM must be initiated to denote analytes that do not meet the RSD or r^2 criteria.

RLCCV Criteria: For any analyte associated with a calibration or minimum response factor, an RLCCV must be analyzed with each subsequent clock. The analytes must be detected in the RLCCV analysis to be reported.
*An NCM must be initiated to denote this situation. Any positive results must be noted as estimated.

Readback Criteria:

EPA 8260C: All calibration levels be within +/- 30% of the true concentration.

EPA 8260D: Lowest calibration level must within 50% of the true concentration. All other levels must be within 30% of the true concentration.

9.2.3.2.3 EPA 624 and 624.1 ICAL

The relative standard deviation of the calibration standards must be <35% for the initial calibration curve to be acceptable. If one or more compounds do not meet the %RSD criterion of <35%, the next option is to evaluate a regression curve. The regression coefficient (r^2) of the regression curve must be greater than 0.990 for the initial calibration curve to be acceptable.

Readback Criteria:
The method does not state a requirement for readback. The laboratory requires that the low-level standard recover within 50% of true value and that all other calibration levels be within +/- 30% of the true concentration.

Note: EPA 624.1 allows a regression coefficient (r^2) of greater than 0.920 for the initial calibration curve to be acceptable. The lab has chosen to use stricter criteria. The regression coefficient (r^2) of the regression curve must be greater than 0.990 for the initial calibration curve to be acceptable.

9.2.3.2.4 SM6200B ICAL:

The relative standard deviation of the calibration standards must be <20% for the initial calibration curve to be acceptable. If one or more compounds do not meet the %RSD criterion of <20%, the next option is to evaluate a regression curve. The regression coefficient (r^2) of the regression curve must be greater than 0.994 for the initial calibration curve to be acceptable

Readback Criteria:
All points must be within 20% of the true value.

9.2.3.2.5 8260 DOD QSM 5.3 ICAL

The relative standard deviation (RSD) of the calibration standards must be ≤ 15%. If one or more compounds do not meet %RSD of ≤15% then a regression curve must be evaluated. The linear or quadratic regression coefficient (r^2) of the regression curve must be greater than or equal to 0.990 for the initial calibration curve to be acceptable.

Read-back Evaluation must be evaluated based on the source method utilized at login.

If EPA 8260B method version is utilized, the CCC and SPCC criteria must also be met.

If the method version requires RRF or RLCCV analysis, these additional requirements must be met (ie EPA 8260C and EPA 8260D).

DOD QSM5.3 does not address analytes that may be considered poor/erratic performers. The laboratory allows a **30%D** for these analytes listed in Attachment 11 only if the compounds are not identified as critical components of concern by the client.

9.2.4 Second Source Initial Calibration Verification (ICV)

The calibration curve must be verified initially – prior to any sample analyses – in accordance with SOP SA-QA-016 with a standard obtained from a second source.

The initial calibration verification standard concentration currently in use in the laboratory is equivalent to the CCV concentration. Refer to Attachment 10 for the standard preparation instructions. Another standard concentration may be used provided it is mid-level and fully documented in accordance with SOP SA-AN-041.

9.2.4.1 EPA 8260B ICV Criteria

All CCC compounds must be within 20% D with remaining analytes within 30%D for the ICV to be acceptable.

The analytes listed in Attachment 11 behave erratically and/or are poor performers. These analytes are allowed a %D >30%, but must be <50% to be acceptable. An NCM must be initiated to denote this situation.

9.2.4.2 EPA 8260C and EPA 8260D ICV Criteria

All compounds must be within 30%D for the ICV to be acceptable.

The analytes listed in Attachment 11 behave erratically and/or are poor performers. These analytes are allowed a %D >30%, but must be <50% to be acceptable.
*An NCM must be initiated to denote this situation.

9.2.4.3 EPA 624 and EPA 624.1 ICV Criteria

The initial calibration verification (ICV) for EPA 624 is acceptable if each analyte meets the criteria outlined in Attachment 12. The initial calibration verification (ICV) for EPA 624.1 is acceptable if each analyte meets the criteria outline in Attachment 13.

9.2.4.4 SM6200B ICV Criteria

All compounds must be within 30%D for the ICV to be acceptable. Analysis may not proceed until ICV criteria is met.

9.2.4.5 8260 DOD QSM 5.3 ICV Criteria

All compounds must be within 20%D for the ICV to be acceptable. Analysis may not proceed until ICV criteria is met.

DOD QSM5.3 does not address analytes that may be considered poor/erratic performers. The laboratory allows a **30%D** for these analytes listed in Attachment 11 only if the compounds are not identified as critical components of concern by the client.

9.2.5 **Continuing Calibration Verification (CCV)**

The initial calibration curve must be verified at the beginning of each clock with a mid-level standard.

The continuing calibration verification standard concentration currently in use in the laboratory is specified in Section 9.2.3. Refer to Attachment 10 for the standard preparation instructions. Another standard concentration may be used provided it is mid-level and fully documented in accordance with SOP SA-AN-041.

9.2.5.1 EPA 8260B CCV Criteria

For EPA 8260B, the CCC and SPCC criteria (Attachment 5) must be met for the CCV to be acceptable. For non-CCC and non-SPCC compounds, %D must be within 50 %D or the CCV to be acceptable.

Note: The lab will report samples with a reduced list of analytes with CCC analyte(s) outside 20%D provided that ALL reported analytes are ≤ 20%D in the CCV.
* An NCM will be initiated to denote this situation.

Note: The SPCC criteria must be met even if the regression curve option is used for quantitation.

9.2.5.2 EPA 8260C and EPA 8260D CCV Criteria

Response Factor Criteria:
EPA 8260C only, the minimum response factor criteria (RRF) as specified in Attachment 6 must be met. For EPA 8260D, the minimum response factor criteria are recommended and not required.
If the minimum RRF criteria are not met, sample analysis may proceed after the analysis of an RLCCV to demonstrate sensitivity.
*An NCM must be initiated for any detections associated with an RRF failure.

%D Criteria:
All analytes must be ≤ 20%D.

The reference method makes the exception, however, such that due to the large numbers of analytes in each CCV, some analytes may not meet these criteria.

-If more than 20% of the analytes in a CCV exceed the %D criteria, the CCV is unacceptable, and re-analysis is required.

- If less than 20% of the analytes in a CCV exceed the %D criteria, analyze an RLCCV, a standard whose concentration is at or below the reporting limit.
  Analytes that do not meet CCV %D criteria may be reported if the affected analytes are qualitatively identified in the RL CCV.
  - If the affected analyte is not detected in the associated client sample, the result is reported without qualification.
  - If the affected analyte is detected in the associated client sample, the result must be reported as estimated.
  - **Note: An NCM must be initiated to denote this situation.**

If the CCV recovery is outside 20%D, high biased, the laboratory will report data if analytes are not-detected in samples.
* An NCM must be initiated to denote this situation.

Note: SW-846 does not put a cap on an individual analyte's %D; however, the laboratory has the requirement that no individual analyte can exceed 50%D.

9.2.5.2 SM6200B CCV Criteria

Compounds must be within 30% of the true value (%difference for average or %drift for regression fits). Compounds identified as gases must be within +/- 40% D.
SM6200B also requires a CCV at the end of the clock to demonstrate instrument performance at the end of the batch. The capping CCV (CCVC) has the same limits as the opening CCV.

Note: The CCV concentration analyzed must be varied over the range of the calibration. Minimum concentration utilized should be two times the reporting limit (RL).

9.2.5.4 EPA 624 and EPA 624.1 CCV Criteria

For EPA Method 624, all target analytes must meet the criteria outlined in Attachment 12 to be acceptable. For EPA 624.1, all target analytes must meet the criteria outlined in Attachment 13.

9.2.5.5 DOD QSM 5.3 CCV Criteria

A CCV is required before sample analysis; after every 12 hours of analysis time; and at the end of the analytical batch run.
For the opening CCV, all target analytes and surrogates must within 20% of the true value.
For the closing CCV (CCCV), all target analytes and surrogates must be within 50%D.

If utilizing 8260C or 8260D, the response factor criteria must be met as defined in section 9.2.5.2.

Corrective Action if CCV does not meet acceptance: Immediately analyze two additional consecutive CCVs. If both pass, samples may be reported without re-analysis. If either fails or if consecutive CCVs cannot be run, repeat analysis of all associated samples.

9.2.5.5 CCV Poor Performers- EPA 8260B, EPA 8260C, EPA 8260D

Refer to Attachment 11 for the identification of poor and/or erratic performing analytes. These analytes are allowed to exceed the limit of 50%D , but must have a maximum %D limits of 100%D. If there are poor performers that exceed 50%D, the data may be reported and results are noted as estimated. The poor performers must be counted in the total number of allowances within a CCV analysis
****An NCM must be initiated to denote this situation**.

Note: DOD QSM 5.3 does not identify or address analytes that may have erratic response and be considered poor performers. The laboratory allows a 30%D for these analytes listed in Attachment 11 only if the compounds are not identified as critical components of concern by the client.

9.3 Internal Standard (ISTD)

9.3.1 EPA 8260B, EPA 8260C, EPA 8260D, EPA 624, EPA 624.1:

This procedure is an internal standard (ISTD) procedure. Fluorobenzene, 1,4-dichlorobenzene-d4, and chlorobenzene-d5 are the internal standards.

Prior to analysis, the internal standards must be added to all standards, samples, and QC items. The concentration of the internal standards must be the same in all calibration samples, field samples, and QC samples. A concentration of 50ug/L is used for each of the ISTD analytes.

The response of the ISTD in the ICV/CCV must be within -50% to +100% of the response of the ISTD in the CCV-level standard in the initial calibration sequence (denoted as ICIS), If the response is outside of this range, the analysis of the CCV must be repeated and any samples associated with the CCV must also be re-analyzed. Repeated failure of the ISTD response will require re-calibration.

The response of the ISTD in the samples and batch QC items must be within -50% to +100% of the response of the CCVIS. If the response is outside of this range, corrective action must be taken.

9.3.2 DOD QSM 5.3 ISTD Criteria

The response of the ISTD in the ICV/CCV must be within -50% to +100% of the response of the ISTD in the CCV-level standard in the initial calibration sequence which is denoted as ICIS.

The ISTD must elute within +/- 10 seconds from the retention time of the midpoint standard of the calibration, which is denoted as ICIS.

If the response or retention time is outside of this range, the analysis of the CCV must be repeated and any samples associated with the CCV must also be re-analyzed. Repeated failure of the ISTD response will require re-calibration.

On days when ICAL is not performed, the daily CCV, denoted as CCVIS, is used. The response of the ISTD in the samples and batch QC items must be within -50% to +100% of the response of the CCVIS. The ISTD must elute within +/10 seconds from the retention time of the CCVIS. If the response or retention time is outside of this range, corrective action must be taken.

9.3.3 SM6200B ISTD criteria:

This procedure is an internal standard (ISTD) procedure. Fluorobenzene, 1,4-dichlorobenzene-d4, and chlorobenzene-d5 are the internal standards.

Prior to analysis, the internal standards must be added to all standards, samples, and QC items. The concentration of the internal standards must be the same in all calibration samples, field samples, and QC samples. A concentration of 50ug/L is used for each of the ISTD analytes.

The response of the ISTD in the ICV/CCV must be within 70% to 130% of the response of the average ISTD in the List One initial calibration sequence (denoted as ICISAV), If the response is outside of this range, the analysis of the CCV must be repeated and any samples associated with the CCV must also be re-analyzed. Repeated failure of the ISTD response will require re-calibration.

9.4 Surrogates

This procedure uses surrogates to evaluate the analytical process.
Dibromofluoromethane, 1,2-dichloroethane-d4, toluene-d8, and p-BFB are the surrogates routinely used.

Prior to preparation, the surrogates must be added to all samples and QC items. The concentration of the surrogates must be the same in all field samples and QC samples. A concentration of 50ug/L is used for each of the surrogate analytes.

Note: The EPA 8260_AK method, which requires a medium-level methanol extraction for soils, requires an additional surrogate to be incorporated prior to sample collection. This surrogate, f_3-toluene, is supplied in the outgoing methanol vial.

The percent recovery of the surrogate in all field samples and QC samples must be within the limits listed in the Method Limit Groups (MLGs) in LIMS. If the percent recovery is outside of this range, the analysis of the sample must be repeated.
Repeated failure of the surrogate percent recovery may indicate re-calibration or maintenance is necessary.

9.5 **Corrective Action for Out-of-Control Data**

When the quality control parameters do not meet the criteria set forth in this SOP, corrective action must be taken in accordance with SOP SA-QA-005: *Preventive and Corrective Action Procedures* the QC Summary Table in Attachment 3. SOP SA-QA-005 provides contingencies for out-of-control data and gives guidance for exceptionally permitting departures from approved policies and procedures. Nonconformance Memos must be initiated to document all instances where QC criteria are not met and all departures from approved policies and procedures.

10.0 PROCEDURE

10.1 Preparation

10.1.1 Aqueous Sample Preparation

Remove the samples from the refrigerator and allow them to come to room temperature.

Several of the instruments in use are able to automatically add ISSU to the samples during the sample transfer step on the autosampler. For the instruments that can spike automatically, the room temperature vials are placed directly onto the instrument with no additional steps necessary.

For the instruments that are unable to spike the vials, using a 50uL syringe, add 43uL of the ISSU to the sample, injecting the solution through the vial septum. Invert the vial several times to mix. Transfer to the instrument.

10.1.2 Aqueous QC Preparation

The method blank and LCS are prepared in reagent water using a 50mL volumetric flask.

- For the method blank, add 50uL ISSU to 50mL reagent water. Pour into a VOA vial and place on the instrument.
- For the LCS, add 50uL ISTD, 50uL Mega Mix, and 150uL MeOH to 50mL reagent water. Pour into a VOA vial and place on the instrument.
- For the MS/MSD, add 43uL ISTD, 43uL Mega Mix, and 129uL MeOH into each sample vial selected for the MS and MSD. Place on the instrument.

10.1.3 Soil Sample Preparation

Refer to SOP SA-VO-001: *Preparation, Screening, and Storage of Volatiles Samples* for the soil specific sample preparation procedures. This information is summarized below:

For routine soil samples, three pre-weighed TerraCore kits and one bulk container are supplied for each soil sample. Two of the TerraCore kits contain water and are used for low level analysis. The remaining TerraCore kit contains methanol to be used for a medium-level analysis if the low-level samples exceed the calibration range. The bulk container is used for screening, and for the percent solids determination.

For EPA 8260_AK soil samples, one pre-weighed 4oz jar and is supplied for each soil sample along with a 40mL VOA vial containing 25mL of the methanol/surrogate solution. A 25g sample is collected into the jar, and then the methanol/surrogate solution is added.

At the time of analysis, the following steps are taken:

For routine low-level samples, 5mL reagent water and 5uL ISSU are added directly to the sample vial. The sample is then ready for analysis.

For routine medium-level samples, a dilution is prepared and analyzed. The default dilution is a DF40 (i.e., using 125uL of sample). 5uL VOA Surrogate Mix is added directly to the sample vial (if 5mL of MeOH were used) or 10uL VOA Surrogate Mix is added (if 10mL of MeOH were used). The sample is then ready for analysis on a soil instrument with a heated purge.

For EPA 8260_AK soil samples, a dilution is prepared and analyzed. The default dilution is a DF40 (i.e., prepared using 1.25mL of sample into 50mL water). 50uL ISSU is added directly to the volumetric flask before being transferred to the 40mL VOA vial. The sample is then ready for analysis on a liquid instrument with an ambient purge.

10.1.4 Soil QC Preparation

For routine low-level samples, the method blank and LCS are prepared using blank sand directly in a VOA vial.

- For the method blank, add 5g sand, a stir bar, 10mL reagent water, and 5uL ISSU.
- For the LCS, add 5g sand, a stir bar, 10mL reagent water, 5uL ISTD, 5uL Mega Mix, and 15uL methanol.
- For the MS/MSD, add 5mL water, 5uL ISTD, 5uL Mega Mix, and 15uL methanol directly to the selected sample.

For routine medium-level samples, the method blank and LCS are prepared using Ottawa sand directly in a VOA vial.

- For the method blank, add 5g blank sand and 5mL methanol. Remove a 125uL aliquot and add to 10mL reagent water spiked with 5uL ISSU.
- For the LCS, add 5g blank sand and 5mL methanol. Remove a 125uL aliquot and add to 10mL reagent water spiked with 5uL ISTD, 5uL Gas Intermediate, and 5uL Mega Mix.
- For the MS/MSD, remove a 125uL aliquot and add to 10mL reagent water spiked with 5uL ISTD, 5uL Gas Intermediate, and 5uL Mega Mix.

For EPA 8260_AK soil samples, the method blank and LCS are prepared using Ottawa sand directly in a VOA vial.

- For the method blank, add 25g blank sand and 25mL methanol. Remove a 1.25mL aliquot and add to 50mL reagent water spiked with 50uL ISSU.
- For the LCS, add 25g blank sand and 25mL methanol. Remove a 1.25mL aliquot and add to 50mL reagent water spiked with 50uL ISTD and 50uL Mega Mix.
- For the MS/MSD, remove a 1.25mL aliquot and add to 50mL reagent water spiked with 50uL ISTD and 50uL Mega Mix.

10.2 **Analysis:**

10.2.1 Instrument Operating Conditions

The instrument conditions listed in this SOP are provided for guidance purposes. The purge time must be 11 minutes. All other parameters are optimized by the lab for the target compounds and documented in the maintenance log. Therefore, the actual conditions used by the laboratory may be slightly different from those listed here and must be documented in the instrument maintenance log, data system, and/or run log.

Instrument maintenance must be performed in accordance with Attachment 4 of this SOP.

The goal is to have maximum separation between the target compounds in the shortest run time while maintaining sufficient sensitivity to detect the target compounds at the reporting limit and MDL (if required).

GC Parameters
Column: 20m x 0.18mm ID x 1.0um
Column: Flow: 0.5-1.0mL/min
Injector: Split/Splitless operated in the split mode with 1mm ID quartz insert
Split ratio (desorb to column flow): 40:1 to 80:1

MS Parameters
Mass spectrometer interface: 240°C (direct column interface)
Mass spectrometer source temperature: 250°C
Mass scan range: 35-300amu, with a minimum scan cycle of 1 scan per second
Injector: 250°C
Temperature Program:
Initial Temperature: 50°C
Program Rate: 18°C/minute
Final Temperature: 200°C

Example Purge and Trap Conditions
The purge and trap conditions listed in this section are for guidance. The lab must document the actual conditions used. The purge time must be 11 minutes. Other parameters may be varied to optimize the detection of the target compounds.

VOCARB 3000 trap
Purge Time: 11 minutes
Purge temperature: aqueous-ambient; soils-heated 40°C
Desorb time: 0.50 minutes
Desorb temperature: 250°C
Bake time: 4 to 6 minutes at 260°C
Purge flow: Approximately 30-40mL/minute
Valve temperature: 150°C
Transfer line: 150°C

The purge flow must be balanced for adequate sensitivity of the target compounds. If the purge flow is too high, the response of the gases will be low and not reproducible. The SPCC criteria for chloromethane may not be achieved if the purge flow is too high. If the purge flow is too low, the response of the more water-soluble targets - ketones, ethers, bromoform - may be low and the reporting limit may not be achieved on a routine basis.

10.2.2 Internal Standard (ISTD)

Prior to analysis, internal standard must be added to all standards, samples, and QC items. The concentration of the internal standard must be the same in all calibration samples, field samples, and QC samples. Instructions for the addition of the internal standard spiking mix are given in the previous sections.

10.2.3 BFB Tune Check

A BFB tune check must be analyzed at the beginning of each clock as a check on the "tune" of the mass spectrometer. This check must meet the criteria described given in Section 9.2.1.

EPA 8260B, EPA 8260C, EPA 8260D, EPA 624, EPA 624.1

If the instrument is configured for direct injection, inject 2uL of the 25ng/uL solution into the GC and analyze the 4-BFB using the instrument conditions listed in Section 10.1. A solvent delay should be set to allow the methanol to pass through the mass spectrometer while the MS is "off".

EPA 8260B, EPA 8260C, EPA 8260D, EPA 624, EPA 624.1

If the purge and trap is used to introduce the 4-BFB into the MS, add 1uL of the 50ng/uL solution to 5.0mL of reagent water. Transfer the 5.0mL to the purge and trap device and purge the sample using the same conditions used for sample analysis.

SM6200B:

If the instrument is configured for direct injection, inject 1uL of the 25ng/uL solution into the GC and analyze the 4-BFB using the instrument conditions listed in Section 10.1. A solvent delay should be set to allow the methanol to pass through the mass spectrometer while the MS is "off".

If the purge and trap is used to introduce the 4-BFB into the MS, add 1uL of the 25ng/uL solution to 20mL of reagent water. Transfer to the purge and trap device and purge the sample using the same conditions used for sample analysis.

10.2.4 Trap Check Standard

The trap check standard is used to evaluate the condition of the trap by monitoring the formation of chloromethane and bromomethane. Chloromethane and bromomethane may be formed on a degraded trap by thermal decomposition of halogenated compounds. This check must meet the criteria described given in Section 9.2.2.

10.2.5 Initial and Continuing Calibration

Calibrate the instrument using the standards and criteria described given in Section 9.2.3. Once the calibration has been established and verified with an ICV in accordance with Section 9.2.4, sample analysis may proceed.

Verify the calibration curve with a continuing calibration verification using the standards and criteria described given in Section 9.2.5

10.2.6 Sample Analysis

The sample/extract must be injected using the same injection volume used for the calibration standards. The samples can only be analyzed after the tune, the trap check, the calibration (initial or continuing), and the method blank and LCS criteria have been met. Samples that are known to be relatively clean should be analyzed first. Samples suspected of containing high concentrations should be analyzed last. Instrument blanks may be analyzed after suspected high concentration samples to allow the detector response to stabilize.

10.2.7 Example Analytical Sequence

A typical example analytical sequence is listed below.

Description	Comments
Blank	
BFB	Clock time begins with injection of the BFB
Initial Calibration	
ICV	Second Source
BFB	Clock time begins with injection of the BFB
CCV	
LCS	
LCSD	Required if insufficient sample provided for MS/MSD
Trap Check	
MB	
Samples & Batch QC Items	
Closing CCV	Applies to DOD QSM and SM6200B
BFB	Clock time begins with injection of the BFB
CCV	
LCS	
LCSD	Required if insufficient sample provided for MS/MSD
Trap Check	
MB	
Samples & Batch QC Items	
Closing CCV	Applies to DODQSM and SM6200B.

11.0 CALCULATIONS/DATA REDUCTION

11.1 Data Reduction

Data evaluation must be performed in accordance with SA-QA-008: *Evaluation of Chromatographic Data.* This SOP includes specific information regarding the evaluation of chromatographic data, including the requirements for performing manual integrations and the evaluation of retention times.

Data review and reporting must be performed in accordance with SA-QA-002: *Data Generation and Review.*

11.1.1 Target Analyte Identification

A target compound is identified by the visual comparison of the sample mass spectrum with the mass spectrum of the target compound from a reference spectrum of the target compound stored in a library from a standard spectral library such as the NIST/NBS.

11.1.1.1 Two criteria must be met in order to identify a target compound:

- Elution of the sample component within ±0.06 Relative Retention Time (RRT) units of the daily standard containing that compound. The RRT is calculated as follows:

$$RRT = \frac{retention\ time\ of\ the\ target\ compound}{retention\ time\ of\ the\ associated\ internal\ standard}$$

- Correspondence of the target compound spectrum and the standard component mass spectrum

11.1.1.2 All ions present in the standard component mass spectrum at a relative intensity greater than 10% (most abundant ion = 100%) must be present in the sample component mass spectrum. Other ions may be present in the sample component. Coelution of a non-target compound with a target compound will make the identification of the target compound more difficult. The ions due to the non-target compound should be subtracted from the sample component spectrum as part of the background to account for the discrepancy between the sample spectrum and the standard spectrum.

11.1.1.3 The relative intensities of the ions present in the sample component spectrum must agree within ± 30% of the relative intensities of the ions in the standard reference spectrum. For example, an ion with an abundance of 50% in the reference spectrum should have a corresponding abundance between 20% and 80% in the sample component spectrum. The intensities of the ions in the standard reference spectrum are updated utilizing the continuing calibration standard. If the relative intensities are outside 30% in sample, the data will be flagged with an R by CHROM.

11.1.1.4 If the above criteria are not met exactly, the analyst should seek help from a senior analyst or the Department Manager. If there is sufficient evidence to support the identification of the component, then the component is identified, quantified, and reported.

11.1.2 Dilutions

Samples may be screened prior to analysis as outlined in SOP SA-VO-001. This screening data provides qualitative information that is used to determine if dilutions are required for analysis.

Unless otherwise specified by a client QAPP, results from a single analysis are reported as long as the largest target analyte (when multiple analytes are present) is in the upper half if the calibration range. When reporting results from dilutions, appropriate data flags must be used or qualification in a case narrative provided to the client.

For clients who require we provide lower detection limits, a general guide would be to report the dilution detailed above and one additional run at a dilution factor 1/10 of the dilution with the highest target in the upper half of the calibration curve. For example, if samples analyzed at a 1/50 dilution resulted in a target in the upper half of the calibration curve, the sample would be analyzed at a dilution factor of 1/5 to provide lower reporting limits.

11.1.3 Historical Data

Many of the laboratory's clients submit samples for repeat monitoring purposes. Prior to analysis, verify LIMS Worksheet Notes or use the LIMS Historical Data Tracker feature to determine if historical data is available for review.

11.1.4 Chemical Relationships

Benzene, toluene, ethylbenzene, and the xylenes are generally present together in samples and indicate the presence of gasoline

m/p-Xylenes are generally higher than o-xylene

Hydrocarbons present is samples containing gasoline generally contain mass 43 and may co-elute with target analytes with mass 43 as the quant or confirmation ion or may skew the spectrum of a compound with mass 43 as part of the spectrum.

Cis-isomers are generally more prevalent than the trans- isomers

Pay particular attention to the retention time of isomer because the only way to positively identify them is by retention time. The isomers are:

1,1-dichloroethane and 1,2-dichloroethane
1,1-dichloroethene, cis-1,2-dichloroethene, and trans-1,2-dichloroethene
1,1,1-trichloroethane and 1,1,2-trichloroethane
ethyl benzene, m/p-xylene, and o-xylene
1,3-dichlorobenzene, 1,4-dichlorobenzene, and 1,2-dichlorobenzene
1,1-dichloropropene, cis-1,2-dichloropropene, and trans-1,2-dichloropropene
2-chlorotoluene and 4-chlorotoluene
1,2,3-trichlorobenzene and 1,2,4-trichlorobenzene
1.3.5-trimethylbenzene and 1,2,4-trimethylbenzene
4-methyl-2-pentanone (MIBK) and 2-hexanone
n-butylbenzene, sec-butylbenzene, tert-butylbenzene, and isopropylbenzene

Higher chlorinated alkanes and alkenes may have lower chlorinated alkanes or alkenes present due to degradation. The following table lists some common chlorinated compounds and their degradation products. Look for the degradation product(s) when the concentration of the compound in the left column is present at high concentrations.

Analyte	Degration Product
1,1,2,2-tetrachloroethane	trichloroethene (TCE) cis-1,2-dichloroethene (c-1,2-DCE) trans-1,2-dichloroethene (t-1,2-DCE) vinyl chloride 1,1,2-trichloroethane (1,1,2-TCA) 1,2-dichloroethane (1,2-DCA) Chloroethane
1,1,2-trichloroethane (1,1,2-TCA)	1,2-dichloroethane (1,2-DCA) Chloroethane
1,1,1-trichloroethane (1,1,1-TCA)	1,1-dichloroethene (1,1-DCE) 1,1-dichloroethane (1,1-DCA) Chloroethane
Carbon tetrachloride	Chloroform Methylene chloride Chloromethane
Tetrachloroethene (PCE) (PCE = perchloroethylene which is a common name for tetrachloroethene)	trichloroethene (TCE) cis-1,2-dichloroethene (c-1,2-DCE) trans-1,2-dichloroethene (t-1,2-DCE) Chloroethene
1,2,4-trichlorobenzene	1,4-dichlorobenzene (1,4-DCB) 1,2-dichlorobenzene (1,2-DCB) Chlorobenzene

11.1.5 Dry Weight/Moisture Content Correction

All routine soil samples are reported on a dry weight basis.
As outlined in EPA 8000D, for samples collected in methanol preserved containers; however, there is an additional methanol dry weight correction required. Since water and methanol are miscible, the nominal volume of methanol needs to be adjusted for moisture content as well.

11.2 Calculations

11.2.1 The calculations associated with batch QC determinations are given in SOP SA-QA-017. Applicable calculations include accuracy (% recovery) and precision (%RPD).

11.2.2 The calculations associated with initial and continuing calibrations and are given in SOP SA-QA-016. Applicable calculations include determination for: calibration factor, standard deviation, relative standard deviation, relative response factor, and relative standard deviation.

11.2.3 The calculation to determine final concentration is given as follows:

$$Final Concentration = CONC_{Sample} \otimes \frac{F}{I \times dw} \otimes D$$

Where:
$CONC_{Sample}$= Concentration of the sample extract (at the instrument)
F = Final volume of the extract
I = Initial volume/weight
dw = % Solids decimal equivalent
D = Dilution factor

All dry weight corrections are performed automatically in LIMS.

Note: This formula assumes all unit conversion factors are applied.
Note: Procedures for entering soil preparation and dilution data into LIMS and CHROM are given in Attachment 15 of this SOP.

11.2.4 Acceptance Criteria Independent of Calibration Model – Method 8000D

Method 8000D in SW-846 recommends the use of either the Percent Error or Relative Standard Error to evaluate the calibration model as described below.

Note: For South Carolina work, one of these methods of evaluation must be used.

11.2.4.1. Percent Error: The percent error assesses the refitting of the calibration data back to the model. The percent error between the calculated and expected amounts of an analyte must be ≤ 30% for all standards except for the lowest data point which must be ≤ 50%. Unless specified in the Analysis method, this limit will be utilized.

Calculation of the Percent Error

$$\%Error = \frac{x_i - x_i'}{x_i} \times 100$$

Where:

x'_i = Measured amount of analyte at calibration level *i*, in mass or concentration units

x'_i = True amount of analyte at calibration level i, in mass or concentration units

Relative Standard Error (RSE): Relative standard error provides a measure of the fit of the calibration that is independent of the calibration model. RSE is calculated using the following equation:

$$RSE = 100 \times \sqrt{\frac{\sum_{i=1}^{N}\left(\frac{x'_i - x_i}{x_i}\right)^2}{(n-p)}}$$

Where:

x'_i = Measured amount of analyte at calibration level *i*, in mass or concentration units

x_i = True amount of analyte at calibration level *i*, in mass or concentration units

p = Number of terms in the fitting equation *(average = 1, linear = 2, quadratic = 3, cubic = 4)*

n = Number of calibration terms

The RSE acceptance limit criterion is the same as the RSD limit.

12.0 METHOD PERFORMANCE:

12.1 Lower Limit of Quantitation (LLOQ)

The laboratory shall establish the LLOQ as the lowest concentration at which the laboratory has demonstrated target analytes can be reliably measured and reported with a certain degree of confidence, which must be ≥ the lowest point in the calibration curve. The LLOQ is equivalent to the standard reporting limit (RL). The required LLOQ verification is performed at a concentration of 0.5-2 times the LLOQ (or RL).

12.2 Reporting Limit Verification (RLV) aka Lower Limit of Quantitation Verification (LLOQV)

At a minimum, RLVs/LLOQVs must be performed initially upon method set-up in accordance with SOP SA-QA-007: *Determination and Verification of Detection and Reporting Limits*.

For analytes and methods certified by DOD ELAP, RLVs/LLOQVs must also be performed quarterly thereafter. For all compounds analyzed by Method 8260, LLOQVs must be performed annually for compliance with Method 8000D, SW-846 Update V.

Recovery of target analytes in the LLOQV must be within established in-house limits. Until the laboratory has sufficient data to determine acceptance limits, the LCS criteria ± 20% may be used for the LLOQ.

Exceptions may be made for project-specific non-routine analytes.

12.3 Method Detection Limit (MDL) Study

The MDL is the lowest concentration that can be detected for a given analytical method and sample matrix with 99% confidence that the analyte is present. MDLs reflect a calculated (statistical) value determined under ideal laboratory conditions in a clean matrix and may not be achievable in all environmental matrices. The current MDLs associated with this procedure are given in the Method Limit Group (MLG) in LIMS.

At a minimum, MDL Studies must be performed initially upon method set-up in accordance with SOP SA-QA-007: *Determination and Verification of Detection and Reporting Limits*.

Note: MDL Studies are not required for non-routine analytes provided results are not reported below the RL (i.e., MDL equals RL in LIMS).

12.4 Method Detection Limit Verification (MDLV)

At a minimum, MDLVs must be performed initially upon method set-up in accordance with SOP SA-QA-007: *Determination and Verification of Detection and Reporting Limits*.

For analytes and methods certified by DOD ELAP, MDLVs must also be performed quarterly thereafter. For all other analytes and methods, MDLVs must also be performed annually thereafter.

Note: MDLVs are not required for non-routine analytes provided results are not reported below the RL (i.e., MDL equals RL in LIMS).

12.5 QC Limit Generation, Control Charting, and Trend Analysis

12.5.1 EPA 624 and EPA 624.1

The control limits for the batch QC items (LCS/LCSD and MS/MSD) for this procedure are specified in the reference method and cannot be broadened; therefore, the laboratory defaults to the method-defined limits and does not utilize in-house or laboratory-derived limits for the evaluation of batch QC items.

12.5.2 EPA 8260B, EPA 8260C and EPA 8260D

The control limits for the batch QC items (LCS/LCSD and MS/MSD) for this procedure are not specified by the reference method; therefore, the laboratory defaults to in-house and/or laboratory-derived limits for the evaluation of batch QC items.

12.5.3 SM6200B

The control limits for the batch QC items (LCS/LCSD and MS/MSD) for this procedure are specified in the reference method and cannot be broadened; therefore, the laboratory defaults to the method-defined limits and does not utilize in-house or laboratory-derived limits for the evaluation of batch QC items.

Control charting is a useful tool and is performed to assess analyte recoveries over time to evaluate trends. Control charting must be performed periodically (at a minimum annually) in accordance with SOP SA-QA-017: *Evaluation of Batch QC Data.*

12.6 Demonstrations of Capability

Initial and continuing demonstration of capability must be performed in accordance with SOP SA-QA-006*: Training Procedures.*

Prior to performing this procedure unsupervised, each new analyst who performs this analysis must demonstrate proficiency per method/analyte combination by successful completion of an initial demonstration of capability (IDOC). The IDOC is performed by the analysis of 4 consecutive LCSs that meet the method criteria for accuracy and precision. The IDOC must be documented and routed to the QA Department for filing.

Annual continuing demonstrations of capability (CDOCs) are also required per analyst per method/analyte combination. The CDOC requirement may be met by the consecutive analysis of four LCS all in the same batch, by the analysis of four LCS analyzed in four consecutive batches (in different batches on different days), via acceptable results on a PT study, or by the analysis of client samples with statistically indistinguishable results when compared to another certified analyst. The CDOC must be documented and routed to the QA Department for filing.

The laboratory utilizes DOD QSM limits were applicable for IDOC criteria.

12.7 Training Requirements

All training must be performed and documented in accordance with SOP SA-QA-006: *Training Procedures*.

Note: The SOPs listed in the Reference/Cross-Reference Section are applicable to this procedure. All employees performing this procedure must also be trained on these SOPs, and/or have a general understanding of these procedures, as applicable.

13.0 POLLUTION CONTROL

It is Eurofins TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (e.g., examining recycling options, ordering chemicals based on quantity needed, preparing reagents based on anticipated usage and reagent stability, etc.). Employees must abide by the policies in Section 13 of the Environmental Health and Safety Manual.

This procedure has been evaluated for opportunities to minimize the waste generated. Where reasonably feasible, pollution control procedures have been incorporated.

14.0 WASTE MANAGMENT

Waste management practices must be conducted consistent with all applicable federal, state, and local rules and regulations. All waste (i.e., excess reagents, samples, and method process wastes) must be disposed of in accordance with Section 9 of the Eurofins TestAmerica Savannah Addendum to the EHSM. Waste description rules and land disposal restrictions must be followed.

14.1 Waste Streams Produced by the Method

The following waste streams are produced when this method is carried out:

- Excess samples, reagents, and standards must be disposed in accordance with the Eurofins TestAmerica Savannah Addendum to the EHSM.
- Methanolic waste from rinsings and standards – Transfer to satellite container for methanolic (flammable) waste. Transfer to hazardous waste section when satellite container is full.
- Excess aqueous samples – Dispose according to characterization on the sample disposal sheets. Neutralize non-hazardous samples before disposal into drain/sewer. Transfer hazardous samples (identified on disposal sheets) to the waste department for disposal.
- Excess soil and solid samples – Dispose according to characterization on sample disposal sheets. Transfer non-hazardous and RCRA hazardous samples to the yard box. Transfer any PCB hazardous or Foreign Soil hazardous to the waste coordinator.
- Excess Soils with Methanol- Samples should be disposed via Drum marked as “Soil Vials with Methanol”.

15.0 REFERENCES/CROSS REFERENCES

- SOP SA-AN-041: *Reagent and Standard Materials Traceability*
- SOP SA-AN-100: *Laboratory Support Equipment (Verification and Use)*
- SOP SA-QA-002: *Data Generation and Review*
- SOP SA-QA-005: *Preventive and Corrective Action Procedures*
- SOP SA-QA-006*: Training Procedures*
- SOP SA-QA-007: *Determination and Verification of Detection and Reporting Limits*
- SOP SA-QA-008: *Evaluation of Chromatographic Data*
- SOP SA-QA-015: *Homogenization, Compositing, and Segregation of Samples*
- SOP SA-QA-016: *Evaluation of Calibration Curves*
- SOP SA-QA-017: *Evaluation of Batch QC Data*
- SOP SA-VO-001: *Preparation, Screening, and Storage of Volatiles Samples*
- Eurofins TestAmerica Savannah Quality Assurance Manual
- Eurofins TestAmerica Environmental Health and Safety Manual

- Eurofins TestAmerica Savannah Addendum to the Environmental Health and Safety Manual.
- EPA Method 8000B*: Determinative Chromatographic Separations.* SW-846 Update 3, Revision 2. December 1996.
- EPA Method 8000D*: Determinative Chromatographic Separations.* SW-846 Update VI, Revision 5. March 2018.
- EPA Method 5030B*: Purge and Trap for Aqueous Sample.* SW-846 Update 3, Revision 2. December 1996.
- EPA Method 5030C*: Purge and Trap for Aqueous Sample*. U.S. EPA 2003, Revision 3, May 2003.
- EPA Method 5035*: Closed System Purge-and-Trap and Extraction for Volatile Organics in Soil and Waste Samples*. SW-846 Update 3, Revision 3.
- EPA Method 5035A: *Closed System Purge-and-Trap and Extraction for Volatile Organics in Soil and Waste Samples*. Revision 1, July 2002.
- EPA Method 8260B: *Volatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS).* SW-846 Update 3, Revision 2, December 1996.
- EPA Method 8260C*: Volatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS).* U.S. EPA, Revision 3, August 2006.
- EPA Method 8260D*: Volatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS).* SW-846 Update VI , Revision 4, June 2018
- EPA Method 624*: Purgeables.* 40 CFR Part 136, Appendix A, July 1, 1995.
- EPA Method 624.1: Purgeables by GC/MS. 40 CFR Part 136, Appendix A, July 1, 1995
- SM6200B: Volatile Organic Compounds . Revision 22nd ED 2011.
- DOD Quality Systems Manual Version 5.3 , 2019

16.0 METHOD MODIFICATIONS AND CLARIFICATIONS

16.1 Incorporation of Other Matrices

This procedure may be modified to analyze other matrices (e.g., wipe, waste, and TCLP/SPLP leachate samples) based on the needs of the client. This will need to be arranged by the Project Manager at the initiation of the project.

Wipe and waste matrices are non-routine, and the laboratory is not currently NELAC certified for these matrices. The laboratory uses its routine soil RLs (converted for initial and final volumes, etc.) and default QC limits to evaluate waste samples and its routine water RLs (converted for initial and final volumes, etc.) and default QC limits to evaluate wipe samples. Soil and/or water DOCs can be used to satisfy analyst demonstrations of capability for these types of non-routine matrices, as applicable. The laboratory uses its routine aqueous RLs (converted for initial and final volumes, etc.) and default QC limits to evaluate TCLP/SPLP leachate samples. Water DOCs can be used to satisfy analyst demonstrations of capability for TCLP/SPLP matrices. Ottawa sand is used as the blank matrix for wipes and wastes unless specifically requested otherwise by the project.

16.1.1 Collection and Handling Procedures

Waste (oil) samples are routinely collected in 40mL VOA vials. Waste (oil) samples must be iced at the time of collection and maintained at 0-6°C (i.e., less than 6°C with no frozen samples) until time of preparation. Samples must be analyzed within 14 days of collection.

Wipe samples are routinely collected in 40mL VOA vials. Wipe samples must be iced at the time of collection and maintained at 0-6°C (i.e., less than 6°C with no frozen samples) until time of preparation. Samples must be analyzed within 14 days of collection.

Once the TCLP/SPLP extraction procedure has been performed, the leachate is transferred to a Tedlar bag. TCLP/SPLP leachates must be stored at 0-6°C (i.e., less than 6°C with no frozen samples) until time of preparation. The leachate sample must be analyzed within 14 days of completion of the TCLP/SPLP extraction.

16.1.2 Preparation and Analytical Procedures

Wipe samples are analyzed in the same fashion as water samples. Refer to Work Instruction FQA088: *Wipe Tests: Sampling and Analysis* for additional information on wipe procedures.

Waste samples are prepared and analyzed in the same fashion as medium-level soil samples. Note: Waste samples often require large dilutions.

TCLP/SPLP matrices are prepared and analyzed in the same manner as routine water samples; however, a default dilution factor of 20 is used.

16.2 Other Considerations

16.2.1 EPA Method 8260B does not contain calibration verification criteria for non-CCC analytes nor does it require non-CCC analytes to be evaluated for response; however, the laboratory has adopted in-house criteria for non-CCC analytes as outlined in this SOP.

16.2.2 EPA Method 8260B does not require the analysis of an ICV. NELAC requires an ICV; however, it does not list specific criteria. The laboratory has adopted in-house criteria for ICVs for EPA 8260B as outlined in this SOP. The laboratory satisfies this requirement via the ICV performed with each ICAL.

16.2.3 EPA Method 8260B, EPA 8260C and EPA 8260D allows alternate criteria to be used for the BFB evaluation. As such the laboratory has incorporated criteria from the CLP OLMO4.0 (January 1998) method.

16.2.4 The laboratory has defined the analytes listed in Attachment 11 as poor or erratic performers and allows for exceptions to the ICV, CCV, LCS/LCSD, MS/MSD, and Sporadic Marginal Exceedance criteria for these analytes as outlined in this SOP. **These exceptions are not permitted for QC associated with samples originating in SC**.

16.2.5 It has been determined that increased methanol concentrations can suppress the response of the gas compounds, leading to erratic recovery and reduced linearity. As such, the laboratory normalizes the volume of methanol in all standards and QC items to minimize this effect. By introducing a constant volume of methanol to initial calibration standards, CCV/ICV, LCS, and MS/MSD, better sensitivity and recovery of these analytes is achieved.

16.2.6 The 7-day holding time for samples with pH>2 is listed in EPA 624 and EPA 624.1 but is not included in the SW-846 methods. The laboratory has adopted this as internal guidance for EPA 8260B, EPA 8260C and EPA 8260D as is common industry practice and will make every effort to analyze samples with pH>2 within 7 days.

16.2.7 Historical standard practice for most laboratories was to combine all analytes into one analysis using a single acid preserved container. This practice is still acceptable in those cases where the compounds of interest are not adversely affected by the addition of the hydrochloric acid (HCl) preservative; however, EPA 624, EPA 624.1, and Chapter 4 of SW-846 all list special preservation requirements for acrolein, acrylonitrile, and 2-chloroethylvinyl ether (2-CEVE). Although these analytes are rarely found in the environment, preservation at pH<2 (as achieved using HCl preservative) breaks down these analytes and may result in a significantly low bias and/or non-detect value.

The reference methods suggest to preserve these analytes to a pH between 4-5. Achieving this narrow pH range is problematic; therefore, the only other alternative is to use an unpreserved container for these analytes. As such, if acrolein, acrylonitrile, and/or 2-CEVE are on the requested target analyte list, the laboratory now defaults to using an unpreserved container. The other target analytes reported for these methods are not adversely affected by using an unpreserved container provided they are analyzed within the shorter, method-prescribed holding times (HT) defined for unpreserved samples. Specific information on VOA holding times, preservation requirements, and analyte-specific requirements is given in Attachment 2.

16.2.8 The 40mL VOA vials used by the laboratory have been demonstrated to actually contain approximately 43mL. As such, standard spiking amounts have been adjusted accordingly to accommodate for this volume (e.g., spiking 43uL instead of 40uL).

16.2.9 The laboratory has incorporated the batch QC items as outlined in Section 9.1. Some additional QC items are performed which differ from those specified in the reference methods (e.g., LCSD or MSD) to satisfy common state regulatory requirements and/or client requests for precision data and/or to facilitate scheduling and data evaluation. The method-specified batch QC items are as follows:

EPA 8260B, EPA 8260C, EPA 8260D and SM6200B :
Method blank, LCS, MS, and sample duplicate or MSD

EPA 624 and EPA 624.1
Method blank, LCS, MS per 10% of samples analyzed

16.2.10 EPA Method 624 specifies a required concentration range criteria for the evaluation of QC items (i.e., the Q-Table). The laboratory has converted these values into percent recovery limits to be used to assess CCV, ICV, LCS/LCSD, and MS/MSD.

16.2.11 The EPA 8260B reference method specifies a 1-week expiration date for the gas standards. The laboratory prepares a 25mL volume of MegaMix standard and splits it into five 5mL Mini-nert vials. Each vial is assigned a 1-week expiration date from the date that vial is opened, not to exceed 14 days from the preparation date of the 25mL volume. **Note: This expiration date is a maximum. Shorter expiration dates may be used based on evidence of gas compound volatility as evaluated via CCVs.**

16.2.12 Due to client requests, the analyte list utilized by the laboratory may be different than those listed in the reference methods.

16.2.13 The minimum response factors (RFs) required for Acetone, 2-Butanone (MEK), 4-Methyl-2-pentanone (MIBK), and 2-Hexanone have been expanded from those listed in the EPA 8260C reference method.

16.2.14 The EPA 8260B reference method specifies that the reference mass spectra must be generated by the laboratory using the conditions of this method. The reference spectra utilized by the laboratory in CHROM for peak identification is generated by the NIST library. The mass ratios for the ions are updated by the continuing calibration analysis.

16.2.15 The EPA 624 reference method specifies a desorb time of 4 minutes. The laboratory employs a desorb time of about 0.50 minutes. The EPA Methods Update Rule and subsequent memo allow a laboratory the flexibility to modify CWA methods. This memo states that if the underlying chemistry and determinative technique were the same as the unmodified method, then the modified method is considered an equivalent and acceptable alternative; thus this desorb time modification is an allowable modification for EPA 624 under this rule.

16.2.16 SM6200B reference method specifies an Internal Standard Concentration of 1.0ug/L. The laboratory is using a concentration of 50ug/L.

17.0 ATTACHMENTS:

The following Tables, Diagrams, and/or Validation Data are included as Attachments:

Attachment 1: SOP Summary
Attachment 2: Sample Collection, Preservation, and Holding Time Table
Attachment 3: QC Summary
Attachment 4: Preventative Maintenance and Troubleshooting
Attachment 5: EPA 8260B Calibration Criteria: SPCCs and CCCs
Attachment 6: EPA 8260C Minimum Response Factors
Attachment 7: BFB Tune Criteria
Attachment 8: Target Compound Information: Ions and ISTDs
Attachment 9: Instrument Configurations
Attachment 10: Standard Information and Recipes
Attachment 11: Poor Performers
Attachment 12: EPA 624 CCV & ICV Criteria (Q-Table)
Attachment 13: EPA 624.1 CCV & ICV Criteria (Q-Table)
Attachment 14: Glassware Cleaning Procedures
Attachment 15: Data Entry Procedure Work Instruction
Attachment 16: DOD QSM 5.3 B-4 Tables

Attachment 1:
SOP Summary

Sample Preparation Summary
Aqueous samples are routinely purged at ambient conditions; however, a heated purge may be used if required by the project. A 5mL purge volume is used as the default. The calibration standards and the associated QC must be analyzed under the same conditions and volume.

Low-level soil samples are purged at 40°C in a purge and trap instrument designed to add water and internal standards to the vial containing the sample without breaking the seal. The sample is stirred during purging to thoroughly mix the soil and water. The calibration standards and associated QC are purged under the same conditions.

Medium-level soils, waste samples, and soil samples for EPA 8260_AK are extracted with methanol. An aliquot of the methanol extract is injected into reagent water. The methanol extract/reagent water is purged at a heated temperature (i.e., 40□C) for routine soils and at ambient temperature for wastes and EPA 8260_AK, using the same instrument conditions and calibration used for aqueous samples.

Sample Analysis Summary
VOCs are purged from the sample matrix with nitrogen. The VOCs are transferred from the sample matrix to the vapor phase. The vapor is swept through a sorbent tube where the VOCs are trapped. After the purging is completed, the trap is heated and backflushed with helium to desorb the VOCs onto a GC column. The GC is temperature-programmed to separate the VOCs, which are then detected by a mass spectrometer. Qualitative identification of the target compounds in the sample is based on the relative retention time and the mass spectra of the characteristic masses (ions) determined from standards analyzed on the same GC/MS under the same conditions. Quantitative analysis is performed using the internal standard technique with a single characteristic ion.

Description	Comments
Blank	
BFB	Clock time begins with injection of the BFB
Initial Calibration	
ICV	Second Source
BFB	Clock time begins with injection of the BFB
CCV	
LCS	
LCSD	Required if insufficient sample is provided for MS/MSD
Trap Check	
MB	
Samples & Batch QC Items	
CCVC	EPA 8260C, EPA 8260D and SM6200
BFB	Clock time begins with injection of the BFB
CCV	
LCS	
LCSD	Required if insufficient sample is provided for MS/MSD
Trap Check	
MB	
Samples & Batch QC Items	
CCVC	EPA 8260C, EPA 8260D and SM6200B

Attachment 2:
Sample Collection, Preservation, and Holding Time Table

Aqueous samples are routinely collected in three 40mL VOA vials. The table below outlines the laboratory's default procedures for aqueous samples:
:

Method	Acrolein, Acrylonitrile, or 2-CEVE Requested?	Chemical Preservation	Thermal Preservation	Holding Time
EPA 8260B EPA 8260C	Yes	None	0-6°C	7 days
EPA 8260B EPA 8260C	No	HCl (pH<2)	0-6°C	14 days
EPA 624 EPA 624.1	Yes	None	0-6°C	3 days*
EPA 624 EPA 624.1	No	HCl (pH<2)	0-6°C	14 days

*Note: 3-day HT is specific to acrolein. If acrolein is not requested, 7 days is used for the remaining target compounds.

- **Samples originating in SC for which acrolein and/or acrylonitrile are requested, must be preserved to a pH of 4-5 SU unless the Quality Assurance Project Plan (QAPP) or site sampling plan specify otherwise.**

The table below outlines the sample collection options for soil samples:

Sample Container	Chemical Preservation	Thermal Preservation	Holding Time
TerraCore Kit (Routine): 1 x 5g TerraCore sampler, 2 x pre-weighed 40mL VOA vial w/ H2O, 1x pre-weighed 40mL VOA vial w/ MeOH, 1 x 4oz bulk jar	H_2O & MeOH (added in field)	<-10°C	48 hours to freeze, 14 days to analyze
EPA 8260_AK Soils: 1 x 4oz bulk jar, 1 x 40mL VOA vial w/ 25mL MeOH + Surrogate	MeOH (+ Surrogate; added in field)	0-6°C	14 days to analyze
EnCore Kit (Non-Routine) 3 x 5g EnCore samplers, 3 x 40mL VOA vial, 1 x 4oz bulk jar	$NaHSO_4$, H_2O, or MeOH (added in lab)	0-6°C	48 hours to preserve, 14 days to analyze
EnCore Kit (Non-Routine) 3 x 5g EnCore samplers, 2 x pre-weighed 40mL VOA vial w/ NaHSO4, 1 x pre-weighed 40mL VOA w/ MeOH, 1 x 4oz bulk jar	$NaHSO_4$ or MeOH (added in field)	0-6°C	48 hours to preserve, 14 days to analyze

Attachment 3:
QC Summary

QC Item	Frequency	Criteria	Corrective Action
Clock Time	EPA 8260B, EPA 8260C, and EPA 8260D and SM6200B : 12 hours EPA 624: 24 hours EPA 624 Cluster Rule (Chloroform Only): 8 Hours EPA 624.1: 12 hours (time starts after analysis of the MB but may not exceed 14 hours total)	Clock time starts with the injection of the BFB. Analysis of samples and QC items must conclude within expiration of clock time. Subsequent analysis requires new BFB.	Not applicable
Tune Standard (BFB)	At beginning of each clock	Refer to Attachment 7.	- Perform instrument maintenance - Re-tune.
Trap Check Standard (Optional)	At beginning of each clock	<RL (Chloromethane & Bromomethane)	- Perform instrument maintenance. - Change trap.

QC Item	Frequency	Criteria	Corrective Action
Initial Calibration (ICAL) - Minimum of 3 points for EPA 624 and 624.1. - Minimum of 5 points for EPA 8260B, EPA 8260C and EPA 8260D and SM6200B.	Upon instrument set-up, and after unsuccessful CCV	<u>EPA 624 and EPA 624.1</u>: %RSD < 35% If %RSD > 35%, use curve fit w/ r^2>0.990. <u>EPA 8260B:</u> - CCC: %RSD < 30% - SPCC: RRF_{avg} > Attachment 5 - If %RSD >15%, use curve fit with r^2 > 0.990. <u>EPA 8260C and EPA 8260D</u> -5-point Minimum RRF per Attachment 6. %RSD < 20%. If %RSD > 20%, use curve fit w/ r^2 > 0.990; EPA 8260C- Read back criteria for all levels of +/- 30% of true value. EPA 8260D- Readback criteria is +/- 50% for RL level and +/- 30% for all others. <u>SM6200B</u> - 5pt minimum RSD<20%. If >20% use curve fit with r^2 > 0.994.	-Reanalyze standard(s) -Prepare new standard(s) and reanalyze -Perform injector port maintenance and reanalyze standards -Retune and reanalyze standards -Replace column and reanalyze standards -Clean source and reanalyze standards

QC Item	Frequency	Criteria	Corrective Action
Initial Calibration Verification (ICV) - Second Source	After each ICAL	EPA 624: Attachment 12 EPA 624.1: Attachment 13 EPA 8260B: CCC ≤ 20%D All other analytes ≤ 30%D Poor Performer %D ≤50%-as per Attachment 11 EPA 8260C and EPA 8260D: %D ≤ 30% Poor Performer %D <50% as per Attachment 11. SM6200B %D ≤30%	-Reanalyze standard -Prepare new standard and reanalyze -Recalibrate

QC Item	Frequency	Criteria	Corrective Action
Continuing Calibration Verification (CCV)	After BFB	EPA 624: Attachment 12 EPA 624.1 Attachment 13 EPA 8260B: - CCC: %D < 20% - SPCC: RRF > Attachment 5 - Non-CCC and non-SPCC: %D <60%; EPA 8260C and EPA 8260D: EPA 8260C -Meet minimum RF criteria in Attachment 6. EPA 8260D does not mandate minimum RF criteria. All Analytes %D ≤ 20%, no analyte >50%D. Poor Performer %D may be >50%D as per Attachment 11. RLCCV is required to report NDs for analytes outside 20%D or RRF criteria. An NCM is required to denote analytes outside criteria. SM6200B: %D < 30%D CCV required at beginning and end of sequence with alternating levels used.	-If CCV %D is above control limits, except 8260B CCC analytes, and there are no detections in the samples, the laboratory will report the non-detect results with an NCM -Reanalyze standard -Prepare new standard and reanalyze -Recalibrate

QC Item	Frequency	Criteria	Corrective Action
Internal Standards (ISTD)	Spiked in all CCVIS, samples, and batch QC items	EPA 8260B/C/D, EPA 624, EPA 624.1 CCVIS: - Area -50% to +100% CCV level in ICAL. - RT +/-10 seconds from ICAL. Samples & batch QC items: - Area within -50% to +100% of previous CCVIS. SM6200B Area +/- 30% from mean of calibration	-Evaluate chromatogram, spectra, and integrations -Reanalyze extract -Perform instrument maintenance and reanalyze extract -Re-extract and reanalyze if sufficient sample available
Surrogate Compounds	Spiked in all samples and batch QC items.	Within MLG limits	-Evaluate chromatogram, spectra, and integrations -Reanalyze sample, if sufficient sample available
Analytical Batch Definition	Analyzed together w/in 24-hr timeframe; not to exceed 20 field samples	Not Applicable	Not Applicable
Method Blank (MB)	One per batch	No analytes detected ≥ ½ RL or > 1/10th the amount measured in a sample.	Refer to SOP SA-QA-017

QC Item	Frequency	Criteria	Corrective Action
Laboratory Control Sample (LCS)	One per batch	Within LIMS MLG Limits[1]	Refer to SOP SA-QA-017
Laboratory Control Sample Duplicate (LCSD)	One per batch, when insufficient sample is provided for MS/MSD	Within LIMS MLG Limits[1]	Refer to SOP SA-QA-017
Matrix Spike (MS)	EPA 8260B, EPA 8260C, EPA 8260D and EPA624.1: One per batch EPA 624: 1 per 10% of samples	Within LIMS MLG Limits	Refer to SOP SA-QA-017
Matrix Spike Duplicate (MSD)	One per batch	Within LIMS MLG Limits	Refer to SOP SA-QA-017
Initial Demonstration of Capability (IDOC)	Initially, per analyst, per analyte/method/matrix combination	Refer to SOP SA-QA-006	Refer to SOP SA-QA-006 Note: Unsupervised work must not begin until acceptable IDOC is obtained.
Continuing Demonstration of Capability (CDOC)	Annually, per analyst, per analyte/method/matrix combination	Refer to SOP SA-QA-006	Refer to SOP SA-QA-006

QC Item	Frequency	Criteria	Corrective Action
Reporting Limit Verification (RLV)	Upon method/instrument set-up, per analyte/method/matrix combination. Then quarterly thereafter (for DOD ELAP) or annually thereafter (for non-DOD ELAP)	Refer to SOP SA-QA-007	Refer to SOP SA-QA-007
Method Detection Limit Study (MDL)	Upon method/instrument set-up, per analyte/method/matrix combination	Refer to SOP SA-QA-007	Refer to SOP SA-QA-007
MDL Verification (MDLV)	Upon method/instrument set-up, per analyte/method/matrix combination. Then quarterly thereafter (for DOD ELAP) or annually thereafter (for non-DOD ELAP)	Refer to SOP SA-QA-007	Refer to SOP SA-QA-007

[1]South Carolina requires 70-130% LCS recovery limits for all target analytes with the exception of poor performers, which have acceptance criteria of 60-140%. All target analytes must pass LCS criteria.
For South Carolina UST program, all analytes must meet accuracy limits of 70-130% and precision of <20%. The use of poor performer limits is not permitted.

Attachment 4:
Preventative Maintenance and Troubleshooting

Instrument Labeling

Each instrument must be labeled with its name or ID (e.g., MSA, ICP-D, etc.). Additionally, non-operational instruments must be isolated from service or marked as being out of service. Each piece of equipment has an "Operational / Not Operational" sticker that is used for this purpose.

Maintenance Log

A maintenance log must be established for each piece of equipment used in the laboratory. All maintenance that is performed on the instrument must be recorded in the log including:

- type of maintenance performed (Note: This includes preventative/routine maintenance; non-routine maintenance; maintenance performed by an external vendor; updates to software versions; etc.)
- analyst or technician performing the maintenance
- date the maintenance was performed
- detailed explanation of the reason for the maintenance
- resolution of the problem and return to control
- all service calls from instrument representatives
- resolution of the problem (i.e., the Return to Control)

Preventive Maintenance

LABORATORY EQUIPMENT PREVENTIVE MAINTENANCE SCHEDULE								
	Service Interval							
EQUIPMENT ITEM	D	W	M	Q	SA	A	AN	SERVICE LEVEL
Injector Port							X	Replace septum, sleeve, inlet seal, and washer (Recommend every 2 weeks)
Sparge Tubes							X	Clean (Recommend every 3 months)
Column							X	Change column (Recommend annually)

D=daily; W=Weekly; M=monthly; Q=Quarterly; SA=semi-annually; A=annually; AN=as needed

Troubleshooting

Troubleshooting must be documented as outlined above. If possible, troubleshooting is best performed in a step-wise manner to systematically isolate instrument components. Refer to the instrument manufacturer's guides for specific information and strategies. Enlist assistance from technical and/or department management as needed.

Contingency Plan

Close contact is maintained with service personnel to ensure optimal instrument functioning. An extensive spare parts inventory is maintained for routine repairs. Since instrumentation is standardized throughout the laboratory network, spare parts and components can be readily exchanged among the network.

In general, the laboratory has at least one backup unit for each critical unit. In the event of instrument failure, portions of the sample load may be diverted to duplicate instrumentation, the analytical technique switched to an alternate approved technique (such as manual colorimetric determination as opposed to automated colorimetric determination), or samples shipped to another properly certified or approved Eurofins TestAmerica location.

Attachment 5:
EPA 8260B Calibration Criteria: SPCCs and CCCs

Calibration Check Compounds (CCCs)

Initial Calibration	Continuing Calibration
<=30% RSD	<=20% difference from initial calibration

System Performance Check Compounds (SPCCs)

SPCC	Minimum RRF
Chloromethane	0.10
1,1-Dichloroethane	0.10
Chlorobenzene	0.30
Bromoform	>0.10
1,1,2,2-Tetrachloroethane	0.30

Note: The CCC and SPCC criteria must be met even if the calibration curve option is used for quantitation. If the CCC and SPCC criteria do not pass, a new calibration curve must be prepared and analyzed.

Calibration Check Compounds (CCCs)
Vinyl chloride
1,1-Dichloroethene
Chloroform
1,2-Dichloropropane
Toluene
Ethylbenzene

System Performance Check Compounds (SPCCs)
Chloromethane
1,1-Dichloroethane
Chlorobenzene
Bromoform
1,1,2,2-Tetrachloroethane

Attachment 6:
EPA 8260C Minimum Response Factors

Compound	Minimum RRF
Dichlorodifluoromethane	0.100
Chloromethane	0.100
Vinyl Chloride	0.100
Bromomethane	0.100
Chloroethane	0.100
Trichlorofluoromethane	0.100
1,1-Dichloroethene	0.100
Freon-113	0.100
Acetone	0.050*
Carbon disulfide	0.100
Methyl acetate	0.100
Methylene chloride	0.100
trans-1,2-Dichloroethene	0.100
Cis-1,2-Dichloroethene	0.100
MTBE	0.100
1,1-Dichloroethane	0.200
2-Butanone	0.050*
Chloroform	0.200
1,1,1-Trichloroethene	0.100
Cyclohexanone	0.100
Carbon tetrachloride	0.100
Benzene	0.500
1,2-Dichloroethane	0.100
Trichloroethene	0.200
Methylcyclohexane	0.100
1,2-Dichloropropane	0.100
Bromodichloromethane	0.200
Cis-1,3-Dichloropropene	0.200
Trans-1,3-Dichloropropene	0.100
4-Methyl-2-pentanone	0.050*
Toluene	0.400
1,1,2-Trichloroethane	0.100
Tetrachloroethene	0.200
2-Hexanone	0.050*
Dibromochloromethane	0.100
1,2-Dibromoethane	0.100
Chlorobenzene	0.500
Ethylbenzene	0.100
m,p-Xylene	0.100
o-xylene	0.300
Styrene	0.300
Bromoform	0.100

Isopropylbenzene	0.100
1,1,2,2-Tetrachloroethane	0.300
Compound	**Minimum RRF**
1,3-Dichlorobenzene	0.600
1,4-Dichlorobenzene	0.500
1,2-Dichlorobenzene	0.400
1,2-Dibromo-3-chloropropane	0.050
1,2,4-Trichlorobenzene	0.200

*These RRFs are different than those listed in the EPA 8260C reference method.

Attachment 7:
BFB Tune Criteria

m/e	Abundance Criteria
50	15.0-40.0% of mass 95
75	30.0-60.0% of mass 95
95	Base peak, 100% relative abundance
96	5.0-9.0% of mass 95
173	<2.0% of mass 174
174	>50% of mass 95
175	5.0-9.0% of mass 174
176	95.0-101.0% of mass 174
177	5.0-9.0% of mass 176

Attachment 8:
Target Compound Information: Ions and ISTDs

Analyte	Quant Ion	Secondary Ions		ISTD
1,1,1,2-Tetrachloroethane	131	133	119	2
1,1,1-Trichloroethane	97	99	61	1
1,1,2,2-Tetrachloroethane	83	85	168	2
1,1,2-Trichloro-1,2,2-trifluoroethane	101	151	103	1
1,1,2-Trichloroethane	83	97	99	1
1,1-Dichloroethane	63	65	83	1
1,1-Dichloroethene	96	61	98	1
1,1-Dichloropropene	75	110	77	1
1,2,3-Trichlorobenzene	180	182		3
1,2,3-Trichloropropane	110	112		2
1,2,4-Trichlorobenzene	180	182	145	3
1,2,3-Trimethylbenzene	105	120	77	3
1,2,4-Trimethylbenzene	105	120	77	3
1,2-Dibromo-3-Chloropropane	157	75	155	3
1,2-Dichlorobenzene	146	148	111	3
1,2-Dichloroethane	62	49	64	1
1,2-Dichloroethene, Total	Sum of cis and trans isomers			
1,2-Dichloropropane	63	76	65	1
1,3,5-Trimethylbenzene	105	120	77	2
1,3,5-Trichlorobenzne	180	182	184	3
1,3-Dichlorobenzene	146	148	111	3
1,3-Dichloropropane	76	78	41	1
1,3-Dichloropropene, Total	Sum of cis and trans isomers			
1,4-Dichlorobenzene	146	148	111	3
1-Chlorohexane	41	43		2
1-Methylnaphthalene	142	141		3
2,2-Dichloropropane	77	41		1
2-Butanone (MEK)	43	72		1
2-Chloro-1,3-butadiene	53	88		1
2-Chloroethyl vinyl ether	63	65	106	2
2-Chlorotoluene	126	91	63	2
2-Hexanone	43	58		1
2-Methylnaphthalene	142	141		3
2-Nitropropane	43	41	39	2
3,3-Dimethyl-1-Butanol	57	69	41	1
3-Chloro-1-propene	76	41		1
3-Methylhexane	43	71		2
4-Chlorotoluene	126	91	63	2
4-Isopropyltoluene	119	134	91	3
4-Methyl-2-pentanone (MIBK)	43	57	58	1
Acetone	58	43		1
Acetonitrile	41	40		1

	56	55		1
Acrylonitrile	53	52	51	1
Amyl acetate	70	61		3
Benzene	78	50		1
Analyte	**Quant Ion**	**Secondary Ions**		**ISTD**
Benzyl chloride	91	63		3
Bromobenzene	156	77	158	2
Bromoform	173	171	175	2
Bromomethane	96	94	79	1
Butadiene	54	39		1
Carbon disulfide	76	78		1
Carbon tetrachloride	117	119	121	1
Chlorobenzene	112	77	51	2
Chlorobromomethane	49	128	130	1
Chlorodibromomethane	129	127	131	1
Chloroethane	64	66		1
Chloroform	83	85	47	1
Chloromethane	50	52		1
Chloroprene	53	88		1
cis-1,2-Dichloroethene	96	61	98	1
cis-1,3-Dichloropropene	75	77	110	1
Cyclohexane	56	69	84	1
Cyclohexanone	55	42	98	2
Dibromomethane	93	174	95	1
Dichlorobromomethane	83	85	129	1
Dichlorodifluoromethane	85	87	101	1
Dichlorofluoromethane	67	69		1
Ethyl acetate	43	61	70	1
Ethyl acrylate	55	73		1
Ethanol	45	45		1
Ethyl ether (diethyl ether)	59	74	45	1
Ethyl methacrylate	69	41	39	1
Ethylbenzene	91	106	51	2
Ethylene Dibromide	107	109		1
Ethylene oxide	43	44		1
Furan	68	39		1
Hexachlorobutadiene	225	223	190	3
Hexane	57	41	43	1
Iodomethane	142	127		1
Isobutyl alcohol	43	41		1
Isopropyl acetate	61	59	87	1
Isopropyl ether	45	43		1
Isopropylbenzene	105	120	77	2
m,p-Xylene	106	91	77	2
Methacrylonitrile	67	52		1

Methyl acetate	43	74		1
Methyl acrylate	55	85		1
Methyl methacrylate	69	41		1
Methyl styrene	118	103	91	3
Methyl tert-butyl ether	73	57		1
Methylcyclohexane	83	55	98	1
Methylene Chloride	84	49	86	1
Naphthalene	128	102	51	3
Analyte	**Quant Ion**	**Secondary Ions**		**ISTD**
n-Butanol	56	41	43	1
n-Butyl acetate	43	56		1
n-Butyl acrylate	55	56		2
n-Butylbenzene	91	92	134	3
n-Heptane	43	57	71	1
N-Propylbenzene	120	91	65	2
o-Xylene	106	91	77	2
Pentachloroethane	167	130		3
Propene oxide	58	43		1
Propionitrile	54	55		1
sec-Butylbenzene	105	134	91	2
Styrene	104	78	103	2
T-Amyl Alcohol	59	73	55	1
Tert-amyl methyl ether	73	55	87	1
Tert-butyl alcohol	59	41		1
tert-Butylbenzene	119	91	134	2
Tert-butyl ethyl ether	59	87		1
Tert-Butyl Formate	59	57	41	1
Tetrachloroethene	164	166	168	1
Tetrahydrofuran	42	71		1
Toluene	92	91	65	1
trans-1,2-Dichloroethene	96	61	98	1
trans-1,3-Dichloropropene	75	77	110	1
trans-1,4-Dichloro-2-butene	53	88	89	2
Trichloroethene	130	95	132	1
Trichlorofluoromethane	101	103	105	1
Trichlorotrifluoroethane (Freon 113)	101	151	103	1
Trihalomethanes, Total	Sum of chloroform, dichlorobromomethane, dibromochloromethane, and bromoform			
Vinyl acetate	43	86		1
Vinyl chloride	62	64		1
Xylenes, Total	Sum of m/p- and o- isomers			

Analyte	Quant Ion	Secondary Ions		ISTD
Surrogates				
Dibromofluoromethane	113	81	111	1
Toluene-d8	98	100	70	2
p-Bromofluorobenzene	95	174	176	3
1,2-Dichloroethane-d4	65	67	102	1
Internal Standards				
Fluorobenzene	96	77		1
Chlorobenzene-d5	82	117	119	2
1,4-Dichlorobenzene-d4	152	115	150	3

Note: For a complete list of target analytes for each method, refer to the LIMS Method Limit Groups (MLGs).

Attachment 9:
Instrument Configurations

System	GC	MS	P/T	Autosampler	Trap	GC/MS Data System	Methods
CMSA2	Agilent 6890	Agilent 5973	EST Evolution	EST Centurion	Supelco VOCARB 3000	Agilent Chemstation	EPA 624/624.1 EPA 8260B/C/D AQ 524.2 EPA 8260_AK
CMSB	Agilent 6890	Agilent 5973	ATOMX XYZ	ATOMX XYZ	Supelco VOCARB 3000	Agilent Chemstation	EPA 624/624.1 EPA 8260B/C/D AQ EPA 8260_AK
CMSC	Agilent 7890	Agilent 5975	ATOMX XYZ	ATOMX XYZ	Supelco VOCARB 3000	Agilent Chemstation	EPA 8260B/C/D SO EPA 8260_AK
CMSAB	Agilent 6890	Agilent 5973	OI Eclipse 4760	OI 4100	OI Trap	Agilent Chemstation	EPA 624/624.1 EPA 8260B/C/D AQ
CMSAD	Agilent 6890	Agilent 5973	EST Encon	EST Centurion	Supelco VOCARB 3000	Agilent Chemstation	8260 Screener instrument
CMSAG	Agilent 6890	Agilent 5973	EST Evolution	EST Centurion	Supelco VOCARB 3000	Agilent Chemstation	524.2 624.1/624 8260 B/C/D AQ

System	GC	MS	P/T	Autosampler	Trap	GC/MS Data System	Methods
CMSS	Agilent 6890	Agilent 5973	OI Eclipse 4760	OI 4100	OI Trap	Agilent Chemstation	EPA 624/624.1 EPA 8260B/C/D AQ
CMSO	Agilent 6890	Agilent 5973	EST Encon	EST Centurion	Supelco VOCARB 3000	Agilent Chemstation	EPA 624/624.1 EPA 8260B/C/D AQ EPA 8260_AK SM6200B
CMSP	Agilent 6890	Agilent 5973	EST Encon	EST Centurion	Supelco VOCARB 3000	Agilent Chemstation	EPA 8260B/C/D SO EPA 8260_AK
CMSAH	Agilent 6890	Agilent 5973	EST Encon	EST Centurion	Supelco VOCARB 3100	Agilent Chemstation	EPA 624/624.1 EPA 8260B/C/D AQ EPA 8260_AK
CMSAA	Agilent 6890	Agilent 5973	EST Encon	EST Centurion	Supelco VOCARB 3000	Agilent Chemstation	EPA 624/624.1 EPA 8260B/C/D AQ EPA 8260_AK
CMSU	Agilent 6890	Agilent 5973	EST Encon	EST Centurion	Supelco VOCARB 3000	Agilent Chemstation	EPA 624 /624.1 EPA 8260B/C/D AQ

Attachment 10
Standard Information and Recipes

Store standards in glass vials fitted with Teflon-lined caps in standards-only refrigerator at <-10C. Choose a container volume that will have minimal headspace when standard is added. Mark the level on the outside of the container so that evaporation of the solvent will be apparent.

The expiration dates represent the maximum time a standard can be maintained. Standards that exhibit obvious signs of degradation must be replaced sooner.

Routine Target Compounds

Stock Standard Mixes

Stock/Mix	LIMS ID	Vendor/Part Number	Concentration (ug/mL)
8260 List 1/ Std #1 Mega Mix	571992	Restek 571992	2500 (majority of compounds)
8260 List 1/ Std #2 Ketones	569721	Restek 569721	12,500
8260 List 1/Std #3 Gases	569722	Restek 569722	2500
8260 List 1/ Std #4 2-CEVE	569723	Restek 569723	2500
8260 List 1/ Std #5 Acrolein	568720	Restek 568720	20,000
8260 List 1/Std #6 Vinyl Acetate	569724	Restek 569724	5000
8260 IS/Surrogate Mix	567650	Restek 56750	2500

Expiration:
Un-opened ampuls: manufacturer's expiration date
Opened ampuls: 1 month (Note: These ampuls are used to prepare the Working Standards and are typically consumed/disposed upon opening.)

8260 Working ISTD
(LIMS ID: DoD ISTD)

Stock/Mix	Initial Volume (uL)	Final Volume (mL)	Final Concentration (ug/mL)
8260 IS/Surrogate Mix	500	25	50

Solvent: P/T Methanol
Expiration: 1 Month

8260 List 1
(Mega Mix Working Standard)
(LIMS ID: 8260 MMix)

Stock/Mix	Initial Volume (uL)	Final Volume (mL)	Final Concentration (ug/mL)
8260 List 1/ Std #1 Mega Mix	500	25	50
8260 List 1/ Std #2 Ketones	500		250
8260 List 1/ Std #4 2-CEVE	500		50
8260 List 1/Std #6 Vinyl Acetate	500		100
8260 Surrogate Standard	500		50

Expiration: 2 weeks from date prepared

(LIMS ID: VM_GASINT)

Stock/Mix	Initial Volume (uL)	Final Volume (mL)	Final Concentration (ug/mL)
8260 List 1/Std #3 Gases	500	25	50

Expiration: 1 week from date prepared

(LIMS ID: VM_Acrolein)

Stock/Mix	Initial Volume (uL)	Final Volume (mL)	Final Concentration (ug/mL)
Acrolein	500	10	1000

Expiration: 2 weeks from date prepared

Solvent: P/T Methanol
(Note: The 25mL final volume is split between five 5mL Mini-nert vials. Due to the volatility of the gas analytes, each vial is assigned a 1-week expiration date from the date that vial was opened, not to exceed 14 days from the preparation date.)

Calibration Standards

Stock/Mix		1	2	3	4	5	6	7	8
		Aliquot (uL) to prepare CAL standard							
8260 MMix		2	4	10	20	40	100	200	400
VM_GASINT		2	4	10	20	40	100	200	400
8260 List 1/ Std #5 Acrolein		2	4	10	20	40	100	200	400
8260 ISTD		100	100	100	100	100	100	100	100
Volume of MeOH		796	792	780	760	720	600	400	0

100mL final volume for all levels. Expiration: 24 hours from date prepared

EPA 8260_AK Calibration Standards

Stock/Mix	1	2	3	4	5	6	7
	Aliquot (uL) to prepare CAL standard						
8260 MMix	0.50	1.0	2.0	5.0	10	25	50
8260 List 1/ Std #5 Acrolein	0.50	1.0	2.0	5.0	10	25	50
VM_GASINT	0.5	1.0	2.0	5.0	10	25	50
8260 ISTD	25	25	25	25	25	25	25
Volume of MeOH	99	98	96	90	80	50	0

50mL final volume for all levels. Expiration: 24 hours

The laboratory normalizes the methanol content of the initial calibration standards to minimize methanol suppression of the early eluting analytes caused by varying amounts of methanol contained in the working standards. For example, the high level initial calibration standard is created by the addition of 450uL of the stock standards brought up to 50mL with reagent water. This contributes 450uL of methanol to the standard. The low level initial calibration standard is created by the addition of 54uL of the stock standards bought up to 50mL with reagent water. This contributes 54uL of methanol to the standard. The laboratory adds an additional 396uL of methanol to the low level standard to normalize its methanol content with that of the high level standard (i.e., for a total of 450uL). This methanol normalization is a common industry practice and allows for more consistent responses of those analytes co-elute with methanol.

8260 List 2 (Non-routine)

Stock Standard Mixes

Stock/Mix	LIMS ID	Vendor/Part Number	Concentration (ug/mL)
8260 List 2/Std #2	569726	Restek 569726	2500
8260 List 2/Std # 3 Cyclohexanone	567648	Restek 567648	2000
8260 List 2/ Std #6	570808	Restek 570808	2500-62500
8260 List 2/ Std #7	570809	Restek 570809	2500-5000
8260 L3/ Std #1	569728	Restek 569728	2500-100000

8260 Working ISTD
(LIMS ID: 8260 ISTD)

Stock/Mix	Aliquot Volume (uL)	Final Volume (mL)	Final Concentration (ug/mL)
8260 IS/Surrogate Mix	5000	25	50

Solvent: P/T Methanol
Expiration: 1 month

8260 List 2
(LIMS ID: 8260L2)

Stock/Mix	Aliquot Volume (uL)	Final Volume (mL)	Final Concentration (ug/mL)
8260 List 2/Std #2	500	25	50-2500
8260 List 2/ Std #3	625		500
8260 List 2/ Std #6	500		50-1250
8260 List 2/ Std #7	500		50-100
8260 List 3/ Std #1	500		50-2000

Solvent: P/T Methanol
Expiration: 2 weeks from date prepared.

Calibration Standards

Stock/Mix	1	2	3	4	5	6	7
	Aliquot (uL) to prepare CAL standard						
8260 List 2	1	5	10	20	50	100	200
8260 ISTD	50	50	50	50	50	50	50
Volume of water (mL)	50	50	50	50	50	50	50
Target Compounds (ng) (majority of compounds)	5	25	50	100	250	500	1000
Internal Standards (ng)	250	250	250	250	250	250	250

Note: Record calibration standard preparation in A/D batch.
Expiration: 24 hours

SM6200B List 1
(Mega Mix Working Standard)
(LIMS ID: 6200 MMix 1)

Stock/Mix	Initial Volume (uL)	Final Volume (mL)	Final Concentration (ug/mL)
8260 List 1/ Std #1 Mega Mix	200	10	50
8260 List 1/ Std #2 Ketones	400		500
8260 List 1/Std #6 Vinyl Acetate	400		200
8260 Surrogate Standard	200		50

Expiration: 2 weeks from date prepared

(LIMS ID: 6200 MMIX 3G)

Stock/Mix	Initial Volume (uL)	Final Volume (mL)	Final Concentration (ug/mL)
8260 List 1/Std #3 Gases	100	5	50

Expiration: 1 week from date prepared

SM6200B List 2

(Mega Mix 2 Working Standard)
(LIMS ID: 6200 MMix 2)

Stock/Mix	Initial Volume (uL)	Final Volume (mL)	Final Concentration (ug/mL)
8260 List 3/ Std #1 Polar Additions	100	10	250
8260 Ethanol Standard	400		4000
Custom 6200 Oxygenate 1	1000		1000
Custom 6200 Oxygenate 2	250		250

Expiration: 2 weeks from date prepared

Solvent: P/T Methanol
(Note: The 10mL final volume is split between two 5mL Mini-nert vials. Due to the volatility of the gas analytes, each vial is assigned a 1-week expiration date from the date that vial was opened, not to exceed 14 days from the preparation date.)

SM6200B Calibration Standards List 1

Stock/Mix	1	2	3	4	5	6	7	8
	Aliquot (uL) to prepare CAL standard							
6200 MMIX 1	1	2	5	10	20	40	50	100
6200 MMIX 3G	1	2	5	10	20	40	50	100
8260 ISTD	100	100	100	100	100	100	100	100
Volume of MeOH	790	786	784	780	760	720	700	600

100mL final volume for all levels. Expiration: 24 hours

SM6200B Calibration Standards List 2

Stock/Mix	1	2	3	4	5	6	7	8
	Aliquot (uL) to prepare CAL standard							
6200 MMIX 2	2	4	10	20	40	80	100	200
8260 ISTD	100	100	100	100	100	100	100	100
Volume of MeOH	0	0	0	0	0	0	0	0

100mL final volume for all levels. Expiration: 24 hours

Attachment 11
Poor Performers

As indicated in EPA 8260B/C/D via assessment of laboratory control sample recoveries and control charts, the compounds listed below are poor performers and/or behave erratically.

** Note for South Carolina the poor performers must recover with 60-140%. For SouthCarolina UST work all analytes must recover 70-130%.

Note: An NCM must be initiated to denote this situation.

Poor Performer List

1,1,2-Trichloro-1,2,2-Trifluoroethane
1,2-Dibromo-3-Chloropropane
1,2,3-Trichlorobenzene
1,4-Dioxane
2-Butanone
2-Chloroethyl vinyl ether
2-Hexanone
2-Methyl-2-Propanol (t-Butyl Alcohol)
4-Methyl-2-Pentanone
2-Nitropropane
2-Propanol
Acetone
Acetonitrile
Acrolein
Acrylonitrile
Allyl Chloride
Carbon Disulfide
Bromoform
Bromomethane
Chloroethane
Chloromethane
Cis-1,4-Dichloro2-butene
Cyclohexanone
Dichlorodifluoromethane
Dichlorofluoromethane
Ethanol
Ethyl Acetate
Ethyl Methacrylate
Iodomethane
Isobutyl Alcohol
Isopropyl Alcohol
n-Butanol
Naphthalene
Pentachloroethane
Propionitrile
Trans-1,4-Dichloro2-butene
Trichlorotrifluoromethane
Vinyl Acetate
Methyl Methacrylate
Methyl Acetate
Methyl Cyclohexane
Methacrylonitrile

Attachment 12
EPA 624 CCV and ICV Criteria
(Q Table)

Analyte	Criteria* (ug/L)
Benzene	12.8-27.2
Bromodichloromethane	13.1-26.9
Bromoform	14.2-25.8
Bromomethane	2.8-37.2
Carbon tetrachloride	14.6-25.4
Chlorobenzene	13.2-26.8
Chloroethane	7.6-32.4
2-chloroethyl vinyl ether	D-44.8*
Chloroform	13.5-26.5
Chloromethane	D-40.8
Dibromochloromethane	13.5-26.5
1,2-Dichlorobenzene	12.6-27.4
1,3-Dichlorobenzene	14.6-25.4
1,4-Dichlorobenzene	12.6-27.4
1,1-Dichloroethane	14.5-25.5
1,2-Dichloroethane	13.6-26.4
trans-1,2-Dichloroethene	13.9-26.1
1,1-Dichloroethene	10.1-29.9
1,2-Dichloropropane	6.8-33.2
cis-1,3-Dichloropropene	4.8-35.2
trans-1,3-Dichloropropene	10.0-30.0
Ethylbenzene	11.8-28.2
Methylene chloride	12.1-27.9
1,1,2,2-Tetrachloroethane	12.1-27.9
Tetrachloroethene	14.7-25.3
Toluene	14.9-25.1
1,1,1-Trichloroethane	15.-25.0
1,1,2-Trichloroethane	14.2-25.8
Trichloroethene	13.3-26.7
Trichlorotrifluoromethane	9.6-30.4
Vinyl chloride	0.8-39.2

*These values are given in ug/L (i.e., concentration ranges). The laboratory has converted these values into percent recovery limits to be used to assess CCV, ICV, LCS/LCSD, and MS/MSD.

Attachment 13
EPA 624.1 CCV and ICV Criteria
(Q Table)

Analyte	CCV/LCS/ICV Criteria (% Recovery)
Acrolein	60-140
Acrylonitrile	60-140
Benzene	65-135
Bromodichloromethane	65-135
Bromoform	70-130
Bromomethane	15-185
Carbon tetrachloride	70-130
Chlorobenzene	65-135
Chloroethane	40-160
2-chloroethyl vinyl ether	>0-225
Chloroform	70-135
Chloromethane	>0-205
Dibromochloromethane	70-135
1,2-Dichlorobenzene	65.135
1,3-Dichlorobenzene	70-130
1,4-Dichlorobenzene	65-135
1,1-Dichloroethane	70-130
1,2-Dichloroethane	70-130
trans-1,2-Dichloroethene	70-130
1,1-Dichloroethene	50-150
1,2-Dichloropropane	35-165
cis-1,3-Dichloropropene	25-175
trans-1,3-Dichloropropene	50-150
Ethylbenzene	60-140
Methylene chloride	60-140
1,1,2,2-Tetrachloroethane	60-140
Tetrachloroethene	70-130
Toluene	70-130
1,1,1-Trichloroethane	70-130
1,1,2-Trichloroethane	70-130
Trichloroethene	65-135
Trichlorotrifluoromethane	50-150
Vinyl chloride	5-195

*Analytes not listed in the above table have a default % recovery of 60-140.

Attachment 14
Glassware Cleaning Procedures

GLASSWARE CLEANING PROCEDURES

VOLATILES DEPARTMENT

1. Rinse glassware 3 times thoroughly with DI water.
2. Rinse once with methanol and then rinse once more with DI water.
3. Place glassware, top-down, within storage rack and allow to air dry.
4. If glassware was used to prepare waste sample, use FL-70 and water to scrub glassware and follow previous steps.

Attachment 15
Data Entry Procedures

eurofins | Environment Testing TestAmerica

Work Instruction
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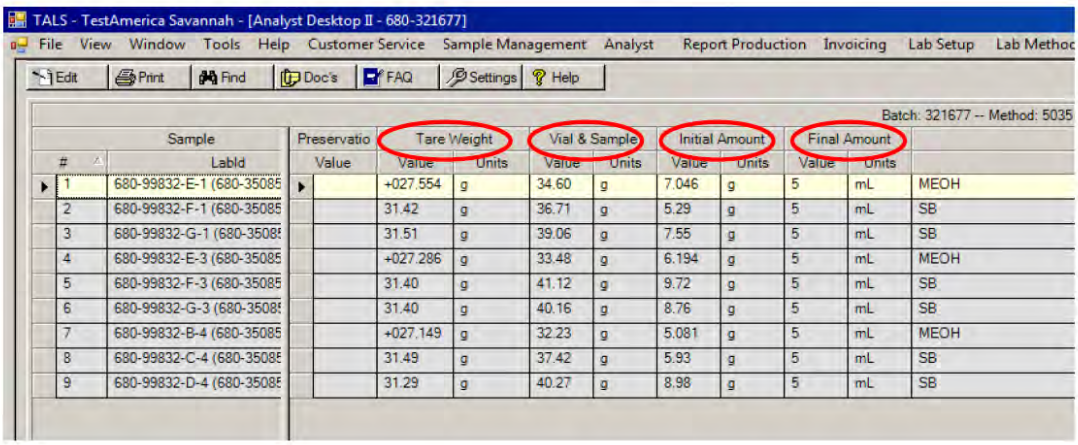
Data Entry Procedures for Soil Preparations and Dilutions (EPA 8260B, EPA 8260C, and EPA 8015C_GRO)

Summary
For EPA 8260B, EPA 8260C, and EPA 815C_GRO, the majority of soil samples are collected in 3 pre-weighed, pre-preserved vials. Two vials contain DI water or sodium bisulfate to be used for low-level analyses (i.e., analyzed straight). One vial contains methanol to be used for medium-level analyses (i.e., analyzed at dilution factor of 20 to 40, or higher). A preparation batch (i.e., a weight batch) is utilized to capture initial/final weights and volumes. Dilutions are captured via CHROM and are automatically transferred to the TALS analytical batch.

Note: The initial and final weight/volume entered in the TALS analytical batch must always be 5g and 5mL. The dilution factor is used to adjust calculations for Medium-Level soil samples. Adjustment/entry of initial volumes in TALS to reflect the MeOH sample aliquot (e.g., 125uL for a DF40) must not be performed as this will result in double-calculation of the dilution factor and miscalculation of final results.

Entry of Values in to the Weight Batch

1. Record the tare weight, which is found on the vial label, in the "Tare Weight" column.
2. Weigh the vial to determine the combined weight of the vial and sample. Enter into "Vial & Sample" column.
3. TALS automatically calculates Initial Amount by subtracting the Tare Weight from the Vial & Sample weight.
4. Enter purge volume in the "Final Amount" column. Note: Purge volume is always equivalent to 5mL.
5. Record preservative type in Worksheet Notes section - i.e., DI Water (DI), sodium bisulfate (SB), or Methanol (MEOH).



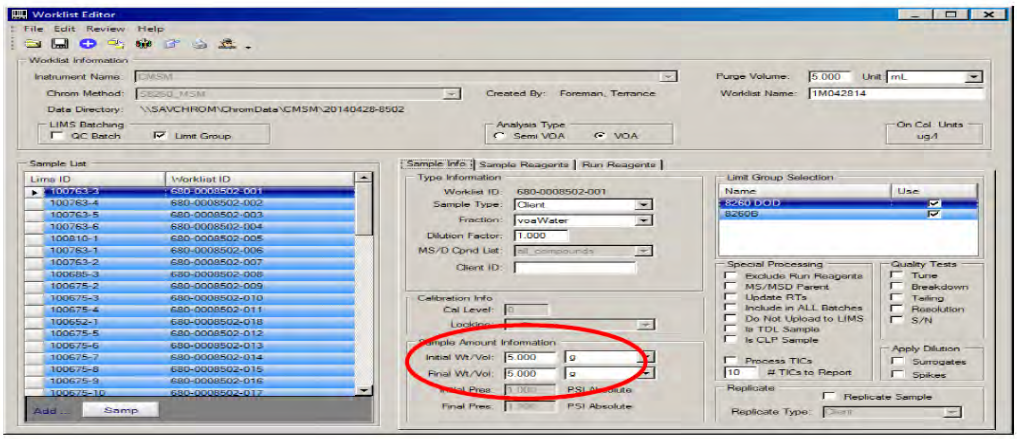
#	Sample LabId	Preservatio Value	Tare Weight Value	Tare Weight Units	Vial & Sample Value	Vial & Sample Units	Initial Amount Value	Initial Amount Units	Final Amount Value	Final Amount Units	
1	680-99832-E-1 (680-35085		+027.554	g	34.60	g	7.046	g	5	mL	MEOH
2	680-99832-F-1 (680-35085		31.42	g	36.71	g	5.29	g	5	mL	SB
3	680-99832-G-1 (680-3508		31.51	g	39.06	g	7.55	g	5	mL	SB
4	680-99832-E-3 (680-35085		+027.286	g	33.48	g	6.194	g	5	mL	MEOH
5	680-99832-F-3 (680-35085		31.40	g	41.12	g	9.72	g	5	mL	SB
6	680-99832-G-3 (680-3508		31.40	g	40.16	g	8.76	g	5	mL	SB
7	680-99832-B-4 (680-35085		+027.149	g	32.23	g	5.081	g	5	mL	MEOH
8	680-99832-C-4 (680-3508		31.49	g	37.42	g	5.93	g	5	mL	SB
9	680-99832-D-4 (680-3508		31.29	g	40.27	g	8.98	g	5	mL	SB

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Low-Level Soil Procedure

Note: Vials designated as DI or SB are used for the Low-Level analysis.

In the CHROM Worklist, enter 5g for the Initial Wt./Vol. and 5g as the Final Wt./Vol. (as shown below). This information is automatically transferred to the worksheet section of the analytical batch in TALS.



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Medium-Level Soil Procedure

Note: Vials designated as MEOH are used for the Medium-Level analysis. An aliquot of sample is taken from the MeOH vial and diluted in water.

The default (i.e., lowest) dilution for Medium-Level analyses is prepared as follows:

Method	Sample Aliquot (uL)	Final Volume (mL)	Dilution Factor
EPA 8260B / EPA 8260C	125	5	40
EPA 8015C_GRO	250	10	20*

Higher dilutions are prepared as follows:

Method	Sample Aliquot (uL)	Final Volume (mL)	Dilution Factor
EPA 8260B / EPA 8260C	100	5	50
EPA 8260B / EPA 8260C	50	5	100
EPA 8260B / EPA 8260C	25	5	200
EPA 8260B / EPA 8260C	10	5	500
EPA 8260B / EPA 8260C	5	5	1000
EPA 8015C_GRO	100	10	50*
EPA 8015C_GRO	50	10	100*
EPA 8015C_GRO	20	10	250*
EPA 8015C_GRO	10	10	500*
EPA 8015C_GRO	5	10	1000*
EPA 8015C_GRO	1	10	5000*
EPA 8015C_GRO	0.5	10	10000*

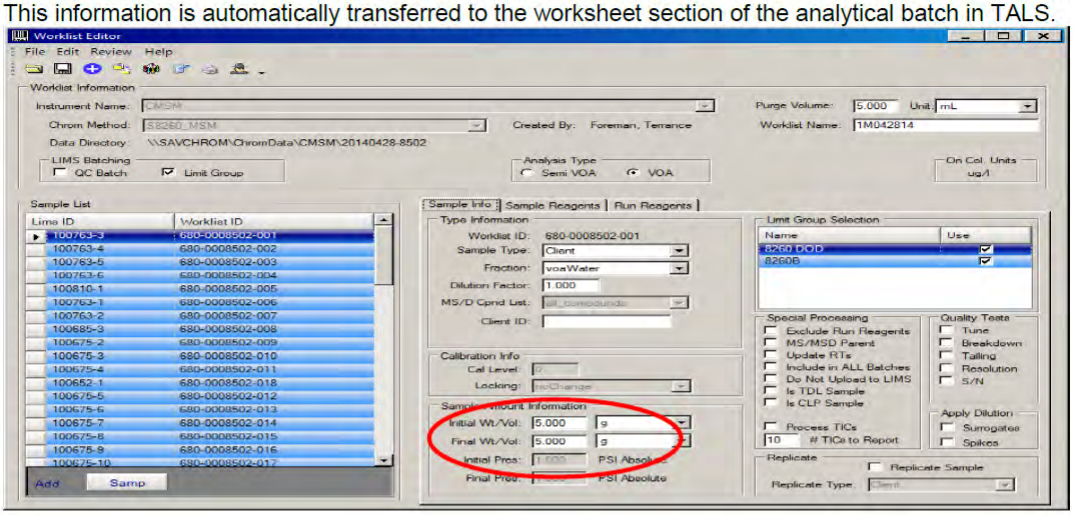
* = Due to differences in the autosampler types utilized for EPA 8260B and EPA 8260C versus EPA 8015C_GRO, there is a difference in the water volume added to the sample to prepare the dilution. The autosamplers utilized for EPA 8260B and EPA 8260C add 5mL of water upon sampling of the vial. The autosamplers utilized for EPA 8015C_GRO do not perform this action. As such, an additional 5mL of water is added to the EPA 8015C_GRO sample vial upon preparation of the dilution (i.e., the dilution is prepared in 10mL of water).

Note: The dilution factor obtained is based on the 5mL purge volume. (e.g., for EPA 8015C_GRO, a 250uL MeOH sample aliquot diluted into 10mL of water is equivalent to a dilution factor of 20.)

Note: Dilutions larger than those listed in the table require the use of a serial dilution.

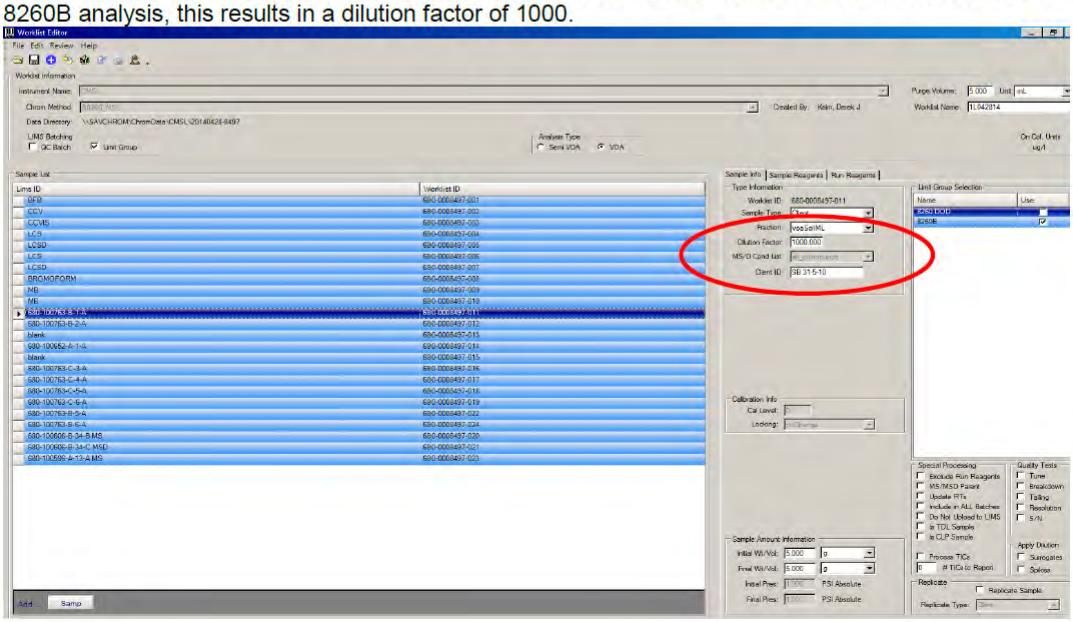
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In the CHROM Worklist, enter 5g for the Initial Wt./Vol. and 5g as the Final Wt./Vol. (as shown below). This information is automatically transferred to the worksheet section of the analytical batch in TALS.



Enter the dilution factor in CHROM to reflect the dilution prepared (as shown below). This information is automatically transferred to the worksheet section of the analytical batch in TALS.

In this example, a 5uL aliquot was taken from the MeOH vial and added to 5mL of water. For EPA 8260B analysis, this results in a dilution factor of 1000.



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Attachment 16:

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Table B-4. Organic Analysis by Gas Chromatography/Mass Spectrometry (GC/MS)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Tune Check	Prior to ICAL and prior to each 12-hour period of sample analysis.	Specific ion abundance criteria of BFB or DFTPP from method.	Retune instrument and verify.	Flagging is not appropriate.	No samples shall be analyzed without a valid tune.
Performance Check (Method 8270 only)	At the beginning of each 12-hour period, prior to analysis of samples.	Degradation ≤ 20% for DDT. Benzidine and pentachlorophenol shall be present at their normal responses and shall not exceed a tailing factor of 2.	Correct problem, then repeat performance checks.	Flagging is not appropriate.	No samples shall be analyzed until performance check is within criteria. The DDT breakdown and Benzidine/ pentachlorophenol tailing factors are considered overall system checks to evaluate injector port inertness and column performance and are required regardless of the reported analyte list.
Initial calibration (ICAL) for all analytes (including surrogates)	At instrument set-up and after ICV or CCV failure, prior to sample analysis.	Each analyte must meet one of the three options below: Option 1: RSD for each analyte ≤ 15%; Option 2: linear least squares regression for each analyte: $r^2 \geq 0.99$; *(continued next page)*	Correct problem then repeat ICAL.	Flagging is not appropriate.	Minimum 5 levels for linear and 6 levels for quadratic. No samples shall be analyzed until ICAL has passed.

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Table B-4. Organic Analysis by Gas Chromatography/Mass Spectrometry (GC/MS)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Initial calibration (ICAL) for all analytes (including surrogates) *(Continued)*		Option 3: non-linear least squares regression (quadratic) for each analyte: $r^2 \geq 0.99$.			If the specific version of a method requires additional evaluation (e.g., RFs or low calibration standard analysis and recovery criteria) these additional requirements must also be met.
Retention Time window position establishment	Once per ICAL and at the beginning of the analytical sequence.	Position shall be set using the midpoint standard of the ICAL curve when ICAL is performed. On days when ICAL is not performed, the initial CCV is used.	NA.	NA.	Calculated for each analyte and surrogate.
Evaluation of Relative Retention Times (RRT)	With each sample.	RRT of each reported analyte within ± 0.06 RRT units.	Correct problem, then rerun ICAL.	NA.	After maintenance is performed which may affect retention times, RRTs may be updated based on the daily CCV. RRTs shall be compared with the most recently updated RRTs.
Initial Calibration Verification (ICV)	Once after each ICAL, analysis of a second source standard prior to sample analysis.	All reported analytes within ± 20% of true value.	Correct problem. Rerun ICV. If that fails, repeat ICAL.	Flagging is not appropriate.	No samples shall be analyzed until calibration has been verified with a second source.

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Table B-4. Organic Analysis by Gas Chromatography/Mass Spectrometry (GC/MS)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Continuing Calibration Verification (CCV)	Daily before sample analysis; after every 12 hours of analysis time; and at the end of the analytical batch run.	All reported analytes and surrogates within ± 20% of true value. All reported analytes and surrogates within ± 50% for end of analytical batch CCV.	Immediately analyze two additional consecutive CCVs. If both pass, samples may be reported without reanalysis. If either fails or if two consecutive CCVs cannot be run, perform corrective action(s) and repeat CCV and all associated samples since last successful CCV. Alternately, recalibrate if necessary; then reanalyze all associated samples since the last acceptable CCV.	If reanalysis cannot be performed, data must be qualified and explained in the Case Narrative. Apply Q-flag to all results for the specific analyte(s) in all samples since last acceptable calibration verification.	Results may not be reported without valid CCVs. Flagging is only appropriate in cases where the samples cannot be reanalyzed. If the specific version of a method requires additional evaluation (e.g., average RFs) these additional requirements must also be met.
Internal Standards (IS)	Every field sample, standard, and QC sample.	Retention time within ± 10 seconds from retention time of the midpoint standard in the ICAL; EICP area within – 50% to +100% of ICAL midpoint standard. On days when ICAL is not performed, the daily initial CCV can be used.	Inspect mass spectrometer and GC for malfunctions and correct problem. Reanalysis of samples analyzed while system was malfunctioning is mandatory.	If corrective action fails in field samples, data must be qualified and explained in the Case Narrative. Apply Q-flag to analytes associated with the non-compliant IS. Flagging is not appropriate for failed standards.	NA.

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Table B-4. Organic Analysis by Gas Chromatography/Mass Spectrometry (GC/MS)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Method Blank (MB)	One per preparatory batch.	No analytes detected > ½ LOQ or > 1/10th the amount measured in any sample or 1/10th the regulatory limit, whichever is greater. Common contaminants must not be detected > LOQ.	Correct problem. If required, reprep and reanalyze MB and all QC samples and field samples processed with the contaminated blank.	If reanalysis cannot be performed, data must be qualified and explained in the Case Narrative. Apply B-flag to all results for the specific analyte(s) in all samples in the associated preparatory batch.	Results may not be reported without a valid Method Blank. Flagging is only appropriate in cases where the samples cannot be reanalyzed.
Laboratory Control Sample (LCS)	One per preparatory batch.	A laboratory must use the DoD/DOE QSM Appendix C Limits for batch control if project limits are not specified. If the analyte(s) are not listed, use in-house LCS limits if project limits are not specified.	Correct problem, then reprep and reanalyze the LCS and all samples in the associated preparatory batch for failed analytes if sufficient sample material is available.	If reanalysis cannot be performed, data must be qualified and explained in the Case Narrative. Apply Q-flag to specific analyte(s) in all samples in the associated preparatory batch.	Must contain all surrogates and all analytes to be reported. Results may not be reported without a valid LCS. Flagging is only appropriate in cases where the samples cannot be reanalyzed.

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Table B-4. Organic Analysis by Gas Chromatography/Mass Spectrometry (GC/MS)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Matrix Spike (MS)	One per preparatory batch.	A laboratory must use the DoD/DOE QSM Appendix C Limits for batch control if project limits are not specified. If the analyte(s) are not listed, use in-house LCS limits if project limits are not specified.	Examine the project-specific requirements. Contact the client as to additional measures to be taken.	For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met and explain in the Case Narrative.	Must contain all surrogates and all analytes to be reported. For matrix evaluation only. If MS results are outside the limits, the data shall be evaluated to determine the source(s) of difference, i.e., matrix effect or analytical error.
Matrix Spike Duplicate (MSD) or Matrix Duplicate (MD)	One per preparatory batch.	A laboratory must use the DoD/DOE QSM Appendix C Limits for batch control if project limits are not specified. If the analyte(s) are not listed, use in-house LCS limits if project limits are not specified. MSD or MD: RPD of all analytes ≤ 20% (between MS and MSD or sample and MD).	Examine the project-specific requirements. Contact the client as to additional measures to be taken.	For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met and explain in the Case Narrative.	MSD: Must contain all surrogates and all analytes to be reported. The data shall be evaluated to determine the source of difference. For Sample/MD: RPD criteria only apply to analytes whose concentration in the sample is greater than or equal to the LOQ.

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Table B-4. Organic Analysis by Gas Chromatography/Mass Spectrometry (GC/MS)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Surrogate Spike	All field and QC samples.	QC acceptance criteria specified by the project if available; otherwise use DoD/DOE QSM Appendix C limits or in-house LCS limits if analyte(s) are not listed.	Correct problem, then reprep and reanalyze all failed samples for all surrogates in the associated preparatory batch if sufficient sample material is available. If obvious chromatographic interference is present, reanalysis may not be necessary, but the client must be notified prior to reporting data and the failures must be discussed in the Case Narrative.	Apply Q-flag to all associated analytes if acceptance criteria are not met and explain in the Case Narrative.	Alternative surrogates are recommended when there is obvious chromatographic interference.

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Revision History

Summary of Changes for Revision 11:

- Revised Attachment 3 and 11 to include the requirement that all target analytes must be evaluated for precision and accuracy.
- Revised Attachment 3 and 11 that South Carolina UST must meet 70-130% limits for LCS/LCSD/MS/MSD.

Summary of Changes for Revision 10A:

- Removed South Carolina state specific requirements for poor performers.

Summary of Changes for Revision 10:

- Added reference to Attachment 8 in Section 1.0. FLDOH Review 2021
- Updated Attachment 9 for current instrument configurations. FLDOH Review 2021.
- Updated ICAL readback criteria in section 9.2.3.2.3 for EPA 624 and 624.1 to be consistent with SOP SA-QA-006.

Summary of Changes in Revision 9:

- Revised limit of poor performers from 60%D to 50%D for CCV and ICV. Poor performers for DOD QSM 5.3 have a 30%D limit unless identified as compounds of concern.
- Updated ICV and CCV language regarding Poor Performer Exceptions.
- DOD QSM 5.3 criteria has been added to all applicable sections.
- DOD QSM 5.3 Table B-4 has been added as Attachment 16 for laboratory reference.
- DOD QSM 5.3 criteria has been added to applicable sections of the SOP.
- Updated language in SOP from “should” to “must” in applicable sections.
- Updated calibration concentrations in Section 9.2.3 to levels currently utilized.
- Added the Readback criteria for calibrations to Section 9.2.3.
- Added 8260B CCV criteria to Section 9.2.5.1 for reduced list of analytes.
- Added to Section 9.2.5.2 that the CCV for SM6200B must be varied of the range of the calibration.
- Added Maximum %D limit for poor performers of 100%D to section 9.2.5.5.
- Revised section 10.2.3 for BFB Tune for SM6200B. 20ml volume is needed for a purge of BFB.
- Removed requirement for Closing CCV for EPA 860C and 8260D in Section 10.27.
- Added Section 12.5.3 to address control limits for SM6200B.
- Revised References in Section 15 in order to incorporate SW846 Updates and Revisions.
- Updated Attachment 2 with Preservation and Holding Times.
- Revised Attachment 3 to include Readback criteria.
- Removed requirement for closing CCV from Attachment 3 for EPA 8260C and EPA 8260D. Closing CCV required for DOD QSM5.3.
- Revised Poor Performer List in Attachment 11.
- Added comment to Section 16 that a concentration of 50ug/L is used for SM6200B.

Summary of Changes from Previous Revision 8:

- Added reference and criteria for SM6200B in all applicable sections of this SOP.
- Added information regarding the Master Equipment listing to Section 6.2.

Summary of Changes from Previous Revision 7:

- Minor editorial, grammatical, and/or formatting changes made.
- Updated SOP signatories to reflect current responsibilities and titles.
- Updated company name and logo where applicable.
- Updated ISTD references in Attachment 9 to reflect ISTD references in CHROM method.
- Updated mechanism of ISTD addition in sections 6.4 and 10.1.1.
- Clarified CCV criteria in section 9.2.5.1 and 9.2.5.2 for poor performers. Updated Attachment 10 for standard preparation to reflect current procedures.

Quality Assurance Manual

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1.0 Title Page

Quality Assurance Manual
Approval Signatures



02/16/2021

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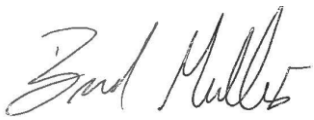
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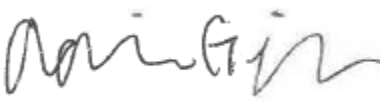
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3.0 INTRODUCTION, SCOPE AND APPLICABILITY

3.1 Introduction and Compliance References

Eurofins TestAmerica Savannah's Quality Assurance (QA) Manual is a document prepared to define the overall policies, organization objectives and functional responsibilities for achieving the laboratory's QA Program. Governing SOPs are in place within the organization to ensure the proper execution of this QA Manual (refer to Appendix 1). This manual and referenced documents are required reading for all personnel.

The laboratory is a team of people who work together to serve the health and environmental needs of society through science and technology. We offer comprehensive expertise in environmental laboratory applications and client relations and maintain a national perspective in terms of quality.

As such, this QA Manual has been prepared to assure compliance with The NELAC Institute (TNI) Standard, dated 2009 and 2016; ISO/IEC Guide 17025:2005 and 2017; and the Department of Defense (DoD) Quality Systems Manual (QSM). The policies and procedures listed in Appendix 1 are compliant with the National Divisional Support Center (NDSC) Quality Management Plan (QMP) for Eurofins TestAmerica and the various accreditation and certification programs which are held by the laboratory to support environmental work (Appendix 2). The QMP provides a summary of Eurofins TestAmerica's quality and data integrity system. It contains requirements and general guidelines under which all Eurofins TestAmerica facilities shall conduct their operations.

Refer to Appendix 3 for a list of additional references for which this QA Manual is compliant; and Appendix 4 for the compliance crosswalk of this manual to the TNI and ISO/IEC Guide 17025 requirements.

3.2 Terms and Definitions

A QA Program is a system designed to ensure that data produced by the laboratory conforms to the standards set by state and/or federal regulations. The program functions at the local management level through company goals, from guidance at the executive management level, and at the analytical level through Standard Operating Procedures (SOPs) and quality control.

Our program is designed to minimize systematic error, encourage constructive, documented problem solving, and provide a framework for continuous improvement within the organization to better serve our clients.

Refer to Appendix 5 for a list of Terms and Acronyms.

3.3 Scope / Fields of Testing

The laboratory analyzes a broad range of environmental and industrial samples. Sample matrices vary among drinking water, effluent water, groundwater, hazardous waste, sludge, and soils. The

QA Program contains specific procedures and methods to test samples of differing matrices for chemical and physical parameters. The Program also contains guidelines on maintaining documentation of analytical processes, reviewing results, servicing clients, and tracking samples through the laboratory. The technical and service requirements of all analytical requests are thoroughly evaluated before commitments are made to accept the work. Measurements are made using published reference methods or methods developed and validated by the laboratory.

The methods covered by this manual include the most frequently requested methodologies needed to provide analytical services in the United States and its territories. The specific list of test methods used by the laboratory can be found in the methods Listing housed in the laboratory's information management system (i.e., LIMS). Our areas of expertise include Volatiles, Semivolatiles, Metals, Pesticides/PCBs/Herbicides, Petroleum Hydrocarbons, Waste Characterization, Non-Potable Water Testing, Drinking Water Testing, Soil and Surface Water Testing, Sediment and Tissue Testing, and Method Development.

Note: All current certificates and scopes of accreditation are available on the laboratory's website.

The approach of this manual is to define the minimum level of quality assurance and quality control necessary to meet these requirements. All methods performed by the laboratory shall meet these criteria as appropriate. In some instances, quality assurance project plans (QAPPs), project specific data quality objectives (DQOs) or local regulations may require criteria other than those contained in this manual. In these cases, the laboratory will abide by the requested criteria following review and acceptance of the requirements by the Laboratory Director, Business Unit Manager (BUMa), and/or the Quality Assurance (QA) Manager. In some cases, QAPPs and DQOs may specify less stringent requirements. The Laboratory Director and/or Business Unit Manager and the QA Manager must determine if it is in the lab's best interest to follow the less stringent requirements.

3.4 Management of the Manual

3.4.1 Review Process

The template on which this manual is based is reviewed annually by Eurofins National Divisional Support Center (NDSC) which houses the Quality Assurance leadership team for Eurofins Environment Testing America. NDSC QA will assure that the template remains in compliance with Section 3.1. This manual itself is reviewed annually by senior laboratory management to assure that it reflects current practices and meets the requirements of the laboratory's clients and regulators as well as the QMP. Occasionally, the manual may need changes in order to meet new or changing regulations and operations. The QA Manager will review the changes in the normal course of business and incorporate changes into revised sections of the document. All updates will be reviewed by the senior laboratory management staff. The laboratory updates and approves such changes according to our document control procedures (refer to SOP SA-QA-001.

4.0 MANAGEMENT REQUIREMENTS

4.1 Overview

Eurofins TestAmerica Savannah is a local operating unit of Eurofins Environment Testing America. The laboratory's operational and support staff have the day-to-day independent operational authority under the direction of the Laboratory Director and Business Unit Manager and is supported by the NDSC QA team. The laboratory management staff includes department managers and supervisors. The organizational chart of the management staff is presented in Figure 4-1. Individual departmental staff lists are maintained in the laboratory's internal intranet.

4.2 Roles and Responsibilities

In order for the QA Program to function properly, all members of the staff must clearly understand and meet their individual responsibilities as they relate to the quality program. The responsibility for quality resides with every employee of the laboratory. All employees have access to the QA Manual, are trained to this manual, and are responsible for upholding the standards therein. Each person carries out his/her daily tasks impartially and in a manner consistent with the goals and in accordance with the procedures in this manual and the laboratory's SOPs. The following descriptions briefly define each role in its relationship to the Quality Assurance Program.

4.2.1 Vice President of Quality and Environmental Health and Safety (VP-QA/EHS)

The Vice President (VP) of QA/EHS reports directly to Eurofins Environment Testing America Chief Operating Officer (COO). With the aid of the NDSC Quality Team Members, Business Unit Managers, and Laboratory Directors, the VP-QA/EHS has the responsibility for the establishment, general overview and maintenance of the Quality Assurance and EH&S Programs within Eurofins Environment Testing America. Additional responsibilities include:

- Review of QA/QC and EHS aspects of NDSC Official Documents, national projects and expansions or changes in services.
- Work with various organizations outside of the laboratory to further the development of quality standards and represent the laboratory at various trade meetings.
- Prepare monthly reports for quality and EH&S metrics across the environmental testing laboratories and a summary of any quality and EH&S related initiatives and issues.
- With the assistance of the Executive Management and the EHS Managers, maintenance and implementation of the Eurofins Environment Testing America Environmental, Health and Safety Program.

4.2.2 Quality Directors

The Quality Directors within NDSC report directly to the VP-QA/EHS. The Quality Directors have oversight of the general overview and maintenance of the QA Program within the Eurofins Environment Testing America laboratories. Supported tasks include:

- Monitors laboratory internal audit findings;
- Identifies common laboratory weaknesses and monitors corrective action closures.
- Develops NDSC quality guidance documents and management tools for ensuring and improving compliance;
- Monitors and communicates DoD/DoE requirements;
- Monitors and communicates regulatory and certification requirements;
- Training and OnBoarding
- Laboratory assessments, mentoring, and interventions
- Track/drive root cause investigations and corrective action plans
- Builds knowledge base for preventive actions

4.2.3 Quality Information Manager

The Quality Information Manager works directly with the NDSC Quality Directors and EHS Managers, and reports directly to the VP-QA/EHS. The Quality Information Manager is responsible for the management of:

- NDSC Official Documents
- TALS/LIMS Certification Module Data
- Company's Intranet website
- Company's Regulatory Limits Database
- Subcontract laboratory and approved vendor information
- Internal and External client support for various company groups (e.g., Client Services, EH&S, Legal, IT, Sales) for both quality and operational functions
- Communication of regulatory information and lists

4.2.4 Environmental Health and Safety (EH&S) Managers

The EH&S Managers within NDSC report directly to the VP-QA/EHS. The EH&S Managers have oversight of the general overview and maintenance of the EH&S Program within the Eurofins Environment Testing America laboratories. Supported tasks include:

- Consolidation and tracking all safety and health-related information and reports for the company, and managing compliance activities for Eurofins Environment Testing America locations.
- Coordination/preparation of the Environmental Health and Safety Manual Template that is used by each laboratory to prepare its own laboratory-specific Safety Manual/CHP.
- Preparation of information and training materials for laboratory EHS Coordinators.
- Assistance in the coordination of employee exposure and medical monitoring programs to insure compliance with applicable safety and health regulations.
- Serving as Department of Transportation (DOT) focal point and providing technical assistance to location management.
- Serving as Hazardous Waste Management main contact and providing technical assistance to location management.

4.2.5 Ethics and Compliance Officers (ECOs)

The NDSC VP-QA/EHS and Corporate Counsel are the designated Ethics and Compliance Officers (ECOs). Each ECO acts as a back-up to the other ECO and both are involved when data investigations occur. Each ECO has a direct line of communication to the entire executive management personnel and lab management staff.

The ECOs monitor and audit procedures to determine compliance with policies and to make recommendations for policy enhancements to the President, COO, Laboratory Director or other appropriate individuals within the laboratory. The ECO will assist the laboratory QA Manager in the coordination of internal auditing of ethical policy related activities and processes within the laboratory, in conjunction with the laboratory's regular internal auditing function.
The ECOs will also participate in investigations of alleged violations of policies and work with the appropriate internal departments to investigate misconduct, remedy the situation, and prevent recurrence of any such activity.

4.2.6 Laboratory Director

The Laboratory Director is responsible for the overall quality, safety, financial, technical, human resource and service performance of the whole laboratory and reports to their business unit President. The Laboratory Director is also responsible for any service centers connected with their laboratory that perform analytical tests, such as short holding time analyses for pH. The Laboratory Director provides the resources necessary to implement and maintain an effective and comprehensive Quality Assurance and Data Integrity Program.
Specific responsibilities include, but are not limited to:

- Provides one or more Technical Managers for the appropriate fields of testing. If the Technical Manager is absent for a period of time exceeding 15 consecutive calendar days, the Laboratory Director must designate another full time staff member meeting the qualifications of the Technical Manager to temporarily perform this function. If the absence exceeds 35 consecutive calendar days, the primary accrediting authority must be notified in writing.
- Ensures that all analysts and supervisors have the appropriate education and training to properly carry out the duties assigned to them and ensures that this training has been documented. Works with Eurofins Environment Testing Human Resources for hiring of new personnel.
- Ensures that personnel are free from any commercial, financial and other undue pressures which might adversely affect the quality of their work.
- Ensures company human resource policies are adhered to and maintained.
- Ensures that sufficient numbers of qualified personnel are employed to supervise and perform the work of the laboratory. Assesses laboratory capacity and workload.
- Ensures that appropriate corrective actions are taken to address analyses identified as requiring such actions by internal and external performance or procedural audits. Procedures that do not meet the standards set forth in the QA Manual or laboratory SOPs may be temporarily suspended by the Laboratory Director.
- May review and approve SOPs prior to their implementation and ensures all approved SOPs are implemented and adhered to.

- Pursues and maintains appropriate laboratory certification and contract approvals. Supports ISO 17025 requirements.
- Ensures client specific reporting and quality control requirements are met.
- Contributes to the continuous improvement of the laboratory operations.
- Maintains an awareness of technical developments and regulatory requirements.
- Captains the management team, consisting of the QA Manager, the Technical Manager(s), and the Operations Manager as direct reports.

4.2.7 Quality Assurance (QA) Manager

The QA Manager has responsibility and authority to ensure the continuous implementation of the quality system at the laboratory where they work based on ISO/IEC 17025, DoD ELAP, and TNI. The QA Manager is also responsible for any service centers connected with their laboratory that perform analytical tests, such as short holding time analyses for pH.

The QA Manager is independent of production and reports directly to the Laboratory Director and their NDSC Quality Director. This position is able to evaluate data objectively and perform assessments without outside (e.g., managerial) influence. The NDSC QA Team may be used as a resource in dealing with regulatory requirements, certifications, and other quality assurance related items. The QA Manager directs the activities of the QA Department to accomplish specific responsibilities, which include, but are not limited to:

- Serving as the focal point for QA/QC in the laboratory.
- Having functions independent from laboratory operations for which he/she has quality assurance oversight.
- Having documented training and/or experience in QA/QC procedures and the laboratory's Quality System.
- Having a general knowledge of the analytical test methods for which data audit/review is performed (and/or having the means of obtaining this information when needed).
- Arranging for or conducting internal audits on quality systems and the technical operation
- Notifying laboratory management of deficiencies in the quality system and ensuring corrective action is taken.
- Maintaining and updating the QA Manual.
- Monitoring and evaluating laboratory certifications; scheduling proficiency testing samples.
- Monitoring and communicating regulatory changes that may affect the laboratory to management.
- Training and advising the laboratory staff on quality assurance/quality control procedures that are pertinent to their daily activities.
- Maintaining records of all ethics-related training, including the type and proof of attendance.
- Maintaining, improving, and evaluating the corrective action database and the corrective and preventive action systems.
- Objectively monitoring standards of performance in quality control and quality assurance without outside (e.g., managerial) influence.
- Coordinating of document control of SOPs, MDLs, control limits, and miscellaneous forms and information.
- Performing technical data audits and method audits to ensure consistency and compliance with regulatory requirements.

- Reviewing external audit reports and data validation requests.
- Follow-up with audits to ensure client QAPP requirements are met.
- Establishing reporting schedule and preparation of various quality reports for the Laboratory Director, clients and/or NDSC QA Team.
- Developing suggestions and recommendations to improve quality systems.
- Researching current state and federal requirements and guidelines.
- Participating in the vendor and supplier approval process, including subcontractors.
- Managing the QA team to enable communication and to distribute duties and responsibilities.
- Communicating to relevant regulatory authorities when there are management or facility changes that impact the laboratory.
- Ensuring communication and monitoring standards of performance to ensure that systems are in place to produce the level of quality as defined in this document.
- Evaluating the thoroughness and effectiveness of training.
- Ensuring compliance with ISO 17025 and the DoD QSM.

4.2.8 Technical Manager

The Technical Manager reports directly to the Laboratory Director. The Technical Manager is accountable for all analyses and analysts under their experienced supervision and for compliance with the ISO 17025:2017 Standard and DoD QSM. The scope of responsibility ranges from the new-hire process and existing technology through the ongoing training and development programs for existing analysts and new instrumentation. Specific responsibilities include, but are not limited to:

- Exercising day-to-day supervision of laboratory operations for the appropriate field of accreditation and reporting of results. Coordinating, writing, and reviewing preparation of all test methods (i.e., SOPs) with regard to quality, integrity, regulatory requirements, and optimum and efficient production techniques, and subsequent analyst training and interpretation of the SOPs for implementation and evaluation of unusual project samples. The Technical Manager ensures the SOPs are properly managed and adhered to at the bench. He develops standard costing of SOPs to include supplies, labor, overhead, and capacity (design vs. demonstrated versus first-run yield) utilization.
- Reviewing and approving, with input from the QA Manager, proposals from marketing, in accordance with an established procedure for the review of requests and contracts.
- Monitoring the validity of the analyses performed and data generated in the laboratory. This activity begins with reviewing and supporting all new business contracts, insuring data quality, analyzing internal and external non-conformances to identify root cause issues and implementing the resulting corrective and preventive actions, facilitating the data review process (training, development, and accountability at the bench), and providing technical and troubleshooting expertise on routine and unusual or complex problems.
- Providing training and development programs to applicable laboratory staff as new hires and, subsequently, on a scheduled basis. Training includes instruction on calculations, instrumentation management to include troubleshooting and preventive maintenance.
- Enhancing efficiency and improving quality through technical advances and improved LIMS utilization. Capital forecasting and instrument life cycle planning for second generation methods and instruments as well as asset inventory management.

- Coordinating sample management from “cradle to grave,” insuring that no time is lost in locating samples.
- Scheduling all QA/QC-related requirements for compliance, e.g., MDLs, etc.
- Managing department personnel to communicate quality, technical, personnel, and instrumental issues for a consistent team approach.
- Coordinating audit responses with the QA Manager.
- Ensuring compliance with ISO 17025 and the DoD QSM.

4.2.10 Project Manager (PM)

Members of the laboratory Client Services/Project Management Group are responsible for organizing and managing client projects. Clients are assigned a project manager who serves as their primary contact at the laboratory. It is the PM’s responsibility to act as the client advocate by communicating client requirements to laboratory personnel and ensuring that clients provide complete information needed by the laboratory to meet those requirements – including all verbal communications. Responsibilities include:

- Scheduling sample submissions, sample container orders and sample pick-up via the laboratory courier service.
- Confirming certification status
- Coordinating and communicating turnaround time (TAT) requirements for high priority samples/projects.
- Answering common technical questions, facilitating problem resolution and coordinating technical details with the laboratory staff.
- Ensuring that client specifications, when known, are met by communicating project and quality assurance requirements to the laboratory.
- Notifying the supervisors of incoming projects and sample delivery schedules.
- Accountable to clients for communicating sample status reports or results prior to receipt of the final report.
- Monitoring the status of all data package projects in-house to ensure timely and accurate delivery of reports.
- Informing clients of data package-related problems and resolve service issues.

4.2.11 Department Supervisors

Supervisors are responsible to:

- Ensure that analysts in their department adhere to applicable SOPs and the QA Manual. They perform frequent SOP and QA Manual review to determine if analysts are in compliance and if new, modified, and optimized measures are feasible and should be added to these documents.
- With regard to analysts, participates in the selection, training, development of performance objectives and standards of performance, appraisal (measurement of objectives), scheduling, counseling, discipline, and motivation of analysts and documents these activities in accordance with systems developed by the QA and Human Resources Departments. They evaluate staffing sufficiency and overtime needs. Training consists of familiarization with SOP, QC, Safety, and computer systems.

- Encourage the development of analysts to become cross-trained in various methods and/or operate multiple instruments efficiently while performing maintenance and documentation, self-supervise, and function as a department team.
- Provide guidance to analysts in resolving problems encountered daily during sample prep/analysis in conjunction with the Technical Manager, Laboratory Director, and/or QA Manager.
- Perform data review and documentation of non-conformance issues, the timely and accurate completion of performance evaluation samples and MDLs, for his department.
- Ensure all logbooks are maintained, current, and properly labeled or archived.
- Report all non-conformance conditions to the QA Manager, Technical Manager, and/or Laboratory Director.
- Ensure that preventive maintenance is performed on instrumentation as detailed in the QA Manual or SOPs. He is responsible for developing and implementing a system for preventive maintenance, troubleshooting, and repairing or arranging for repair of instruments.
- Maintain adequate and valid inventory of reagents, standards, spare parts, and other relevant resources required to perform daily analysis.
- Achieve optimum turnaround time on analyses and compliance with holding times.
- Conduct efficiency and cost control evaluations on an ongoing basis to determine optimization of labor, supplies, overtime, first-run yield, capacity (designed vs. demonstrated), second- and third-generation production techniques/instruments, and long-term needs for budgetary planning.
- Develop, implement, and enhance calibration programs.
- Provide written responses to external and internal audit issues.

4.2.12 Laboratory Analysts

Laboratory analysts are responsible for conducting analysis and performing all tasks assigned to them by the group leader or supervisor. The responsibilities of the analysts are listed below:

- Perform analyses by adhering to analytical and quality control protocols prescribed by current SOPs, this QA Manual, and project-specific plans honestly, accurately, timely, safely, and in the most cost-effective manner.
- Document standard and sample preparation, instrument calibration and maintenance, data calculations, sample matrix effects, and any observed non-conformance on worklists, bench sheets, lab notebooks and/or the Non-Conformance database.
- Report all non-conformance situations, instrument problems, matrix problems and QC failures, which might affect the reliability of the data, to their supervisor, the Technical Manager, and/or the QA Manager or member of QA staff.
- Perform 100% review of the data generated prior to entering and submitting for secondary level review.
- Suggest method improvements to their supervisor, the Technical Manager, and the QA Manager. Ideas for the optimum performance of their assigned area, for example, through the proper cleaning and maintenance of the assigned instruments and equipment, are encouraged.
- Work cohesively as a team in their department to achieve the goals of accurate results, optimum turnaround time, cost effectiveness, cleanliness, complete documentation, and personal knowledge of environmental analysis.

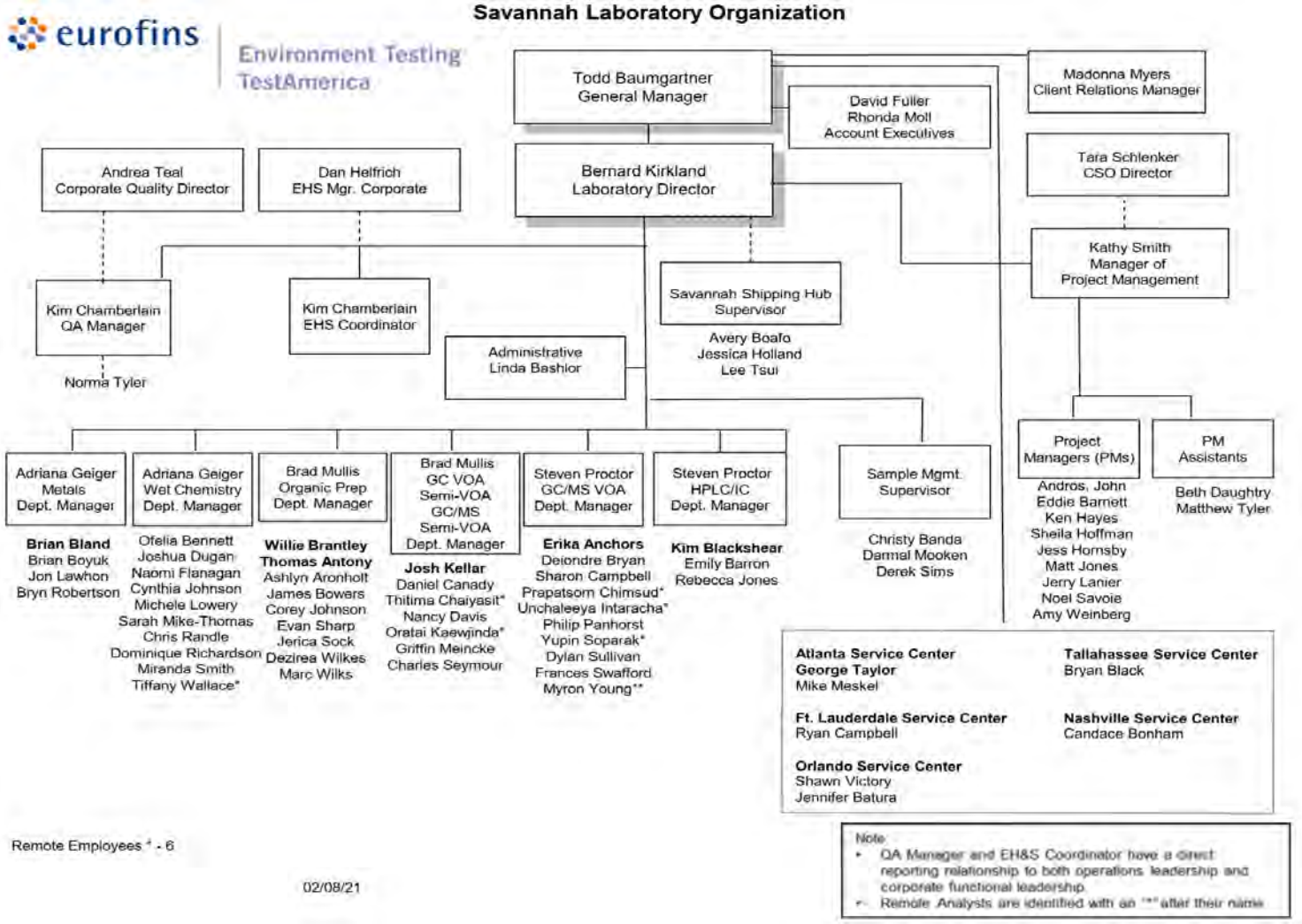
4.3 Business Continuity and Contingency Plans and Deputy Assignments

Various policies and practices are in place to address continuity of business and contingency plans to ensure continued operations or minimal disruption in operations should unplanned events (natural disasters, unexpected management changes, etc.) occur. Deputies are identified for all key management personnel. Deputies would temporarily fill a role if the primary is absent for more than 15 consecutive calendar days. The deputies must meet the same qualifications as the primary person should they be required to take on the responsibilities. The QA Manager communicates to the relevant regulatory authorities when there are management or facility changes that impact the laboratory. Changes in the technical director must be communicated within a period of time and in the manner dictated by each regulatory authority.

The following table defines who assumes the responsibilities of key personnel in their absence:

Key Personnel	Deputy
Laboratory Director	QA Manager
QA Manager	Laboratory Director
Department Manager	Laboratory Director
Technical Manager	Laboratory Director
EHS Coordinator	Laboratory Director

Figure 4-1. Laboratory Organization Charts



5.0 PERSONNEL

5.1 Overview

The laboratory's management believes that its highly qualified and professional staff is the single most important aspect in assuring a high level of data quality and service. The staff consists of professionals and support personnel.

All personnel must demonstrate competence in the areas where they have responsibility. Any staff that is undergoing training shall have appropriate supervision until they have demonstrated their ability to perform their job function on their own. Staff shall be qualified for their tasks based on appropriate education, training, experience and/or demonstrated skills as required.

The laboratory employs sufficient personnel with the necessary education, training, technical knowledge and experience for their assigned responsibilities.

All personnel are responsible for complying with all QA/QC requirements that pertain to the laboratory and their area of responsibility. Each staff member must have a combination of experience and education to adequately demonstrate a specific knowledge of their particular area of responsibility. Technical staff must also have a general knowledge of lab operations, test methods, QA/QC procedures and records management.

Laboratory management is responsible for formulating goals for lab staff with respect to education, training, and skills, and ensuring that the laboratory has a policy and procedures for identifying training needs and providing training of personnel. The training shall be relevant to the present and anticipated responsibilities of the lab staff.

The laboratory only uses personnel that are employed by or under contract to, the laboratory. Contracted personnel, when used, must meet competency standards of the laboratory and work in accordance to the laboratory's quality system.

5.2 Education and Experience Requirements for Technical Personnel

The laboratory makes every effort to hire analytical staffs that possess a college degree (e.g., AA, BA, BS) in an applied science with some chemistry in the curriculum. Exceptions can be made based upon the individual's experience and ability to learn. Selection of qualified candidates for laboratory employment begins with documentation of minimum education, training, and experience prerequisites needed to perform the prescribed task. Minimum education and training requirements for laboratory employees are outlined in job descriptions maintained by the Eurofins Environment Testing America Human Resources Department.

Experience and specialized training are occasionally accepted in lieu of a college degree (basic lab skills such as using a balance, pipette, etc., are also considered).

As a general rule for analytical staff:

Specialty	Education	Experience
Extractions, Digestions, some electrode methods (pH, DO, Redox, etc.), or Titrimetric and Gravimetric Analyses	H.S. Diploma	On the job training (OJT)
CVAA, Single component or short list Chromatography (e.g., Fuels, BTEX-GC, IC)	A college degree in an applied science or 2 years of college and at least 1 year of college chemistry	Or 2 years prior analytical experience is required
ICP, ICPMS, Long List or complex chromatography (e.g., Pesticides, PCB, Herbicides, HPLC, etc.), GCMS	A college degree in an applied science or 2 years of college chemistry	Or 5 years of prior analytical experience
Spectra Interpretation	A college degree in an applied science or 2 years of college chemistry	And 2 years relevant experience Or 5 years of prior analytical experience
Technical Manager (General)	Bachelor's Degree in an applied science or engineering with 24 semester hours in chemistry An advanced (MS, PhD.) degree may substitute for one year of experience	And 2 years' experience in environmental analysis of representative analytes for which they will oversee
Technical Manager (Wet Chem Only) (No advanced instrumentation)	Associates degree in an applied science or engineering or 2 years of college with 16 semester hours in chemistry	And 2 years relevant experience

Specialty	Education	Experience
Technical Manager (Microbiology)	Bachelor's degree in applied science with at least 16 semester hours in general microbiology and biology An advanced (MS, PhD.) degree may substitute for one year of experience	And 2 years of relevant experience

When an analyst does not meet these requirements, they can perform a task under the direct supervision of a qualified analyst, peer reviewer or Technical Manager, and are considered an analyst in training. The person supervising an analyst in training is accountable for the quality of the analytical data and must review and approve data and associated corrective actions.

5.3 Training

The laboratory is committed to furthering the professional and technical development of employees at all levels.

Orientation to the laboratory's policies and procedures, in-house method training, and employee attendance at outside training courses and conferences all contribute toward employee proficiency. Below are examples of various areas of required employee training:

Required Training	Time Frame	Employee Type
Environmental Health & Safety	Prior to lab work	All
Ethics (Comprehensive)	60 days of hire	All
Data Integrity	60 days of hire	Technical and PMs
Quality Assurance	90 days of hire	All
Ethics (Comprehensive Refresher)	Annually	All
Initial Demonstration of Capability (IDOC)	Prior to unsupervised method performance	Technical

The laboratory maintains records of relevant authorization/competence, education, professional qualifications, training, skills and experience of technical personnel (including contracted personnel) as well as the date that approval/authorization was given. These records are kept on file at the laboratory. Also refer to Demonstration of Capability information in Section 19.

The training of technical staff is kept up to date by:

- Each employee must have documentation in their training file that they have read, understood and agreed to follow the most recent version of the laboratory QA Manual and SOPs in their area of responsibility. This documentation is updated as SOPs are updated.
- Documentation from any training courses or workshops on specific equipment, analytical techniques or other relevant topics.
- Documentation of proficiency (refer to Section 19).
- An Ethics Agreement signed by each staff member (renewed each year) and evidence of annual ethics training.
- A Confidentiality Agreement signed by each staff member signed at the time of employment.
- Human Resources maintains documentation and attestation forms on employment status and records; benefit programs; timekeeping/payroll; and employee conduct (e.g., ethics violations). This information is maintained in the employee's secured personnel file.

Evidence of successful training could include such items as:

- Adequate documentation of training within operational areas, including one-on-one technical training for individual technologies, and particularly for people cross-trained.
- Analyst's knowledge to refer to QA Manual for quality issues.
- Analysts following SOPs, i.e., practice matches SOPs.
- Analysts regularly communicate to supervisors and QA if SOPs need revision, rather than waiting for auditors to find problems.

Further details of the laboratory's training program are described in SOP SA-QA-006*: Training Procedures.*

5.4 Data Integrity and Ethics Training Program

The laboratory's Ethics and Data Integrity Program is discussed in Section 6.2. Employees are trained as to the legal and environmental repercussions that result from data misrepresentation.

Key topics covered in the presentation include:

- Organizational mission and its relationship to the critical need for honesty and full disclosure in all analytical reporting.
- Ethics Policy
- How and when to report ethical/data integrity issues. Confidential reporting.
- Record keeping.
- Discussion regarding data integrity procedures.
- Specific examples of breaches of ethical behavior (e.g. peak shaving, altering data or computer clocks, improper macros, etc., accepting/offering kickbacks, illegal accounting practices, unfair competition/collusion)
- Internal monitoring. Investigations and data recalls.
- Consequences for infractions including potential for immediate termination, debarment, or criminal prosecution.
- Importance of proper written narration / data qualification by the analyst and project manager with respect to those cases where the data may still be usable but are in one sense or another partially deficient.

Additionally, a data integrity hotline (1-800-736-9407) is maintained by the NDSC and can be used for anonymous reporting.

6.0 ACCOMMODATIONS AND ENVIRONMENTAL CONDITIONS

6.1 Overview

The laboratory is a 55,000 ft^2 secure laboratory facility with controlled access and designed to accommodate an efficient workflow and to provide a safe and comfortable work environment for employees. All visitors sign in and are escorted by laboratory personnel. Access is controlled by various measures.

The laboratory is equipped with structural safety features. Each employee is familiar with the location, use, and capabilities of general and specialized safety features associated with their workplace. The laboratory provides and requires the use of protective equipment including safety glasses, protective clothing, gloves, etc., OSHA and other regulatory agency guidelines regarding required amounts of bench and fume hood space, lighting, ventilation (temperature and humidity controlled), access, and safety equipment are met or exceeded.

Traffic flow through sample preparation and analysis areas is minimized to reduce the likelihood of contamination. Adequate floor space and bench top area is provided to allow unencumbered sample preparation and analysis space. Sufficient space is also provided for storage of reagents and media, glassware, and portable equipment. Ample space is also provided for refrigerated sample storage before analysis and archival storage of samples after analysis. Laboratory HVAC and deionized water systems are designed to minimize potential trace contaminants.

The laboratory is separated into specific areas for sample receiving, sample preparation, volatile organic sample analysis, non-volatile organic sample analysis, inorganic sample analysis, and administrative functions

6.2 Environment

Laboratory accommodation, test areas, energy sources, and lighting are adequate to facilitate proper performance of tests. The facility is equipped with heating, ventilation, and air conditioning (HVAC) systems appropriate to the needs of environmental testing performed at this laboratory.

The environment in which these activities are undertaken does not invalidate the results or adversely affect the required accuracy of any measurements.

The laboratory provides for the effective monitoring, control, and recording of environmental conditions that may affect the results of environmental tests as required by the relevant specifications, methods, and procedures.

When any of the method or regulatory required environmental conditions change to a point where they may adversely affect test results, analytical testing will be discontinued until the environmental conditions are returned to the required levels.

Environmental conditions of the facility housing the computer network and LIMS are regulated to protect against raw data loss.

When the laboratory performs laboratory activities at sites or facilities outside its permanent control, it shall ensure that the requirements related to facilities and environmental conditions of this document are met.

Specific requirements for facility and environmental conditions, as well as periodic monitoring of conditions, are given in the Environmental Health and Safety Manual plus each laboratory's Facility Addendum.

6.3 Work Areas

There is effective separation between neighboring areas when the activities therein are incompatible with each other. Examples include:

- Volatile organic chemical handling areas, including sample preparation and waste disposal, and volatile organic chemical analysis areas.
- Access to and use of all areas affecting the quality of analytical testing is defined and controlled by secure access to the laboratory building as described below in the Building Security section.
- Adequate measures are taken to ensure good housekeeping in the laboratory and to ensure that any contamination does not adversely affect data quality. These measures include regular cleaning to control dirt and dust within the laboratory. Work areas are available to ensure an unencumbered work area. Work areas include:
- Access and entryways to the laboratory.
- Sample receipt areas.
- Sample storage areas.
- Chemical and waste storage areas.
- Data handling and storage areas.
- Sample processing areas.
- Sample analysis areas.

6.4 Responding to Emergencies

Employees must be aware of procedures to respond to all emergencies that might occur in the workplace. Employees must be familiar with the location and proper operation of all emergency equipment, evacuation routes and designated assembly areas for all areas where they work. Refer to the NDSC EH&S Manual Document No. CW-E-M-001. Sec. 7 and the laboratory's local EH&S Addendum for complete details. These documents provide direction for situations where normal operations of the laboratory are not possible (e.g., electrical failures, heating/air conditioning failures, fire/building evacuation, computer failures, hazardous material spills, injury to employees, pandemic flu, disruption of phone service, etc.)

In the event that the building or information technology (IT) systems would be severely challenged, a designated disaster recovery team, which includes Facility Management, Maintenance, Safety, Laboratory/Executive Management, Public Relations, IT, QA and other applicable personnel depending on the scope of the disaster, would assemble at a designated area to assess the situation and formulate a plan.

6.5 Building Security

The laboratory is considered a secure facility. All outside doors are locked during normal business hours to prevent unauthorized entry. Building keys and alarm codes are distributed to employees as necessary. Employees wear photographic identification name cards while on the premises.

All visitors to the laboratory must sign in and out in a visitor's logbook. A visitor is defined as any person who visits the laboratory who is not an employee of the laboratory. In addition to signing into the laboratory, the Environmental Health and Safety Manual contains requirements for visitors and vendors. There are specific safety forms that must be reviewed and signed.

Visitors (with the exception of company employees) are escorted by laboratory personnel at all times, or the location of the visitor is noted in the visitor's logbook.

7.0 QUALITY SYSTEM

7.1 Quality Policy Statement

The Quality Policy statement gives employees clear requirements for the production of analytical data. As an organization, all personnel are committed to high quality professional practice, testing and data, and service to our clients.

We strive to provide the highest quality data achievable by:

- Reading and understanding all of the quality documents applicable to each position and implementing the process in our work.
- Following all recordkeeping requirements; describing clearly and accurately all activities performed; recording "real time" as the task is carried out; understanding that it is never acceptable to "back date" entries and should additional information be required at a later date, the actual date and by whom the notation is made must be documented.
- Ensuring data integrity through the completeness, consistency, impartiality and accuracy of the data generated. Data is attributable, legible, contemporaneously recorded, original or a true copy, and accurate (ALCOA). This applies to manual paper documentation and electronic records.
- Providing accountability and traceability for each sample analyzed through proper sample handling, labeling, preparation, instrument calibration/qualification/validation, analysis, and reporting; establishing an audit trail (the who, what, when, and why) that identifies date, time, analyst, instrument used, instrument conditions, quality control samples (where appropriate and/or required by the method), and associated standard material.
- Emphasizing a total quality management process which provides impartiality, accuracy, and strict compliance with agency regulations and client requirements, giving the highest degree of confidence; understanding that meeting the requirements of the next employee in the work flow process is just as important as meeting the needs of the external client.
- Providing thorough documentation and explanation to qualify reported data that may not meet all requirements and specifications, but is still of use to the client; understanding this occurs only after discussion with the client on the data limitations and acceptability of this approach.
- Responding immediately to indications of questionable data, out-of-specification occurrences, equipment malfunctions, and other types of laboratory problems, with investigation and applicable corrective action; documenting these activities completely, including the reasons for the decisions made.
- Providing a work environment that ensures accessibility to all levels of management and encourages questions and expression of concerns on quality issues to management. Eurofins recognizes that the implementation of a quality assurance program requires management's commitment and support as well as the involvement of the entire staff
- Continually improve systems and manage risk to support quality improvement efforts in laboratory, administrative and managerial activities

Every staff member at the laboratory plays an integral part in quality assurance and is held responsible and accountable for the quality of their work. It is, therefore, required that all laboratory personnel are trained and agree to comply with applicable procedures and requirements established by this document, the ISO/IEC 17025:2017(E) International Standard,

the 2009 TNI Standard, and the DOD/DOE QSM, and to continually improve the effectiveness of the quality management system.

7.2 Ethics and Data Integrity

Eurofins Environment Testing America is committed to ensuring the integrity of its data and meeting the quality needs of its clients. The laboratory operates our Ethics and Data Integrity program under the guidance of Eurofins Key Guidance Document (KGD).

The elements of the our program include:

- An Ethics Policy (NDSC Document No. CW-L-P-004) and Employee Ethics Statements.
- Ethics and Compliance Officer/s (ECOs).
- A Training Program.
- Self-governance through disciplinary action for violations.
- A confidential mechanism for anonymously reporting alleged misconduct and a means for conducting internal investigations of all alleged misconduct. (NDSC Document No. CW-L-S-002).
- Procedures and guidance for recalling data if necessary (NDSC Document No. CW-Q-S-005).
- Effective external and internal monitoring system that includes procedures for internal audits (Section 17).
- Production of results, which are accurate and include QA/QC information that meets client pre-defined Data Quality Objectives (DQOs).
- Presentation of services in a confidential, honest and forthright manner.
- Provide employees with guidelines and an understanding of the Ethical and Quality Standards of our Industry.
- Provide procedures and guidance to ensure the impartiality and confidentiality of all data and customer information.
- Operate our facilities in a manner that protects the environment and the health and safety of employees and the public.
- Obey all pertinent federal, state and local laws and regulations and encourage other members of our industry to do the same.
- Educate clients as to the extent and kinds of services available.
- Assert competency only for work for which adequate personnel and equipment are available and for which adequate preparation has been made.
- Promote the status of environmental laboratories, their employees, and the value of services rendered by them.

7.3 Quality System Documentation

The laboratory's Quality System is communicated through a variety of documents.

- Quality Assurance (QA) Manual – Each laboratory has a lab-specific QA manual.
- NDSC Official Documents – Each laboratory may use the Guidance (instructional use) documents at their discretion. Template documents are process documents that the laboratories need to implement locally by using the document as is or as an outline to define their internal practices that meet the minimum requirements of the template. Required documents need to be implemented as is and listed in the laboratory's document control list.

- Key Guidance Documents (KGDs) - Documents compiled at the Group Service Centre (GSC) level by Functional Leaders (document owners) aimed at providing specific Eurofins groups of employees with guidelines necessary for the good conduct of their respective work.
- Laboratory SOPs and Policies – General and Technical
- Work Instructions – A subset of procedural steps, tasks or forms associated with an operation of a management system (e.g., checklists, preformatted bench sheets, forms).

7.3.1 Order of Precedence

In the event of a conflict or discrepancy between policies, the order of precedence is as follows:

- Quality Management Plan (QMP)
- NDSC Guidance Documents
- KGDs
- Laboratory Quality Assurance Manual (QA Manual)
- Laboratory SOPs and Policies
- Other (Work Instructions (WI), memos, flow charts, etc.)

Note: The laboratory has the responsibility and authority to operate in compliance with regulatory requirements of the jurisdiction in which the work is performed. Where the QMP conflicts with those regulatory requirements, the regulatory requirements of the jurisdiction shall hold primacy. The laboratory's QA Manual shall take precedence over the QMP in those cases.

7.4 QA/QC Objectives for the Measurement of Data

Quality Assurance (QA) involves developing planned activities whose purpose is to provide assurance to all levels of management that a quality program is in place within the laboratory, and that it is functioning in an effective manner that is consistent with the requirements of NELAP, ISO 17025, DoD, and any other regulatory agencies (i.e., states) in which the laboratory maintains accreditation.

Quality Control (QC) is generally understood to be limited to the analyses of samples and to be synonymous with the term *"analytical quality control"*. QC refers to the routine application of statistically based procedures to evaluate and control the accuracy of results from analytical measurements. The QC program includes procedures for estimating and controlling precision and bias and for determining reporting limits.

Request for Proposals (RFPs) and Quality Assurance Project Plans (QAPP) provide a mechanism for the client and the laboratory to discuss the data quality objectives in order to ensure that analytical services closely correspond to client needs. In order to ensure the ability of the laboratory to meet the Data Quality Objectives (DQOs) specified in the QAPP, clients are advised to allow time for the laboratory to review the QAPP before being finalized. The client is responsible for developing the QAPP; however, the laboratory will provide support to the client for developing the sections of the QAPP that concern laboratory activities.

Historically, laboratories have described their QC objectives in terms of precision, accuracy, representativeness, comparability, completeness, selectivity and sensitivity (PARCCSS). Each laboratory SOP defines the required QC indicators.

7.4.1 Precision

The objective is to meet the performance for precision demonstrated for the methods on similar samples and to meet DQOs of the EPA and/or other regulatory programs. Precision is defined as the degree of reproducibility of measurements under a given set of analytical conditions (exclusive of field sampling variability). Precision is documented on the basis of replicate analysis, usually duplicate or matrix spike (MS) duplicate samples.

7.4.2 Accuracy

The objective is to meet the performance for accuracy demonstrated for the methods on similar samples and to meet DQOs of the EPA and/or other regulatory programs. Accuracy is defined as the degree of bias in a measurement system. Accuracy may be documented through the use of laboratory control samples (LCS) and/or MS. A statement of accuracy is expressed as an interval of acceptance recovery about the mean recovery.

7.4.3 Representativeness

The objective is to provide data which is representative of the sampled medium. Representativeness is defined as the degree to which data represent a characteristic of a population or set of samples and is a measurement of both analytical and field sampling precision. The representativeness of the analytical data is a function of the procedures used in procuring and processing the samples. The representativeness can be documented by the relative percent difference between separately procured, but otherwise identical samples or sample aliquots.

The representativeness of the data from the sampling sites depends on both the sampling procedures and the analytical procedures. Refer to laboratory SOPs for subsampling and homogenization techniques appropriate to the analytical method.

7.4.4 Comparability

The objective is to provide analytical data for which the accuracy, precision, representativeness, and reporting limit statistics are similar to these quality indicators generated by other laboratories for similar samples, and data generated by the laboratory over time.
Comparability is documented by inter-laboratory studies carried out by regulatory agencies or carried out for specific projects or contracts, by comparison of periodically generated statements of accuracy, precision, and reporting limits with those of other laboratories.

7.4.5 Completeness

The completeness objective for data is 90% (or as specified by a particular project), expressed as the ratio of the valid data to the total data over the course of the project. Data will be

considered valid if they are adequate for their intended use. Data usability will be defined in a QAPP, project scope, or regulatory requirement. Data validation is the process for reviewing data to determine its usability and completeness. If the completeness objective is not met, actions will be taken internally and with the data user to improve performance. This may take the form of an audit to evaluate the methodology and procedures as possible sources for the difficulty or may result in a recommendation to use a different method.

7.4.6 Selectivity

Selectivity is defined as the capability of a test method or instrument to respond to a target substance or constituent in the presence of non-target substances. Target analytes are separated from non-target constituents and subsequently identified/detected through one or more of the following, depending on the analytical method: extractions (separation), digestions (separation), interelement corrections (separation), use of matrix modifiers (separation), specific retention times (separation and identification), confirmations with different columns or detectors (separation and identification), specific wavelengths (identification), specific mass spectra (identification), and specific electrodes (separation and identification).

7.4.7 Sensitivity

Sensitivity refers to the amount of analyte necessary to produce a detector response that can be reliably detected (above the Method Detection Limit) or quantified (above the Reporting Limit).

7.5 Criteria for Quality Indicators

The laboratory maintains Method Limit Groups in LIMS that summarize the precision and accuracy acceptability limits for performed analyses. This summary includes an effective date, is updated each time new limits are generated, and are managed by the laboratory's QA Department. Unless otherwise noted, limits within these tables are laboratory generated. Some acceptability limits are derived from US EPA methods when they are required. Where US EPA method limits are not required, the laboratory has developed limits from evaluation of data from similar matrices. Criteria for development of control limits are contained in SOP SA-QA-017: *Evaluation of Batch QC Data*.

7.6 Statistical Quality Control

Statistically-derived precision and accuracy limits are required by selected methods (such as SW-846) and programs. The laboratory routinely utilizes statistically-derived limits to evaluate method performance and determine when corrective action is appropriate. The analysts use the current limits entered into LIMS. The QA department maintains an archive of all limits used within the laboratory. If a method defines the QC limits, the method limits are used.

If a method requires the generation of historical limits, the lab develops such limits from recent data in the QC database of the LIMS following the guidelines described in Section 26. All calculations and limits are documented and dated when approved and effective. On occasion, a client requests contract-specified limits for a specific project.

Current QC limits are entered and maintained in the LIMS analyte database. As sample results and the related QC are entered into LIMS, the sample QC values are compared with the limits in LIMS to determine if they are within the acceptable range. The analyst then evaluates if the sample needs to be rerun or re-extracted/rerun or if a comment should be added to the report explaining the reason for the QC outlier.

7.6.1 QC Charts

Control charting is a useful tool and is performed to assess analyte recoveries over time to evaluate trends. Control charting must be performed periodically (recommended annually) in accordance with SOP SA-QA-017: *Evaluation of Batch QC Data.* The QA Manager evaluates control charts to determine if adjustments need to be made or for corrective actions to methods. All findings are documented and kept on file.

7.7 Quality System Metrics

In addition to the QC parameters discussed above, the entire Quality System is evaluated on a monthly basis through the use of specific metrics (refer to Section 18). These metrics are used to drive continuous improvement in the laboratory's Quality System.

8.0 DOCUMENT CONTROL

8.1 Overview

The QA Department is responsible for the control of documents used in the laboratory to ensure that approved, up-to-date documents are in circulation and out-of-date (obsolete) documents are archived or destroyed. The following documents, at a minimum, must be controlled:

- Laboratory Quality Assurance (QA) Manual
- Laboratory Standard Operating Procedures (SOP)
- Laboratory Policies
- Work Instructions and Forms
- NDSC Documents[1]
- KGDs[1]

[1]Includes locally implemented documents that are document controlled within the laboratory's document control system.

The NDSC and/or KGD documents are only considered controlled when they are read on the intranet site. Printed copies are considered uncontrolled unless the laboratory physically distributes them as controlled documents. A detailed description of the procedure for issuing, authorizing, controlling, distributing, and archiving NDSC Official Documents is found in document No. CW-Q-S-001: *NDSC Document Control and Archiving*. The laboratory's internal document control procedure is defined in SOP No. SA-QA-001.

The laboratory QA Department also maintains access to various references and document sources integral to the operation of the laboratory. This includes reference methods and regulations. Instrument manuals (hard or electronic copies) are also maintained by the laboratory.

The laboratory maintains control of records for raw analytical data and supporting records such as audit reports and responses, logbooks, standard logs, training files, MDL studies, Proficiency Testing (PT) studies, certifications and related correspondence, and corrective action reports.

Raw analytical data consists of bound logbooks, instrument printouts, any other notes, magnetic media, electronic data, and final reports.

8.2 Document Approval and Issue

The pertinent elements of the document control system includes a unique document title and number, pagination, the total number of pages of the item or an 'end of document' page, the effective date, revision number, and the laboratory's name. The QA personnel are responsible for the maintenance of this system.

Controlled documents are authorized by the QA Department. In order to develop a new document, an employee submits an electronic draft to the QA Department for suggestions and approval before use. Upon approval, QA personnel add the identifying version information to the document and retain that document as the official document on file. That document is then

provided to all applicable operational units. Controlled documents are identified as such and records of their distribution are kept by the QA Department. Document control may be achieved by either electronic or hardcopy distribution.
The QA Department maintains a list of the official versions of controlled documents.

Quality System Policies and Procedures will be reviewed at a minimum of every year and revised as appropriate. Changes to documents occur when a procedural change warrants.

8.3 Procedures for Document Control Policy

For changes to the QA Manual, refer to SOP SA-QA-001: *Document Control Program.* Uncontrolled copies must not be used within the laboratory. Previous revisions and back-up data are stored by the QA Department. Electronic controlled pies are stored on the Public server in the QA folder.

For changes to SOPs, refer to SOP SA-QA-001: *Document Control Program.*
Electronic copies of current documents (including QA Manuals, SOPs, Forms, Work Instructions, etc.) are maintained by the QA Department and distributed electronically via the LIMS File System Shares.

8.4 Obsolete Documents

All invalid or obsolete documents are removed, or otherwise prevented from unintended use. The laboratory has specific procedures as described above to accomplish this. In general, obsolete documents are collected from employees according to distribution lists and are marked obsolete on the cover or destroyed. At least one copy of the obsolete document is archived according to SOP SA-QA-001: *Document Control Program.*

9.0 SERVICE TO THE CLIENT

9.1 Overview

The laboratory has established procedures for the review of work requests and contracts, oral or written. The procedures include evaluation of the laboratory's capability and resources to meet the contract's requirements within the requested time period. All requirements, including the methods to be used, must be adequately defined, documented and understood. For many environmental sampling and analysis programs, testing design is site or program specific and does not necessarily fit into a standard laboratory service or product. It is the laboratory's intent to provide both standard and customized environmental laboratory services to our clients.

A thorough review of technical and QC requirements contained in contracts is performed to ensure project success. The appropriateness of requested methods, and the lab's capability to perform them must be established. Projects, proposals, and contracts are reviewed for adequately defined requirements and the laboratory's capability to meet those requirements. Alternate test methods that are capable of meeting the clients' requirements may be proposed by the lab. A review of the lab's capability to analyze non-routine analytes is also part of this review process.

All projects, proposals and contracts are reviewed for the client's requirements in terms of compound lists, test methodology requested, sensitivity (detection and reporting levels), accuracy, and precision requirements (% Recovery and RPD). The reviewer ensures that the laboratory's test methods are suitable to achieve these requirements and that the laboratory holds the appropriate certifications and approvals to perform the work. The laboratory and any potential subcontract laboratories must be certified, as required, for all proposed tests.

Electronic or hard copy deliverable requirements are evaluated against the laboratory's capacity for production of the documentation.

If the laboratory cannot provide all services but intends to subcontract such services, whether to another Eurofins facility on the same LIMS or to an outside firm, this will be documented and discussed with the client prior to contract approval. (Refer to Section 10 for Subcontracting Procedures.)

The laboratory informs the client of the results of the review if it indicates any potential conflict, deficiency, lack of accreditation, or inability of the lab to complete the work satisfactorily. Any discrepancy between the client's requirements and the laboratory's capability to meet those requirements is resolved in writing before acceptance of the contract. It is necessary that the contract be acceptable to both the laboratory and the client. Amendments initiated by the client and/or Eurofins are documented in writing.

All contracts, QAPPs, Sampling and Analysis Plans (SAPs), contract amendments, and documented communications become part of the project record.

The same contract review process used for the initial review is repeated when there are amendments to the original contract by the client, and the participating personnel are informed of the changes.

9.2 Review Sequence and Key Personnel

Appropriate personnel will review the work request at each stage of evaluation.

For routine projects and other simple tasks, a review by the Project Manager (PM) is considered adequate. The PM confirms that the laboratory has any required certifications, that it can meet the clients' data quality and reporting requirements, and that the lab has the capacity to meet the clients turn around needs.

For new, complex or large projects, the proposed contract is given to the Sale Directors, who will decide which lab will receive the work based on the scope of work and other requirements, including certification, testing methodology, and available capacity to perform the work. The contract review process is outlined in NDSC Document CA-L-P-002: *Contract Compliance Policy.*

This review encompasses all facets of the operation. The scope of work is distributed to the appropriate personnel, as needed based on scope of contract, to evaluate all of the requirements shown above.

The Laboratory Director reviews the formal laboratory quote and makes final acceptance for their facility.

The Sales Director, Contract Administrator, Account Executive or Proposal Coordinator then submits the final proposal to the client.

In the event that one of the designated personnel is not available to review the contract, his back-up will fulfill the review requirements.

9.3 Balancing Laboratory Capacity and Workload

Evaluating laboratory capacity to perform specific projects is the responsibility of the Business Unit Manager and Laboratory Director. Many analysts are cross-trained to perform a variety of tests, and there is redundant equipment available in case of malfunctions. This minimizes the need to evaluate small and medium size projects against capacity available to complete them. Large and complex projects are reviewed against capacity estimates before bids are submitted to ensure that the client's analysis schedule is met. Regularly scheduled meetings are held between laboratory management, Client Services, and QA personnel to review progress with current projects, as well as special requirements of new work scheduled for the laboratory. Laboratory capacity and backlog is tracked on a continuous basis using information from the Laboratory Information Management System (LIMS) including turnaround time, and work in-house.

9.4 Documentation

The Contracts Department maintains copies of all signed contracts. Locally, the Quality Assurance Specialist maintains copies.

Appropriate records are maintained for every contract or work request. All stages of the contract review process are documented and include records of any significant changes. These records are maintained by the Quality Assurance Specialist.

The contract will be distributed to and maintained by the appropriate sales/marketing personnel and the Account Executive. A copy of the contract and formal quote will be filed with the laboratory PM and the Laboratory Director.

Records are maintained of pertinent discussions with a client relating to the client's requirements or the results of the work during the period of execution of the contract.

9.4.1 Project-Specific Quality Planning

Communication of contract specific technical and QC criteria is an essential activity in ensuring the success of site specific testing programs. To achieve this goal, a PM is assigned to each client. It is the PM's responsibility to ensure that project-specific technical and QC requirements are effectively evaluated and communicated to the laboratory personnel before and during the project. QA department involvement may be needed to assist in the evaluation of custom QC requirements.

PMs are the primary client contact and they ensure resources are available to meet project requirements. They coordinate opportunities and work with laboratory management and supervisory staff to ensure available resources are sufficient to perform work for the client's project.

Prior to work on a new project, the dissemination of project information and/or project opening meetings may occur to discuss schedules and unique aspects of the project. Items to be discussed may include the project technical profile, turnaround times, holding times, methods, analyte lists, reporting limits, deliverables, sample hazards, or other special requirements. The PM introduces new project information to maximize production and client satisfaction, while maintaining quality. Project notes may be associated with each sample batch as a reminder upon sample receipt and analytical processing.

Any change that may occur within an active project is agreed upon between the client/regulatory agency and the PM/laboratory. These changes (e.g., use of a non-standard method or modification of a method) and approvals must be documented prior to implementation. Documentation pertains to any document (e.g., letter, e-mail, variance, contract addendum), which has been signed by both parties.

Such changes are also communicated to the laboratory during production meetings. The laboratory staff is then introduced to the modified requirements via the PM or the individual laboratory Technical Manager.

The laboratory strongly encourages client visits to the laboratory and for formal/informal information sharing session with employees in order to effectively communicate ongoing client needs as well as project specific details for customized testing programs.

9.5 Special Services

The laboratory cooperates with clients and their representatives to monitor the laboratory's performance in relation to work performed for the client. It is the laboratory's goal to meet all client requirements in addition to statutory and regulatory requirements. The laboratory has procedures to ensure confidentiality to clients (Section 17 and 25).

The laboratory's standard procedures for reporting data are described in Section 25. Special services are also available and provided upon request. These services include:

- Reasonable access for our clients or their representatives to the relevant areas of the laboratory for the witnessing of tests performed for the client.
- Assisting client-specified third party data validators as specified in the client's contract.
- Supplemental information pertaining to the analysis of their samples. Note: An additional charge may apply for additional data/information that was not requested prior to the time of sample analysis or previously agreed upon.

When the client requests a statement of conformity to a specification or standard based on the analysis performed by the laboratory (e.g., pass/fail, in-tolerance/out-of-tolerance), the decision rule shall be clearly defined. Unless inherent in the requested specification or standard, the decision rule selected shall be communicated to the client. Associated reporting requirements are addressed in Section 25.2.18.

9.6 Client Communication

PMs are the primary communication link to the clients. They shall inform their clients of any delays in project completion as well as any non-conformances in either sample receipt or sample analysis. Project management will maintain ongoing client communication throughout the entire client project.

Technical Managers are available to discuss any technical questions or concerns that the client may have.

9.7 Reporting

The laboratory works with our clients to produce any special communication reports required by the contract.

9.8 Client Feedback and Surveys

The laboratory assesses both positive and negative client feedback. The results are used to improve overall laboratory quality and client service. Eurofins Sales and Marketing teams periodically develop lab and client specific surveys to assess client satisfaction.

When a complaint is received, we determine, to the best of our ability, the extent of the issue and what data is in question. The person receiving the complaint documents this information and promptly forwards it to the appropriate management personnel where the work in question was performed. If a data reporting error is discovered, the final report and/or data must be regenerated with the correct value(s).

The QA Department is responsible for entering client concerns into ICAT where a CAR is initiated to address and document the situation. While an individual issue may not warrant a formal investigation, QA monitors these issues for potential trends and will issue a CAR if a trend is evident.

10.0 SUBCONTRACTING OF TESTS

The laboratory may subcontract tests to other laboratories if the requested testing is not routinely performed in our facility. To a lesser extent, samples may also need to be subcontracted to an overflow laboratory to ensure holding times and/or turn-around-times (TAT) are met.

Testing is only subcontracted with the client's knowledge and approval. The Project Manager must notify the client in writing when any of their requested analyses will be subcontracted to another lab. Client approval must be obtained in writing before samples are shipped.

Subcontract laboratories are selected based on their qualifications and accreditations. The subcontractor is requested to sign a Laboratory Analytical Services Subcontract. Refer to form Q-EQA-FRM6867 to review details of the contract terms and information requested from the subcontract laboratory. If projects require a specific agency certification (e.g., individual state agencies, NELAP, DoD, ISO 17025, etc.), only an appropriately accredited laboratory is used. The client may also have a list of laboratories to be used for subcontracting. In these cases, the evaluation of the subcontract laboratory is made by the client.

Data obtained from subcontract laboratories is clearly marked as such when reported by the laboratory. The data are submitted to the client in the format obtained from the subcontractor.

10.1 Overview

For the purpose of this quality manual, the phrase subcontract laboratory refers to a laboratory external to the Eurofins laboratories and those not on TALS. The phrase "work sharing" refers to internal transfers of samples between the Eurofins laboratories on TALS. The term outsourcing refers to the act of subcontracting tests.

When contracting with our clients, the laboratory makes commitments regarding the services to be performed and the data quality for the results to be generated. When the need arises to outsource testing for our clients because project scope, changes in laboratory capabilities, capacity, or unforeseen circumstances, we must be assured that the subcontractors or work sharing laboratories understand the requirements and will meet the same commitments we have made to the client. Refer to the NDSC Documents on Subcontracting Procedures (CW-L-S-004) and the Work Sharing Process (CA-C-S-001).

When outsourcing analytical services, the laboratory will assure, to the extent necessary, that the subcontract or work sharing laboratory maintains a program consistent with the requirements of this document, the requirements specified in TNI/ISO 17025 and/or the client's Quality Assurance Project Plan (QAPP). All QC guidelines specific to the client's analytical program are transmitted to the subcontractor and agreed upon before sending the samples to the subcontract facility. Additionally, work requiring accreditation will be placed with an appropriately accredited laboratory. The laboratory performing the subcontracted work will be identified in the final report, as will non-TNI accredited work where required.

PMs or other responsible Client Service members, for the export lab (i.e., the Eurofins TestAmerica laboratory that transfers samples to another laboratory) are responsible for obtaining client approval prior to subcontracting any samples. The laboratory will advise the client of a subcontract arrangement in writing and when possible approval from the client shall be obtained and retained in the project folder. Standard Eurofins TestAmerica Terms & Conditions include the flexibility to subcontract samples within the Eurofins TestAmerica laboratories. Therefore, additional advance notification to clients for intra-laboratory subcontracting is not necessary unless specifically required by a client contract.

Note: In addition to the client, some regulating agencies (e.g., USDA) or contracts (e.g., DoD projects) require notification prior to placing such work**.**

10.2 Qualifying and Monitoring Subcontractors

Whenever a PM becomes aware of a client requirement or laboratory need where samples must be outsourced to another laboratory, the other laboratory(s) shall be selected based on the following:

- Subcontractors specified by the client - In these circumstances, the client assumes responsibility for the quality of the data generated from the use of a subcontractor.
- Subcontractors reviewed by Eurofins TestAmerica – Firms which have been reviewed by the company and are known to meet standards for accreditations (e.g., State, TNI and DoD/DOE); technical specifications; legal and financial information.

A listing of vendors is available on the Eurofins TestAmerica intranet site.

All Eurofins TestAmerica laboratories are pre-qualified for work sharing provided they hold the appropriate accreditations and can adhere to the project/program requirements. Client approval is not necessary unless specifically required by the contract. In these cases, the client must provide acknowledgement that the samples can be sent to that facility (an e-mail is sufficient documentation or if acknowledgement is verbal, the date, time, and name of person providing acknowledgement must be documented). The originating laboratory is responsible for communicating all technical, quality, and deliverable requirements as well as other contract needs. (NDSC Document No. CA-C-S-001, Work Sharing Process)

10.2.1 When the potential subcontract laboratory has not been previously approved, Account Executives or PMs may nominate a laboratory as a subcontractor based on need. The decision to nominate a laboratory must be approved by the Client Relations Manager (CRM) or Laboratory Director. The CRM or Laboratory Director requests that the PM begins the process of approving the subcontract laboratory as outlined in NDSC Document No. CW-L-S-004, Subcontracting Procedures.

Once the appropriate accreditation and legal information is received by the laboratory, it is evaluated for acceptability and forwarded to the NDSC Quality Information Manager (QIM) for review. After the NDSC QIM reviews the documents for completeness, the information is forwarded to the Finance Department for formal signature and contracting with the laboratory. The approved vendor will be added to the approved subcontractor list on the intranet site, and the finance group is concurrently notified.

The client will assume responsibility for the quality of the data generated from the use of a subcontractor they have requested the lab to use. The qualified subcontractors on the intranet site are known to meet minimal standards. Eurofins TestAmerica does not certify laboratories. The subcontractors on our approved list can only be recommended to the extent that we would use them.

10.3 Oversight and Reporting

The status and performance of qualified subcontractors will be monitored by NDSC QA Department, and includes an annual review process (see NDSC Document No. SOP CW-L-S-004). Any problems identified will be brought to the attention of NDSC QA and Procurement personnel.

- Complaints shall be investigated. Documentation of the complaint, investigation, and corrective action will be maintained in the subcontractor's file on the intranet site. Complaints are posted using the Vendor Performance Report.
- Information shall be updated on the intranet when new information is received from the subcontracted laboratories.
- Subcontractors in good standing will be retained on the intranet listing. Client Service personnel will notify all TestAmerica laboratories, NDSC QA and Contracts Departments if any laboratory requires removal from the intranet site. This notification will be posted on the intranet site and e-mailed to all Client Service Personnel, Laboratory Directors, QA Managers, and Sales Personnel.

Prior to initially sending samples to the subcontracted laboratory, the PM confirms their certification status to determine if it's current and scope-inclusive. The information is documented within the project records.

The laboratory's certifications can be viewed on the company's website.

10.3.1 All subcontracted samples must be accompanied by a Eurofins TestAmerica Chain-of-Custody (COC). A copy of the original COC sent by the client must be available in LIMS for all samples workshares within Eurofins TestAmerica. Client COCs are only forwarded to external subcontractors when samples are shipped directly from the project site to the subcontractor lab. Under routine circumstances, client COCs are not provided to external subcontractors.

Through communication with the subcontracted laboratory, the PM monitors the status of the subcontracted analyses, facilitates successful execution of the work, and ensures the timeliness and completeness of the analytical report.

Non-TNI accredited work must be identified in the subcontractor's report as appropriate. If TNI accreditation is not required, the report does not need to include this information.

Reports submitted from subcontractor laboratories are not altered and are included in their original form in the final project report. This clearly identifies the data as being produced by a subcontractor facility. If subcontract laboratory data is incorporated into the laboratory's EDD (i.e., imported), the report must explicitly indicate which lab produced the data for which methods and samples.

Note: The results submitted by a Eurofins TestAmerica work sharing laboratory may be transferred electronically and the results reported by the Eurofins TestAmerica work sharing lab are identified on the final report. The report must explicitly indicate which lab produced the data for which methods and samples. The final report must include a copy of the completed COC for all work sharing reports.

10.4 Contingency Planning

The full qualification of a subcontractor may be waived to meet emergency needs. This decision and justification must be documented in the project files, and the 'Purchase Order Terms And Conditions For Subcontracted Laboratory Services' must be sent with the samples and COC.

In the event this provision is utilized, the laboratory (e.g., PM) will be required to verify and document the applicable accreditations of the subcontractor. All other quality and accreditation requirements will still be applicable, but the subcontractor need not have signed a subcontract agreement with Eurofins TestAmerica at this time.

The use of any emergency subcontractor will require the PM to complete a New Vendor Add Form in order to process payment to the vendor and add them to LIMS. This form requires the user to define the subcontractor's category/s of testing and the reason for testing.

10.5 Use of NELAP and A2LA Logo

It is not laboratory policy to use these logos on any company letterhead, including analytical reports.

11.0 PURCHASING SERVICES AND SUPPLIES

11.1 Overview

Evaluation and selection of suppliers and vendors is performed, in part, on the basis of the quality of their products, their ability to meet the demand for their products on a continuous and short term basis, the overall quality of their services, their past history, and competitive pricing. This is achieved through evaluation of objective evidence of quality furnished by the supplier, which can include certificates of analysis, recommendations, and proof of historical compliance with similar programs for other clients. To ensure that quality critical consumables and equipment conform to specified requirements, which may affect quality, all purchases from specific vendors are approved by a member of the supervisory or management staff. Capital expenditures are made in accordance with NDSC Document No. CW-F-S-007, Eurofins TestAmerica's Fixed Asset Acquisition, Retention and Safeguarding.

Contracts will be signed in accordance with the laboratory's authorization matrix, NDSC Document No. CW-F-P-002, Company-Wide Authorization Matrix Policy. Requests for Proposals (RFPs) will be issued where more information is required from the potential vendors than just price. Process details are available in NDSC Document No. CW-F-P-004, Guidance on Procurement and Contracts Policy. RFPs allow the laboratory to determine if a vendor is capable of meeting requirements such as supplying all of the laboratory facilities, meeting required quality standards and adhering to necessary ethical and environmental standards. The RFP process also allows potential vendors to outline any additional capabilities they may offer.

11.2 Glassware

Glassware used for volumetric measurements must be Class A or verified for accuracy according to laboratory procedure. Pyrex (or equivalent) glass should be used where possible. For safety purposes, thick-wall glassware should be used where available.

11.3 Reagents, Standards, and Supplies

Purchasing guidelines for equipment, consumables, and reagents must meet the requirements of the specific method and testing procedures for which they are being purchased. Solvents and acids are pre-tested in accordance with NDSC Document No. CA-Q-S-001 *Solvent and Acid Lot Testing and Approval.* Approval information for the solvents and acids tested under Document No. C-Q-S-001 is stored on the laboratory's shared files under Solvent Approvals. A master list of all tested materials, as well as the certificates of analysis for the materials, is stored in the same location.

11.3.1 Purchasing

Chemical reagents, solvents, glassware, and general supplies are ordered as needed to maintain sufficient quantities on hand. Materials used in the analytical process must be of a known quality. The wide variety of materials and reagents available makes it advisable to specify recommendations for the name, brand, and grade of materials to be used in any determination. This information is contained in the method SOP.

11.3.2 Receiving

It is the responsibility of the shipping/receiving department to receive the shipment. It is the responsibility of the analyst who ordered the materials to document the date materials were received. Once the ordered reagents or materials are received, the analyst compares the information on the label or packaging to the original order to ensure that the purchase meets the quality level specified. This is documented through the addition of the received date and initials to the information present on the order log.

The department verifies the lot numbers of received solvents and acids against the pre-approval lists. If a received material is listed as unapproved, or is not listed, it is sequestered and returned to the vendor. Alternatively, the laboratory may test the material for the intended use, and if it is acceptable, document the approval on the approval list. Records of any testing performed locally are maintained on the shared "public" folder on the computer network.
Materials may not be released for use in the laboratory until they have been inspected, verified as suitable for use, and the inspection/verification has been documented.

Safety Data Sheets (SDSs) are available online through the Company's intranet website. Anyone may review these for relevant information on the safe handling and emergency precautions of on-site chemicals.

11.3.3 Specifications

Methods used in the laboratory specify the grade of reagent that must be used in the procedure. If the quality of the reagent is not specified, analytical reagent grade will be used. It is the responsibility of the analyst to check the procedure carefully for the suitability of grade of reagent.

Chemicals must not be used past the manufacturer's expiration date and must not be used past the expiration time noted in a method SOP. If expiration dates are not provided, the laboratory may contact the manufacturer to determine an expiration date.

The laboratory assumes a five year expiration date on inorganic dry chemicals and solvents unless noted otherwise by the manufacturer or by the reference source method.

Chemicals/solvents should not be used past the manufacturer's or SOP expiration date unless verified as outlined below.

- An expiration date cannot be extended if the dry chemical/solvent is discolored or appears otherwise physically degraded. In this case, the dry chemical/solvent must be discarded.
- Expiration dates can be extended if the dry chemical/solvent is found to be satisfactory based on acceptable performance of quality control samples (Continuing Calibration Verification (CCV), Blanks, Laboratory Control Sample (LCS), etc.).
- If the dry chemical/solvent is used for the preparation of standards, the expiration dates can be extended 6 months if the dry chemical/solvent is compared to an unexpired independent source in performing the method and the performance of the dry chemical/solvent is found to be satisfactory. The comparison must show that the dry chemical/solvent meets CCV limits. The comparison studies are maintained on-file and available for review.

Wherever possible, standards must be traceable to national or international standards of measurement or to national or international reference materials. Records to that effect are available to the user.

Compressed gases in use are checked for pressure and secure positioning daily. To prevent a tank from going to dryness, or introducing potential impurities, the pressure should be closely watched as it decreases to approximately 15% of the original reading, at which point it should be replaced. For example, a standard sized laboratory gas cylinder containing 3,000 psig of gas should be replaced when it drops to approximately 500 psig. The quality of the gases must meet method or manufacturer specification or be of a grade that does not cause any analytical interference.

Water used in the preparation of samples, standards or reagents must have a specific conductivity of less than 1-µmho/cm (or specific resistivity of greater than 1.0 megohm-cm) at 25ºC. The specific conductivity is checked and recorded daily. If the water's specific conductivity is greater than the specified limit, the Facility Manager and appropriate Technical Managers must be notified immediately in order to notify all departments, decide on cessation (based on intended use) of activities, and make arrangements for correction.

The laboratory may purchase reagent grade (or other similar quality) water for use in the laboratory. This water must be certified clean by the supplier for all target analytes or otherwise verified by the laboratory prior to use. This verification is documented.

Standard lots are verified before first time use if the laboratory switches manufacturers or has historically had a problem with the type of standard.

Purchased bottleware used for sampling must be certified clean and the certificates must be maintained. If uncertified sampling bottleware is purchased, all lots must be verified clean prior to use. This verification must be maintained.

Records of manufacturer's certification and traceability statements are maintained in files or binders in each laboratory section. These records include date of receipt, lot number (when applicable), and expiration date (when applicable). Incorporation of the item into the record indicates that the analyst has reviewed the certificate.

11.3.4 Storage

Reagent and chemical storage is important from the aspects of both integrity and safety. Light-sensitive reagents may be stored in brown-glass containers. Storage conditions are per the NDSC Environmental Health & Safety Manual, Document No. CW-E-M-001, the local laboratory EHSM addendum and method SOPs or manufacturer instructions.

11.4 Purchase of Equipment / Instruments / Software

When a new piece of equipment is needed, either for additional capacity or for replacing inoperable equipment, the analyst or supervisor makes a supply request to the Technical Manager and/or the Laboratory Director. If they agree with the request, the procedures outlined in NDSC Document No. CA-T-P-001, Qualified Products List, are followed. A decision is made as to which piece of equipment can best satisfy the requirements. The appropriate written requests are completed and purchasing places the order.
Upon receipt of a new or used piece of equipment, an identification name is assigned and added to the equipment list. IT must also be notified so that they can synchronize the instrument for back-ups. Its capability is assessed to determine if it is adequate or not for the specific application. For instruments, a calibration curve is generated, followed by MDLs, Demonstration of Capabilities (DOCs), and other relevant criteria (refer to Section 20). For software, its operation must be deemed reliable and evidence of instrument verification must be retained by the IT Department. Software certificates supplied by the vendors are filed with the LIMS Administrator. The manufacturer's operation manual is retained electronically.

11.5 Services

Service to analytical instruments (except analytical balances) is performed on an as needed basis. Routine preventative maintenance is discussed in Section 22. The need for service is determined by analysts and/or Technical Managers. The service providers that perform the services are approved by the Technical Manager.

Analytical balances are serviced and calibrated annually in accordance with SOP SA-AN-100: *Laboratory Support Equipment.* The calibration and maintenance services are performed on-site, and the balances are returned to use immediately following successful calibration. Calibration certificates are filed for reference. If the calibration was unsuccessful, the balance is immediately removed from service and segregated pending either further maintenance or disposal.

Calibration services for support equipment such as thermometers, weight sets, autopipettors, etc., are obtained from vendors with current and valid ISO 17025 accreditation for calibration of the specific piece of equipment. Prior to utilizing the vendor's services, the vendor's accreditation status is verified. Once the equipment has been calibrated, the calibration certificates are reviewed by the QA Department, and documentation of the review is filed with the calibration certificates. The equipment is then returned to service within the laboratory

11.6 Suppliers

The laboratory selects vendors through a competitive proposal / bid process, strategic business alliances or negotiated vendor partnerships (contracts). This process is defined in the NDSC Document No. CW-F-P-004, Procurement & Contracts Policy. The level of control used in the selection process is dependent on the anticipated spending amount and the potential impact on the laboratory's business. Vendors that provide test and measuring equipment, solvents, standards, certified containers, instrument related service contracts or subcontract laboratory services shall be subject to more rigorous controls than vendors that provide off-the-shelf items

of defined quality that meet the end use requirements. The purchasing system includes all suppliers/vendors that have been approved for use.

Evaluation of suppliers is accomplished by ensuring the supplier ships the product or material ordered and that the material is of the appropriate quality. This is documented by signing off on packing slips or other supply receipt documents. The purchasing documents contain the data that adequately describe the services and supplies ordered.

Any issues of vendor performance are to be reported immediately by the laboratory staff to the Purchasing Group by completing a Vendor Performance Report.
The Purchasing Group will work through the appropriate channels to gather the information required to clearly identify the problem and will contact the vendor to report the problem and to make any necessary arrangements for exchange, return authorization, credit, etc.

Suppliers are subject to re-evaluation, as deemed appropriate, through the use of Vendor Performance Reports used to summarize and review to determine corrective action necessary, or service improvements required by vendors

The laboratory has access to a listing of all approved suppliers of critical consumables, supplies and services. This information is provided through the purchasing system.

11.6.1 New Vendor Procedure

Laboratory employees who wish to request the addition of a new vendor must complete a Vendor Add Request Form.

New vendors are evaluated based upon criteria appropriate to the products or services provided as well as their ability to provide those products and services at a competitive cost. Vendors are also evaluated to determine if there are ethical reasons or potential conflicts of interest with laboratory employees that would make it prohibitive to do business with them as well as their financial stability. The QA Department and/or the Technical Management are consulted with vendor and product selection that have an impact on quality.

12.0 COMPLAINTS

12.1 Overview

The laboratory considers an effective client complaint handling processes to be of significant business and strategic value. Listening to and documenting client concerns captures client knowledge that enables our operations to continually improve processes and client satisfaction. An effective client complaint handling process also provides assurance to the data user that the laboratory will stand behind its data, service obligations and products.

A client complaint is any expression of dissatisfaction with any aspect of our business services (e.g., communications, responsiveness, data, reports, invoicing and other functions) expressed by any party, whether received verbally or in written form. Client inquiries, complaints or noted discrepancies are documented, communicated to management, and addressed promptly and thoroughly.

The laboratory has procedures for addressing both external and internal complaints with the goal of providing satisfactory resolution to complaints in a timely and professional manner.
The nature of the complaint is identified, documented and investigated, and an appropriate action is determined and taken. In cases where a client complaint indicates that an established policy or procedure was not followed, the QA Department must evaluate whether a special audit must be conducted to assist in resolving the issue. A written confirmation or letter to the client, outlining the issue and response taken is recommended as part of the overall action taken.
The process of complaint resolution and documentation utilizes the procedures outlined in Section 14 (Corrective Actions) and is documented following SOP SA-QA-005: *Preventive and Corrective Action.* A copy of this procedure will be made available to any interested party on request.

12.2 External Complaints

An employee that receives a complaint initiates the complaint resolution process by first documenting the complaint according to SOP SA-CA-005.
Complaints fall into two categories: correctable and non-correctable. An example of a correctable complaint would be one where a report re-issue would resolve the complaint. An example of a non-correctable complaint would be one where a client complains that their data was repeatedly late. Non-correctable complaints should be reviewed for preventive action measures to reduce the likelihood of future occurrence and mitigation of client impact.

The general steps in the complaint handling process are:
- Receiving and documenting complaints
- Acknowledging receipt of complaint, whenever possible
- Complaint investigation and service recovery
- Process improvement

The laboratory shall inform the initiator of the complaint of the results of the investigation and the corrective action taken, if any.

12.3 Internal Complaints

Internal complaints include, but are not limited to: errors and non-conformances, training issues, internal audit findings, and deviations from methods. Corrective actions may be initiated by any staff member who observes a nonconformance and shall follow the procedures outlined in Section 14. In addition, Executive Management, Sales and Marketing, and IT may initiate a complaint by contacting the laboratory or through the corrective action system described in Section 14.

12.4 Management Review

The number and nature of client complaints is reported by the QA Manager, Laboratory Director, and Quality Director in the QA Monthly report. Monitoring and addressing the overall level and nature of client complaints and the effectiveness of the solutions is part of the Annual Management Systems Review (Section 18).

13.0 CONTROL OF NON-CONFORMING WORK

13.1 Overview

When data discrepancies are discovered or deviations and departures from laboratory SOPs, policies and/or client requests have occurred, corrective action is taken immediately. First, the laboratory evaluates the significance of the nonconforming work. Then, a corrective action plan is initiated based on the outcome of the evaluation. If it is determined that the nonconforming work is an isolated incident, the plan could be as simple as adding a qualifier to the final results and/or making a notation in the case narrative. If it is determined that the nonconforming work is a systematic or improper practices issue, the corrective action plan could include a more in depth investigation and a possible suspension of an analytical method. In all cases, the actions taken are documented using the laboratory's corrective action system (refer to Section 14).

Due to the frequently unique nature of environmental samples, sometimes departures from documented policies and procedures are needed. When an analyst encounters such a situation, the problem is presented to the Department Manager for resolution. The Department Manager may elect to discuss it with the Technical Manager or have a representative contact the client to decide on a logical course of action. Once an approach is agreed upon, the analyst documents it using the laboratory's corrective action system described in Section 12. This information can then be supplied to the client in the form of a footnote or a case narrative with the report.

Project Management may encounter situations where a client requests that a special procedure be applied to a sample that is not standard laboratory practice. Based on a technical evaluation, the laboratory may accept or reject the request based on technical or ethical merit. Such a request would need to be approved by laboratory management and documented in the project files. Deviations to standard operating procedures must be noted in the final report.

13.2 Responsibilities and Authorities

Under certain circumstances, the Laboratory Director, a Technical Manager, or a member of the QA team may authorize departures from documented procedures or policies. The departures may be a result of procedural changes due to the nature of the sample; a one-time procedure for a client; QC failures with insufficient sample to reanalyze, etc. In most cases, the client will be informed of the departure prior to the reporting of the data. Any departures must be well documented using the laboratory's corrective action procedures. This information may also be documented in logbooks and/or data review checklists as appropriate. Any impacted data must be referenced in a case narrative and/or flagged with an appropriate data qualifier.

Any misrepresentation or possible misrepresentation of analytical data discovered by any laboratory staff member must be reported to facility Senior Management within 24 hours. The Senior Management staff is comprised of the Laboratory Director, the QA Manager, and the Technical Managers. The reporting of issues involving alleged violations of the company's Data Integrity or Manual Integration procedures must be conveyed to an ECO (e.g., the VP-QA/EHS) and the laboratory's Quality Director within 24 hours of discovery.

Whether an inaccurate result was reported due to calculation or quantitation errors, data entry errors, improper practices, or failure to follow SOPs, the data must be evaluated to determine the possible effect.
The Laboratory Director, QA Manager, ECOs, VP of Operations and the Quality Directors have the authority and responsibility to halt work, withhold final reports, or suspend an analysis for due cause as well as authorize the resumption of work.

13.3 Evaluation of Significance and Actions Taken

For each nonconforming issue reported, an evaluation of its significance and the level of management involvement needed is made. This includes reviewing its impact on the final data, whether or not it is an isolated or systematic issue, and how it relates to any special client requirements.

NDSC Document No. CW-Q-S-005 is the procedure to be followed when it is discovered that erroneous or biased data may have been reported to clients or regulatory agencies.

The NDSC Document entitled Internal Investigations (CW-L-S-002) is the procedure to be followed for investigation and correction of situations involved alleged incidents of misconduct or violation of the company's ethics policy.

Laboratory level decisions are documented and approved using the laboratory's standard nonconformance/corrective action reporting in lieu of the data recall determination form contained in NDSC Document No.CW-Q-S-005.

13.4 Prevention of Nonconforming Work

If it is determined that the nonconforming work could recur, further corrective actions must be made following the laboratory's corrective action system. Periodically as defined by the laboratory's preventive action schedule, the QA Department evaluates non-conformances to determine if any nonconforming work has been repeated multiple times. If so, the laboratory's corrective action process may be followed.

13.5 Method Suspension / Restriction (Stop Work Procedures)

In some cases, it may be necessary to suspend/restrict the use of a method or target analyte which constitutes significant risk and/or liability to the laboratory. Suspension/restriction procedures can be initiated by any of the persons noted in Section 13.2, Paragraph 5.
Prior to suspension/restriction, confidentiality will be respected, and the problem with the required corrective and preventive action will be stated in writing and presented to the Laboratory Director.

The Laboratory Director shall arrange for the appropriate personnel to meet with the QA Manager as needed. This meeting shall be held to confirm that there is a problem, that suspension/restriction of the method is required and will be concluded with a discussion of the steps necessary to bring the method/target or test fully back on line. In some cases, that may not be necessary if all appropriate personnel have already agreed there is a problem and there

is agreement on the steps needed to bring the method, target or test fully back on line. The QA Manager will also initiate a corrective action report as described in Section 14 if one has not already been started. A copy of any meeting notes and agreed upon steps should be e-mailed by the laboratory to their Quality Director. This e-mail acts as notification of the incident.

After suspension/restriction, the lab will hold all reports to clients pending review. No faxing, mailing or distributing through electronic means may occur. The report must not be posted for viewing on the internet. It is the responsibility of the Laboratory Director to hold all reporting and to notify all relevant laboratory personnel regarding the suspension/restriction (e.g., Project Management, Log-in, etc.). Clients will NOT generally be notified at this time. Analysis may proceed in some instances depending on the non-conformance issue.

Within 72 hours, the QA Manager will determine if compliance is now met and reports can be released, or determine the plan of action to bring work into compliance, and release work. A team, with all principals involved (e.g., Laboratory Director, Technical Manager, QA Manager) can devise a start-up plan to cover all steps from client notification through compliance and release of reports. Project Management and the Directors of Client Services and Sales and Marketing must be notified if clients must be notified or if the suspension/restriction affects the laboratory's ability to accept work. The QA Manager must approve start-up or elimination of any restrictions after all corrective action is complete.

14.0 CORRECTIVE ACTION

14.1 Overview

A major component of the laboratory's (QA) Program is the problem investigation and feedback mechanism designed to keep the laboratory staff informed on quality related issues and to provide insight to problem resolution. When nonconforming work or departures from policies and procedures in the quality system or technical operations are identified, the corrective action procedure provides a systematic approach to assess the issues, restore the laboratory's system integrity, and prevent reoccurrence. The laboratory employs two systems to manage non-conformances. Issues suspected of being systematic in nature and for which root cause analysis and a formal Corrective Action Report (CAR) are documented in the Incident Corrective Action Tracking (ICAT) database. Routine batch non-conformances, events that are understood to be isolated in nature, are documented in the LIMS non-conformance memo (NCM) system.

14.2 General

Problems within the quality system or within analytical operations may be discovered in a variety of ways, such as QC sample failures, internal or external audits, proficiency testing (PT) performance, client complaints, staff observation, etc.

The purpose of a corrective action system is to:

- Identify non-conformance events and assign responsibility for investigating.
- Resolve non-conformance events and assign responsibility for any required corrective action.
- Identify systematic problems before they become serious.
- Identify and track client complaints and provide resolution.

14.2.1 Non-Conformance Memo (NCM)

NCMs are used to document the following types of corrective actions:

- Deviations from an established procedure or SOP
- QC outside of limits
- Isolated reporting / calculation errors
- Client complaints
- Discrepancies in materials / goods received vs. manufacturer packing slips (Forms of documentation other than NCMs in LIMS are also acceptable)

14.2.2 Corrective Actions Documented In the ICAT Database

The ICAT database is used to document background information, track the results of corrective action investigations and root cause analysis, and to provide reports of corrective action plans. The following types of issues are documented in ICAT:

- Internal and external audit findings
- Failed or unacceptable PT results
- Identified poor process or method performance trends
- Systematic reporting / calculation errors

- Data recall investigations
- Questionable trends that are found in the review of NCMs.
- Client complaints
- Excessive revised reports

14.3 Closed Loop Corrective Action Process

Any employee in the company can initiate a corrective action. There are four main components to a closed-loop corrective action process once an issue has been identified: Cause Analysis, Selection and Implementation of Corrective Actions (both short and long term), Monitoring of the Corrective Actions, and Follow-up.

14.3.1 Cause Analysis

Upon discovery of a non-conformance event, the event must be defined and documented. An entry into the ICAT system must be initiated, someone is assigned to investigate the issue and the event is investigated for cause. Table 12-1 provides some general guidelines on determining responsibility for assessment.

The cause analysis step is the key to the process as a long term corrective action cannot be determined until the cause is determined.

If the cause is not readily obvious, the Technical Manager, Laboratory Director, or QA Manager (or QA designee) is consulted.

14.3.2 Selection and Implementation of Corrective Actions

Where corrective action is needed, the laboratory shall identify potential corrective actions. The action(s) most likely to eliminate the problem and prevent recurrence are selected and implemented. Responsibility for implementation is assigned.

Corrective actions shall be to a degree appropriate to the magnitude of the problem identified through the cause analysis.

Whatever corrective action is determined to be appropriate, the laboratory shall document and implement the changes. The ICAT record is used for this documentation.

14.3.3 Root Cause Analysis

Root Cause Analysis is a class of problem solving (investigative) methods aimed at identifying the basic or causal factor(s) that underlie variation in performance or the occurrence of a significant failure. The root cause may be buried under seemingly innocuous events, many steps preceding the perceived failure. At first glance, the immediate response is typically directed at a symptom and not the cause. Typically, root cause analysis would be best with three or more incidents to triangulate a weakness. NDSC Document No. CA-Q-S-009: Root Cause Analysis provides guidance on this procedure.

Systematically analyze and document the root causes of the more significant problems that are reported. Identify, track, and implement the corrective actions required to reduce the likelihood of recurrence of significant incidents. Trend the root cause data from these incidents to identify root causes that, when corrected, can lead to dramatic improvements in performance by eliminating entire classes of problems.
Identify the one event associated with problem and ask why this event occurred. Brainstorm the root causes of failures; for example, by asking why events occurred or conditions existed; and then why the cause occurred consecutive times until you get to the root cause. For each of these sub events or causes, ask why it occurred. Repeat the process for the other events associated with the incident.

Root cause analysis does not mean the investigation is over. Look at technique or other systems outside the normal indicators. Often creative thinking will find root causes that ordinarily would be missed and continue to plague the laboratory or operation.

14.3.4 Monitoring of the Corrective Actions

The Technical Manager and QA Manager are responsible to ensure that the corrective action taken was effective.

Ineffective actions are documented and re-evaluated until acceptable resolution is achieved. Technical Managers are accountable to the Laboratory Director to ensure final acceptable resolution is achieved and documented appropriately.

The QA Manager reviews monthly NCM and ICAT records for trends. Highlights are included in the QA monthly report (refer to Section 18). If a significant trend develops that adversely affects quality, an audit of the area is performed and corrective action implemented.

Any out-of-control situations that are not addressed acceptably at the laboratory level may be reported to the NDSC Quality Director by the QA Manager, indicating the nature of the out-of-control situation and problems encountered in solving the situation.

14.3.5 Follow-up Audits

Follow-up audits may be initiated by the QA Manager and shall be performed as soon as possible when the identification of a nonconformance casts doubt on the laboratory's compliance with its own policies and procedures, or on its compliance with state or federal requirements.

These audits often follow the implementation of the corrective actions to verify effectiveness. An additional audit would only be necessary when a critical issue or risk to business is discovered. (Also refer to Section 17.1.4, Special Audits.)

14.4 Technical Corrective Actions

In addition to providing acceptance criteria and specific protocols for technical corrective actions in the method SOPs, the laboratory has general procedures to be followed to determine when departures from the documented policies and procedures and quality control have occurred (refer to Section 13). The documentation of these procedures is through the use of an NCM or record in the ICAT system.

Table 14-1 includes examples of general technical corrective actions. For specific criteria and corrective actions, refer to SOP SA-QA-017: *Evaluation of Batch QC Data* and the analytical SOPs.

Table 14-1 provides some general guidelines for identifying the individual(s) responsible for assessing each QC type and initiating corrective action. The table also provides general guidance on how a data set should be treated if associated QC measurements are unacceptable. Specific procedures are included in method SOPs, Work Instructions, QA Manual Sections 19 and 20. All corrective actions are reviewed monthly, at a minimum, by the QA Manager and highlights are included in the QA monthly report.

To the extent possible, samples shall be reported only if all quality control measures are acceptable. If the deficiency does not impair the usability of the results, data will be reported with an appropriate data qualifier and/or the deficiency will be noted in the case narrative. Where sample results may be impaired, the PM is notified by an NCM and appropriate corrective action (e.g., reanalysis) is taken and documented.

14.5 Basic Corrections

When mistakes occur in records, each mistake shall be crossed-out, [not obliterated (e.g. no white-out)], and the correct value entered alongside. All such corrections shall be initialed (or signed) and dated by the person making the correction. In the case of records stored electronically, the original uncorrected file must be maintained intact and a second corrected file is created. This same process applies to adding additional information to a record. All additions made later than the initial must also be initialed (or signed) and dated. When corrections are due to reasons other than obvious transcription errors, the reason for the corrections (or additions) shall also be documented.

Table 14-1. General Corrective Action Procedures

QC Activity ***(Individual Responsible for Initiation/Assessment)***	**Acceptance Criteria**	**Recommended Corrective Action**
Instrument Blank *(Analyst)*	- Criteria in analytical SOP	- Prepare and analyze another blank. - If same response, determine cause of contamination: reagents, environment, instrument equipment failure, etc.
Initial Calibration Standards *(Analyst)*	- Criteria in analytical SOP	- Reanalyze standards. - If still unacceptable, remake standards and recalibrate instrument.
Initial Calibration Verification (Second Source ICV) *(Analyst)*	- Criteria within analytical SOP	- Remake and reanalyze standard. - If still unacceptable, then remake calibration standards or use new primary standards and recalibrate instrument.
Continuing Calibration Verification (CCV) *(Analyst)*	- Criteria within analytical SOP	- Reanalyze standard. - If still unacceptable, then recalibrate and rerun affected samples.
Matrix Spike / Matrix Spike Duplicate (MS/MSD) *(Analyst)*	- Criteria in LIMS MLGs	- If matrix interferences are present, evaluate the LCS. - If the LCS is within acceptable limits the batch is acceptable.
Laboratory Control Sample (LCS) *(Analyst)*	- Criteria in LIMS MLGs and SOP SA-QA-017	- Reanalyze LCS. - Batch must be re-prepared and/or re-analyzed.
Surrogates *(Analyst)*	- Criteria in LIMS MLGs	- Individual sample must be repeated, unless obvious matrix interference is noted.
Method Blank *(Analyst)*	<1/2RL	- Reanalyze blank. - Determine source of contamination. - Re-prepare/re-analyze batch.

15.0 PREVENTIVE ACTION / IMPROVEMENT

15.1 Overview

The laboratory's preventive action programs improve or eliminate potential causes of nonconforming product and/or nonconformance to the quality system. This preventive action process is a proactive and continuous process of improvement activities that can be initiated through feedback from clients, employees, business providers, and affiliates. The QA Department has the overall responsibility to ensure that the preventive action process is in place, and that relevant information on actions is submitted for management review.

Dedicating resources to an effective preventive action system emphasizes the laboratory's commitment to its QA Program. It is beneficial to identify and address negative trends before they develop into complaints, problems and corrective actions. Additionally, the laboratory continually strives to improve customer service and client satisfaction through continuous improvements to laboratory systems.

Opportunities for improvement may be discovered through any of the following:

- review of the monthly QA Metrics Report,
- trending NCMs,
- review of control charts and QC results,
- trending proficiency testing (PT) results,
- performance of management system reviews,
- trending client complaints,
- review of processing operations, or
- staff observations.

The monthly Management Systems Metrics Report shows performance indicators in all areas of the laboratory and quality system. These areas include revised reports, corrective actions, audit findings, internal auditing and data authenticity audits, client complaints, PT samples, holding time violations, SOPs, ethics training, etc. The metrics report is reviewed monthly by the laboratory management, the NDSC QA Team, and Executive Management. These metrics are used in evaluating the management and quality system performance on an ongoing basis and provide a tool for identifying areas for improvement.

Items identified as continuous improvement opportunities to the management system may be issued as goals from the annual management systems review, recommendations from internal audits, white papers, Lessons Learned, Technical Services audit report, Technical Best Practices, or as Executive or management initiatives.

The laboratory's corrective action process is integral to implementation of preventive actions. A critical piece of the corrective action process is the implementation of actions to prevent further occurrence of a non-compliance event. Historical review of corrective action and non-conformances provides a valuable mechanism for identifying preventive action opportunities.

15.1.1 The following elements are part of a preventive action/process improvement system:

- Identification of an opportunity for preventive action or process improvement.
- Process for the preventive action or improvement.
- Define the measurements of the effectiveness of the process once undertaken.
- Execution of the preventive action or improvement.
- Evaluation of the plan using the defined measurements.
- Verification of the effectiveness of the preventive action or improvement.
- Close-Out by documenting any permanent changes to the Quality System as a result of the Preventive Action or Process Improvement. Documentation of Preventive Action/process Improvement is incorporated into the monthly QA reports, corrective action process and management review.

15.1.2 Any preventive actions/process improvement undertaken or attempted shall be taken into account during the annual Management Systems Review (Section 16). A highly detailed report is not required; however, a summary of successes and failures within the preventive action program is sufficient to provide management with a measurement for evaluation.

15.2 Management of Change

The Management of Change process is designed to manage significant events and changes that occur within the laboratory. Through these procedures, the potential risks inherent with a new event or change are identified and evaluated. The risks are minimized or eliminated through pre-planning and the development of preventive measures. The types of changes covered under this system include: facility changes, major accreditation changes, addition or deletion to capabilities or instrumentation, key personnel changes, and laboratory information management system (LIMS) changes, etc. This process is discussed in further detail within the laboratory SOPs.

16.0 CONTROL OF RECORDS

The laboratory maintains a records management system appropriate to its needs and that complies with applicable standards or regulations as required. The system produces unequivocal, accurate records that document all laboratory activities. The laboratory retains all original observations, calculations and derived data, calibration records and a copy of the analytical report for a minimum of five years after it has been issued. Exceptions for programs with longer retention requirements are discussed in Section 14.1.2.

16.1 Overview

The laboratory has established procedures for identification, collection, indexing, access, filing, storage, maintenance and disposal of quality and technical records. A record index is listed in Table 16-1. More detailed information on retention of specific records is provided in NDSC Document Nos. CW-L-P-001, Records Retention Policy and CW-L-WI-001, Records Retention/Storage Schedule. Quality records are maintained by the QA Department on the Q-Drive, which is backed up as part of the regular laboratory backup. Technical records are maintained in the Data Archival folder on the Public_QA Drive and are backed up as part of the regular network backup. Records are of two types; either electronic or hard copy paper formats depending on whether the record is computer or hand generated (some records may be in both formats).

Table 16-1. Record Index

	Record Types [1]:	Retention Time:
Technical Records	- Raw Data - Logbooks[2] - Standards - Certificates - Analytical Records - MDLs/IDLs/DOCs - Lab Reports	- 5 Years from analytical report issue*
Official Documents	- Quality Assurance Manual (QAM) - Work Instructions - Policies - SOPs - Published Methods	- Indefinitely
QA Records	- Certifications - Method & Software Validation/Verification Data	- Indefinitely
QA Records	- Internal & External Audits/Responses - Corrective/Preventive Actions - Management Reviews - Data Investigation	- 5 Years from archival* - **Data Investigation:** 5 years or the life of the affected raw data storage whichever is greater (beyond 5 years if ongoing project or pending investigation)
Project Records	- Sample Receipt & COC Documentation - Contracts and Amendments - Correspondence - QAPP & SAP - Lab Reports	- 5 Years from analytical report issue*
Administrative Records	Financial and Business Operations	Refer to CW-L-WI-001
	EH&S Manual and Permits	Indefinitely
	Disposal Records	Indefinitely
	Employee Handbook	Indefinitely
	Personnel files, Employee Signature & Initials, Administrative Training Records (e.g., Ethics)	Refer to HR Manual
	Administrative Policies	Indefinitely
	Technical Training Records	7 years
	Legal Records	Indefinitely
	HR Records	Refer to CW-L-WI-001
	IT Records	Refer to CW-L-WI-001
	Corporate Governance Records	Refer to CW-L-WI-001
	Sales & Marketing	5 years
	Real Estate	Indefinitely

[1] Record Types encompass hardcopy and electronic records.
[2] Examples of Logbook types: Maintenance, Instrument Run, Preparation (standard and samples), Standard and Reagent Receipt, Archiving, Balance Calibration, Temperature (hardcopy or electronic records).
* Exceptions listed in Table 16-2.

16.1.1 All records are stored and retained in such a way that they are secure and readily retrievable at the laboratory facility that provides a suitable environment to prevent damage or deterioration and to prevent loss**.** All records shall be protected against fire, theft, loss, environmental deterioration, and vermin. In the case of electronic records, electronic or magnetic sources, storage media are protected from deterioration caused by magnetic fields and/or electronic deterioration.

Access to the data is limited to laboratory and company employees and shall be documented with an access log**.** Records are maintained for a minimum of five years unless otherwise specified by a client or regulatory requirement.

For raw data and project records, record retention shall be calculated from the date the project report is issued. For other records, such as NDSC and/or KGD documents, QA, or administrative records, the retention time is calculated from the date the record is formally retired. Records related to the programs listed in Table 16-2 have lengthier retention requirements and are subject to the requirements in Section 16.1.3.

16.2 Programs with Longer Retention Requirements

Some regulatory programs have longer record retention requirements than the standard record retention time. These are detailed in Table 16-2 with their retention requirements. In these cases, the longer retention requirement is enacted. If special instructions exist such that client data cannot be destroyed prior to notification of the client, the container or box containing that data is marked as to who to contact for authorization prior to destroying the data.

Table 16-2. Special Record Retention Requirements

Program	[1]Retention Requirement
Drinking Water – All States	10 years (lab reports and raw data) 10 years - Radiochemistry (project records)
Drinking Water Lead and Copper Rule	12 years (project records)
Commonwealth of MA – All environmental data 310 CMR 42.14	10 years
FIFRA – 40 CFR Part 160	Retain for life of research or marketing permit for pesticides regulated by EPA
Housing and Urban Development (HUD) Environmental Lead Testing	10 years
Alaska	10 years
Louisiana – All	10 years
Michigan Department of Environmental Quality – all environmental data	10 years
Navy Facilities Engineering Service Center (NFESC)	10 years
Ohio VAP	10 years and State contacted prior to disposal
OSHA	30 years

[1]Note: Extended retention requirements must be noted with the archive documents or addressed in facility-specific records retention procedures.

16.2.1 The laboratory has procedures to protect and back-up records stored electronically and to prevent unauthorized access to or amendment of these records. All analytical data is maintained as hard copy or in a secure readable electronic format. For analytical reports that are maintained as copies in PDF format, refer to Section 19.15.1 for more information.

Electronic records are maintained in the Data Archival Folder on the Public_QA drive, or in another applicable drive (such as Q-drive). Refer to SOP SA-QA-001: Document Control Program for specific information on the archival, storage, and back-up of records.

16.2.2 The record keeping system allows for historical reconstruction of all laboratory activities that produced the analytical data, as well as rapid recovery of historical data. The history of the sample from when the laboratory took possession of the samples must be readily understood through the documentation. This shall include inter-laboratory transfers of samples and/or extracts.

- The records include the identity of personnel involved in sampling, sample receipt, preparation, or testing. All analytical work contains the initials (at least) of the personnel involved. The chain of custody would indicate the name of the sampler.
- All information relating to the laboratory facilities' equipment, analytical test methods, and related laboratory activities, such as sample receipt, sample preparation, or data verification are documented.
- The recordkeeping system facilitates the retrieval of all working files and archived records for inspection and verification purposes (e.g., set format for naming electronic files, set format for what is included with a given analytical data set, etc. as per SOP SA-QA-001:

Document Control Program). Instrument data is stored sequentially by instrument. A given day's analyses are maintained in the order of the analysis. Where an analysis is performed without an instrument, LIMS sheets, bound logbooks, bench sheets, or spreadsheets are used to record and file data. Standard and reagent information is recorded in LIMS for each method.

- Changes to hardcopy records shall follow the procedures outlined in Section 12 and 19. Changes to electronic records in LIMS or instrument data are recorded in audit trails.
- The reason for a signature or initials on a document is clearly indicated in the records such as "sampled by," "prepared by," "reviewed by", or "analyzed by".
- All generated data except those that are generated by automated data collection systems, are recorded directly, promptly and legibly in permanent dark ink.
- Hard copy data may be scanned into PDF format for record storage as long as the scanning process can be verified in order to ensure that no data is lost and the data files and storage media must be tested to verify the laboratory's ability to retrieve the information prior to the destruction of the hard copy that was scanned.
- Also refer to Section 19.15.1 'Computer and Electronic Data Related Requirements'.

16.3 Technical and Analytical Records

16.3.1 The laboratory retains records of original observations, derived data and sufficient information to establish an audit trail, calibration records, staff records and a copy of each analytical report issued, for a minimum of five years unless otherwise specified by a client or regulatory requirement. The records for each analysis shall contain sufficient information to enable the analysis to be repeated under conditions as close as possible to the original. The records shall include the identity of laboratory personnel responsible for performance of each analysis and reviewing results.

16.3.2 Observations, data and calculations are recorded real-time and are identifiable to the specific task.

16.3.3 Changes to hardcopy records shall follow the procedures outlined in Section 12 and 19. Changes to electronic records in LIMS or instrument data are recorded in audit trails.

The essential information to be associated with analysis, such as strip charts, tabular printouts, computer data files, analytical notebooks, and run logs, include:

- Laboratory sample ID code;
- Date of analysis; time of analysis is also required if the holding time is seventy-two (72) hours or less, or when time critical steps are included in the analysis (e.g., drying times, incubations, etc.); instrumental analyses have the date and time of analysis recorded as part of their general operations. Where a time critical step exists in an analysis, location for such a time is included as part of the documentation in a specific logbook or on a benchsheet.
- Instrumentation identification and instrument operating conditions/parameters. Operating conditions/parameters are typically recorded in instrument maintenance logs where available.
- analysis type;
- all manual calculations and manual integrations;
- analyst's or operator's initials/signature;
- sample preparation including cleanup, separation protocols, incubation periods, ID codes,

volumes, weights, instrument printouts, meter readings, calculations, reagents;
- test results;
- standard and reagent origin, receipt, preparation, and use;
- calibration criteria, frequency and acceptance criteria;
- data and statistical calculations, review, confirmation, interpretation, assessment and reporting conventions;
- quality control protocols and assessment;
- electronic data security, software documentation and verification, software and hardware audits, backups, and records of any changes to automated data entries; and
- Method performance criteria including expected quality control requirements. These are indicated both in LIMS and on specific analytical report formats.

16.3.4 All logbooks used during receipt, preparation, storage, analysis, and reporting of samples or monitoring of support equipment shall undergo a periodic, documented supervisory or peer review.

16.4 Laboratory Support Activities

In addition to documenting all the above-mentioned activities, the following are retained QA records and project records (previous discussions in this section relate where and how these data are stored):
- all original raw data, whether hard copy or electronic, for calibrations, samples and quality control measures, including analysts' work sheets and data output records (chromatograms, strip charts, and other instrument response readout records);
- a written description or reference to the specific test method used which includes a description of the specific computational steps used to translate parametric observations into a reportable analytical value;
- copies of final reports;
- archived SOPs;
- correspondence relating to laboratory activities for a specific project;
- all corrective action reports, audits and audit responses;
- proficiency test results and raw data; and
- results of data review, verification, and crosschecking procedures

16.4.1 Sample Handling Records

Records of all procedures to which a sample is subjected while in the possession of the laboratory are maintained. These include but are not limited to records pertaining to:
- sample preservation including appropriateness of sample container and compliance with holding time requirement;
- sample identification, receipt, acceptance or rejection and login;
- sample storage and tracking including shipping receipts, sample transmittal / COC forms; and
- procedures for the receipt and retention of samples, including all provisions necessary to protect the integrity of samples.

16.5 Administrative Records

The laboratory also maintains the administrative records in either electronic or hard copy form. Refer to Table 16-1.

16.6 Records Management, Storage and Disposal

All records (including those pertaining to test equipment), certificates and reports are safely stored, held secure and in confidence to the client. Certification related records are available upon request.

All information necessary for the historical reconstruction of data is maintained by the laboratory. Records that are stored only on electronic media must be supported by the hardware and software necessary for their retrieval.

Records that are stored or generated by computers or personal computers have hard copy, write-protected backup copies, or an electronic audit trail controlling access.

The laboratory has a record management system (a.k.a., document control) for control of laboratory notebooks, instrument logbooks, standards logbooks, and records for data reduction, validation, storage and reporting. Laboratory notebooks are issued on a per analysis basis, and are numbered sequentially. All data are recorded sequentially within a series of sequential notebooks. Bench sheets are filed sequentially. Standards are maintained in the LIMS. Records are considered archived when noted as such in the records management system.

16.6.1 Transfer of Ownership

In the event that the laboratory transfers ownership or goes out of business, the laboratory shall ensure that the records are maintained or transferred according to client's instructions. Upon ownership transfer, record retention requirements shall be addressed in the ownership transfer agreement and the responsibility for maintaining archives is clearly established. In addition, in cases of bankruptcy, appropriate regulatory and state legal requirements concerning laboratory records must be followed. In the event of the closure of the laboratory, all records will revert to the control of NDSC. Should the entire company cease to exist, as much notice as possible will be given to clients and the accrediting bodies who have worked with the laboratory during the previous 5 years of such action.

16.6.2 Records Disposal

Records are removed from the archive and destroyed after 5 years unless otherwise specified by a client or regulatory requirement. On a project specific or program basis, clients may need to be notified prior to record destruction. Records are destroyed in a manner that ensures their confidentiality such as shredding, mutilation or incineration. (Refer to Tables 14-1 and 14-2).

Electronic copies of records must be destroyed by erasure or physically damaging off-line storage media so no records can be read.

If a third party records management company is hired to dispose of records, a “Certificate of Destruction” is required.

17.0 AUDITS

17.1 Internal Audits

Internal audits are performed to verify that laboratory operations comply with the requirements of the lab's quality system and with the external quality programs under which the laboratory operates. Audits are planned and organized by the QA staff. Personnel conducting the audits should be independent of the area being evaluated. Auditors will have sufficient authority, access to work areas, and organizational freedom necessary to observe all activities affecting quality and to report the assessments to laboratory management and, when requested, to Executive Management.

Audits are conducted and documented as described in the NDSC document on performing Internal Auditing, SOP No. CW-Q-S-003. The types and frequency of routine internal audits are described in Table 17-1. Special or ad hoc assessments may be conducted as needed under the direction of the QA staff.

Table 17-1. Types of Internal Audits and Frequency

Description	Performed by	Frequency
Quality Systems Audits	QA Department, QA approved designee, or NDSC QA	All areas of the laboratory annually
Method Audits / QA Technical Audits	Joint Responsibility: a) QA Manager or designee, b) Technical Manager or designee (Refer to SOP CW-Q-S-003)	QA Technical Audits Frequency: 50% of methods annually
SOP Method Compliance	Joint Responsibility: a) QA Manager or designee, b) Technical Manager or designee (Refer to SOP CW-Q-S-003)	SOP Compliance Review Frequency: - Every 2 years (non-DoD SOPs) Every year (Drinking Water SOPs) - 100% of SOPs annually (DoD/DOE SOPs)
Special	QA Department or Designee	Surveillance or spot checks performed as needed, e.g., to confirm corrective actions from other audits.
Performance Testing	Analysts with QA oversight	Two successful per year for each TNI field of testing or as dictated by regulatory requirements

17.1.1 Annual Quality Systems Audit

An annual quality systems audit is required to ensure compliance to analytical methods and SOPs, Eurofins Data Integrity and Ethics Policies (see Sec. 7.2), TNI quality systems, client and state requirements, and the effectiveness of the internal controls of the analytical process, including but not limited to data review, quality controls, preventive action and corrective action. The completeness of earlier corrective actions is assessed for effectiveness & sustainability. The audit is divided into sections for each operating or support area of the lab, and each section is comprehensive for a given area. The area audits may be performed on a rotating schedule throughout the year to ensure adequate coverage of all areas. This schedule may change as situations in the laboratory warrant.

17.1.2 QA Technical Audits

QA technical audits assess data authenticity and analyst integrity. These audits are based on client projects, associated sample delivery groups, and the methods performed. Reported results are compared to raw data to verify the authenticity of results. The validity of calibrations and QC results are compared to data qualifiers, footnotes, and case narratives. Documentation is assessed by examining run logs and records of manual integrations. Manual calculations are checked. Where possible, electronic audit miner programs (e.g., Chrom AuditMiner) are used to identify unusual manipulations of the data deserving closer scrutiny. QA technical audits will include all methods within a two-year period.

17.1.3 SOP Method Compliance

Compliance of all SOPs with the source methods and compliance of the operational groups with the SOPs will be assessed by the Technical Manager or qualified designee at least every year. It is also recommended that the work of each newly hired analyst is assessed within 3 months of working independently, (e.g., completion of method IDOC). In addition, as analysts add methods to their capabilities, (new IDOC) reviews of the analyst work products will be performed.

17.1.4 Special Audits

Special audits are conducted on an as needed basis, generally as a follow up to specific issues such as client complaints, corrective actions, PT results, data audits, system audits, validation comments, regulatory audits or suspected ethical improprieties. Special audits are focused on a specific issue, and report format, distribution, and timeframes are designed to address the nature of the issue.

17.1.5 Performance Testing

Single blind performance audits are employed for several reasons. One purpose is to provide corrective action for parameters judged to be unacceptable on external or internal performance audits. Periodic internal performance audits are also used to test parameters that are not routinely tested by external performance audits. Finally, single blind performance audits are employed to satisfy certain certification requirements, to satisfy auditors' specific requests for

performance audit samples, or to provide additional evidence of data quality to clients with specific questions regarding laboratory performance.

The laboratory participates semi-annually in performance audits conducted through the analysis of PT samples provided by a third party. The laboratory generally participates in the following types of PT studies: Drinking Water, Non-Potable Water, and Soil.

These PT studies are performed approximately six months apart. The first study of the year is usually performed in January, and the second study is usually performed in July. The PT results are submitted to certification agencies directly from the PT Provider. Remedial PT studies can be performed, as required, for any analytes scored as unacceptable. Root cause investigation into any unacceptable results must be initiated. Written responses to unacceptable PT results are required. In some cases it may be necessary for blind QC samples to be submitted to the laboratory to show a return to control.

It is Eurofins policy that PT samples be treated as typical samples in the production process. Furthermore, where PT samples present special or unique problems, in the regular production process they may need to be treated differently, as would any special or unique request submitted by any client. The QA Manager must be consulted and in agreement with any decisions made to treat a PT sample differently due to some special circumstance.
Written investigations for unacceptable PT results are required. In some cases it may be necessary for blind QC samples to be submitted to the laboratory to show a return to control.

17.2 External Audits

External audits are performed when certifying agencies or clients conduct on-site inspections or submit performance testing samples for analysis. It is Eurofins policy to cooperate fully with regulatory authorities and clients. The laboratory makes every effort to provide the auditors with access to personnel, documentation, and assistance. Laboratory supervisors are responsible for providing corrective actions to the QA Manager who coordinates the response. Audit responses are due in the time allotted by the client or agency performing the audit.

The laboratory cooperates with clients and their representatives to monitor the laboratory's performance in relation to work performed for the client. The client may only view data and systems related directly to the client's work. All efforts are made to keep other client information confidential.

17.2.1 Confidential Business Information (CBI) Considerations

During on-site audits, auditors may come into possession of information claimed as business confidential. A business confidentiality claim is defined as "a claim or allegation that business information is entitled to confidential treatment for reasons of business confidentiality or a request for a determination that such information is entitled to such treatment." When information is claimed as business confidential, the laboratory must place on (or attach to) the information at the time it is submitted to the auditor, a cover sheet, stamped or typed legend or other suitable form of notice, employing language such as "trade secret", "proprietary" or "company confidential". Confidential portions of documents otherwise non-confidential must be

clearly identified. CBI may be purged of references to client identity by the responsible laboratory official at the time of removal from the laboratory. However, sample identifiers may not be obscured from the information. Additional information regarding CBI can be found in within the 2009 and 2016 TNI standards.

17.3 Audit Findings

Audit findings are documented using the corrective action process and database (see Section 14). The laboratory's corrective action responses may include action plans that could not be completed within a predefined timeframe. In these instances, a completion date must be set and agreed to by operations management and the QA Manager.

Developing and implementing corrective actions to findings is the responsibility of the Department Manager where the finding originated. Findings that are not corrected by specified due dates are reported monthly to management in the QA monthly report.

If any audit finding casts doubt on the effectiveness of the operations or on the correctness or validity of the laboratory's test results, the laboratory shall take timely corrective action, and shall notify clients in writing if the investigations show that the laboratory results have been affected. Once corrective action is implemented, a follow-up audit is scheduled to ensure that the problem has been corrected.

Clients must be notified promptly in writing of any event such as the identification of defective measuring or test equipment that casts doubt on the validity of results given in any test report or amendment to a test report. The investigation must begin within 24 hours of discovery of the problem and all efforts are made to notify the client within two weeks after the completion of the investigation.

18.0 MANAGEMENT REVIEWS

18.1 Quality Assurance Report

The QA Department is responsible for preparing a comprehensive monthly metrics report to Management to keep them apprised of current quality issues. This report fosters communication, review, and refinement of the QA system to evaluate the suitability of policies and procedures to meet both regulatory and laboratory quality objectives.

The NDSC QA team compiles information from all of the Environment Testing laboratories monthly metrics reports for the Executive Management team. This report includes notable information and concerns regarding the laboratories QA program and a listing of new regulations that may potentially impact the laboratories.

18.2 Annual Management Review

The senior management team conducts a review annually of its quality systems to ensure its continuing suitability and effectiveness in meeting client and regulatory requirements and to introduce any necessary changes or improvements. It will also provide a platform for defining goals, objectives and action items that feed into the laboratory planning system. N DSC personnel is be included in this meeting at the discretion of the Laboratory Director. The LIMS review consists of examining any audits, complaints or concerns that have been raised through the year that are related to LIMS. The laboratory will summarize any critical findings that cannot be solved by the lab and report them to Corporate IT.

This management systems review (NDSC Document No. CW-Q-S-004 and Work Instruction No. CW-Q-WI-003) uses information generated during the preceding year to assess the “big picture” by ensuring that routine actions taken and reviewed on a monthly basis are not components of larger systematic concerns. The monthly review should keep the quality systems current and effective, therefore, the annual review is a formal senior management process to review specific existing documentation. Significant issues from the following documentation are compiled or summarized by the QA Manager prior to the review meeting:

- Matters arising from the previous annual review.
- Prior monthly QA Reports issues.
- Laboratory QA Metrics.
- Review of report reissue requests.
- Review of client feedback and complaints.
- Issues arising from any prior management or staff meetings.
- Minutes from prior senior lab management meetings. Issues that may be raised from these meetings include:
  - Adequacy of staff, equipment and facility resources.
  - Adequacy of policies and procedures.
  - Future plans for resources and testing capability and capacity.
- The annual internal double blind PT program sample performance (if performed),
- Compliance to the Ethics Policy and Data Integrity Plan. Including any evidence/incidents of inappropriate actions or vulnerabilities related to data Integrity.

- Evaluation of overall risk, including risks to impartiality, confidentiality, reporting statements of conformity, and nonconforming work.

A report is generated by the QA Manager and management. The report is distributed to the Laboratory Manager, BUMa, and the Quality Director. The report includes, but is not limited to:

- The date of the review and the names and titles of participants.
- A reference to the existing data quality related documents and topics that were reviewed.
- Quality system or operational changes or improvements that will be made as a result of the review [e.g., an implementation schedule including assigned responsibilities for the changes (Action Table)].

Changes to the quality systems requiring update to the laboratory QA Manual shall be included in the next revision of the QA Manual.

18.3 Potential Integrity Related Managerial Reviews

Potential integrity issues (data or business related) must be handled and reviewed in a confidential manner until such time as a follow-up evaluation, full investigation, or other appropriate actions have been completed and issues clarified. The NDSC Document No. CW-L-S-002: *Internal Investigations* shall be followed. All investigations that result in finding of inappropriate activity are documented and include any disciplinary actions involved, corrective actions taken, and all appropriate notifications of clients.

The Eurofins Environment Testing America Presidents, Business Unit Managers, Laboratory Directors, and NDSC Team are informed of any current data integrity or data recall investigations via the monthly metrics report.

19.0 TEST METHODS AND METHOD VALIDATION

19.1 Overview

The laboratory uses methods that are appropriate to meet our clients' requirements and that are within the scope of the laboratory's capabilities. These include sampling, handling, transport, storage and preparation of samples, and, where appropriate, an estimation of the measurement of uncertainty as well as statistical techniques for analysis of environmental data.

Instructions are available in the laboratory for the operation of equipment as well as for the handling and preparation of samples. All instructions, Standard Operating Procedures (SOPs), reference methods and manuals relevant to the working of the laboratory are readily available to all staff. Deviations from published methods are documented (with justification) in the laboratory's approved SOPs. SOPs are submitted to clients for review at their request. Significant deviations from published methods require client approval and regulatory approval where applicable.

19.2 Standard Operating Procedures (SOPs)

The laboratory maintains SOPs that accurately reflect all phases of the laboratory such as assessing data integrity, corrective actions, handling customer complaints as well as all analytical methods and sampling procedures. The method SOPs are derived from the most recently promulgated/approved, published methods and are specifically adapted to the laboratory facility. Modifications or clarifications to published methods are clearly noted in the SOPs. All SOPs are controlled in the laboratory.

- All SOPs contain a revision number, effective date, and appropriate approval signatures. Controlled copies are available to all staff.
- Procedures for writing an SOP are incorporated by reference to Eurofins TestAmerica's Corporate SOP entitled 'Writing a Standard Operating Procedure', No. CW-Q-S-002 or the laboratory's SOP SA-QA-001: *Document Control*.
- SOPs are reviewed at a minimum annually and, where necessary, revised to ensure continuing suitability and compliance with applicable requirements.

19.3 Laboratory Methods Manual

For each test method, the laboratory shall have available the published referenced method as well as the laboratory developed SOP.

Note: If more stringent standards or requirements are included in a mandated test method or regulation than those specified in this manual, the laboratory shall demonstrate that such requirements are met. If it is not clear which requirements are more stringent, the standard from the method or regulation is to be followed. Any exceptions or deviations from the referenced methods or regulations are noted in the specific analytical SOP.

The laboratory maintains an SOP Index for both technical and non-technical SOPs. Technical SOPs are maintained to describe a specific test method. Non-technical SOPs are maintained to describe functions and processes not related to a specific test method.

19.4 Selection of Methods

Since numerous methods and analytical techniques are available, continued communication between the client and laboratory is imperative to assure the correct methods are utilized. Once client methodology requirements are established, this and other pertinent information is summarized by the PM. These mechanisms ensure that the proper analytical methods are applied when the samples arrive for log-in. For non-routine analytical services (e.g., special matrices, non-routine compound lists), the method of choice is selected based on client needs and available technology. The methods selected should be capable of measuring the specific parameter of interest, in the concentration range of interest, and with the required precision and accuracy.

19.4.1 Sources of Methods

Routine analytical services are performed using standard EPA-approved methodology. In some cases, modification of standard approved methods may be necessary to provide accurate analyses of particularly complex matrices. When the use of specific methods for sample analysis is mandated through project or regulatory requirements, only those methods shall be used.

When clients do not specify the method to be used or methods are not required, the methods used will be clearly validated and documented in an SOP and available to clients and/or the end user of the data. Refer to Appendix 6 for a list of the currently accepted U.S. EPA analytical method references used by the laboratory.

The laboratory reviews updated versions to all the aforementioned references for adaptation based upon capabilities, instrumentation, etc., and implements them as appropriate. As such, the laboratory strives to perform only the latest versions of each approved method as regulations allow or require.

Other reference procedures for non-routine analyses may include methods established by specific states (e.g., Underground Storage Tank methods), ASTM or equipment manufacturers. Sample type, source, and the governing regulatory agency requiring the analysis will determine the method utilized.

The laboratory shall inform the client when a method proposed by the client may be inappropriate or out of date. After the client has been informed, and they wish to proceed contrary to the laboratory's recommendation, it will be documented.

19.4.1.1 Client Supplied Methods

Most of the client-supplied method requirements presented to us involve achieving specific quality control criteria, limits of quantitation (LOQ), and/or method detection limits (MDL) using standard EPA methods. These requirements are communicated to the appropriate technical groups prior to the project start up. Each technical group evaluates the scope of work and the requirements to ensure the criteria can be met using the standard EPA method. The data is

monitored to ensure the criteria are met throughout the project. The PM notifies the client if there is a more appropriate method available or if the client's criteria cannot be achieved on a certain sample matrix (i.e., due to matrix or dilutions).

Occasionally, we are asked to transfer a non-standardized method from a client into our lab or to develop a new method, when one is not available. In the case of a method transfer, we set up the client's method and perform some initial evaluation. After the initial evaluation, we may make recommendations on how to improve method performance. If the method appears to be adequate, we determine linearity, specificity, precision, accuracy, MDL, and LOQ by performing calibrations, analyzing method blanks, and carrying out method detection limit and IDOC studies.

In the case of method development, we work with the client and/or data user to determine the level of validation required ensuring that the method meets its intended purpose. In addition to the elements above, we also determine standard and sample stability and robustness depending on the scope of the project. Typically, a standard operating procedure is written and submitted to the client with the results of the validation. These steps are completed prior to analysis of field samples. Data related to the setup of the method are archived.

19.4.1.2 Procedural Deviations

Analysts are required to follow a documented method for all tests performed; and any deviations from analytical methods must be documented, approved, and justified in an appropriate and consistent manner. We classify method deviations as either being a planned deviation or an unplanned deviation. In general, the following information is captured to document both types of situations:

- Description of the situation
- Reason or justification for the deviation
- Impact the deviation had on the testing
- Signature/date of analyst performing the test
- Signature/date of QA and Laboratory management approving the deviation
- Signature/date of client approval, if necessary

Deviations to written procedures are documented in raw data records or through ICAT system. Both types of documentation require management and QA review and approval.

19.4.2 Demonstration of Capability

Before the laboratory may institute a new method and begin reporting results, the laboratory shall confirm that it can properly operate the method. In general, this demonstration does not test the performance of the method in real world samples, but in an applicable and available clean matrix sample. If the method is for the testing of analytes that are not conducive to spiking, demonstration of capability may be performed on quality control samples.

A demonstration of capability (DOC) is performed whenever there is a change in instrument type (e.g., new instrumentation), matrix, method or personnel (e.g., analyst has not performed the test within the last 12 months).

Note: The laboratory shall have a DOC for all analytes included in the methods that the laboratory performs, and proficiency DOCs for each analyst shall include all analytes that the laboratory routinely performs. Addition of non-routine analytes does not require new DOCs for all analysts if those analysts are already qualified for routine analytes tested using identical chemistry and instrument conditions.

The initial demonstration of capability must be thoroughly documented and approved by the Technical Manager and QA Manager prior to independently analyzing client samples. All associated documentation must be retained in accordance with the laboratory's archiving procedures.

The laboratory must have an approved SOP, demonstrate satisfactory performance, and conduct an MDL study (when applicable). There may be other requirements as stated within the published method or regulations (e.g., retention time window study).

Note: In some instances, a situation may arise where a client requests that an unusual analyte be reported using a method where this analyte is not normally reported. If the analyte is being reported for regulatory purposes, the method must meet all procedures outlined within this QA Manual (SOP, MDL, and Demonstration of Capability). If the client states that the information is not for regulatory purposes, the result may be reported as long as the following criteria are met:

- The instrument is calibrated for the analyte to be reported using the criteria for the method and ICV/CCV criteria are met (unless an ICV/CCV is not required by the method or criteria are per project DQOs).
- The laboratory's nominal or default reporting limit (RL) is equal to the quantitation limit (QL), must be at or above the lowest non-zero standard in the calibration curve and must be reliably determined. Project RLs are client specified reporting levels which may be higher than the QL. Results reported below the QL must be qualified as estimated values. Also see Section 19.6.1.3, Relationship of Limit of Detection (LOD) to Quantitation Limit (QL).
- The client request is documented and the lab informs the client of its procedure for working with unusual compounds. The final report must be footnoted: *Reporting Limit based on the low standard of the calibration curve.*

19.4.3 Initial Demonstration of Capability (IDOC) Procedures

Refer to SOP SA-QA-006: *Training Procedures* for information on performing Initial Demonstrations of Capability (IDOC).

A certification statement (refer to Figure 19-1) can be used to document the completion of each initial demonstration of capability. A copy of the certification is archived in the analyst's training folder.

Note: Results of successive LCS analyses can be used to fulfill the DOC requirement.

19.5 Laboratory Developed Methods and Non-Standard Methods

Any new method developed by the laboratory must be fully defined in an SOP and validated by qualified personnel with adequate resources to perform the method. Method specifications and

the relation to client requirements must be clearly conveyed to the client if the method is a non-standard method (not a published or routinely accepted method). The client must also be in agreement to the use of the non-standard method.

19.6 Validation of Methods

Validation is the confirmation by examination and the provision of objective evidence that the particular requirements for a specific intended use are fulfilled.

All non-standard methods, laboratory designed/developed methods, standard methods used outside of their scope, and major modifications to published methods must be validated to confirm they are fit for their intended use. The validation will be as extensive as necessary to meet the needs of the given application. The results are documented with the validation procedure used and contain a statement as to the fitness for use.

19.6.1 Method Validation and Verification Activities for All New Methods

While method validation can take various courses, the following activities can be required as part of method validation. Method validation records are designated QC records and are archived accordingly.
When changes are made to any validated methods, the influence of such changes shall be documented and, if appropriate, a new validation shall be performed.

19.6.1.1 Determination of Method Selectivity – Method selectivity is the demonstrated ability to discriminate the analyte(s) of interest from other compounds in the specific matrix or matrices from other analytes or interference. In some cases to achieve the required selectivity for an analyte, a confirmation analysis is required as part of the method.

19.6.1.2 Determination of Method Sensitivity – Sensitivity can be both estimated and demonstrated. Whether a study is required to estimate sensitivity depends on the level of method development required when applying a particular measurement system to a specific set of samples. Detection limit studies are conducted as described in Section 19.7. Where other protocols for estimations and/or demonstrations of sensitivity are required by regulation or client agreement, these shall be followed.

19.6.1.3 Relationship of Limit of Detection (LOD) to the Limit of Quantitation (LOQ) – An important characteristic of expression of sensitivity is the distinction between the LOD and the LOQ. The LOD is the minimum level at which the presence of an analyte can be reliably concluded. The LOQ is the minimum concentration of analyte that can be quantitatively determined with acceptable precision and bias, equivalent to the laboratory's routine reporting limit (RL). For most instrumental measurement systems, there is a region where semi-quantitative data is generated around the LOD (both above and below the estimated MDL or LOD) and below the LOQ. In this region, detection of an analyte may be confirmed but quantification of the analyte is unreliable within the accuracy and precision guidelines of the measurement system. When an analyte is detected below the LOQ, and the presence of the analyte is confirmed by meeting the qualitative identification criteria for the analyte, the analyte can be reliably reported, but the amount of the analyte can only be estimated. If data is to be

reported in this region, it must be done so with a qualification that denotes the semi-quantitative nature of the result.

19.6.1.4 Determination of Interferences – A determination that the method is free from interferences in a blank matrix is performed.

19.6.1.5 Determination of Range – Where appropriate to the method, the quantitation range is determined by comparison of the response of an analyte in a curve to established or targeted criteria. Generally the upper quantitation limit is defined by highest acceptable calibration concentration. The lower quantitation limit or QL cannot be lower than the lowest non-zero calibration level, and can be constrained by required levels of bias and precision.

19.6.1.6 Determination of Accuracy and Precision – Accuracy and precision studies are generally performed using replicate analyses, with a resulting percent recovery and measure of reproducibility (standard deviation, relative standard deviation) calculated and measured against a set of target criteria.

19.6.1.7 Documentation of Method – The method is formally documented in an SOP. If the method is a minor modification of a standard laboratory method that is already documented in an SOP, an SOP Attachment describing the specific differences in the new method is acceptable in place of a separate SOP.

19.6.1.8 Continued Demonstration of Method Performance – Continued demonstration of method performance is addressed in the SOP. Continued demonstration of method performance is generally accomplished by batch specific QC samples such as LCS, method blanks or PT samples.

19.7 Method Detection Limit (MDL) / Limits of Detection (LOD)

The MDL is the minimum measured quantity of a substance that can be reported with 99% confidence that the concentration is distinguishable from method blank results, consistent with 40CFR Part 136 Appendix B, August, 2017. The MDL is equivalent to the TNI LOD or DL, and is also equivalent to the DoD/DOE Quality Systems Manual (QSM) DL. The working or final MDL is the higher of the MDL value determined from spikes (MDLs) and the MDL value determined from blanks (MDLb). An initial MDL study shall be performed during the method validation process and when the method is altered in a way that can reasonably be expected to change its sensitivity. On-going data are collected during each quarter in which samples are being analyzed. At least once every 13 months the MDLs and MDLb are re-calculated and re-evaluated using data collected during the preceding period. Details of the laboratory's procedure for conducting MDL studies are given in NDSC Document No.CA-Q-S-006).

19.8 Verification of Detection Limits

If it is found during the re-evaluation of detection limit results that more than 5% of the spiked samples do not return positive numeric results that meet all method qualitative identification criteria, then then spiking level shall be increased and the initial MDL study pre-performed at the

new spiking concentration. For DoD labs, additional guidance provided in NDSC Document No. No. CA-Q-S-006 - Attachment 2.

19.9 Instrument Detection Limits (IDL)

The IDL is sometimes used to assess the reasonableness of the MDL or in some cases required by the analytical method or program requirements. IDLs are most commonly used in metals analyses but may be useful in demonstration of instrument performance in other areas. IDLs are calculated to determine an instrument's sensitivity independent of any preparation method. IDLs are calculated either using 7 replicate spike analyses, like MDL but without sample preparation, or by the analysis of 10 instrument blanks and calculating 3 x the absolute value of the standard deviation.

19.10 Limit of Quantitation

The LOQ shall be at a concentration equivalent to the lowest calibration standard concentration, with the exception of methods using a single-point calibration, and shall be greater than the MDL. The LOQ is verified by preparing and analyzing spikes at concentrations 1-2X the selected LOQ, employing the complete analytical process. For DoD labs, additional requirements provided in NDSC Document No. CA-Q-S-006.

When the laboratory establishes a quantitation limit, it must be initially verified by the analysis of a low level standard or QC sample at 1-2 times the reporting limit or by a DL check samples at or below the LOQ. The LOQ is verified annually thereafter. The annual requirement is waived for methods that have an annually verified MDL. The laboratory will comply with any regulatory requirements.

19.11 Retention Time Windows

Most organic analyses and some inorganic analyses use chromatography techniques for qualitative and quantitative determinations. For every chromatography analysis or as specified in the reference method, each analyte will have a specific time of elution from the column to the detector. This is known as the analyte's retention time. The variance in the expected time of elution is defined as the retention time window. As the key to analyte identification in chromatography, retention time windows must be established on every column for every analyte used for that method. These records are kept on-file and available for review. Complete details are available in the laboratory SOPs.

19.12 Evaluation of Selectivity

The laboratory evaluates selectivity by following the checks within the applicable analytical methods, which include mass spectral tuning, second column confirmation, ICP interelement interference checks, and chromatography retention time windows.

19.13 Estimation of Uncertainty of Measurement

19.13.1 Uncertainty is "a parameter associated with the result of a measurement, that characterizes the dispersion of the values that could reasonably be attributed to the measurand" (as defined by the International Vocabulary of Basic and General Terms in Metrology, ISO Geneva, 1993, ISBN 92-67-10175-1). Knowledge of the uncertainty of a measurement provides additional confidence in a result's validity. Its value accounts for all the factors which could possibly affect the result, such as adequacy of analyte definition, sampling, matrix effects and interferences, climatic conditions, variances in weights, volumes, and standards, analytical procedure, and random variation. Some national accreditation organizations require the use of an "expanded uncertainty" defined as the range within which the value of the measurand is believed to lie within at least a 95% confidence level with the coverage factor k=2.

19.13.2 Uncertainty is not error. Error is a single value (i.e., the difference between the true result and the measured result). On environmental samples, the true result is never known. The measurement is the sum of the unknown true value and the unknown error. Unknown error is a combination of systematic error, or bias, and random error. Bias varies predictably, constantly, and independently from the number of measurements. Random error is unpredictable, assumed to be Gaussian in distribution, and reducible by increasing the number of measurements.

19.13.3 The minimum uncertainty associated with results generated by the laboratory can be determined by using the Laboratory Control Sample (LCS) accuracy range for a given analyte. The LCS limits are used to assess the performance of the measurement system since they take into consideration all of the laboratory variables associated with a given test over time (except for variability associated with the sampling and the variability due to matrix effects). The percent recovery of the LCS is compared either to the method-required LCS accuracy limits or to the statistical, historical, in-house LCS accuracy limits.

19.13.4 To calculate the uncertainty for the specific result reported, multiply the result by the decimal of the lower end of the LCS range percent value for the lower end of the uncertainty range, and multiply the result by the decimal of the upper end of the LCS range percent value for the upper end of the uncertainty range. These calculated values represent uncertainties at approximately the 99% confidence level with a coverage factor of k = 3. As an example, for a reported result of 1.0 mg/L with an LCS recovery range of 50 to 150%, the estimated uncertainty in the result would be 1.0 +/- 0.5 mg/L.

Refer to SOP SA-QA-017: *Evaluation of Batch QC Data* for more information on this topic.

19.13.5 In the case where a well-recognized test method specifies limits to the values of major sources of uncertainty of measurement (e.g., EPA 524.2, EPA 525, etc.) and specifies the form of presentation of calculated results, no further discussion of uncertainty is required.

19.14 Sample Reanalysis Guidelines

Because there is a certain level of uncertainty with any analytical measurement, a sample re-preparation (where appropriate) and subsequent analysis (hereafter referred to as 'reanalysis') may result in either a higher or lower value from an initial sample analysis. There are also

variables that may be present (e.g., sample homogeneity, analyte precipitation over time, etc.) that may affect the results of a reanalysis. Based on the above comments, the laboratory will reanalyze samples at a client's request with the following caveats. Client specific Contractual Terms & Conditions for reanalysis protocols may supersede the following items.

- Homogenous samples: If a reanalysis agrees with the original result to within the RPD limits for MS/MSD or Duplicate analyses, or within ± 1 reporting limit for samples ≤ 5x the reporting limit, the original analysis will be reported. At the client's request, both results may be reported on the same report but not on two separate reports.
- If the reanalysis does not agree (as defined above) with the original result, then the laboratory will investigate the discrepancy and reanalyze the sample a third time for confirmation if sufficient sample is available.
- Any potential charges related to reanalysis are discussed in the contract terms and conditions or discussed at the time of the request. The client will typically be charged for reanalysis unless it is determined that the lab was in error.
- Due to the potential for increased variability, reanalysis may not be applicable to Non-homogenous, Encore, and Sodium Bisulfate preserved samples. See the Area Supervisor or Laboratory Director if unsure.

19.15 Control of Data

The laboratory has policies and procedures in place to ensure the authenticity, integrity, and accuracy of the analytical data generated by the laboratory.

19.15.1 Computer and Electronic Data Related Requirements

The three basic objectives of our computer security procedures and policies are shown below. The laboratory is currently using the Eurofins LIMS system, which has been highly customized to meet the needs of the laboratory.

19.15.1.1 Maintain the Database Integrity – Assurance that data is reliable and accurate through data verification (review) procedures, password-protecting access, anti-virus protection, data change requirements, as well as an internal LIMS permissions procedure.

- LIMS Database Integrity is achieved through data input validation, internal user controls, documentation of system failures and corrective actions taken, and data change requirements.
- Spreadsheets and other software developed in-house must be verified with documentation through hand calculations prior to use. Cells containing calculations must be lock-protected and controlled.
- Instrument hardware and software adjustments are safeguarded through maintenance logs, audit trails and controlled access.

19.15.1.2 Ensure Information Availability – Protection against loss of information or service is ensured through scheduled back-ups, stable file server network architecture, secure storage of media, line filter, Uninterruptible Power Supply (UPS), and maintaining older versions of software as revisions are implemented.

19.15.1.3 Maintain Confidentiality – Ensure data confidentiality through physical access controls such as password protection or website access approval when electronically transmitting data.

19.15.2 Data Reduction

The complexity of the data reduction depends on the analytical method and the number of discrete operations involved (e.g., extractions, dilutions, instrument readings and concentrations). The analyst calculates the final results from the raw data or uses appropriate computer programs to assist in the calculation of final reportable values.

For manual data entry, e.g., Wet Chemistry, the data is reduced by the analyst and then verified by the Department Manager or alternate analyst prior to approving the data in LIMS.

Manual integration of peaks will be documented and reviewed and the raw data will be flagged using NDSC Document No. CA-Q-S-002, *Acceptable Manual Integration Practices* and SOP SA-QA-008: *Evaluation of Chromatographic Data.*

Analytical results are reduced to appropriate concentration units specified by the analytical method, taking into account factors such as dilution, sample weight or volume, etc. Blank correction will be applied only when required by the method or per manufacturer's indication; otherwise, it should not be performed. Calculations are independently verified by appropriate laboratory staff. Calculations and data reduction steps for various methods are summarized in the respective analytical SOPs or program requirements.

19.15.2.1 All raw data must be retained in the worklist folder, computer file (if appropriate), and/or runlog. All criteria pertinent to the method must be recorded. The documentation is recorded at the time observations or calculations are made and must be signed or initialed/dated (month/day/year). It must be easily identifiable who performed which tasks if multiple people were involved.
19.15.2.2 In general, concentration results are reported in milligrams per liter (mg/l) or micrograms per liter (µg/l) for liquids and milligrams per kilogram (mg/Kg) or micrograms per kilogram (µg/Kg) for solids. For values greater than 10,000 mg/L, results can be reported in percent, i.e., 10,000 mg/L = 1**%.** Units are defined in each lab SOP.

19.15.2.3 If final calculations are performed external to LIMS, the results should be entered in LIMS with at least three significant figures. In general, results are reported to 2 significant figures on the final report.

19.15.2.4 For those methods that do not have an instrument printout or an instrumental output compatible with the LIMS, the raw results and dilution factors are entered directly into LIMS by the analyst, and the software calculates the final result for the analytical report. LIMS has a formatter for significant figure criterion for each analyte.

19.15.2.5 The laboratory strives to import data directly from instruments or calculation spreadsheets to ensure that the reported data are free from transcription and calculation errors. For those analyses with an instrumental output compatible with the LIMS, the raw results and dilution factors are transferred into LIMS electronically. Electronic data from instruments are saved electronically in a daily folder on the system (CHROM or instrument computer). For

instruments that print out calibrations and concentrations, the data are retained with the data file. The data file is stored in the Archival Folder on the Public_QA. These files are transferred to the server daily, and eventually, to a tape file.

19.15.3 Logbook / Worksheet Use Guidelines

Logbooks and worksheets are filled out 'real time' and have enough information on them to trace the events of the applicable analysis/task. (e.g. calibrations, standards, analyst, sample ID, date, time on short holding time tests, temperatures when applicable, calculations are traceable, etc.)

- Corrections are made following the procedures outlined in Section 14.
- Logbooks are controlled by the QA department. A record is maintained of all logbooks in the lab.
- Unused portions of pages must be "Z"'d out, signed and dated.
- Worksheets are created with the approval of the Technical Manager/QA Manager at the facility. The QA Manager controls all worksheets following the procedures in Section 6.

19.15.4 Review / Verification Procedures

Data review procedures are outlined in the analytical SOPs and SOP SA-QA-002: *Data Generation and Review* and ensure that data reported are free from calculation and transcription errors and that QC parameters have been reviewed and evaluated before data is reported. The laboratory also has an SOP discussing manual integrations to ensure the authenticity of the data (SOP SA-QA-008). The general review concepts are discussed below; more specific information can be found in the SOPs.

19.15.4.1 Log-In Review - The data review process starts at the sample receipt stage. Sample control personnel review chain-of-custody forms and project instructions from the project management group. This is the basis of the sample information and analytical instructions entered into the LIMS. The log-in instructions are reviewed by the personnel entering the information, and a second level review is conducted by the project management staff.

19.15.4.2 First Level Data Review - The next level of data review occurs with the analysts. As data are generated, analysts review their work to ensure that the results meet project and SOP requirements. First level reviews include inspection of all raw data (e.g., instrument output for continuous analyzers, chromatograms, spectra, and manual integrations), evaluation of calibration/calibration verification data in the day's analytical run, evaluation of QC data, and reliability of sample results. The analyst transfers data into LIMS, data qualifiers are added as needed. All first level reviews are documented.

19.15.4.3 Second Level Data Review – All analytical data are subject to review by a second qualified analyst or supervisor. Second level reviews include inspection of all raw data (e.g., instrument output, chromatograms, and spectra) including 100% of data associated with any changes made by the primary analyst, such as manual integrations or reassignment of peaks to different analytes, or elimination of false negative analytes. The second review also includes evaluation of initial calibration/calibration verification data in the day's analytical run, evaluation of QC data, reliability of sample results, qualifiers and NCM narratives. Manual calculations are checked in second level review. All second level reviews are documented.

Issues that deem further review include the following:

- QC data are outside the specified control limits for accuracy and precision
- Reviewed sample data does not match with reported results
- Unusual detection limit changes are observed
- Samples having unusually high results
- Samples exceeding a known regulatory limit
- Raw data indicating some type of contamination or poor technique
- Inconsistent peak integration
- Transcription errors
- Results outside of calibration range

19.15.4.4 Unacceptable analytical results may require reanalysis of the samples. Any problems are brought to the attention of the Laboratory Director, Project Manager, Quality Director/Manager, Technical Manager, or Supervisor for further investigation. Corrective action is initiated whenever necessary.

19.15.4.5 The results are then entered or directly transferred into the computer database and a pdf is printed for the client.

19.15.4.6 As a final review prior to the release of the report, the Project Manager reviews the results for appropriateness and completeness. This review and approval ensures that client requirements have been met and that the final report has been properly completed. The process includes, but is not limited to, verifying that the COC is followed, cover letters / narratives are present, flags are appropriate, and project specific requirements are met. The Project Manager may also evaluate the validity of results for different test methods given expected chemical relationships.

- Total Results are > Dissolved results (e.g. metals)
- Total Solids (TS) > Total Dissolved Solids (TDS) or Total Suspended Solids (TSS)
- TKN > Ammonia
- Total Phosphorus > Orthophosphate
- COD > TOC
- Total Cyanide > Amenable Cyanide
- TDS > individual anions

19.15.4.7 Any project that requires a data package is subject to a tertiary data review for transcription errors and acceptable quality control requirements. The Project Manager then signs the final report.

19.15.4.8 A brief overview of sample flow and information through the laboratory, as well as data review and validation, is presented in Figure 19-1.

19.15.5 Manual Integrations

Computerized data systems provide the analyst with the ability to re-integrate raw instrument data in order to optimize the interpretation of the data. Though manual integration of data is an invaluable tool for resolving variations in instrument performance and some sample matrix problems, when used improperly, this technique would make unacceptable data appear to meet quality control acceptance limits. Improper re-integrations lead to legally indefensible data, a poor reputation, or possible laboratory decertification. Because guidelines for re-integration of data are not provided in the methods and most methods were written prior to widespread implementation of computerized data systems, the laboratory trains all analytical staff on proper manual integration techniques using NDSC Document No. CA-Q-S-002, Acceptable Manual Integration Practices as the guideline for our internal SOP No. SA-QA-008, entitled *Evaluation of Chromatographic Data.*

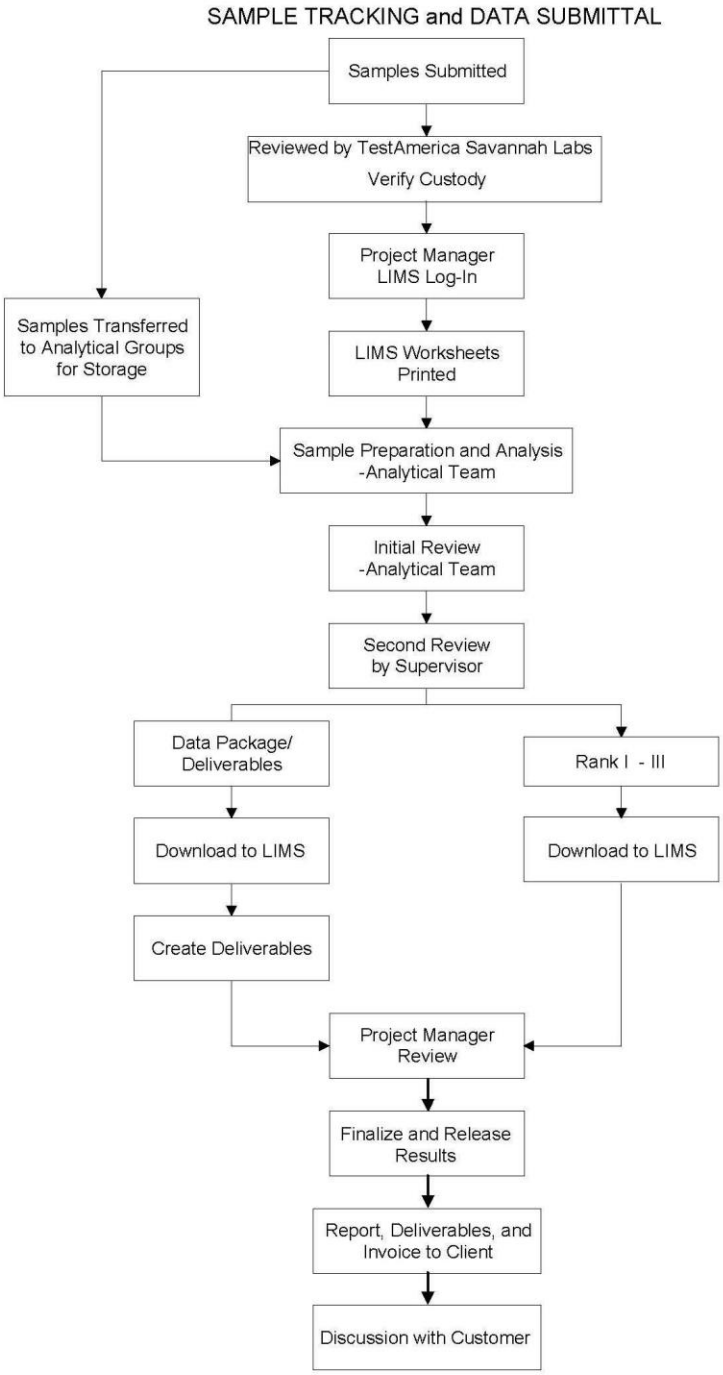
19.15.5.1 The analyst must adjust baseline or the area of a peak in some situations, for example when two compounds are not adequately resolved or when a peak shoulder needs to be separated from the peak of interest. The analyst must use professional judgment to determine when manual integrating is required. Analysts are encouraged to ask for assistance from a senior analyst or manager when in doubt.

19.15.5.2 Analysts shall not increase or decrease peak areas for the sole purpose of achieving acceptable QC recoveries that would have otherwise been unacceptable. The intentional recording or reporting of incorrect information (or the intentional omission of correct information) is against company principles and policy and is grounds for immediate termination.

19.15.5.3 Client samples, performance evaluation samples, and quality control samples are all treated equally when determining whether or not a peak area or baseline should be manually adjusted.

19.15.5.4 All manual integrations receive a second level review. Manual integrations must be indicated on expanded scale “before” and “after” chromatograms such that the integration performed can be easily evaluated during data review. Expanded scale “before” chromatograms are also required for all manual integrations on QC parameters (calibrations, calibration verifications, laboratory control samples, internal standards, surrogates, etc.) unless the laboratory has another documented NDSC approved procedure in place that can demonstrate an active process for detection and deterrence of improper integration practices.

Figure 19-1. Work Flow



20.0 EQUIPMENT and CALIBRATIONS

20.1 Overview

The laboratory purchases the most technically advanced analytical instrumentation for sample analyses. Instrumentation is purchased on the basis of accuracy, dependability, efficiency and sensitivity. Each laboratory is furnished with all items of sampling, preparation, analytical testing and measurement equipment necessary to correctly perform the tests for which the laboratory has capabilities. Each piece of equipment is capable of achieving the required accuracy and complies with specifications relevant to the method being performed. Before being placed into use, the equipment (including sampling equipment) is calibrated and checked to establish that it meets its intended specification. The calibration routines for analytical instruments establish the range of quantitation. Calibration procedures are specified in laboratory SOPs. A list of available laboratory instrumentation is presented in Table 20-1.

Equipment is only operated by authorized and trained personnel. Manufacturer's instructions for equipment use are readily accessible to all appropriate laboratory personnel.

20.2 Préventive Maintenance

The laboratory follows a well-defined maintenance program to ensure proper equipment operation and to prevent the failure of laboratory equipment or instrumentation during use. This program of preventive maintenance helps to avoid delays due to instrument failure.

Routine preventive maintenance procedures and frequency, such as cleaning and replacements, should be performed according to the procedures outlined in the manufacturer's manual. Qualified personnel must also perform maintenance when there is evidence of degradation of peak resolution, a shift in the calibration curve, loss of sensitivity, or failure to continually meet one of the quality control criteria.

Scheduled routine maintenance is defined in each method SOP. It is the responsibility of each Department Manager to ensure that instrument maintenance logs are kept for all equipment in his/her department. Preventative maintenance procedures are also outlined in analytical SOPs or instrument manuals. (Note: for some equipment, the log used to monitor performance is also the maintenance log. Multiple pieces of equipment may share the same log as long as it is clear as to which instrument is associated with an entry.)

Instrument maintenance logs are controlled and are used to document instrument problems, instrument repair and maintenance activities. Maintenance logs shall be kept for all major pieces of equipment. Instrument maintenance logs may also be used to specify instrument parameters.

- Documentation must include all major maintenance activities such as contracted preventive maintenance and service and in-house activities such as the replacement of electrical components, lamps, tubing, valves, columns, detectors, cleaning and adjustments.
- Each entry in the instrument log includes the analyst's initials, the date, a detailed description of the problem (or maintenance needed/scheduled), a detailed explanation of the solution or maintenance performed, and a verification that the equipment is functioning properly (state what was used to determine a return to control. e.g. CCV run on *'date'* was acceptable, or

instrument recalibrated on 'date' with acceptable verification, etc.) must also be documented in the instrument records.
- When maintenance or repair is performed by an outside agency, service receipts detailing the service performed can be affixed into the logbooks adjacent to pages describing the maintenance performed.

If an instrument requires repair (subjected to overloading or mishandling), gives suspect results, or otherwise has shown to be defective or outside of specified limits it shall be taken out of operation and tagged as out-of-service or otherwise isolated until such a time as the repairs have been made and the instrument can be demonstrated as operational by calibration and/or verification or other test to demonstrate acceptable performance. The laboratory shall examine the effect of this defect on previous analyses.

In the event of equipment malfunction that cannot be resolved, service shall be obtained from the instrument vendor manufacturer, or qualified service technician, if such a service can be tendered. If on-site service is unavailable, arrangements shall be made to have the instrument shipped back to the manufacturer for repair. Back up instruments, which have been approved, for the analysis shall perform the analysis normally carried out by the malfunctioning instrument.

If the back-up is not available and the analysis cannot be carried out within the needed timeframe, the samples shall be subcontracted.

At a minimum, if an instrument is sent out for service or transferred to another facility, it must be recalibrated and the laboratory MDL verified (using an MDLV) prior to return to lab operations.

20.3 Support Equipment

This section applies to all devices that may not be the actual test instrument, but are necessary to support laboratory operations. These include but are not limited to: balances, ovens, refrigerators, freezers, incubators, water baths, temperature measuring devices, and volumetric dispensing devices if quantitative results are dependent on their accuracy, as in standard preparation and dispensing or dilution into a specified volume. All raw data records associated with the support equipment are retained to document instrument performance.

20.3.1 Weights and Balances

The accuracy of the balances used in the laboratory is checked every working day, before use. All balances are placed on stable counter tops.

Each balance is checked prior to initial serviceable use with at least two certified ASTM type 1 weights spanning its range of use (weights that have been calibrated to ASTM type 1 weights may also be used for daily verification). ASTM type 1 weights used only for calibration of other weights (and no other purpose) are inspected for corrosion, damage or nicks at least annually and if no damage is observed, they are calibrated at least every 5 years by an outside calibration laboratory. Any weights (including ASTM Type 1) used for daily balance checks or other purposes are recalibrated/recertified annually to NIST standards (this may be done internally if laboratory maintains "calibration only" ASTM type 1 weights).

All balances are serviced annually by a qualified service representative, who supplies the laboratory with a certificate that identifies traceability of the calibration to the NIST standards.

All of this information is recorded in logs, and the recalibration/recertification certificates are kept on file.

20.3.2 pH, Conductivity, and Turbidity Meters

The pH meters used in the laboratory are accurate to ± 0.1 pH units, and have a scale readability of at least 0.05 pH units. The meters automatically compensate for the temperature, and are calibrated with at least two working range buffer solutions before each use.

Conductivity meters used in the laboratory are capable or measuring conductivity with an error not exceeding 1% or one umhos/cm, whichever is greater. The meters are also calibrated before each use with a known standard.

Turbidity meters are also calibrated before each use. All of this information is documented in logs.

Consult pH, Conductivity, and Turbidity SOPs for further information.

20.3.3 Thermometers

All thermometers are calibrated on an annual basis with a NIST-traceable thermometer.

- If the temperature measuring device is used over a range of 10°C or less, then a single point verification within the range of use is acceptable;
- If the temperature measuring device is used over a range of greater than 10°C, then the verification must bracket the range of use.

IR thermometers, digital probes and thermocouples are calibrated quarterly. IR Thermometers should be calibrated over the full range of use, including ambient, iced (4°C) and frozen (0°C to -5°C), per the Drinking Water Manual.

The mercury NIST thermometer is recalibrated every three years (unless thermometer has been exposed to temperature extremes or apparent separation of internal liquid) by an approved outside service and the provided certificate of traceability is kept on file. The NIST thermometer(s) have increments of 1 degree (0.5 degree or less increments are required for drinking water microbiological laboratories), and have ranges applicable to method and certification requirements. The NIST traceable thermometer is used for no other purpose than to calibrate other thermometers.

All of this information is documented electronically. Monitoring method-specific temperatures, including incubators, heating blocks, water baths, and ovens, is documented in equipment-specific logbooks or LIMS sample batches. More information on this subject can be found in SOP SA-AN-100: *Laboratory Support Equipment (Verification and Use)*.

20.3.4 Refrigerators/Freezer Units, Water baths, Ovens and Incubators

The temperatures of all refrigerator units and freezers used for sample and standard storage are monitored each working day – including holidays and weekends (i.e., 7 days a week).
Ovens, water baths and incubators are monitored on days of use.

All of this equipment has a unique identification number, and is assigned a thermometer for monitoring.

Sample storage refrigerator temperatures are kept between > 0ºC and ≤ 6 ºC.

Specific temperature settings/ranges for other refrigerators, ovens, water baths, and incubators can be found in method specific SOPs.

All of this information is documented in Daily Temperature Logbooks and procedure-specific logbooks.

20.3.5 Autopipettors, Dilutors, and Syringes

Mechanical volumetric dispensing devices including burettes (except Class A Glassware and glass microliter syringes) are given unique identification numbers and the delivery volumes are verified gravimetrically, at a minimum, on a quarterly basis (monthly for DoD).

For those dispensers that are not used for quantitative measurements, a label shall be applied to the device stating that it is not calibrated. Any device not regularly verified cannot be used for any quantitative measurements.

Glass micro-syringes are considered the same as Class A glassware provided they are purchased with a manufacturer's certificate attesting to their accuracy. Micro-syringes are routinely purchased from Hamilton Company. The laboratory keeps on file an "Accuracy and Precision Statement of Conformance" from Hamilton attesting established accuracy.

20.4 Instrument Calibrations

Calibration of analytical instrumentation is essential to the production of quality data. Strict calibration procedures are followed for each method. These procedures are designed to determine and document the method detection limits, the working range of the analytical instrumentation and any fluctuations that may occur from day to day.

Sufficient raw data records are retained to allow an outside party to reconstruct all facets of the initial calibration. Records contain, but are not limited to, the following: calibration date, method, instrument, analyst(s) initials or signatures, analysis date, analytes, concentration, response, and type of calibration (Avg RF, curve, or other calculations that may be used to reduce instrument responses to concentration.)

Sample results must be quantitated from the initial calibration and may not be quantitated from any continuing instrument calibration verification unless otherwise required by regulation, method or program.

If the initial calibration results are outside of the acceptance criteria, corrective action is performed and any affected samples are reanalyzed if possible. If the reanalysis is not possible, any data associated with an unacceptable initial calibration will be reported with appropriate data qualifiers (refer to Section 14).

Note: Instruments are calibrated initially and as needed after that and at least annually.

20.4.1 Calibration Standards

Calibration standards are prepared using the procedures indicated in the Reagents and Standards section of the determinative method SOP. If a reference method does not specify the number of calibration standards, a minimum of 3 calibration points will be used.
Standards for instrument calibration are obtained from a variety of sources. All standards are traceable to national or international standards of measurement, or to national or international standard reference materials.

The lowest concentration calibration standard that is analyzed during an initial calibration must be at or below the stated reporting limit for the method based on the final volume of extract (or sample).

The other concentrations define the working range of the instrument/method or correspond to the expected range of concentrations found in actual samples that are also within the working range of the instrument/method. Results of samples not bracketed by initial instrument calibration standards (within calibration range to at least the same number of significant figures used to report the data) must be reported as having less certainty, e.g., defined qualifiers or flags (additional information may be included in the case narrative). The exception to these rules are standard ICP and ICPMS methods which define the working range with periodic linear dynamic range studies, rather than through the range of concentrations of daily calibration standards.

All initial calibrations are verified with a standard obtained from a second source and traceable to a national standard, when available (or vendor certified different lot if a second source is not available). For unique situations, such as air analysis where no other source or lot is available, a standard made by a different analyst at a different time or a different preparation would be considered a second source. This verification occurs immediately after the calibration curve has been analyzed, and before the analysis of any samples.

20.4.1.1 Calibration Verification

The calibration relationship established during the initial calibration must be verified initially and at least daily as specified in the laboratory method SOPs in accordance with the referenced analytical methods and in the 2009 and 2016 TNI Standard. The process of calibration verification applies to both external standard and internal standard calibration techniques, as well as to linear and non-linear calibration models. Initial calibration verification (ICV) is with a standard source secondary (second source standard) to the calibration standards, but

continuing calibration verifications (CCV) may use the same source standards as the calibration curve.

Note: The process of calibration verification referred to here is fundamentally different from the approach called "calibration" in some methods. As described in those methods, the calibration factors or response factors calculated during calibration are used to update the calibration factors or response factors used for sample quantitation. This approach, while employed in other EPA programs, amounts to a daily single-point calibration.

All target analytes and surrogates, including those reported as non-detects, must be included in periodic calibration verifications for purposes of retention time confirmation and to demonstrate that calibration verification criteria are being met, i.e., RPD, per 2009 and 2016 TNI Std. EL-V1M4 Sec. 1.7.2.

All samples must be bracketed by periodic analyses of standards that meet the QC acceptance criteria (e.g., calibration and retention time). The frequency is found in the determinative methods or SOPs.

Note: If an internal standard calibration is being used then bracketing calibration verification standards may not be are not required, only daily verifications are needed, unless specified by the reference method. The results from these verification standards must meet the calibration verification criteria and the retention time criteria (if applicable).

Generally, the calibrations must be verified by an ICV analyzed immediately following initial calibration and before sample analysis. The ICV may be used as the first bracketing CCV, if criteria for both are met.

A continuing instrument calibration verification (CCV) is generally analyzed at the beginning of each 12-hour analytical shift during which samples are analyzed. The 12-hour analytical shift begins with the injection of the calibration verification standard (or the MS tuning standard in MS methods). The shift ends after the completion of the analysis of the last sample, QC, or standard that can be injected within 12-hours of the beginning of the shift. For methods that have quantitation by external calibration models, a CCV is analyzed at the end of each analytical sequence. Some methods have more or less frequent CCV requirements. See specific SOPs. Most inorganic methods require the CCV to be analyzed after ever 10 samples or injections, including matrix or batch QC samples.

Note: If an internal standard calibration is being used (e.g., GCMS) then bracketing standards are not required, only daily verifications are needed, except as specified by program or method requirements.

If the results of a CCV are outside the established acceptance criteria and analysis of a second consecutive (and immediate) CCV fails to produce results within acceptance criteria, corrective action shall be performed. Once corrective actions have been completed and documented, the laboratory shall demonstrate acceptable instrument / method performance by analyzing two consecutive CCVs, or a new initial instrument calibration shall be performed.

Sample analyses and reporting of data may not occur or continue until the analytical system is calibrated or calibration verified. However, data associated with an unacceptable calibration verification may be fully useable reported based upon discussion and approval of the client under the following special conditions:

a) when the acceptance criteria for the CCV are exceeded high (i.e., high bias) and the associated samples within the batch are non-detects, then those non-detects may be reported case narrative comment explaining the high bias. Otherwise the samples affected by the unacceptable CCV shall be re-analyzed after a new calibration curve has been established, evaluated and accepted; or

b) when the acceptance criteria for the CCV are exceeded low (i.e., low bias), those sample results may be reported if they exceed a maximum regulatory limit/decision level. Otherwise the samples affected by the unacceptable CCV shall be re-analyzed after a new calibration curve has been established, evaluated and accepted.

Samples reported by the 2 conditions identified above will be appropriately flagged.

20.4.1.2 Verification of Linear and Non-Linear Calibrations

Calibration verification for calibrations involves the calculation of the percent drift or the percent difference of the instrument response between the initial calibration and each subsequent analysis of the verification standard. (These calculations are available in the laboratory method SOPs.) Verification standards are evaluated based on the % Difference from the average CF or RF of the initial calibration or based on % Drift or % Recovery if a linear or quadratic curve is used.

Regardless of whether a linear or non-linear calibration model is used, if initial verification criterion is not met, then no sample analyses may take place until the calibration has been verified or a new initial calibration is performed that meets the specifications listed in the method SOPs. If the calibration cannot be verified after the analysis of a single verification standard, then adjust the instrument operating conditions and/or perform instrument maintenance, and analyze another aliquot of the verification standard. If the calibration cannot be verified with the second standard, then a new initial calibration is performed.

- When the acceptance criteria for the calibration verification are exceeded high, i.e., high bias, and there are associated samples that are non-detects, then those non-detects may be reported. Otherwise, the samples affected by the unacceptable calibration verification shall be reanalyzed after a new calibration curve has been established, evaluated and accepted.
- When the acceptance criteria for the calibration verification are exceeded low, i.e., low bias, those sample results may be reported if they exceed a maximum regulatory limit/decision level. Otherwise, the samples affected by the unacceptable verification shall be reanalyzed after a new calibration curve has been established, evaluated and accepted. Alternatively, a reporting limit standard may be analyzed to demonstrate that the laboratory can still support non-detects at their reporting limit.

20.5 Tentatively Identified Compounds (TICs) – GC/MS Analysis

For samples containing components not associated with the calibration standards, a library search may be made for the purpose of tentative identification. The necessity to perform this type of identification will be determined by the purpose of the analyses being conducted. For example, the RCRA permit or waste delisting requirements may require the reporting of non-target analytes. Only after visual comparison of sample spectra with the nearest library searches may the analyst assign a tentative identification. Data system library search routines should not use normalization routines that would misrepresent the library or unknown spectra when compared to each other. Additional details are provided in NDSC Document No. CA-Q-QM-001, Tentatively Identified Compounds (TICS) - GCMS Analysis.

Note: If the TIC compound is not part of the client target analyte list but is calibrated by the laboratory and is both qualitatively and/or quantitatively identifiable, it should not be reported as a TIC. If the compound is reported on the same form as true TICs, it should be qualified and/or narrated that the reported compound is qualitatively and quantitatively (if verification in control) reported compared to a known standard that is in control (where applicable).

20.6 GC/MS Tuning

Prior to any GC/MS analytical sequence, including calibration, the instrument parameters for the tune and subsequent sample analyses within that sequence must be set.

Prior to tuning/auto-tuning the mass spectrometer, the parameters may be adjusted within the specifications set by the manufacturer or the analytical method. These generally do not need any adjustment but it may be required based on the current instrument performance. If the tune verification does not pass it may be necessary to clean the source or perform additional maintenance. Any maintenance is documented in the maintenance log.

Table 20-1. Instrumentation List

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Eurofins TestAmerica Savannah Instrument List

Equipment/ Instrument	Manufacturer	Model Number	Serial Number	Year Put into Service	Condition When Received
ICP	Varian (ICP E)	730-ES	IP0712M054	2008	New
ICP	Varian (ICP F)	730-ES	1P0803M118	2012	Used
ICP/MS	Agilent (ICP/MS C)	Agilent 7700x G3281A	JP10390615	2011	New
ICP/MS	Agilent (ICP/MS D)	Agilent 7900 G8403A	JP16071309	2016	New
CVAA	Leeman (2)	HYDRA AA II	00024	2011	New
GC/MS Semivolatiles	Hewlett-Packard (CMS D)	5973/6890	US82311451	1999	New
GC/MS Semivolatiles	Hewlett-Packard (CMS E)	5973/6890	US82311455	1999	New
GC/MS Semivolatiles	Hewlett-Packard (CMS G)	5973/6890	US82311571	1999	New
GC/MS Semivolatiles	Hewlett-Packard (CMS K)	5972/5890	3307A00233	2005	New
GC/MS Semivolatiles	Hewlett-Packard CMS (N)	5973/6890	US72010580	1998	New
GC/MS Semivolatiles	Hewlett-Packard (CMS R)	5973/6890N	21842170	2002	New
GC/MS Semivolatiles	Hewlett-Packard (CMS T)	5973/6890	US33246115	2003	New
GC/MS Semivolatiles	Agilent (CMS W)	5975/6890N	US10608004	2006	New
GC/MS Semivolatiles	Hewlett-Packard (CMS X)	5975/6890N	CN10608061	2006	New
GC/MS Semivolatiles	Agilent (CMS Y)	5975/7980A	US80838915	2008	New
GC/MS Semivolatiles	Hewlett-Packard (CMSAE)	5973/6890	US82311503	2015	Used
GC/MS Volatiles	Hewlett-Packard (CMS A2)	5973/6890	US82311453	2000	New
GC/MS Volatiles	Hewlett-Packard (CMS AA)	5973/6890N	US40620567	2013	Used
GC/MS Volatiles	Hewlett-Packard (CMS B)	5973/6890	US82311452	2000	New

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GC/MS Volatiles	Hewlett-Packard (CMS C)	5975/7890	CN10917056	2012	New
GC/MS Volatiles	Hewlett-Packard (CMS O2)	5973/6890	US7200579	1993	New
GC/MS Volatiles	Hewlett-Packard (CMS P2)	5973/6890	US0039011	2000	New
GC/MS Volatiles	Hewlett-Packard (CMS S)	5973/6890	US21843181	2002	New
GC/MS Volatiles	Agilent (CMS U)	5973/6890	US52441057	2005	New
GC/MS Volatiles	Agilent (CMSAD)	5973/6890	US82311485	2014	Transfer
Ion Chromatograph	Dionex (CIC H)	ICS-2000	06080799	2006	New
Ion Chromatograph	Dionex (CIC K)	ICS-2000	0307011	2012	Used
Ion Chromatograph	Dionex (CIC L)	ICS-2000	05120486	2012	Used
GC Semivolatiles	Hewlett-Packard (CSG J)	6890 (ECD)	US00033184	2000	New
GC Semivolatiles	Hewlett-Packard (CSG K)	6890 (ECD)	US10223085	2002	New
GC Semivolatiles	Agilent (CSG Q)	6890N (FID)	CN10521056	2005	New
GC Semivolatiles	Hewlett-Packard (CSG S)	6890 Plus (ECD)	US00024188	2000	New
GC Semivolatiles	Hewlett-Packard (CSG X)	6890N (ECD)	CN10406086	2003	New
GC Semivolatiles	Agilent (CSG Y)	6890N (ECD)	CN10528081	2005	New
GC Semivolatiles	Agilent (CSG Z)	6890N (ECD)	CN10814004	2008	New
GC Semivolatiles	Agilent (CSGAA)	6890 (ECD)	US00031692	2013	Used
GC Semivolatiles	Agilent (CSG AB1)	6890N(FID)	US10224026	2013	Used
GC Semivolatiles	Agilent (CSG AD)	6890N (ECD)	US10326005	2015	Used
GC Semivolatiles	Agilent (CVG G)	6890 (FID)	14921	2007	New
GC Semivolatiles	Agilent (CVG V)	6890 (FID)	CN10619098	2006	New
GC Volatiles	Agilent (CVG U)	6890 (FID)	US10439011	2005	New
GC Volatiles	Agilent (CVG W)	6890 (FID)	CN10603131	2006	New
Liquid Chromatography	Hewlett-Packard (CLC K)	1100	US72102590	2002	New

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Liquid Chromatography	Hewlett-Packard (CLC N)	1100	DE91607527	2008	Used
Liquid Chromatography	Hewlett-Packard (CLC O)	1100	DE90604676	2016	Used
Liquid Chromatography	Hewlett-Packard (CLC P)	1100	JP24020956	2016	Used
Liquid Chromatography	Hewlett-Packard (CLC Q)	1100	DE14903629	2017	Used
Liquid Chromatography	Hewlett-Packard (CLC R)	1100	DE14903835	2017	Used
General Chemistry	Hach (TURB1)	2100 AN	950400000487	1995	New
General Chemistry	Lachat (2)	QC 8500 Series 2	100200001169	2010	New
General Chemistry	Lachat (3)	QuickChem 8000	A8300-1086	2012	Used
General Chemistry	BOD AssayPlus	Version 3.0	270F6XB334	2006	New
General Chemistry	PCTitrate	Version 3.0	270G6XB370	2006	New
General Chemistry	Konelab (1)	Konelab20	M4218134	2000	New
General Chemistry	Konelab (2)	Konelab20	M3118114	2001	New
General Chemistry	Konelab (3)	Konelab20	S2519236	2013	Used
General Chemistry	Konelab (4)	Konelab20	Z1718383	2016	Used
General Chemistry	Multi X 2500	Analyik jena	N1-197/O	2014	New
General Chemistry	EST Analytical	Xplorer-X	2017.175	2017	New
General Chemistry	Shimadzu (TOC 7)	TOC-L-CPH	H54335232053	2015	New
General Chemistry	Hach (SPC7)	DR2800	1359509	2016	New
General Chemistry	Hach (SPC6)	DR2800	1342967	2016	New
General Chemistry	Shimadzu (SPC8)	UV-1800	A11455150447	2017	New
General Chemistry	Mitsubishi	TOX-300	HOAA0748	2019	New

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21.0 MEASUREMENT TRACEABILITY

21.1 Overview

Traceability of measurements shall be assured using a system of documentation, calibration, and analysis of reference standards. Laboratory equipment that are peripheral to analysis and whose calibration is not necessarily documented in a test method analysis or by analysis of a reference standard shall be subject to ongoing certifications of accuracy. At a minimum, these must include procedures for checking specifications of ancillary equipment: balances, thermometers, temperature, Deionized (DI) and Reverse Osmosis (RO) water systems, automatic pipettes and other volumetric measuring devices. (Refer to Section 20.3). With the exception of Class A Glassware and glass microliter syringes, quarterly accuracy checks are performed for all mechanical volumetric devices. Wherever possible, subsidiary or peripheral equipment is checked against standard equipment or standards that are traceable to national or international standards. Class A Glassware and glass microliter syringes should be routinely inspected for chips, acid etching or deformity (e.g., bent needle). If the Class A glassware or syringe is suspect, the accuracy of the glassware will be assessed prior to use.

21.2 NIST-Traceable Weights and Thermometers

Reference standards of measurement shall be used for calibration only and for no other purpose, unless it can be shown that their performance as reference standards would not be invalidated.

For NIST-traceable weights and thermometers, the laboratory requires that all calibrations be conducted by a calibration laboratory accredited by A2LA, NVLAP (National Voluntary Laboratory Accreditation Program), or another accreditation organization that is a signatory to a MRA (Mutual Recognition Arrangement) of one or more of the following cooperation – ILAC (International Laboratory Accreditation Cooperation) or APLAC (Asia–Pacific Laboratory Accreditation Cooperation). A calibration certificate and scope of accreditation is kept on file at the laboratory.

Calibration laboratory's policy for achieving measurement traceability is defined and includes the subsequent elements of uncertainty. The calibration report or certificate submitted contains a traceability statement, the conditions under which the calibrations were made in the context of any potential influence, a compliance statement with an identified metrological specification and the pertinent clauses, a clearly identified record of the quantities and functional test results before and after re-calibration, and no recommendation on the calibration interval. Opinions and interpretations of results are presented along with the basis upon which they were made and identified as such. All calibration reports are filed in the QA Department.

An external certified service engineer services laboratory balances on an annual basis. This service is documented on each balance with a signed and dated certification sticker. Balance calibrations are checked each day of use. All mercury thermometers are calibrated annually against a traceable reference thermometer. Digital thermometers are verified quarterly against a traceable reference thermometer. Temperature readings of ovens, refrigerators, and incubators are checked on each day of use.

21.3 Reference Standards / Materials

Reference standards/materials, where commercially available, are traceable to certified reference materials. Commercially prepared reference standards, to the extent available, are purchased from vendors that are accredited to ISO Guide 34 and ISO/IEC Guide 17025. All reference standards from commercial vendors shall be accompanied with a certificate that includes at least the following information:

- Manufacturer
- Analytes or parameters calibrated
- Identification or lot number
- Calibration method
- Concentration with associated uncertainties
- Purity

If a standard cannot be purchased from a vendor that supplies a Certificate of Analysis, the purity of the standard is documented by analysis. The receipt of all reference standards must be documented. Reference standards are labeled with a unique reagent ID and expiration date. All documentation received with the reference standard is retained as a QC record and references the reagent ID.

All reference, primary and working standards/materials, whether commercially purchased or laboratory prepared, must be checked regularly to ensure that the variability of the standard or material from the true value does not exceed method requirements. The accuracy of calibration standards is checked by comparison with a standard from a second source. In cases where a second standard manufacturer is not available, a vendor certified different lot is acceptable for use as a second source. For unique situations, where no other source or lot is available, a standard made by a different analyst would be considered a second source. The appropriate Quality Control (QC) criteria for specific standards are defined in laboratory SOPs. In most cases, the analysis of an Initial Calibration Verification (ICV) or LCS (where there is no sample preparation) is used as the second source confirmation. These checks are generally performed as an integral part of the analysis method (e.g. calibration checks, laboratory control samples).

All standards and materials must be stored and handled according to method or manufacturer's requirements in order to prevent contamination or deterioration. Refer to the Environmental Health & Safety Manual or laboratory SOPs. For safety requirements, please refer to method SOPs and the laboratory's Environmental Health and Safety Manual.

Standards and reference materials shall not be used after their expiration dates unless their reliability is verified by the laboratory and their use is approved by the Quality Assurance Manager. The laboratory must have documented contingency procedures for re-verifying expired standards.

21.4 Documentation and Labeling of Standards, Reagents, and Reference Materials

Reagents must be at a minimum the purity required in the test method. The date of reagent receipt and the expiration date are documented. The lots for most of the common solvents and acids are tested for acceptability prior to company-wide purchase. (Refer to NDSC Document No. CA-Q-S-001, Solvent and Acid Lot Testing and Approval.)

All manufacturer or vendor supplied Certificate of Analysis or Purity must be retained, stored appropriately, and readily available for use and inspection. These records are maintained electronically. Records must be kept of the date of receipt and date of expiration of standards, reagents and reference materials. In addition, records of preparation of laboratory standards, reagents, and reference materials must be retained, stored appropriately, and be readily available for use and inspection. For detailed information on documentation and labeling, please refer to method specific SOPs.

Commercial materials purchased for preparation of calibration solutions, spike solutions, etc.., are usually accompanied with an assay certificate or the purity is noted on the label. If the assay purity is 96% or better, the weight provided by the vendor may be used without correction. If the assay purity is less than 96% a correction will be made to concentrations applied to solutions prepared from the stock commercial material. Blended gas standard cylinders use a nominal concentration if the certified value is within +/-15%, otherwise the certified values is used for the canister concentration.

21.4.1 All standards, reagents, and reference materials must be labeled in an unambiguous manner. Standards are logged into the laboratory's LIMS, and are assigned a unique identification number. The following information is typically recorded in the electronic database within the LIMS.

- Standard ID
- Description of Standard
- Department
- Preparer's name
- Final volume and number of vials prepared
- Solvent type and lot number
- Preparation Date
- Expiration Date
- Standard source type (stock or daughter)
- Standard type (spike, surrogate, other)
- Parent standard ID (if applicable)
- Parent Standard Analyte Concentration (if applicable)
- Parent Standard Amount used (if applicable)
- Component Analytes
- Final concentration of each analyte
- Comment box (text field)

Records are maintained electronically for standard and reference material preparation. These records show the traceability to purchased stocks or neat compounds. These records also include method of preparation, date of preparation, expiration date and preparer's name or initials. Preparation procedures are provided in the Method SOPs.

21.4.2 All standards, reagents, and reference materials must be clearly labeled with a minimum of the following information:

- Expiration Date (include prep date for reagents)
- LIMS Standard ID
- Special Health/Safety warnings if applicable

Records must also be maintained of the date of receipt for commercially purchased items or date of preparation for laboratory prepared items. Special Health/Safety warnings must also be available to the analyst. This information is maintained electronically.

21.4.3 In addition, the following information may be helpful:

- Date opened (for multi-use containers, if applicable)
- Description of standard (if different from manufacturer's label or if standard was prepared in the laboratory)
- Recommended Storage Conditions
- Concentration (if applicable)
- Initials of analyst preparing standard or opening container

All containers of prepared reagents must include an expiration date and an ID number to trace back to preparation.

Procedures for preparation of reagents can be found in the Method SOPs.

Standard ID numbers must be traceable through associated logbooks, worksheets and preparation/analytical batch records.

All reagents and standards must be stored in accordance to the following priority: 1) with the manufacturer's recommendations; 2) with requirements in the specific analytical methods as specified in the laboratory SOP.

22.0 SAMPLING

22.1 Overview

The laboratory does not provide sampling services. The laboratory's responsibility in the sample collection process lies in supplying the sampler with the necessary coolers, reagent water, sample containers, preservatives, sample labels, custody seals, COC forms, ice, and packing materials required to properly preserve, pack, and ship samples to the laboratory.

22.2 Sampling Containers

The laboratory offers clean sampling containers for use by clients. These containers are obtained from reputable container manufacturers and meet EPA specifications as required. Certificates of cleanliness for bottles and preservatives are provided by the supplier and are maintained at the laboratory. Alternatively, the certificates may be maintained by the supplier and available to the laboratory on-line.

22.2.1 Preservatives

Upon request, preservatives are provided to the client in pre-cleaned sampling containers. In some cases containers may be purchased pre-preserved from the container supplier. Whether prepared by the laboratory or bought pre-preserved, the grades of the preservatives are at a minimum:

- Hydrochloric Acid – Reagent ACS (Certified VOA Free) or equivalent
- Methanol – Purge and Trap grade
- Nitric Acid – Instra-Analyzed or equivalent
- Sodium Bisulfate – ACS Grade or equivalent
- Sodium Hydroxide – Instra-Analyzed or equivalent
- Sulfuric Acid – Instra-Analyzed or equivalent
- Sodium Thiosulfate – ACS Grade or equivalent

22.3 Definition of Holding Time

The date and time of sampling documented on the COC form establishes the day and time zero. As a general rule, when the maximum allowable holding time is expressed in days (e.g., 14 days, 28 days), the holding time is based on calendar day measured. Holding times expressed in hours (e.g., 6 hours, 24 hours, etc.) are measured from date and time zero. Holding times for analysis include any necessary reanalysis. However, there are some programs that determine holding time compliance based on the date and specific time of analysis compared to the time of sampling regardless of how long the holding time is.

22.4 Sampling Containers, Preservation Requirements, Holding Times

The preservation and holding time criteria specified in the laboratory SOPs are derived from the source documents for the methods. If method required holding times or preservation requirements are not met, the reports will be qualified using a flag, footnote or case narrative.

As soon as possible or “ASAP” is an EPA designation for tests for which rapid analysis is advised, but for which neither EPA nor the laboratory have a basis for a holding time.

22.5 Sample Aliquots / Subsampling

Taking a representative sub-sample from a container is necessary to ensure that the analytical results are representative of the sample collected in the field. The size of the sample container, the quantity of sample fitted within the container, and the homogeneity of the sample need consideration when sub-sampling for sample preparation. It is the laboratory’s responsibility to take a representative subsample or aliquot of the sample provided for analysis.

Analysts should handle each sample as if it is potentially dangerous. At a minimum, safety glasses, gloves, and lab coats must be worn when preparing aliquots for analysis.

Guidelines on taking sample aliquots and subsampling are located SOP SA-QA-015: *Homogenization, Compositing, and Segregation of Samples.*

23.0 HANDLING OF SAMPLES

It is the responsibility of the client to send us representative and/or homogeneous and properly preserved samples of the system from which they are drawn. The laboratory assumes that all multiple sample containers with the same designator/description and bottle type contain a homogeneous, representative sample.

The laboratory provides the appropriate sample containers, required preservative, chain-of-custody (COC) forms, shipping containers, labels, and custody seals. The laboratory also provides trip blanks and analyte-free water for field blanks. Preparation of methanol containers for field preservation of volatile soil samples is available.

Sample management procedures at the laboratory ensure that sample integrity and custody are maintained and documented from sampling/receipt through disposal.

23.1 Chain of Custody (COC)

The COC form is the written documented history of any sample and is initiated when bottles are sent to the field, or at the time of sampling. This form is completed by the sampling personnel and accompanies the samples to the laboratory where it is received and stored under the laboratory's custody. The purpose of the COC form is to provide a legal written record of the handling of samples from the time of collection until they are received at the laboratory. It also serves as the primary written request for analyses from the client to the laboratory. The COC form acts as a purchase order for analytical services when no other contractual agreement is in effect.

23.1.1 Field Documentation

The information the sampler needs to provide at the time of sampling on the container label is:

- Sample identification
- Date and time
- Preservative

During the sampling process, the COC form is completed and must be legible. This form includes information such as:

- Client name, address, phone number and fax number (if available)
- Project name and/or number
- The sample identification
- Date, time and location of sampling
- Sample collectors name
- The matrix description
- The container description
- The total number of each type of container
- Preservatives used
- Analysis requested
- Requested turnaround time (TAT)
- Any special instructions

- Purchase Order number or billing information (e.g. quote number) if available
- The date and time that each person received or relinquished the sample(s), including their signed name.

When the sampling personnel deliver the samples directly to the laboratory personnel, the samples are stored in a cooler with ice, as applicable, and remain solely in the possession of the client's field technician until the samples are delivered to the laboratory personnel. The sample collector must assure that each container is in his/her physical possession or in his/her view at all times, or stored in such a place and manner to preclude tampering. The field technician relinquishes the samples in writing on the COC form to the sample control personnel at the laboratory or to a laboratory courier. When sampling personnel deliver the samples through a common carrier (Fed-Ex, UPS), the CoC relinquished date/time is completed by the field personnel and samples are released to the carrier. Samples are only considered to be received by the laboratory when personnel at the fixed laboratory facility have physical contact with the samples.

Note: Independent couriers are not required to sign the COC form. The COC is usually kept in the sealed sample cooler. The receipt from the courier is stored in log-in by date; it lists all receipts each date.

23.1.2 Legal / Evidentiary Chain-of-Custody

If samples are identified for legal/evidentiary purposes on the COC, login will complete the custody seal retain the shipping record with the COC, and initiate an internal COC for laboratory use by analysts and a sample disposal record.

23.2 Sample Receipt

Samples are received at the laboratory by designated sample receiving personnel and a unique laboratory project identification number is assigned. Each sample container shall be assigned a unique sample identification number that is cross-referenced to the client identification number such that traceability of test samples is unambiguous and documented. Each sample container is affixed with a durable sample identification label. Sample acceptance, receipt, tracking and storage procedures are detailed in the laboratory's SOP No. SA-CU-001: *Sample Receipt Procedures* and summarized in the following sections.

23.2.1 Laboratory Receipt

When samples arrive at the laboratory, sample receiving personnel inspect the coolers and samples. The integrity of each sample must be determined by comparing sample labels or tags with the COC and by visual checks of the container for possible damage. Any non-conformance, irregularity, or compromised sample receipt must be documented on the Sample Receipt Checklist in LIMS and brought to the immediate attention of the client. The COC, shipping documents, documentation of any non-conformance, irregularity, or compromised sample receipt, record of client contact, and resulting instructions become part of the project record.

Sample Registration personnel check and document preservation of non-volatile liquid samples after the samples have been entered into the LIMS and before they are released to the laboratory for testing or placed into storage. Any checks of volatile samples and samples for oil and grease are performed and documented at the time of analysis.

23.3 Sample Acceptance Policy

The laboratory has a written sample acceptance policy (Figure 23-1) that clearly outlines the circumstances under which samples shall be accepted or rejected. These include:

- a COC filled out completely;
- samples must be properly labeled;
- proper sample containers with adequate volume for the analysis (Sampling Guide) and necessary QC;
- samples must be preserved according to the requirements of the requested analytical method (Sampling Guide);
- sample holding times must be adhered to (Sampling Guide);
- the project manager will be notified if any sample is received in damaged condition.

Data from samples which do not meet these criteria are flagged and the nature of the variation from policy is defined.

23.3.1 After inspecting the samples, the sample receiving personnel sign and date the COC form, make any necessary notes of the samples' conditions and store them in appropriate refrigerators or storage locations.

23.3.2 Any deviations from these checks that question the suitability of the sample for analysis, or incomplete documentation as to the tests required will be resolved by consultation with the client. If the sample acceptance policy criteria are not met, the laboratory shall either:

- Retain all correspondence and/or records of communications with the client regarding the disposition of rejected samples, or
- Fully document any decision to proceed with sample analysis that does not meet sample acceptance criteria.

23.4 Sample Storage

In order to avoid deterioration, contamination or damage to a sample during storage and handling, from the time of receipt until all analyses are complete, samples are stored in refrigerators, freezers, or protected locations suitable for the sample matrix. In addition, samples to be analyzed for volatile organic parameters are stored in separate refrigerators designated for volatile organic parameters only. Samples are never to be stored with reagents, standards or materials that may create contamination.

To ensure the integrity of the samples during storage, refrigerator blanks are maintained in the volatile sample refrigerators and analyzed every two weeks.

Analysts retrieve the sample container allocated for their analysis from the designated storage location, prepare or analyze the sample, and return the remaining sample to the storage

location from which it originally came. All samples are scanned into and out of the storage locations using the LIMS sample custody program. Empty containers are scanned into the LIMS sample custody program as empty and are properly disposed of. All samples are kept for at least 30 days after the report is sent out, which meets or exceeds most sample holding times. After this time, the samples are properly disposed of in accordance with the Environmental Health and Safety Manual.

Access to the laboratory is controlled such that sample storage need not be locked at all times unless a project specifically demands it. Samples are accessible to laboratory personnel only. Visitors to the laboratory are prohibited from entering the refrigerator and laboratory areas unless accompanied by an employee of Eurofins TestAmerica.

23.5 Hazardous Samples and Foreign Soils

Upon receipt, foreign soil samples are marked with a fluorescent green “FOREIGN SOIL” label prior to distributing to the analytical departments. Once the sample is received by the department, it is stored in a “FOREIGN SOIL ONLY” box segregated from other samples. Non-hazardous foreign soil samples are sent out for incineration by a USDA-approved waste disposal facility. RCRA hazardous foreign soil samples are heat treated at the laboratory. After heat treatment, normal disposal procedures are followed. Refer to the Environmental Health and Safety Manual Addendum for additional information on disposal of hazardous samples. If not classified as hazardous, foreign soil samples are sent out for incineration by a USDA-approved waste disposal facility.

23.6 Sample Shipping

In the event that the laboratory needs to ship samples, the samples are placed in a cooler with enough ice to ensure the samples remain just above freezing and at or below 6.0°C during transit. The samples are carefully surrounded by packing material to avoid breakage (yet maintain appropriate temperature). A trip blank is enclosed for those samples requiring water/solid volatile organic analyses (see Note). The chain-of-custody form is signed by the sample control technician and attached to the shipping paperwork. Samples are generally shipped overnight express or hand-delivered by a Eurofins TestAmerica courier to maintain sample integrity. All personnel involved with shipping and receiving samples must be trained to maintain the proper chain-of-custody documentation and to keep the samples intact and on ice. The laboratory’s Environmental Health and Safety Manual contains additional shipping requirements.

Note: If a client does not request trip blank analysis on the COC or other paperwork, the laboratory will not analyze the trip blanks that were supplied. However, in the interest of good client service, the laboratory will advise the client at the time of sample receipt that it was noted that they did not request analysis of the trip blank; and that the laboratory is providing the notification to verify that they are not inadvertently omitting a key part of regulatory compliance testing.

23.7 Sample Disposal

Samples should be retained for a minimum of 30 days after the project report is sent, however, provisions may be made for earlier disposal of samples once the holding time is exceeded. Some samples are required to be held for longer periods based on regulatory or client requirements (e.g., 60 days after project report is sent). The laboratory must follow the longer sample retention requirements where required by regulation or client agreement. Several possibilities for sample disposal exist: the sample may be consumed completely during analysis, the sample may be returned to the customer or location of sampling for disposal, or the sample may be disposed of in accordance with the laboratory's waste disposal procedures in the Savannah Addendum to the Environmental Health and Safety Manual. All procedures in the laboratory's Environmental Health and Safety Manual are followed during disposal. Samples are normally maintained in the laboratory no longer than three months from receipt unless otherwise requested. Unused portions of samples found or suspected to be hazardous according to state or federal guidelines may be returned to the client upon completion of the analytical work.
If a sample is part of a known litigation, the affected legal authority, sample data user, and/or submitter of the sample must participate in the decision about the sample's disposal. All documentation and correspondence concerning the disposal decision process must be kept on file. Pertinent information includes the date of disposal, nature of disposal (such as sample depletion, hazardous waste facility disposal, return to client), names of individuals who conducted the arrangements and physically completed the task. The laboratory will remove or deface sample labels prior to disposal unless this is accomplished through the disposal method (e.g., samples are incinerated). A Waste Disposal Record should be completed.

Figure 23-1. Sample Acceptance Policy

All incoming work will be evaluated against the criteria listed below. Where applicable, data from any samples that do not meet the criteria listed below will be noted on the laboratory report defining the nature and substance of the variation. In addition the client will be notified either by telephone, fax, or e-mail ASAP after the receipt of the samples.

1) Samples must arrive in good condition with a Chain-of-Custody (COC) filled out completely and legibly. The following information must be captured:
   - Client name, address, and phone number
   - Project name and/or number
   - Sample identification
   - Date, time and location of sampling
   - Sample collector's name
   - Matrix type
   - Container type
   - Total number of each type of container
   - Preservatives used
   - Analysis requested
   - Any special instructions
   - Purchase Order number or billing information (e.g. quote number) if available.
   - Date and time each person received/relinquished the sample(s), including signed name.
   - Date and time of receipt between last person to relinquish samples and person who receives the samples in the lab.

The project manager will be notified if any sample is received in damaged condition. Eurofins TestAmerica will request that a sample be resubmitted for analysis.

2) Samples must be properly labeled and labels must be legible.
   - Use durable labels (labels provided by Eurofins TestAmerica are preferred)
   - Include a unique identification number
   - Include sampling date and time, and sampler ID
   - Include preservative used.
   - Use indelible ink
   - Information must be legible

3) Samples must be in proper containers with adequate volume for the analysis and any necessary sample-specific QC. Aqueous samples submitted for Volatiles analyses must be submitted without headspace. Samples must be dechlorinated, if needed, and submitted with proper chemical preservation (pH) as required by the analytical test method.

4) Most analytical methods require chilling samples to 4°C. These criteria are met if the samples are chilled to below 6°C and above freezing. Note: Samples hand-delivered to the laboratory immediately after collection are only considered acceptable if there is evidence that the chilling process has begun (i.e., arrival on ice). If sample analysis is begun within fifteen (15) minutes of collection, thermal preservation is not required. Thermal preservation is not required in the field if the laboratory receives and refrigerates the sample within fifteen (15) minutes of collection.

5) Samples must be prepared and analyzed with the holding times defined in the analytical test method.

Other Considerations:

- Chemical preservation (pH) will be verified prior to analysis and documented. The Project Manager will be notified immediately if there is a discrepancy. If analyses will still be performed, all affected results will be flagged to indicate improper preservation.

- Eurofins TestAmerica will make every effort to analyze samples within the regulatory holding time. Samples must be received in the laboratory with enough time to perform the sample analysis. Except for short holding time samples (i.e., <48 hours) sample must be received with at least 48 hours (2 working days) remaining on the holding time to ensure analysis. Samples analyzed in the laboratory will be qualified on the final report with an 'H' to indicate holding time exceedance.

- Analyses that are designated as field analyses (e.g., Odor, pH, Dissolved Oxygen, and Residual Chlorine) should be analyzed as soon as possible by the field sampler prior to delivering to the lab (i.e., within 15 minutes of collection).

- All samples submitted for Volatiles analyses must have a Trip Blank submitted at the same time. Eurofins TestAmerica will supply a blank with the bottle order.

- Per State and/or Federal Regulation, the client is responsible to ensure that samples are shipped in accordance with DOT/IATA requirements, and that radioactive materials may only be delivered to licensed facilities. Any samples containing (or suspected to contain) Source, Byproduct, or Special Nuclear Material as defined by 10 CFR should be delivered directly to facilities licensed to handle such radioactive material. Natural material or ores containing naturally occurring radionuclides may be delivered to any Eurofins TestAmerica facility or courier as long as the activity concentration of the material does not exceed 270 pCi/g alpha or 2700 pCi/g beta (49 CFR Part 173).

Recommendations for packing samples for shipment:

- Pack samples in ice rather than "blue" ice packs.
- Soil samples should be placed in plastic zip-lock bags. The containers often have dirt around the top and do not seal very well and are prone to intrusion from the water from melted ice.
- Water samples would be best if wrapped with bubble-wrap or paper (newspaper, or paper towels work) and then placed in plastic zip-lock bags.
- Fill extra cooler space with bubble wrap.

24.0 ASSURING THE QUALITY OF TEST RESULTS

24.1 Overview

In order to assure our clients of the validity of their data, the laboratory continuously evaluates the quality of the analytical process. The analytical process is controlled not only by instrument calibration as discussed in Section 20, but also by routine process quality control measurements (e.g. Blanks, Laboratory Control Samples (LCS), Matrix Spikes (MS), duplicates (DUP), surrogates, Internal Standards (IS)). These quality control checks are performed as required by the method or regulations to assess precision and accuracy. Quality control samples are to be treated in the exact same manner as the associated field samples being tested. In addition to the routine process quality control samples, Proficiency Testing (PT) Samples (concentrations unknown to laboratory) are analyzed to help ensure laboratory performance.

24.2 Controls

Sample preparation or pre-treatment is commonly required before analysis. Typical preparation steps include homogenization, grinding, solvent extraction, sonication, acid digestion, distillation, reflux, evaporation, drying, and ashing. During these pre-treatment steps, samples are arranged into discreet manageable groups referred to as preparation (prep) batches. Prep batches provide a means to control variability in sample treatment. Control samples are added to each prep batch to monitor method performance and are processed through the entire analytical procedure with investigative/field samples.

24.3 Negative Controls

Table 24-1. Negative Controls

Control Type	Details
Method Blank (MB)	Used to assess preparation and analysis for possible contamination during the preparation and processing steps.
	The specific frequency of use for method blanks during the analytical sequence is defined in the specific standard operating procedure for each analysis. Generally it is 1 for each batch of samples; not to exceed 20 environmental samples.
	The method blank is prepared from a clean matrix similar to that of the associated samples that is free from target analytes (e.g., Reagent water, Ottawa sand, glass beads, etc.) and is processed along with and under the same conditions as the associated samples. The method blank goes through all of the steps of the process (including as necessary: filtration, clean-ups, etc.).
	Reanalyze or qualify associated sample results when the concentration of a targeted analyte in the blank is at or above the reporting limit as established by the method or by regulation, AND is greater than 1/10 of the amount measured in the sample.

Table 24-1. Negative Controls

Control Type	Details
Calibration Blanks	Prepared and analyzed along with calibration standards where applicable. They are prepared using the same reagents that are used to prepare the standards. In some analyses the calibration blank may be included in the calibration curve.
Instrument Blanks	Blank reagents or reagent water that may be processed during an analytical sequence in order to assess contamination in the analytical system. In general, instrument blanks are used to differentiate between contamination caused by the analytical system and that caused by the sample handling or sample prep process. Instrument blanks may also be inserted throughout the analytical sequence to minimize the effect of carryover from samples with high analyte content.
Trip Blank [1]	Required to be submitted by the client with each shipment of samples requiring aqueous and solid volatiles analyses (or as specified in the client's project plan). Additionally, trip blanks may be prepared and analyzed for volatile analysis of air samples, when required by the client. A trip blank may be purchased (certified clean) or is prepared by the laboratory by filling a clean container with pure deionized water that has been purged to remove any volatile compounds. Appropriate preservatives are also added to the container. The trip blank is sent with the bottle order and is intended to reflect the environment that the containers are subjected to throughout shipping and handling and help identify possible sources if contamination is found. The field sampler returns the trip blank in the cooler with the field samples.
Field Blanks [1]	Sometimes used for specific projects by the field samplers. A field blank prepared in the field by filling a clean container with pure reagent water and appropriate preservative, if any, for the specific sampling activity being undertaken. (EPA OSWER)
Equipment Blanks [1]	Sometimes created in the field for specific projects. An equipment blank is a sample of analyte-free media which has been used to rinse common sampling equipment to check effectiveness of decontamination procedures.
Holding Blanks	Referred to as refrigerator or freezer blanks, are used to monitor the sample storage units for volatile organic compounds during the storage of VOA samples in the laboratory

[1] When known, these field QC samples should not be selected for matrix QC as it does not provide information on the behavior of the target compounds in the field samples. Usually, the client sample ID will provide information to identify the field blanks with labels such as "FB", "EB", or "TB."

Evaluation criteria and corrective action for these controls are defined in the specific standard operating procedure for each analysis.

24.4 Positive Control

Control samples (e.g., QC indicators) are analyzed with each batch of samples to evaluate data based upon method performance (e.g., Laboratory Control Sample), which entails both the preparation and measurement steps; and matrix effects (e.g., Matrix Spike or Sample Duplicate), which evaluates field sampling accuracy, precision, representativeness, interferences, and the effect of the matrix on the method performed. Each regulatory program and each method within those programs specify the control samples that are prepared and/or analyzed with a specific batch

Note that frequency of control samples vary with specific regulatory, methodology and project specific criteria. Complete details on method control samples are as listed in each analytical SOP.

24.4.1 Method Performance Control - Laboratory Control Sample (LCS)

The LCS measures the accuracy of the method in a blank matrix and assesses method performance independent of potential field sample matrix affects in a laboratory batch.

The LCS is prepared from a clean matrix similar to that of the associated samples that is free from target analytes (for example: Reagent water, Ottawa sand, glass beads, etc.) and is processed along with and under the same conditions as the associated samples. The LCS is spiked with verified known amounts of analytes or is made of a material containing known and verified amounts of analytes, taken through all preparation and analysis steps along with the field samples. Where there is no preparation taken for an analysis (such as in aqueous volatiles), or when all samples and standards undergo the same preparation and analysis process (such as phosphorus), a calibration verification standard is reported as the LCS.

In some instances where there is no practical clean solid matrix available, aqueous LCS's may be processed for solid matrices; final results may be calculated as mg/kg or ug/kg, assuming 100% solids and a weight equivalent to the aliquot used for the corresponding field samples, to facilitate comparison with the field samples.

Certified pre-made reference material purchased from a NIST/A2LA accredited vendor may also be used for the LCS when the material represents the sample matrix or the analyte is not easily spiked (e.g. solid matrix LCS for metals, TDS, etc.).

The specific frequency of use for LCS during the analytical sequence is defined in the specific standard operating procedure for each analysis. It is generally 1 for each batch of samples; not to exceed 20 environmental samples.

If the mandated or requested test method, or project requirements, do not specify the spiking components, the laboratory shall spike all reportable components to be reported in the Laboratory Control Sample (and Matrix Spike) where applicable (e.g. no spike of pH). However, in cases where the components interfere with accurate assessment (such as simultaneously spiking chlordane, Toxaphene and PCBs in Method 608), the test method has an extremely

long list of components or components are incompatible, at a minimum, a representative number of the listed components (see below) shall be used to control the test method. The selected components of each spiking mix shall represent all chemistries, elution patterns and masses, permit specified analytes and other client requested components. However, the laboratory shall ensure that all reported components are used in the spike mixture within a two-year time period.

- For methods that have 1-10 target analytes, spike all components.
- For methods that include 11-20 target analytes, spike at least 10 or 80%, whichever is greater.
- For methods with more than 20 target analytes, spike at least 16 components.
- Exception: Due to analyte incompatibility in pesticides, Toxaphene and Chlordane are only spiked at client request based on specific project needs.
- Exception: Due to analyte incompatibility between the various PCB aroclors, aroclors 1016 and 1260 are used for spiking as they cover the range of all of the aroclors. Specific aroclors may be used by request on a project specific basis.

24.5 Sample Matrix Controls

Table 24.5. Sample Matrix Controls

Control Type	Details	
Matrix Spikes (MS)	Use	Used to assess the effect sample matrix of the spiked sample has on the precision and accuracy of the results generated by the method used;
	Typical Frequency [1]	At a minimum, with each matrix-specific batch of samples processed, an MS is carried through the complete analytical procedure. Unless specified by the client, samples used for spiking are randomly selected and rotated between different client projects. If the mandated or requested test method does not specify the spiking components, the laboratory shall spike all reportable components to be reported in the Laboratory Control Sample and Matrix Spike. Refer to the method SOP for complete details
	Description	Essentially a sample fortified with a known amount of the test analyte(s).
Surrogate	Use	Measures method performance to sample matrix (organics only).
	Typical Frequency [1]	Added to all samples, standards, and blanks, for all organic chromatography methods except when the matrix precludes its use or when a surrogate is not available. The recovery of the surrogates is compared to the acceptance limits for the specific method. Poor surrogate recovery may indicate a problem with sample composition and shall be reported, with data qualifiers, to the client whose sample produced poor recovery.
	Description	Similar to matrix spikes except the analytes are compounds with properties that mimic the analyte of interest and are unlikely to be found in environment samples.
Duplicates[2]	Use	For a measure of analytical precision, with each matrix-specific batch of samples processed, a matrix duplicate (MD or DUP) sample, matrix spike duplicate (MSD), or LCS duplicate (LCSD) is carried through the complete analytical procedure.

Control Type	Details	
	Typical Frequency [1]	Duplicate samples are usually analyzed with methods that do not require matrix spike analysis.
	Description	Performed by analyzing two aliquots of the same field sample independently or an additional LCS.
Internal Standards	Use	Spiked into all environmental and quality control samples (including the initial calibration standards) to monitor the qualitative aspect of organic and some inorganic analytical measurements.
	Typical Frequency [1]	All organic and ICP methods as required by the analytical method.
	Description	Used to correct for matrix effects and to help troubleshoot variability in analytical response and are assessed after data acquisition. Possible sources of poor internal standard response are sample matrix, poor analytical technique or instrument performance.

[1] See the specific analytical SOP for type and frequency of sample matrix control samples.
[2] LCSDs are normally not performed except when regulatory agencies or client specifications require them. The recoveries for the spiked duplicate samples must meet the same laboratory established recovery limits as the accuracy QC samples. If an LCSD is analyzed both the LCS and LCSD must meet the same recovery criteria and be included in the final report. The precision measurement is reported as "Relative Percent Difference" (RPD). Poor precision between duplicates (except LCS/LCSD) may indicate non-homogeneous matrix or sampling.

24.6 Acceptance Criteria (Control Limits)

As mandated by the test method and regulation, each individual analyte in the LCS, MS, or Surrogate Spike is evaluated against the control limits published in the test method. Where there are no established acceptance criteria, the laboratory calculates in-house control limits with the use of control charts or, in some cases, utilizes client project specific control limits. When this occurs, the regulatory or project limits will supersede the laboratory's in-house limits.

Note: For methods, analytes and matrices with very limited data (e.g., unusual matrices not analyzed often), interim limits are established using available data or by analogy to similar methods or matrices.

Once control limits have been established, they are verified, reviewed, and updated if necessary on an annual basis unless the method requires more frequent updating. Control limits are established per method (as opposed to per instrument) regardless of the number of instruments utilized.

Laboratory generated % Recovery acceptance (control) limits are generally established by taking ± 3 Standard Deviations (99% confidence level) from the average recovery of a minimum of 20-30 data points (more points are preferred).

- Regardless of the calculated limit, the limit should be no tighter than the Calibration Verification (ICV/CCV). (Unless the analytical method specifies a tighter limit).

- In-house limits cannot be any wider than those mandated in a regulated analytical method. Client or contract required control limits are evaluated against the laboratory's statistically derived control limits to determine if the data quality objectives (DQOs) can be achieved. If laboratory control limits are not consistent with DQOs, then alternatives must be considered, such as method improvements or use of an alternate analytical method.
- For routine analytes that are not classified as poor performers, the lowest acceptable recovery limit will be 10% (the analyte must be detectable and identifiable). Exception: The lowest acceptable recovery limit for Benzidine will be 5% and the analyte must be detectable and identifiable.
- If either the high or low end of the control limit changes by $\leq$ 5% from previous, the control chart may be visually inspected and, using professional judgment, they may be left unchanged if there is no effect on laboratory ability to meet the existing limits.

24.6.1 The lab must be able to generate a current listing of their control limits and track when the updates are performed. In addition, the laboratory must be able to recreate historical control limits.

The QA Department generates a Method Limit Group (MLG) in the LIMS that contains tables that summarize the precision and accuracy acceptability limits for analyses performed at Eurofins TestAmerica Savannah. The MLG includes an effective date and is updated each time new limits are generated and entered. Unless otherwise noted, limits within these tables are laboratory generated. The LIMS maintains an archive of all limits used within the laboratory.

24.6.2 A LCS that is within the acceptance criteria establishes that the analytical system is in control and is used to validate the process. Samples that are analyzed with an LCS with recoveries outside of the acceptance limits may be determined as out of control and should be reanalyzed if possible. If reanalysis is not possible, then the results for all affected analytes for samples within the same batch must be qualified when reported. The internal corrective action process (see Section 12) is also initiated if an LCS exceeds the acceptance limits.

Sample results may be qualified and reported without reanalysis if:

- The analyte results are below the reporting limit and the LCS is above the upper control limit.
- If the analytical results are above the relevant regulatory limit and the LCS is below the lower control limit.
- If there are an allowable number of Marginal Exceedances (ME):

<11 analytes	0 marginal exceedances are allowed
11 – 30 Analytes	1 marginal exceedance is allowed
31-50 Analytes	2 marginal exceedances are allowed
51-70 Analytes	3 marginal exceedances are allowed
71-90 Analytes	4 marginal exceedances are allowed
> 90 Analytes	5 marginal exceedances are allowed

Marginal exceedances are recovery exceedances between 3 SD and 4 SD from the mean recovery limit (TNI).

Marginal exceedances should be random. If the same analyte exceeds the LCS control limit repeatedly, it is an indication of a systematic problem. The source of the error must be located and corrective action taken.

Though marginal exceedances may be allowed, the data must still be qualified to indicate it is outside of the normal limits.

24.6.3 If the MS/MSDs do not meet acceptance limits, the MS/MSD and the associated spiked sample is reported with a qualifier for those analytes that do not meet limits. If obvious preparation errors are suspected, or if requested by the client, unacceptable MS/MSDs are reprocessed and reanalyzed to prove matrix interference. A more detailed discussion of acceptance criteria and corrective action can be found in the lab's method SOPs and in Section 12.

24.6.4 If a surrogate standard falls outside the acceptance limits, and if there is not obvious chromatographic matrix interference, reanalyze the sample to confirm a possible matrix effect. If the recoveries confirm or there was obvious chromatographic interference, results are reported from the original analysis and a qualifier is added. If the reanalysis meets surrogate recovery criteria, the second run is reported (or both are reported if requested by the client). Under certain circumstances, where all of the samples are from the same location and share similar chromatography, the reanalysis may be performed on a single sample rather than all of the samples and if the surrogate meets the recovery criteria in the reanalysis, all of the affected samples would require reanalysis.

24.7 Additional Procedures to Assure Quality Control

The laboratory has written and approved method SOPs to assure the accuracy of the test method including calibration (see Section 20), use of certified reference materials (see Section 21) and use of PT samples (see Section 17).

A discussion regarding MDLs, Limit of Detection (LOD) and Limit of Quantitation (LOQ) can be found in Section 19.

- Use of formulae to reduce data is discussed in the method SOPs and in Section 20.
- Selection of appropriate reagents and standards is included in Section 11 and 21.
- A discussion on selectivity of the test is included in Section 7.
- Constant and consistent test conditions are discussed in Section 6.
- The laboratories sample acceptance policy is included in Section 23.

25.0 REPORTING RESULTS

25.1 Overview

The results of each test are reported accurately, clearly, unambiguously, and objectively in accordance with State and Federal regulations as well as client requirements. Analytical results are issued in a format that is intended to satisfy customer and laboratory accreditation requirements as well as provide the end user with the information needed to properly evaluate the results. Where there is conflict between client requests and laboratory ethics or regulatory requirements, the laboratory's ethical and legal requirements are paramount, and the laboratory will work with the client during project set up to develop an acceptable solution. Refer to Section 9.

A variety of report formats are available to meet specific needs.

In cases where a client asks for simplified reports, there must be a written request from the client. There still must be enough information that would show any analyses that were out of conformance (QC out of limits) and there should be a reference to a full report that is made available to the client. Review of reported data is included in Section 19.

25.2 Test Reports

Analytical results are reported in a format that is satisfactory to the client and meets all requirements of applicable accrediting authorities and agencies. A variety of report formats are available to meet specific needs. The report is printed on laboratory letterhead, reviewed, and signed by the appropriate project manager. At a minimum, the standard laboratory report shall contain the following information:

25.2.1 A report title (e.g., Analytical Report)
25.2.2 The cover page shall include the laboratory name, address and telephone number.
25.2.3 A unique identification of the report (e.g., Eurofins TestAmerica Job ID #) and on each page an identification in order to ensure the page is recognized as part of the report and a clear identification of the end.

Note: Page numbers of report are represented as page # of ##. Where the first number is the page number and the second is the total number of pages.

25.2.4 A copy of the chain of custody (COC).
- Any COCs involved with Subcontracting are included.

25.2.5 The name and address of client and a project name/number, if applicable.
25.2.6 Client project manager or other contact
25.2.7 Description and unambiguous identification of the tested sample(s) including the client identification code.
25.2.8 Date of receipt of sample, date and time of collection, and date(s) of test preparation and performance, and time of preparation or analysis if the required holding time for either activity is less than or equal to 72 hours.
25.2.9 Date reported or date of revision, if applicable.

25.2.10 Method of analysis including method code (EPA, Standard Methods, etc.).
25.2.11 Reporting limit.
25.2.12 Method detection limits (if requested)
25.2.13 Definition of Data qualifiers and reporting acronyms (e.g. ND).
25.2.14 Sample results.
25.2.15 QC data consisting of method blank, surrogate, LCS, and MS/MSD recoveries and control limits.
25.2.16 Condition of samples at receipt including temperature. This may be accomplished in a narrative or by attaching sample login sheets (Refer to Sec. 25.2.4 – Item 3 regarding additional addenda).
25.2.17 A statement expressing the validity of the results, that the source methodology was followed, and all results were reviewed for error.
25.2.18 A statement to the effect that the results relate only to the items tested and the sample as received by the laboratory, except when information is provided by the client. When data is provided by the client there shall be a clear identification of it, and a disclaimer shall be put in the report when the client supplied data can affect the validity of the test.
25.2.19 A statement that the report shall not be reproduced except in full, without prior express written approval by the laboratory coordinator.
25.2.20 A signature and title of the person(s) accepting responsibility for the content of the report and date of issue. Authorized signatories are qualified Project Managers appointed by the Manager of Project Managers.
25.2.21 When TNI accreditation is required, the lab shall certify that the test results meet all requirements of TNI or provide reasons and/or justification if they do not.
25.2.22 Where applicable, a narrative to the report that explains the issue(s) and corrective action(s) taken in the event that a specific accreditation or certification requirement was not met.
25.2.23 When soil samples are analyzed, a specific identification as to whether soils are reported on a “wet weight” or “dry weight” basis.
25.2.24 Appropriate laboratory certification number for the state of origin of the sample, if applicable.
25.2.25 If only part of the report is provided to the client (client requests some results before all of it is complete), it must be clearly indicated on the report (e.g., partial report, or how your lab identifies it). A complete report must be sent once all of the work has been completed.
25.2.26 Any non-Eurofins subcontracted analysis results are provided as a separate report on the official letterhead of the subcontractor. All Eurofins subcontracting is clearly identified on the report as to which laboratory performed a specific analysis.
25.2.27 A Certification Summary Report, where required, will document that, unless otherwise noted, all analytes tested and reported by the laboratory were covered by the noted certifications.

Note: Refer to the NDSC Document No. CA-I-P-002, Electronic Reporting and Signature Policy, for details on internally applying electronic signatures of approval.

25.2.28 Where the laboratory is responsible for the sampling stage, in addition to the requirements listed above, reports containing the results of sampling shall include the following, where necessary for the interpretation of test results:

- the date of sampling;
- unambiguous identification of the material sampled;
- the location of sampling plan and procedures, and deviations, addition to or exclusions from the sample procedures;
- a reference to the sampling plan and procedure, and deviations, additions to or exclusions from the sample procedures;
- details of any environmental conditions during sampling that affect the interpretation of test results;
- information required to evaluate measurement uncertainty for subsequent testing

25.3 Reporting Level or Report Type

The laboratory offers four levels of quality control reporting. Each level, in addition to its own specific requirements, contains all the information provided in the preceding level. The packages provide the following information in addition to the information described above:

- Level 1 is a report with all of the elements outlined in Section 25.2 above, excluding 25.2.15 (QC data).
- Level II is a Level I report plus summary information, including results for the method blank reported to the laboratory MDL, percent recovery for laboratory control samples and matrix spike samples, and the RPD values for all MSD and sample duplicate analyses.
- Level III contains all the information supplied in Level II, but presented on the CLP-like summary forms, and relevant calibration information. A Level II report is not included, unless specifically requested. No raw data is provided.
- Level IV is the same as Level III with the addition of all raw supporting data.

In addition to the various levels of QC packaging, the laboratory also provides reports in EDD (see below). Initial reports may be provided to clients by facsimile. Procedures used to ensure client confidentiality are outlined in Section 25.6.

25.4 Electronic Data Deliverables (EDDs)

EDDs are routinely offered as part of Eurofins TestAmerica's services in addition to the test report as described in Section 25.2. When NELAP accreditation is required and both a test report and EDD are provided to the client, the official version of the test report will be the combined information of the report and the EDD. Eurofins TestAmerica Savannah offers a variety of EDD formats including Environmental Resources Program Information Management System (ERPIMS), Automated Data Review (ADR), Locus Focus (EIM), EQUIS ESBasic, Environmental Quality Information Systems (EQUIS), Staged Electronic Data Deliverable (SEDD), EPA Region V EDD (EDMAN), and Terrabase.

EDD specifications are submitted to the IT department by the PM for review and undergo the contract review process. Once the facility has committed to providing data in a specific electronic format, the coding of the format may need to be performed. This coding is documented and validated. The validation of the code is retained by the IT staff coding the EDD.

EDDs shall be subject to a review to ensure their accuracy and completeness. If EDD generation is automated, review may be reduced to periodic screening if the laboratory can demonstrate that it can routinely generate that EDD without errors. Any revisions to the EDD format must be reviewed until it is demonstrated that it can routinely be generated without errors. If the EDD can be reproduced accurately and if all subsequent EDDs can be produced error-free, each EDD does not necessarily require a review.

25.5 Supplemental Information for Test

The laboratory identifies any unacceptable QC analyses or any other unusual circumstances or observations such as environmental conditions and any non-standard conditions that may have affected the quality of a result. This is typically in the form of a footnote or a qualifier and/or a narrative explaining the discrepancy in the front of the report.
Numeric results with values outside of the calibration range, either high or low are qualified as estimated.

Where quality system requirements are not met, a statement of compliance/non-compliance with requirements and/or specifications is required, including identification of test results derived from any sample that did not meet TNI sample acceptance requirements such as improper container, holding time, or temperature.

Where applicable, a statement on the estimated uncertainty of measurements; information on uncertainty is needed when a client's instructions so require.

When, as requested by the client and agreed to by Eurofins TestAmerica, the report includes a statement of conformity to specification or standard (see Special Services, Section 7.4), the report shall clearly identify:

- to which results the statement applies,
- which specifications, standard or parts thereof are met or not, and
- the decision rule that was applied (unless the decision rule is inherent in the requested specification or standard, taking into account the level of risk (such as false accept and false reject and statistical assumptions) associated with the decision rule.

Opinions and Interpretations - The test report contains objective information, and generally does not contain subjective information such as opinions and interpretations. If such information is required by the client, the Laboratory Director will determine if a response can be prepared. If so, the Laboratory Director will designate the appropriate member of the management team to prepare a response. The response will be fully documented, and reviewed by the Laboratory Director, before release to the client. There may be additional fees charged to the client at this time, as this is a non-routine function of the laboratory.

Note: Review of data deliverable packages for submittal to regulatory authorities requires responses to non-conforming data concerning potential impact on data quality. This necessitates a limited scope of interpretation, and this work is performed by the QA Department. This is the only form of "interpretation" of data that is routinely performed by the laboratory.

When opinions or interpretations are included in the report, the laboratory provides an explanation as to the basis upon which the opinions and interpretations have been made. Opinions and interpretations are clearly noted as such and where applicable, a comment should be added suggesting that the client verify the opinion or interpretation with their regulator.

25.6 Environmental Testing Obtained From Subcontractors

If the laboratory is not able to provide the client the requested analysis, the samples would be subcontracted following the procedures outlined in NDSC Document No. CW-L-S-004, Subcontracting.

Data reported from analyses performed by a subcontractor laboratory are clearly identified as such on the analytical report provided to the client. Results from a subcontract laboratory outside of Eurofins are reported to the client on the subcontract laboratory's original report stationary and the report includes any accompanying documentation.

25.7 Client Confidentiality

The laboratory will ensure the highest standards of quality and integrity of the data and services provided to our clients

The laboratory is responsible for maintaining in confidence all client information obtained or created. In situations involving the transmission of environmental test results by telephone, facsimile or other electronic means, client confidentiality must be maintained.

The laboratory will not intentionally divulge to any person (other than the client or any other person designated by the client in writing) any information regarding the services provided by the laboratory or any information disclosed to the laboratory by the client. Furthermore, information known to be potentially endangering to national security or an entity's proprietary rights will not be released.

Information about the client obtained from sources other than the client (e.g., complainant, regulators) shall be confidential between client and the laboratory. The source of this information shall be confidential to the laboratory and shall not be shared with the client, unless agreed by the source.

Note: This shall not apply to the extent that the information is required to be disclosed by the laboratory under the compulsion of legal process. The laboratory will, to the extent feasible, provide reasonable notice to the client before disclosing the information.

Note: Authorized representatives of an accrediting authority are permitted to make copies of any analyses or records relevant to the accreditation process, and copies may be removed from the laboratory for purposes of assessment.

25.7.1 Report deliverable formats are discussed with each new client. If a client requests that reports be e-mailed, the reports are to meet all requirements of this document.

25.8 Format of Reports

The format of reports is designed to accommodate each type of environmental test carried out and to minimize the possibility of misunderstanding or misuse.

25.9 Amendments to Test Reports

Corrections, additions, or deletions to reports are only made when justification arises through supplemental documentation. Justification is documented using the laboratory's corrective action system (refer to Section 12).

The revised report is retained in the LIMS, as is the original report. The revised report is stored in the project files under the sample number followed by "Rev#" where # is the number of the report revision.

When the report is re-issued, the revision number is placed on the cover/signature page of the report or at the top of the narrative page. A brief explanation of reason for the re-issue and a reference back to the last final report generated may be included.

25.10 Policies on Client Requests for Amendments

25.10.1 Policy on Data Omissions or Reporting Limit Increases

Fundamentally, our policy is simply to not omit previously reported results (including data qualifiers) or to not raise reporting limits and report sample results as ND. This policy has few exceptions. Exceptions are:

- Laboratory error.
- Sample identification is indeterminate (confusion between COC and sample labels).
- An incorrect analysis (not analyte) was requested (e.g., COC lists EPA Method 8315 but client wanted EPA Method 8310). A written request for the change is required.
- Incorrect limits reported based on regulatory requirements.
- The requested change has absolutely *no possible* impact on the interpretation of the analytical results and there is *no possibility* of the change being interpreted as misrepresentation by anyone inside or outside of our company.

25.10.2 Multiple Reports

The laboratory does not issue multiple reports for the same work order where there is different information on each report (this does not refer to copies of the same report) unless required to meet regulatory needs and approved by QA.

Appendix 1. List of Governing Documents Applicable to the QA Manual

NDSC Doc. No.	Title
CA-C-S-001	Work Sharing Process
CA-I-P-002	Electronic Reporting and Signature Policy
CA-L-P-002	Contract Compliance Policy
CA-Q-M-002	Quality Management Plan
CA-Q-QM-001	Guidance on Tentatively Identified Compounds (TICs) – GC/MS Analysis
CA-Q-S-001	Acid and Solvent-Lot Testing and Approval Program
CA-Q-S-002	Manual Integrations
CA-Q-S-006	Detection and Quantitation Limits
CA-Q-S-009	Root Cause Analysis
CA-T-P-001	Qualified Products List
CW-E-M-001	Environmental Health & Safety Manual
CW-F-P-002	Company-Wide Authorization Matrix
CW-F-P-004	Procurement and Contracts Policy
CW-F-S-007	Fixed Asset Acquisition, Retention and Safeguarding
CW-I-M-001	IT Change Control Procedure Manual
CW-L-P-001	Records Retention Policy
CW-L-P-004	Ethics Policy
CW-L-S-002	Internal Investigation
CW-L-S-004	Subcontracting
CW-Q-S-001	Document Control and Archiving
CW-Q-S-002	Writing a Standard Operating Procedure (SOPs)
CW-Q-S-003	Internal Auditing
CW-Q-S-004	Management Systems Review
CW-Q-S-005	Data Recall Process
CW-Q-S-001	Document Control and Archiving

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Eurofins TestAmerica Savannah - Standard Operating Procedures

Department	Document Number	Revision	Active Date	SOP Title
AN	SA-AN-041	16	09/28/20	Reagent and Standard Materials Procedures
AN	SA-AN-100	13A	09/15/20	Support Equipment Verification and Use
CU	SA-CU-001	17	10/07/20	Sample Receipt Procedures
EX	SA-EX-015	15	07/24/20	Toxicity Compound Leaching Procedure (TCLP) and Synthetic Precipitation Leaching Procedure (SPLP)
EX	SA-EX-030	17	11/24/20	Liquid Extraction Procedures: Continuous Liquid-Liquid
EX	SA-EX-040	16	06/08/20	Soil Extraction Procedures (Microwave and Sonication)
EX	SA-EX-042	11	06/29/20	Waste Dilution Extraction
GE	SA-GE-001	8	11/10/20	Measurement of Analytes Using Konelab Autoanalyzer
GE	SA-GE-010	12	04/17/20	Bomb Combustate Preparation
GE	SA-GE-040	18	11/10/20	Cyanide: Total, Amenable, and Weak Acid Dissociable
GE	SA-GE-085	15	08/25/20	Sulfide: Titrimetric Preparation and Analysis (Total and Acid Soluble)
LC/IC	SA-GE-113	9	05/14/20	Disinfection Byproduct Anions by IC
LC/IC	SA-GE-115	21	05/14/20	Anions by Ion Chromatography
GE	SA-GE-132	13A	08/12/20	Sulfite
GE	SA-GE-133	8C	12/08/20	Total Residual Chlorine by Iodometric Titration
GE	SA-GE-140	11B	09/07/20	Ignitability
EX	SA-GE-157	16A	01/13/21	Oil & Grease and Total Petroleum Hydrocarbons
GE	SA-GE-165	15C	08/11/20	Total Recoverable Phenolics
GE	SA-GE-187	10A	10/26/20	Organic Halides: Adsorbable (AOX) & Total (TOX)
GE	SA-GE-189	11A	10/20/20	UV - Absorbing Organic Constituents (UV-254 & SUVA)
GE	SA-GE-190	12B	11/25/20	Solid / Residue Determinations
GE	SA-GE-191	9B	08/05/20	pH Determination (Soil and Non-Aqueous Wastes)
GE	SA-GE-193	10	08/11/20	Measurement of Analytes using PC Titrate Analyzer
GE	SA-GE-195	12	08/11/20	Chemical Oxygen Demand (COD): Colorimetric Method
GE	SA-GE-196	5B	10/14/20	Density and Specific Gravity
GE	SA-GE-197	9	03/18/20	Color

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Eurofins TestAmerica Savannah - Standard Operating Procedures

Department	Document Number	Revision	Active Date	SOP Title
GE	SA-GE-198	7B	10/14/20	Free Liquids by Paint Filter Liquids Test
GE	SA-GE-201	10	04/06/20	Total Hardness as CaCO3 by Titrimetric EDTA
GE	SA-GE-202	14	08/11/20	Oxygen Demand: Biochemical Oxygen Demand (BOD), Carbonaceous Biochemical Oxygen Demand (CBOD), and Nitrogenous Biochemical Oxygen Demand (NBOD)
LC/IC	SA-GE-204	10	08/11/20	Carbon Content in Water: TC, TIC, and TOC
GE	SA-GE-206	10	07/24/20	Turbidity
GE	SA-GE-208	8A	11/20/20	Nitrate and Nitrate Plus Nitrite Lachat Procedure
GE	SA-GE-210	7	06/08/20	Total Kjedahl Nitrogen and Total Phosphorus via Lachat Autoanalyzer
GE	SA-GE-211	1	04/23/20	Adsorbable Total (TOX)
LC/IC	SA-LC-070	12	09/07/20	Carbamate Pesticides by HPLC
LC/IC	SA-LC-071	9B	05/22/20	Glyphosate by HPLC
LC/IC	SA-LC-073	1	03/26/20	Acrylamide via HPLC
LC/IC	SA-LC-074	1	03/26/20	Acrylic Acid via HPLC
LC/IC	SA-LC-075	1	08/13/20	Carbonyl Compounds by HPLC
ME	SA-ME-028	15A	12/08/20	Mercury Preparation and Analysis
ME	SA-ME-050	22	08/11/20	Digestion Procedures for Liquids for ICP and ICP/MS
ME	SA-ME-051	15	05/16/20	Digestion Procedures for Solids for ICP & ICP-MS
ME	SA-ME-070	19D	12/09/20	Elements by ICP
ME	SA-ME-074	13A	12/09/20	Elements by ICP-MS
PM	SA-PM-001	12A	01/31/21	Project Management
PM	SA-PM-002	0B	08/05/20	
QA	SA-QA-001	9B	08/05/20	Document Control Program
QA	SA-QA-002	9C	08/05/20	Data Generation and Review
QA	SA-QA-005	8B	05/02/20	Preventive and Corrective Action
QA	SA-QA-006	10B	10/07/20	Training Procedures
QA	SA-QA-007	9B	02/22/20	Determination and Verification of Detection and Reporting Limits (RLs, MDLs, and IDLs)
QA	SA-QA-008	11B	09/15/20	Evaluation of Chromatographic Data
QA	SA-QA-010	6B	05/02/20	Validation of New Analytical Capabilities and Instrumentation
QA	SA-QA-015	6F	10/14/20	Homogenization, Compositing, and Segregation of Samples
QA	SA-QA-016	9C	10/20/20	Evaluation of Calibration Curves

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Department	Document Number	Revision	Active Date	SOP Title
QA	SA-QA-017	7C	9/15/20	Analytical Batching and Evaluation of QC Data
SG	SA-SG-045	23	9/28/20	Organochlorine Pesticides and Polychlorinated Biphenyls (PCBs) by GC/ECD
SG	SA-SG-060	17A	2/2/21	Microextractables by GC/ECD
SG	SA-SG-062	12A	2/2/21	Haloacetic Acids by Gas Chromatography
SG	SA-SG-065	18	4/17/20	Chlorinated Herbicides by GC/ECD: Preparation and Analysis
SG	SA-SG-070	16	9/17/20	Diesel Range Organics (DRO), Oil Range Organics (ORO), and Petroleum Product Identification by GC/FID
SM	SA-SM-002	15	9/17/20	Semivolatile Organic Compounds in Drinking Water by GC/MS
SM	SA-SM-033	10	9/28/20	Semivolatile Compounds by GC/MS
VO	SA-VO-001	9A	2/2/21	Preparation, Screening, and Storage of Volatile Samples
VO	SA-VO-002	9C	2/10/21	Volatile Compounds in Drinking Water by GC/MS
VO	SA-VO-003	7	3/6/20	Acetates in the Pharmaceutical Industry by GC/MS
VO	SA-VO-004	7	4/27/20	Volatile Compounds by GC/MS
VO	SA-VO-005	8	10/13/20	Gasoline Range Organics (GRO) by GC/FID
VO	SA-VO-006	7A	2/2/21	Solvents by Direct Aqueous Injection (DAI) Using GC/FID
VO	SA-VO-007	8	2/14/20	Dissolved Gases in Water

Appendix 2. List of Laboratory Certifications, Accreditations, Validations

Eurofins TestAmerica Savannah performs work from clients located throughout the United States, as well as in some foreign countries. Most states and/or federal agencies maintain a laboratory accreditation program that requires a laboratory to obtain certification with their agency. To obtain certification, a laboratory must maintain an effective quality system that meets the requirements of the agency. Common components of the quality system requirements include maintaining up-to-date standard operating procedures (SOPs) and a Quality Assurance Manual (QAM); participating in a Proficiency Testing (PT) program; performing method detection limit (MDL) studies, initial and continuing demonstrations of capability studies (IDOCs/CDOCs), and internal assessments; and completing an annual renewal application. In addition to the requirements needed for certification, many agencies have specific analytical and/or reporting requirements that laboratories must follow.

Many agencies offer certification via reciprocity. Reciprocity is the acknowledgement of another state and/or agency's certification program. The most common types of reciprocity are homestate reciprocity and TNI (NELAC) reciprocity.

Lab Management, Project Management, Sales & Marketing, and the QA Manager may initiate requests for certification or accreditation. The QA staff completes the administrative tasks associated with the application and maintains the related documents in accordance with SOP SA-QA-001: *Document Control Program.*

Laboratory management has the responsibility and authority to ensure that laboratory operations are in compliance with program and regulatory requirements of the jurisdiction for which laboratory certification/accreditation is sought and maintained.

To perform compliance work in a particular state, the laboratory must maintain certification for the reported analytes. Most accrediting authorities will certify laboratories on a matrix/method/analyte level. For example:

Soil / EPA 8260B / benzene
Water / EPA 624 / toluene

Generally, laboratories must apply and submit supporting documentation (SOPs, MDLs, IDOCs, PTs, etc.) for each individual matrix/method/analyte combination.

1.0 Obtaining Certification

1.1 Certification Application Process

Lab Management, Project Management, Sales and Marketing, and the QA Manager may initiate requests for certification or accreditation. The application is obtained, reviewed, and completed by the QA Manager or designee. Sections of the application may be distributed as appropriate to various staff members to assist in completion.

The certifying agency's regulations should be carefully reviewed at the time of application to ensure any non-routine requirements are communicated to the laboratory.

The QA Manager consults with Lab Management, Project Management, and the Sales and Marketing Staff to determine if additional methods should be added to the current laboratory certification in order to support existing and future work as the laboratory's capabilities change.

1.2 Reciprocity

Reciprocity is a means of acknowledging another agency's certification via mutual agreements between certifying agencies. Many certifying agencies offer some type of reciprocal certification. The most common types of reciprocity are based on either TNI (NELAC) certification or homestate certification.

Homestate reciprocity refers to another state's certifying agency allowing a laboratory to perform work in that state, provided that the laboratory maintains accreditation within the state in which it resides.

TNI refers to The NELAP Institute which governs the NELAP Standard document outlining Quality System and laboratory functions and requirements. NELAP refers to the National Environmental Laboratory Accreditation Program. Many states will acknowledge a laboratory's TNI accreditation from another state.

Note: Reciprocal agreements between states do not afford a "blanket" certification. To obtain reciprocal certification, a laboratory must still apply for accreditation, submit all required application materials, and receive notification of certification – usually in the form of a certificate - from the reciprocal agency.

1.3 Records Maintenance

A copy of the original application, certificate, and related materials are maintained in accordance with SA-QA-001: *Document Control Program.* Copies of current certifications are kept in the Certifications folder on the Public_QA drive, which is accessible to all laboratory staff. In addition, copies of current laboratory certifications from Savannah and other Eurofins TestAmerica facilities are maintained in the TALS Certification Module. These documents may be required to support subcontracting and marketing activities.

1.4 Maintaining Certification

Most states require continued evidence of an effective Quality System in order for a laboratory to maintain certification. In addition to annual renewal applications, laboratories are often required to complete bi-annual PT studies with acceptable results obtained for each certifiable matrix/method/analyte combination. Annual MDL and continued demonstrations of analyst capability are also routinely required, in addition to on-site assessments.

1.5 Certification Tools and Records

There are several tools in place to aid laboratory staff in determining what certifications the laboratory maintains and understanding any state-specific analytical and/or reporting requirements.

1.5.1 TALS Certification Module

The TALS Certification Module is a tool that can aid in determining which certifications the laboratory maintains. This tool is useful in the pre-project planning process.

1.5.2 State and Project Requirement Summaries

Some states and/or projects have specific analytical and/or reporting requirements. A summary of these requirements is kept in the State and Project Requirement Summary on the Public-QA Drive. These requirements must be reviewed by project management and laboratory staff prior to initiating work. The Project Manager must clearly note in the LIMS Worksheet Notes and/or Project Plan if the Project Requirement Summary (PRS) is to be followed.

1.6 Information Resources

1.6.1 Agency Information

The QA staff maintains a controlled access database that lists current contact information for the agency that oversees laboratory certification as well as the regulatory programs that are offered for certification by the agency. This information may be provided as a resource to Lab Management, Project Management, Corporate QA, and the Sales and Marketing staff.

1.6.2 Certification Matrix

The QA Department ensures that the certification matrix maintained in TALS is current.

1.6.3 Certification / Accreditation Maintenance Requirements

Laboratory Management is responsible for ensuring that laboratory operations are in compliance with the regulatory and certification program requirements for the jurisdiction in which certification is maintained.

The QA Department is responsible for maintaining up to date applications and program information including program specific regulations and requirements.

Project Management is responsible for verifying certification of analytes and methods requested by the client prior to accepting work and should be familiar with the state-specific requirements of that state.

1.7 Certifications Listing

At the time of this QA Manual revision, the laboratory has accreditation/certification/ licensing with the following organizations:



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Certification List

Authority	Identification
ANAB	L2463
ANAB	L2463.01
Florida	E87052
Illinois	200022
Louisiana	02011
New Jersey	GA769
New York	10842
Pennsylvania	68-00474
Texas	T1047004185-19-14
Virginia	10509
Alabama	41450
Alaska	GA00006
Alaska (UST)	17-016
Arkansas DEQ	19-015-0
California	2939
Connecticut	PH-0161
Georgia	E87052
Georgia (DW)	803
Guam	19-007R
Hawaii	<cert No.>
Indiana	C-GA-02
Iowa	353
Kentucky (UST)	NA
Louisiana (DW)	LA009
Maine	GA00006
Maryland	250
Massachusetts	M-GA006
Michigan	9925
Mississippi	<cert No.>
Nebraska	NE-OS-7-04
New Mexico	GA00006
North Carolina (DW)	13701
North Carolina (WW/SW)	269
Puerto Rico	GA00006
South Carolina	98001
Tennessee	02961

* Certification Valid - Laboratory is Pending Renewal with the Program Authority.

For more information, or to contact a local Eurofins representative nearest you, please visit our website at www.testamericainc.com

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Certification List

Authority	Identification
Washington	C805
West Virginia DEP	094
Wisconsin	999819810
Wyoming	8TMS-L
Texas	T104704185
US Fish & Wildlife	LE058448-0
USDA	P330-18-00313

The certificates and accredited parameter lists are available for each State/Program at www.testamericainc.com under Analytical Services Search – Certifications. Copies of these documents can also be found on the laboratory's public server, in TALS, and in the QA offices.

Appendix 3. References used to prepare the QA Manual

The QAM has been prepared to be consistent with the requirements of the following documents:

- ANSI/ASQC, E4-1994, "Specifications and Guidelines for Quality Management Systems for Environmental Data Collection and Environmental Technology Programs" (American National Standard, January 5, 1995, or most recent version)
- "EPA Requirements for Quality Management Programs" (QA/R-2) (EPA/240/B-01/002, May 31, 2006).
- EPA 600/4-88/039, *Methods for the Determination of Organic Compounds in Drinking Water,* EPA, Revised July 1991.
- EPA 600/R-95/131, *Methods for the Determination of Organic Compounds in Drinking Water,* Supplement III, EPA, August 1995.
- EPA 600/4-79-019, *Handbook for Analytical Quality Control in Water and Wastewater Laboratories,* EPA, March 1979.
- *Test Methods for Evaluating Solid Waste Physical/Chemical Methods (SW846), Third Edition, September 1986, Final Update I, July 1992, Final Update IIA, August 1993, Final Update II, September 1994; Final Update IIB, January 1995; Final Update III, December 1996; Final Update IV, January 2008; Final Update V, August 2015.*
- *U.S. Department of Defense (DoD)/Department of Energy (DOE) Consolidated Quality Systems Manual (QSM) for Environmental Laboratories, Version 5.1.1, dated 2017*
- Federal Register, 40 CFR Parts 136, 141, 172, 173, 178, 179 and 261.
- *Manual for the Certification of Laboratories Analyzing Drinking Water (EPA 815-R-05-004, January 2005) (DW labs only)*

Appendix 4. QA Manual Crosswalk with TNI and ISO/IEC 17025 Standards

Sec. No.	Title	2009 and 2016 TNI Standard Reference	ISO/IEC 17025:2005(E) Reference	ISO/IEC 17025:2017(E) Reference
-	QUALITY ASSURANCE MANUAL	V1M2 Sec. 4.2.8.3		
1.0	Title Page			
2.0	Table of Contents	V1M2 Secs. 4.2.8.3-4.2.8.4		8.1.2, 8.2.1
3.0	Introduction, Scope and Applicability	V1M2 Sec. 4.2.8.4		
3.1	Introduction and Compliance References	V1M2 Secs. 1.1; 1.2; 2.0; 3.2; 4.1.2; 4.2.4	4.1.2; 4.2.4	5.3; 5.4; 8.2.4; 8.3.1
3.2	Terms and Definitions	V1M2 Secs. 3.0; 4.2.4	4.2.4	
3.3	Scope /Field of Testing	V1M2 Secs. 1.2; 4.2.4	4.1.2; 4.2.4	5.3; 5.4; 8.2.1; 8.2.4
3.4	Management of The Manual	V1M2 Secs. 4.2.1; 4.2.7; 4.3.3.2; 4.3.3.3	4.2.1; 4.2.7; 4.3.3.2; 4.3.3.3	5.3
4.0	Management and Responsibilities	V1M2 Sec. 4		8.2.4; 8.2.5
4.1	Overview	V1M2 Secs. 4.1.1, 4.1.3; 4.1.5	4.1.1; 4.1.3; 4.1.5; 4.2.6	5.1; 5.2; 5.5; 5.6 ; 6.2.1; 6.2.4
4.2	Roles and Responsibilities	V1M2 Secs. 4.1.4; 4.1.5; 4.1.6; 4.2.1; 4.2.6; 5.2.4	4.1.3; 4.1.5; 4.1.6; 4.2.1; 4.2.6; 5.2.4	4.1.1 to 4.1.3; 4.1.5; 5.5; 5.6; 6.2.1; 6.2.4; 6.2.6 8.2.2;
4.3	Business Continuity and Contingency Plan *(Prev. Deputies)*	V1M2 Secs. 4.1.5; 4.1.7.2; 4.2.7	4.1.5; 4.2.7	
5.0	PERSONNEL	V1M2 Secs. 5.2; 5.2.1	5.2.1	6.1; 6.2.3
5.1	Overview	V1M2 Secs. 5.2.2; 5.2.3; 5.2.5	5.2.2; 5.2.3; 5.2.5	6.2.2
5.2	Education and Experience Requirements for Technical Staff	V1M2 Secs. 5.2.1; 5.2.3; 5.2.4	5.2.1; 5.2.3; 5.2.4	6.2.2 to 6.2.4
5.3	Training	V1M2 Sec. 5.2.5	5.2.5	4.2.1; 6.2.2; 6.2.4; 6.2.5
5.4	Data Integrity and Ethics Training Program	V1M2 Sec. 4.2.8.1; 5.2.7		4.1.1
6.0	ACCOMMODATIONS AND ENVIRONMENTAL CONDITIONS	V1M2 Sec. 5.3		6.1; 6.3.1
6.1	Overview	V1M2 Secs. 5.3.1; 5.3.3; 5.3.4; 5.3.5	5.3.1; 5.3.3; 5.3.4; 5.3.5	6.3.1
6.2	Environment	V1M2 Secs. 5.3.1; 5.3.2; 5.3.3; 5.3.4; 5.3.5	5.3.1; 5.3.2; 5.3.3; 5.3.4; 5.3.5	6.3.1 to 6.3.5
6.3	Work Area	V1M2 Secs. 5.3.3; 5.3.4; 5.3.5	5.3.3; 5.3.4; 5.3.5	6.3.1
6.4	Responding to Emergencies			
6.5	Building Security	V1M2 Sec. 5.3.4	5.3.4	6.3.4
7.0	QUALITY SYSTEM			6.1; 8.2.4
7.1	Quality Policy	V1M2 Secs. 4.1.5; 4.2.2; 4.2.3; 4.2.8.3	4.1.5; 4.2.2; 4.2.3	8.2.3; 8.6.1
7.2	Ethics and Data Integrity	V1M2 Secs. 4.1.5; 4.16; 4.2.2; 4.2.8.1; 5.2.7	4.1.5; 4.2.2	4.1.1 to 4.1.3; 4.2.1; 6.2.1; 8.2.2; 8.2.3

Sec. No.	Title	2009 and 2016 TNI Standard Reference	ISO/IEC 17025:2005(E) Reference	ISO/IEC 17025:2017(E) Reference
7.3	Quality System Documentation	V1M2 Secs. 4.1.5; 4.2.2; 4.2.5	4.2.2; 4.2.5	8.2.4
7.4	QA/QC Objectives for the Measurement of Data	V1M2 Sec. 4.2.2	4.1.5; 4.2.2	6.2.4
7.5	Criteria for Quality Indicators			
7.6	Statistical Quality Control			
7.7	Quality System Metrics			
8.0	Document Control	V1M2 Secs. 4.2.7; 4.3.1; 4.3.2.2 ; 4.3.3.3; 4.3.3.4	4.2.7; 4.3.1; 4.3.2.2; 4.3.3.3; 4.3.3.4	8.2.4; 8.3.1
8.1	Overview			8.2.5; 8.3.1; 8.3.2
8.2	Document Approval and Issue	V1M2 Secs. 4.3.2; 4.3.2.1-4.3.2.3; 4.3.3.1	4.3.2.1; 4.3.2.2; 4.3.2.3; 4.3.3.1	8.2.5; 8.3.2
8.3	Procedures for Document Control Policy	V1M2 Secs. 4.3.2.1–4.3.2.2; 4.3.3.1	4.3.2.1; 4.3.2.2; 4.3.3.1	8.2.5; 8.3.2
8.4	Obsolete Documents	V1M2 Secs. 4.3.2.1–4.3.2.2	4.3.2.1; 4.3.2.2	8.2.5; 8.3.2
9.0	SERVICE TO THE CLIENT	V1M2 Secs. 4.4.1 - 4.4.4	4.4.1; 4.4.2; 4.4.3; 4.4.4	7.1.1; 7.1.1.4; 7.1.1.5; 7.1.1.8; 7.1.2.1
9.1	Overview	V1M2 Secs. 4.4.5; 4.5.5; 5.7.1	4.4.5; 5.7.1	
9.2	Review Sequence and Key Personnel	V1M2 Sec. 4.4.5	4.4.5	7.1.1.6
9.3	Balancing Laboratory Work Load and Capacity			
9.4	Documentation	V1M2 Sec. 5.7.1	5.7.1	
9.5	Special Services	V1M2 Secs. 4.7.1-4.7.2	4.7.1; 4.7.2	7.1.1.3; 7.1.1.7
9.6	Client Communication	V1M2 Secs. 4.7.1-4.7.2	4.7.1; 4.7.2	7.1.1.7
9.7	Reporting	V1M2 Secs. 4.7.1-4.7.2	4.7.1; 4.7.2	7.1.1.7
9.8	Client Feedback and Surveys	V1M2 Secs. 4.7.1-4.7.2	4.7.1; 4.7.2	7.1.1.7; 8.6.2
10.0	SUBCONTRACTING OF TESTS	V1M2 Secs. 4.4.3; 4.5.4	4.4.3; 4.5.4	
10.1	Overview	V1M2 Secs. 4.5.1 - 4.5.3; 4.5.5; 5.3.1	4.5.1; 4.5.2; 4.5.3; 5.3.1	6.6.1; 7.1.2.1; 7.1.2.2
10.2	Qualifying and Monitoring Subcontractors	V1M2 Secs. 4.5.1; 4.5.2; 4.5.3; 4.5.5	4.5.1; 4.5.2; 4.5.3	6.6.1; 7.1.2.1; 7.1.2.2
10.3	Oversight and Reporting	V1M2 Sec. 4.5.5		
10.4	Contingency Planning			
10.5	Use of NELAP and A2LA Logo			
11.0	PURCHASING SERVICES AND SUPPLIES	V1M2 Sec. 4.6.1	4.6.1	
11.1	Overview	V1M2 Secs. 4.6.2; 4.6.3; 4.6.4	4.6.2; 4.6.3; 4.6.4	6.6.1; 6.6.2
11.2	Glassware	V1M2 Sec. 5.5.13.1		
11.3	Reagents, Standards & Supplies	V1M2 Secs. 4.6.2; 4.6.3; 4.6.4 2016 V1M4 1.7.2.5	4.6.2; 4.6.3; 4.6.4	6.6.1 to 6.6.3
11.4	Purchase of Equipment / Instruments / Software			
11.5	Services			
11.6	Suppliers			

Sec. No.	Title	2009 and 2016 TNI Standard Reference	ISO/IEC 17025:2005(E) Reference	ISO/IEC 17025:2017(E) Reference
12.0	COMPLAINTS	V1M2 Sec. 4.8	4.8	8.6.1; 8.6.2
12.1	Overview			7.9.1 to 7.9.3 8.6.1; 8.6.2;
12.2	External Complaints			7.9.2 to 7.9.7 8.6.1; 8.6.2;
12.3	Internal Complaints			8.6.1; 8.6.2
12.4	Management Review			8.6.1; 8.6.2
13.0	CONTROL OF NON-CONFORMING WORK	V1M2 Secs. 4.9.1; 5.10.5	4.9.1; 5.10.5	7.10.1
13.1	Overview	V1M2 Secs. 4.9.1; 4.11.3; 4.11.5	4.9.1; 4.11.3; 4.11.5	7.10.1
13.2	Responsibilities and Authorities	V1M2 Secs. 4.9.1; 4.11.3; 4.11.5; 5.2.7	4.9.1; 4.11.3; 4.11.5	7.10.1
13.3	Evaluation of Significance and Action Taken	V1M2 Secs. 4.9.1; 4.11.3; 4.11.5	4.9.1; 4.11.3; 4.11.5	4.1.5; 7.10.1; 7.10.2; 8.5.3
13.4	Prevention of Non-Conforming Work	V1M2 Secs. 4.9.4; 4.11.2	4.9.2; 4.11.2	7.10.2; 7.10.3; 8.5.3
13.5	Method Suspension / Restriction (Stop Work Procedures)	V1M2 Secs. 4.9.1; 4.9.2; 4.11.5	4.9.1; 4.9.2; 4.11.5	7.10.1; 7.10.2
14.0	CORRECTIVE ACTION	V1M2 Sec. 4.11		4.1.4; 4.1.5
14.1	Overview	V1M2 Secs. 4.9.2; 4.11.1; 4.11.2	4.9.2; 4.11.1; 4.11.2; 8.7.1; 8.7.3	7.10.2 8.7.1; 8.7.3;
14.2	General	V1M2 Sec. 4.11.2; 4.11.3	4.11.2; 4.11.3	7.7.2; 8.5.3; 8.7.1;
14.3	Closed Loop Correction Action Process	V1M2 Sec. 4.11.2; 4.11.3; 4.11.4; 4.11.6; 4.11.7; 4.12.2	4.11.2; 4.11.3; 4.11.4; 4.12.2	8.5.3; 8.6.1; 8.7.2
14.4	Technical Corrective Actions	V1M2 Sec. 4.11.6		8.7.1
14.5	Basic Corrections	V1M2 Secs. 4.11.1; 4.13.2.3	4.11.1; 4.13.2.3	7.5.2; 8.7.1
15.0	PREVENTIVE ACTION / IMPROVEMENT	V1M2 Secs. 4.10; 4.12.1; 4.12.2	4.10; 4.12.1; 4.12.2	4.1.4
15.1	Overview	V1M2 Secs. 4.15.1; 4.15.2	4.15.1; 4.15.2	8.6.2
15.2	Management of Change			
16.0	CONTROL OF RECORDS	V1M2 Secs. 4.2.7; 4.13.1.1; 4.13.3	4.2.7; 4.13.1.1	8.4.2
16.1	Overview	V1M2 Secs. 4.13.1.1 - 4.13.1.4; 4.13.2.1-4.13.2.3; 4.13.3	4.13.1.1 - 4.13.1.4; 4.13.2.1-4.13.2.3	8.4.1; 8.4.2
16.2	Programs with Longer Retention Requirements			
16.3	Technical and Analytical Records	V1M2 Sec. 4.13.2.2 - 4.13.2.3	4.13.2.2; 4.13.2.3	7.5.1; 8.4.2
16.4	Laboratory Support Activities			7.5.2; 8.4.2
16.5	Administrative Records			8.4.2
16.6	Records Management, Storage and Disposal	V1M2 Sec. 4.13.3		4.2.1; 8.4.2
17.0	AUDITS			

Sec. No.	Title	2009 and 2016 TNI Standard Reference	ISO/IEC 17025:2005(E) Reference	ISO/IEC 17025:2017(E) Reference
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17.2	External Audits	V1M2 Secs.4.14.2; 4.14.3	4.14.2; 4.14.3; 4.14.4	4.2.1; 8.6.1
17.3	Audit Findings	V1M2 Secs. 4.14.2; 4.14.3; 4.14.5		8.6.1
18.0	MANAGEMENT REVIEWS	V1M2 Sec. 4.1.6; 4.15; 4.15.1; 4.15.2	4.1.6; 4.15.1; 4.15.2	4.1.4; 8.5.1; 8.6.1; 8.9.1; 8.9.2
18.1	Quality Assurance Report			8.5.1
18.2	Annual Management Review	V1M2 Sec. 4.2.2; 4.15.3	4.2.2	4.1.1, 4.1.4; 4.2.1; 7.1.1.3; 8.2.2; 8.5.1 to 8.5.3; 8.6.1; 8.9.3
18.3	Potential Integrity Related Managerial Reviews			4.1.5; 8.5.1; 8.6.1
19.0	TEST METHODS AND METHOD VALIDATION	V1M2 Sec. 5.4.1	5.4.1	7.2.1.1; 8.2.5
19.1	Overview	V1M2 Sec. 5.4.1	5.4.1; 5.4.5.1	6.2.3; 7.2.1.1 to 7.2.1.3
19.2	Standard Operating Procedures (SOPs)	V1M2 Secs. 4.2.8.5; 4.3.3.1; 5.4.2	4.3.3.1; 5.4.2	7.2.1.4
19.3	Laboratory Methods Manual	V1M2 Sec. 4.2.8.5 (2019 5.4.4.2)		6.2.3
19.4	Selection of Methods	V1M2 Secs. 4.13.3; 5.4.1; 5.4.2; 5.4.3. (2019 5.4.4) V1M4 Secs. 1.4; 1.5.1; 1.6.1; 1.6.2; 1.6.2.1; 1.6.2.2	5.4.1; 5.4.2; 5.4.3; 5.4.4; 5.4.5.1; 5.4.5.2; 5.4.5.3	7.1.1.2; 7.2; 7.2.1.2; 7.2.1.3; 7.1.2.4 to 7.2.1.7; 7.2.2.1 to 7.2.1.7; 7.2.2.1
19.5	Laboratory Developed Methods and Non-Standard Methods	V1M2 Sec. 5.4.2. (2019 5.4.4.1, 5.4.4.2) V1M4 Sec. 1.5.1	5.4.2; 5.4.4; 5.4.5.2; 5.4.5.3	7.1.1.2; 7.2.1.4 to 7.2.1.7 7.2.2.1; 7.2.2.3; 8.2.5
19.6	Validation of Methods	V1M2 Sec. 5.4.2. (2019 5.4.5.4) V1M4 Secs. 1.5.1; 1.5.2; 1.5.2.1; 1.5.2.2; 1.5.3	5.4.2; 5.4.4; 5.4.5.2; 5.4.5.3	7.1.1.2; 7.1.2.4; 7.2.1.6; 7.2.2.1 to 7.2.2.4
19.7	Method Detection Limits (MDLs) / Limit of Detection (LOD)	V1M2 Sec. 5.9.3. V1M4 Secs. 1.5.2; 1.5.2.1; 1.5.2.2	5.4.5.3	7.2.2.3
19.8	Verification of Detection Limits	V1M2 Sec. 5.9.3		
19.9	Instrument Detection Limits (IDLs)	V1M2 Sec. 5.9.3. V1M4 Sec. 1.5.2.1		
19.10	Limit of Quantitation	V1M2 Sec. 5.9.3		
19.11	Retention Windows	V1M2 Sec. 5.9.3. V1M4 Sec. 1.5.4; (2009 1.7.3.6) (2016 1.7.2.6)		

Sec. No.	Title	2009 and 2016 TNI Standard Reference	ISO/IEC 17025:2005(E) Reference	ISO/IEC 17025:2017(E) Reference
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19.13	Estimation of Uncertainty of Measurement	V1M2 Sec. 5.1.1; 5.1.2; 5.4.6	5.1.1; 5.1.2; 5.4.6.1; 5.4.6.2; 5.4.6.3	7.6.1; 7.6.2; 7.6.3
19.14	Sample Reanalysis Guidelines	V1M2 Sec 5.9.1	5.9.1	
19.15	Control of Data	V1M2 Secs. 5.4.7.1; 5.4.7.2; 5.9.1	5.4.7.1; 5.4.7.2; 5.9.1	7.11.1 to 7.11.6
20.0	EQUIPMENT AND CALIBRATIONS	V1M2 Secs. 5.5.4; 5.5.5; 5.5.6	5.5.4; 5.5.5; 5.5.6; 5.6.1	6.1; 6.4.3; 6.4.6; 6.4.9
20.1	Overview	V1M2 Secs. 5.5.1; 5.5.2; 5.5.3; 5.5.5; 5.5.10	5.5.1; 5.5.2; 5.5.3; 5.5.5; 5.5.10; 5.6.1	6.4.1; 6.4.4 to 6.4.6; 6.4.9; 6.4.11
20.2	Preventive Maintenance	V1M2 Secs. 5.5.1; 5.5.3; 5.5.7; 5.5.9	5.5.1; 5.5.3; 5.5.7; 5.5.9; 5.6.1	6.4.1 to 6.4.3; 6.4.6; 6.4.10
20.3	Support Equipment	V1M2 Secs. 5.5.10; 5.5.11; 5.5.13.1	5.5.10; 5.5.11; 5.6.2.1.2; 5.6.2.2.1; 5.6.2.2.2	6.4.11; 6.4.12; 6.5.1; 6.5.2; 6.5.3
20.4	Instrument Calibrations	V1M2 Secs. 5.5.8; 5.5.10; 5.6.3.1. V1M4 Sec. 1.7.1.1; (2009 1.7.2) (2016 1.7.1.2)	5.5.8; 5.5.9; 5.5.10; 5.6.1; 5.6.2; 5.6.3.1	6.4.2; 6.4.3; 6.4.6 to 6.4.8; 6.4.11; 6.4.13; 6.4.14; 6.5.1; 6.5.2
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Appendix 5. Terms/Glossary and Acronyms (EL-V1M2 Sec. 3.1)

Terms/Glossary:

Acceptance Criteria: Specified limits placed on characteristics of an item, process, or service defined in requirement documents. (ASQC)

Accreditation: The process by which an agency or organization evaluates and recognizes a laboratory as meeting certain predetermined qualifications or standards, thereby accrediting the laboratory.

Accuracy: The degree of agreement between an observed value and an accepted reference value. Accuracy includes a combination of random error (precision) and systematic error (bias) components which are due to sampling and analytical operations; a data quality indicator. (QAMS)

Analyst: The designated individual who performs the "hands-on" analytical methods and associated techniques and who is the one responsible for applying required laboratory practices and other pertinent quality controls to meet the required level of quality.

Analytical Uncertainty: A subset of Measurement Uncertainty that includes all laboratory activities performed as part of the analysis. (TNI)

Anomaly: A condition or event, other than a deficiency, that may affect the quality of the data, whether in the laboratory's control or not.

Assessment: The evaluation process used to measure or establish the performance, effectiveness, and conformance of an organization and/or its systems to defined criteria (to the standards and requirements of laboratory accreditation). (TNI)

Audit: A systematic and independent examination of facilities, equipment, personnel, training, procedures, record-keeping, data validation, data management, and reporting aspects of a system to determine whether QA/QC and technical activities are being conducted as planned and whether these activities will effectively achieve quality objectives. (TNI)

Batch: Environmental samples that are prepared and/or analyzed together with the same process and personnel, using the same lot(s) of reagents. A **preparation batch** is composed of one (1) to twenty (20) environmental samples of the same quality systems matrix, meeting the above mentioned criteria and with a maximum time between the start of processing of the first and last sample in the batch to be twenty-four (24) hours. An **analytical batch** is composed of prepared environmental samples (extracts, digestates or concentrates) which are analyzed together as a group. An analytical batch can include prepared samples originating from various quality system matrices and can exceed twenty (20) samples. (TNI)

Bias: The systematic or persistent distortion of a measurement process, which causes errors in one direction (i.e., the expected sample measurement is different from the sample's true value). (TNI)

Blank: A sample that has not been exposed to the analyzed sample stream in order to monitor contamination during sampling, transport, storage or analysis. The blank is subjected to the usual analytical and measurement process to establish a zero baseline or background value and is sometimes used to adjust or correct routine analytical results. (ASQC)

Calibration: A set of operations that establish, under specified conditions, the relationship between values of quantities indicated by a measuring instrument or measuring system, or values represented by a material measure or a reference material, and the corresponding values realized by standards. (TNI)

1) In calibration of support equipment the values realized by standards are established through the use of reference standards that are traceable to the International System of Units (SI).
2) In calibration according to methods, the values realized by standards are typically established through the use of Reference Materials that are either purchased by the laboratory with a certificate of analysis or purity, or prepared by the laboratory using support equipment that has been calibrated or verified to meet specifications.

Calibration Curve: The mathematical relationship between the known values, such as concentrations, of a series of calibration standards and their instrument response. (TNI)

Calibration Standard: A substance or reference material used to calibrate an instrument (QAMS)

Certified Reference Material (CRM): A reference material accompanied by a certificate, having a value, measurement uncertainty, and stated metrological traceability chain to a national metrology institute. (TNI)

Chain of Custody (COC) Form: Record that documents the possession of the samples from the time of collection to receipt in the laboratory. This record generally includes: the number and types of containers; the mode of collection; the collector; time of collection; preservation; and requested analyses. (TNI)

Compromised Samples: Those samples which are improperly sampled, insufficiently documented (chain of custody and other sample records and/or labels), improperly preserved, collected in improper containers, or exceeding holding times when delivered to a laboratory. Under normal conditions, compromised samples are not analyzed. If emergency situation require analysis, the results must be appropriately qualified.

Confidential Business Information (CBI): Information that an organization designates as having the potential of providing a competitor with inappropriate insight into its management, operation or products. TNI and its representatives agree to safeguard identified CBI and to maintain all information identified as such in full confidentiality.

Confirmation: Verification of the identity of a component through the use of an approach with a different scientific principle from the original method. These may include, but are not limited to Second Column Confirmation; Alternate wavelength; Derivatization; Mass spectral interpretation; Alternative detectors or Additional Cleanup procedures. (TNI)

Conformance: An affirmative indication or judgment that a product or service has met the requirements of the relevant specifications, contract, or regulation; also the state of meeting the requirements. (ANSI/ASQC E4-1994)

Correction: Actions necessary to correct or repair analysis specific non-conformances. The acceptance criteria for method specific QC and protocols as well as the associated corrective actions. The analyst will most frequently be the one to identify the need for this action as a result of calibration checks and QC sample analysis**.** No significant action is taken to change behavior, process or procedure.

Corrective Action: The action taken to eliminate the causes of an existing nonconformity, defect or other undesirable situation in order to prevent recurrence. (ISO 8402)

Data Integrity: The condition that exists when data are sound, correct, and complete, and accurately reflect activities and requirements. (TNI)

Data Audit: A qualitative and quantitative evaluation of the documentation and procedures associated with environmental measurements to verify that the resulting data re of acceptable quality (i.e., that they meet specified acceptance criteria).

Data Reduction: The process of transforming the number of data items by arithmetic or statistical calculations, standard curves, and concentration factors, and collation into a more useable form. (TNI)

Deficiency: An unauthorized deviation from acceptable procedures or practices, or a defect in an item (ASQC), whether in the laboratory's control or not.

Demonstration of Capability: A procedure to establish the ability of the analyst to generate analytical results of acceptable accuracy and precision. (TNI)

Document Control: The act of ensuring that documents (and revisions thereto) are proposed, reviewed for accuracy, approved for release by authorized personnel, distributed properly, and controlled to ensure use of the correct version at the location where the prescribed activity if performed. (ASQC)

Duplicate Analyses: The analyses or measurements of the variable of interest performed identically on two subsamples of the same sample. The results from duplicate analyses are used to evaluate analytical or measurement precision but not the precision of sampling, preservation or storage internal to the laboratory. (EPA-QAD)

Equipment Blank: Sample of analyte-free media which has been used to rinse common sampling equipment to check effectiveness of decontamination procedures.

External Standard Calibration: Calibrations for methods that do not utilize internal standards to compensate for changes in instrument conditions.

Field Blank: Blank prepared in the field by filing a clean container with pure de-ionized water and appropriate preservative, if any, for the specific sampling activity being undertaken (EPA OSWER)

Field of Accreditation: Those matrix, technology/method, and analyte combinations for which the accreditation body offers accreditation.

Holding Times: The maximum time that samples may be held prior to analyses and still be considered valid or not compromised. (40 CFR Part 136)

Internal Standard: A known amount of standard added to a test portion of a sample as a reference for evaluating and controlling the precision and bias of the applied analytical test method. (TNI)

Internal Standard Calibration: Calibrations for methods that utilize internal standards to compensate for changes in instrument conditions.

Instrument Blank: A clean sample (e.g., distilled water) processed through the instrumental steps of the measurement process; used to determine instrument contamination. (EPA-QAD)

Instrument Detection Limit (IDL): The minimum amount of a substance that can be measured with a specified degree of confidence that the amount is greater than zero using a specific instrument. The IDL is associated with the instrumental portion of a specific method only, and sample preparation steps are not considered in its derivation. The IDL is a statistical estimation at a specified confidence interval of the concentration at which the relative uncertainty is ± 100%. The IDL represents a range where qualitative detection occurs on a specific instrument. Quantitative results are not produced in this range.

Laboratory Control Sample (however named, such as laboratory fortified blank, spiked blank, or QC check sample): A sample matrix, free from the analytes of interest, spiked with verified known amounts of analytes or a material containing known and verified amounts of analytes, taken through all preparation and analysis steps of the procedure unless otherwise noted in a reference method. It is generally used to establish intra-laboratory or analyst specific precision and bias or to assess the performance of all or a portion of the measurement system. An LCS shall be prepared at a minimum of 1 per batch of 20 or less samples per matrix type per sample extraction or preparation method except for analytes for which spiking solutions are not available such as total suspended solids, total dissolved solids, total volatile solids, total solids, pH, color, odor, temperature, dissolved oxygen or turbidity. The results of these samples shall be used to determine batch acceptance.

Least Squares Regression (1st Order Curve): The least squares regression is a mathematical calculation of a straight line over two axes. The y axis represents the instrument response (or Response ratio) of a standard or sample and the x axis represents the concentration. The regression calculation will generate a correlation coefficient (r) that is a measure of the "goodness of fit" of the regression line to the data. A value of 1.00 indicates a

perfect fit. In order to be used for quantitative purposes, r must be greater than or equal to 0.99 for organics and 0.995 for inorganics.

Limit(s) of Detection (LOD) [a.k.a., Method Detection Limit (MDL)]: The MDL is the minimum measured quantity of a substance that can be reported with 99% confidence that the concentration is distinguishable from method blank results, consistent with 40CFR Part 136 Appendix B, August, 2017.

Limit(s) of Quantitation (LOQ) [a.k.a., Reporting Limit]: The minimum levels, concentrations, or quantities of a target variable (e.g., target analyte) that can be reported with a specified degree of confidence.

(QS) Matrix: The component or substrate that contains the analyte of interest. For purposes of batch and QC requirement determinations, the following matrix distinctions shall be used:

Aqueous: Any aqueous sample excluded from the definition of Drinking Water or Saline/Estuarine. Includes surface water, groundwater effluents, and TCLP or other extracts.

Drinking Water: Any aqueous sample that has been designated as a potable or potential potable water source.

Saline/Estuarine: Any aqueous sample from an ocean or estuary, or other salt water source such as the Great Salt Lake.

Non-Aqueous Liquid: Any organic liquid with <15% settleable solids.

Biological Tissue: Any sample of a biological origin such as fish tissue, shellfish, or plant material. Such samples shall be grouped according to origin.

Solids: Includes soils, sediments, sludges, and other matrices with >15% settleable solids.

Chemical Waste: A product or by-product of an industrial process that results in a matrix not previously defined.

Air & Emissions: Whole gas or vapor samples including those contained in flexible or rigid wall containers and the extracted concentrated analytes of interest from a gas or vapor that are collected with a sorbant tube, impinger solution, filter, or other device. (TNI)

Matrix Spike (spiked sample or fortified sample): A sample prepared, taken through all sample preparation and analytical steps of the procedure unless otherwise noted in a referenced method, by adding a known amount of target analyte to a specified amount of sample for which an independent test result of target analyte concentration is available. Matrix spikes are used, for example, to determine the effect of the matrix on a method's recovery efficiency.

Matrix Spike Duplicate (spiked sample or fortified sample duplicate): A replicate matrix spike prepared and analyzed to obtain a measure of the precision of the recovery for each analyte.

Method Blank: A sample of a matrix similar to the batch of associated samples (when available) that is free from the analytes of interest and is processed simultaneously with and under the same conditions as samples through all steps of the analytical procedures, and in which no target analytes or interferences are present at concentrations that impact the analytical results for sample analyses.

Method Detection Limit: See Limit of Detection (LOD)

Negative Control: Measures taken to ensure that a test, its components, or the environment do not cause undesired effects, or produce incorrect test results.

Non-conformance: An indication, judgment, or state of not having met the requirements of the relevant specifications, contract, or regulation.

Observation: A record of phenomena that (1) may assist in evaluation of the sample data; (2) may be of importance to the project manager and/or the client, and yet not at the time of the observation have any known effect on quality.

Performance Audit: The routine comparison of independently obtained qualitative and quantitative measurement system data with routinely obtained data in order to evaluate the proficiency of an analyst or laboratory.

Positive Control: Measures taken to ensure that a test and/or its components are working properly and producing correct or expected results from positive test subjects.

Precision: The degree to which a set of observations or measurements of the same property, obtained under similar conditions, conform to themselves; a data quality indicator. Precision is usually expressed as standard deviation, variance or range, in either absolute or relative terms. (TNI)

Preservation: Any conditions under which a sample must be kept in order to maintain chemical and/or biological integrity prior to analysis. (TNI)

Proficiency Testing: A means of evaluating a laboratory's performance under controlled conditions relative to a given set of criteria through analysis of unknown samples provided by an external source. (TNI)

Proficiency Testing Program: The aggregate of providing rigorously controlled and standardized environmental samples to a laboratory for analysis, reporting of results, statistical evaluation of the results and the collective demographics and results summary of all participating laboratories. (TNI)

Proficiency Test Sample (PT): A sample, the composition of which is unknown to the laboratory and is provided to test whether the laboratory can produce analytical results within specified acceptance criteria. (TNI)

Quality Assurance: An integrated system of management activities involving planning, implementation, assessment, reporting and quality improvement to ensure that a process, item or service is of the type of quality needed and expected by the client. (TNI)

Quality Assurance [Project] Plan (QAPP): A formal document describing the detailed quality control procedures by which the quality requirements defined for the data and decisions pertaining to a specific project are to be achieved. (EAP-QAD)

Quality Control: The overall system of technical activities that measures the attributes and performance of a process, item, or service against defined standards to verify that they meet the stated requirements established by the customer; operational techniques and activities that are used to fulfill requirements for quality; also the system of activities and checks used to ensure that measurement systems are maintained within prescribed limits, providing protection against "out of control" conditions and ensuring that the results are of acceptable quality. (TNI)

Quality Control Sample: A sample used to assess the performance of all or a portion of the measurement system. One of any number of samples, such as Certified Reference Materials, a quality system matrix fortified by spiking, or actual samples fortified by spiking, intended to demonstrate that a measurement system or activity is in control. (TNI)

Quality Manual: A document stating the management policies, objectives, principles, organizational structure and authority, responsibilities, accountability, and implementation of an agency, organization, or laboratory, to ensure the quality of its product and the utility of its product to its users. (TNI)

Quality System: A structured and documented management system describing the policies, objectives, principles, organizational authority, responsibilities, accountability, and implementation plan of an organization for ensuring quality in its work processes, products (items), and services. The quality system provides the framework for planning, implementing, and assessing work performed by the organization and for carrying out required QA and QC activities. (TNI)

Raw Data: The documentation generated during sampling and analysis. This documentation includes, but is not limited to, field notes, electronic data, magnetic tapes, untabulated sample results, QC sample results, print outs of chromatograms, instrument outputs, and handwritten records. (TNI)

Record Retention: The systematic collection, indexing and storing of documented information under secure conditions.

Reference Material: Material or substance one or more properties of which are sufficiently homogeneous and well established to be used for the calibration of an apparatus, the assessment of a measurement method, or for assigning values to materials. (TNI)

Reference Standard: Standard used for the calibration of working measurement standards in a given organization or a given location. (TNI)

Sampling: Activity related to obtaining a representative sample of the object of conformity assessment, according to a procedure.

Second Order Polynomial Curve (Quadratic): The 2nd order curves are a mathematical calculation of a slightly curved line over two axis. The y axis represents the instrument response (or Response ratio) of a standard or sample and the x axis represents the concentration. The 2nd order regression will generate a coefficient of determination (COD or r^2) that is a measure of the "goodness of fit" of the quadratic curvature the data. A value of 1.00 indicates a perfect fit. In order to be used for quantitative purposes, r^2 must be greater than or equal to 0.99.

Selectivity: The ability to analyze, distinguish, and determine a specific analyte or parameter from another component that may be a potential interferent or that may behave similarly to the target analyte or parameter within the measurement system. (TNI)

Sensitivity: The capability of a method or instrument to discriminate between measurement responses representing different levels (e.g., concentrations) of a variable of interest. (TNI)

Spike: A known mass of target analyte added to a blank, sample or sub-sample; used to determine recovery efficiency or for other quality control purposes.

Standard: The document describing the elements of laboratory accreditation that has been developed and established within the consensus principles of standard setting and meets the approval requirements of standard adoption organizations procedures and policies. (TNI)

Standard Operating Procedures (SOPs): A written document which details the method for an operation, analysis, or action, with thoroughly prescribed techniques and steps. SOPs are officially approved as the methods for performing certain routine or repetitive tasks. (TNI)

Storage Blank: A blank matrix stored with field samples of a similar matrix (volatiles only) that measures storage contribution to any source of contamination.

Surrogate: A substance with properties that mimic the analyte of interest. It is unlikely to be found in environment samples and is added to them for quality control purposes.
Surrogate compounds must be added to all samples, standards, and blanks, for all organic chromatography methods except when the matrix precludes its use or when a surrogate is not available. Poor surrogate recovery may indicate a problem with sample composition and shall be reported to the client whose sample produced poor recovery. (QAMS)

Systems Audit (also Technical Systems Audit): A thorough, systematic, qualitative on-site assessment of the facilities, equipment, personnel, training, procedures, record keeping, data validation, data management, and reporting aspects of a total measurement system. (EPA-QAD)

Technical Manager: A member of the staff of an environmental laboratory who exercises actual day-to-day supervision of laboratory operations for the appropriate fields of accreditation and reporting of results

Technology: A specific arrangement of analytical instruments, detection systems, and/or preparation techniques.

Traceability: The ability to trace the history, application, or location of an entity by means of recorded identifications. In a calibration sense, traceability relates measuring equipment to national or international standards, primary standards, basic physical constants or properties, or reference materials. In a data collection sense, it relates calculations and data generated throughout the project back to the requirements for the quality of the project. (TNI)

Trip Blank: A blank matrix placed in a sealed container at the laboratory that is shipped, held unopened in the field, and returned to the laboratory in the shipping container with the field samples.

Uncertainty: A parameter associated with the result of a measurement that characterizes the dispersion of the value that could reasonably be attributed to the measured value.

Acronyms:

CAR – Corrective Action Report
CCV – Continuing Calibration Verification
CF – Calibration Factor
CFR – Code of Federal Regulations
COC – Chain of Custody
DOC – Demonstration of Capability
DQO – Data Quality Objectives
DUP - Duplicate
EHS – Environment, Health and Safety
EPA – Environmental Protection Agency
GC - Gas Chromatography
GC/MS - Gas Chromatography/Mass Spectrometry
HPLC - High Performance Liquid Chromatography
ICP - Inductively Coupled Plasma Atomic Emission Spectroscopy
ICP/MS – ICP/Mass Spectrometry
ICV – Initial Calibration Verification
IDL – Instrument Detection Limit
IH – Industrial Hygiene
IS – Internal Standard
LCS – Laboratory Control Sample
LCSD – Laboratory Control Sample Duplicate
LIMS – Laboratory Information Management System
LOD – Limit of Detection
LOQ – Limit of Quantitation
MDL – Method Detection Limit
MDLCK – MDL Check Standard
MDLV – MDL Verification Check Standard
MRL – Method Reporting Limit Check Standard
MS – Matrix Spike
MSD – Matrix Spike Duplicate
SDS - Safety Data Sheet
NELAP - National Environmental Laboratory Accreditation Program
PT – Performance Testing
TNI – The NELAC Institute
QAM – Quality Assurance Manual
QA/QC – Quality Assurance / Quality Control
QAPP – Quality Assurance Project Plan
RF – Response Factor
RPD – Relative Percent Difference
RSD – Relative Standard Deviation
SD – Standard Deviation
SOP – Standard Operating Procedure
TAT – Turn-Around-Time
VOA – Volatiles
VOC – Volatile Organic Compound

Appendix 6. Analytical Method References

Reference methods include:

- *Guidelines Establishing Test Procedures for the Analysis of Pollutants Under the Clean Water Act; Analysis and Sampling Procedures; 40CFR Part 136 as amended by Method Update Rule; August 28, 2017.*
- *Methods for Chemical Analysis of Water and Wastes, EPA 600 (4-79-020), 1983.*
- *Methods for the Determination of Inorganic Substances in Environmental Samples, EPA-600/R-93/100, August 1993.*
- *Methods for the Determination of Metals in Environmental Samples, EPA/600/4-91/010, June 1991. Supplement I: EPA-600/R-94/111, May 1994.*
- *Methods for the Determination of Organic Compounds in Drinking Water, EPA-600/4-88-039, December 1988, Revised, July 1991, Supplement I, EPA-600-4-90-020, July 1990, Supplement II, EPA-600/R-92-129, August 1992. Supplement III EPA/600/R-95/131 - August 1995 (EPA 500 Series) (EPA 500 Series methods)*
- *Technical Notes on Drinking Water Methods, EPA-600/R94-173, October 1994*
- *Standard Methods for the Examination of Water and Wastewater, 18th/19th /20th/ on-line edition; Eaton, A.D. Clesceri, L.S. Greenberg, A.E. Eds; American Water Works Association, Water Pollution Control Federation, American Public Health Association: Washington, D.C.*
- *Test Methods for Evaluating Solid Waste Physical/Chemical Methods (SW846), Third Edition, September 1986, Final Update I, July 1992, Final Update IIA, August 1993, Final Update II, September 1994; Final Update IIB, January 1995; Final Update III, December 1996; Final Update IV, January 2008; Final Update V, August 2015.*
- *Annual Book of ASTM Standards, American Society for Testing & Materials (ASTM), Philadelphia, PA.*
- *National Status and Trends Program, National Oceanographic and Atmospheric Administration, Volume I-IV, 1985-1994.*
- *Manual for the Certification of Laboratories Analyzing Drinking Water (EPA 815-R-05-004, January 2005)*
- *Code of Federal Regulations (CFR) 40, Parts 136, 141, 172, 173, 178, 179 and 261*

Revision History

Summary of Changes for Revision 9:

- This QAM revision reflects a re-work of the local manual to comply with the 2020 QA Manual Template. Most changes are administrative in nature and deal with incorporation of text to accommodate non-TestAmerica Business Units. Titles, roles, and responsibilities have been updated to reflect current Eurofins nomenclature.
  - Updated Quality Policy statement to reflect current Eurofins statement. Section 7.1
  - Clarified NDSC document control nomenclature and included reference to KGDs. Section 7.3
  - Added section on Use of NELAP and A2LA logo. Section 7.5
  - Removed example COC, CAR, and IDOC Form and routine instrument maintenance schedule. Removed sections on sample identifier info and floor plans.
  - Updated Org Chart, Instrument Listing, Certification Listing, and SOP Listing to reflect the current status.
  - Updated Training schedule to reflect current practice. Section 5.3.
  - Added text on Client Supplied Methods. Section 19.4.1.1
  - Added listing of Analytical Method References.

APPENDIX B
Treatability Study Work Plan for Groundwater

GROUNDWATER TREATABILITY STUDY WORK PLAN

Operable Unit 1/Operable Unit 2 Portion of the Anniston PCB Site

Submitted by

Pharmacia LLC/Solutia Inc.

June 2022

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ACRONYMS AND ABBREVIATIONS

ASTM	ASTM International
DQO	data quality objective
EBV	empty bed volume
gpm	gallons per minute
GAC	granular activated carbon
LGAC	liquid-phase granular activated carbon
mm	millimeter(s)
µg/L	micrograms per liter
µm	micron
OU	operable unit
PCB	polychlorinated biphenyl
PDI	predesign investigation
PDIWP	predesign investigation work plan
P/S	Pharmacia LLC/Solutia Inc.
ROD	Record of Decision
RSSCT	rapid small-scale column test
TS	treatability study
TSWP	treatability study work plan
UFP-QAPP	Uniform Federal Policy Quality Assurance Project Plan
USEPA	United States Environmental Protection Agency

1. INTRODUCTION

This groundwater treatability study work plan (Groundwater TSWP), prepared on behalf of Pharmacia LLC/Solutia Inc. (P/S), presents the approach to conduct bench-scale treatability testing and data analysis to support the design of the groundwater remedy component for the Operable Unit 1/Operable Unit 2 (OU-1/OU-2) portion of the Anniston Polychlorinated Biphenyl (PCB) Site (the Site). It is a supporting document to the predesign investigation work plan (PDIWP) for OU-1/OU-2 (OU-1/OU-2 PDIWP) and a deliverable under the OU-1/OU-2 *Remedial Design/Remedial Action Consent Decree* (United States Environmental Protection Agency [USEPA] 2021).

This Groundwater TSWP provides the objectives and technical approach for the planned treatability study (TS) that will be conducted to support the development of the remedial design for groundwater at the T-11 area. The TS is based on the current conceptual approach to the groundwater remedy with specific focus on performance of the carbon adsorption aspect of the planned treatment. The treatability testing outlined in this Groundwater TSWP may be modified in the future, and additional treatability testing may be identified based on the results of the predesign investigation (PDI) or as the remedial design is advanced. If necessary, modifications to the TS program will be documented in an addendum to this work plan.

1.1 Background Information

As described in the Record of Decision (ROD), the groundwater remedy for the T-11 area includes installing and operating a groundwater extraction and treatment (i.e., pump-and-treat) system to capture PCB-impacted groundwater (USEPA 2017). The long-term goal of this system is to restore groundwater to the remedial goal for PCBs (0.5 micrograms per liter [µg/L]). The pump-and-treat system described in the OU-1/OU-2 ROD includes the following elements:

- Two groundwater extraction wells equipped with submersible pumps and controls
- Liquid-phase granular activated carbon (LGAC) filters equipped with pretreatment particulate filters, as needed
- Discharge of treated groundwater to Snow Creek via the permit equivalency process for National Pollutant Discharge Elimination System

Groundwater samples collected from monitoring well T-11 as part of the OU-1/OU-2 remedial investigation exhibited total PCB concentrations in unfiltered samples ranging from 3.3 µg/L (Aroclors) to 17.3 µg/L (homologs), with a concentration of 10.4 µg/L (homologs) reported following redevelopment of the monitoring well that was completed due to high turbidity noted in the initial samples (Environ 2015). As noted in the ROD, the distribution of PCBs in the floodplain area surrounding T-11 appeared to be unique for the Site, characterized by the presence of the lesser-chlorinated PCB compounds (e.g., Aroclor 1221 and homologs monochlorobiphenyl and dichlorobiphenyl). The presence of these lesser-chlorinated PCBs, along with the potential for carbon adsorption demand from naturally occurring organic matter and other geochemical species in this floodplain area form the primary basis for the TS proposed herein.

The groundwater extraction system is anticipated to consist of two extraction wells, each equipped with a submersible pump rated for producing an instantaneous flow rate of approximately 4 to 5 gallons per minute (gpm). The anticipated maximum flow rate for the system during pumping is approximately 8 to 10 gpm. However, the long-term well yields for extraction wells at the OU-3 portion of the Site average approximately 0.1 to 0.5 gpm per well (totaling 0.2 to 1 gpm for a 2 well extraction system). As such, the long-term average flow rate for the system could be less than the maximum capacity. Conveyance piping (including fittings, valves, and pressure gauges) is anticipated to be installed between pump discharge ports and the climate-controlled treatment system enclosure. The treatment system is anticipated to consist of a prefiltration unit (i.e., a 10-micron [µm] bag filter) and two LGAC vessels arranged in series with lead-lag configuration. Each carbon vessel is anticipated to have a maximum carbon capacity of approximately 250 pounds, for a combined system capacity of 500 pounds. The treatment system is anticipated to include pressure gauges, valves, sampling ports, a flow meter, a fan, a heater, lighting, and all other equipment necessary for system operations. Discharge piping for the treated groundwater is anticipated to be routed from the effluent of the lag carbon vessel to Snow Creek.

1.2 Existing Data to Support Treatability Studies

Data generated during previous investigations in OU-1/OU-2 and data collected as part of the planned PDI will provide information regarding groundwater conditions within the T-11 area. The existing information and data will be supplemented with the TS data to support the remedial design.

As discussed in the OU-1/OU-2 feasibility study, the use of GAC filtration for treatment of PCB-impacted groundwater is well proven (Ramboll Environ 2016), and such treatment is being used effectively within the OU-3 portion of the Site (USEPA 2020). Nevertheless, as discussed above, a TS of GAC performance for the T-11 area is recommended because lesser-chlorinated PCBs are present and naturally occurring species in the floodplain area have the potential for carbon adsorption demand.

1.3 Work Plan Organization

The remainder of this Groundwater TSWP is organized as follows:

- Section 2 details the data quality objectives (DQOs) for the groundwater treatability study.
- Section 3 describes the field sampling and treatability testing.
- Section 4 presents the treatability testing reporting and schedule.
- Section 5 lists the references cited in this document.

2. TREATABILITY STUDY DATA QUALITY OBJECTIVES

The overall objective and purpose of the TS is to generate data to use in the design and evaluation of groundwater treatment using GAC filtration. Accordingly, the following TS DQOs have been established as discussed in the PDIWP:

- GW-DQO 3: Determine an efficient and cost-effective GAC media for PCB treatment.
- GW-DQO 4: Determine the estimated time to PCB breakthrough in a full-scale GAC system.

Groundwater samples for the bench-scale treatability tests will be collected from monitoring well T-11 as part of the PDI sampling (Section 3.2). Bench-scale treatability tests will include an isotherm batch test to address GW-DQO 3 and a rapid small-scale column test to address GW-DQO 4. Details of these tests are provided in Section 3 below. In addition, PCB and geochemical analyses will be conducted on groundwater samples from T-11 to establish current groundwater conditions and determine the volume of groundwater needed for the treatability tests.

3. FIELD SAMPLING AND TREATABILITY TESTING

This section describes the roles and responsibilities for the TS, the field sampling, and the treatability testing and analytical program.

3.1 Roles and Responsibilities

P/S is the primary responsible party and, therefore, has overall responsibility for managing the TS. Agency communications will be through P/S unless P/S directs this to other members of the team. USEPA is the lead regulatory agency on the Anniston PCB Site. P/S has retained a technical team to support the design efforts. As part of the technical team, GSI Environmental, Inc., will have the technical responsibility for completing the groundwater TS described herein.

3.2 Field Sampling Plan

Groundwater samples for treatability testing will be collected from monitoring well T-11 in conjunction with the PDI field program. As described in the OU-1/OU-2 PDIWP, the groundwater PDI program at the T-11 area will follow a phased approach.

- Phase 1 will include redeveloping the well, sampling groundwater for laboratory analysis of PCBs and geochemical parameters to supplement existing characterization data, and confirming the hydraulic conductivity of the transmissive zone through aquifer slug testing.
- Phase 2 will involve installing groundwater observation wells to be used in subsequent aquifer pump testing and collecting soil samples for geotechnical analysis.
- Phase 3 will include conducting aquifer pump testing to assess aquifer response and well yield for designing the extraction wells. It is anticipated that a portion of the groundwater discharge from the aquifer pump tests will be collected for use in the TS.

The groundwater samples for treatability testing will be collected and contained in sealable water containers (e.g., 5-gallon plastic carboys and/or 30-gallon plastic drums). Approximately 120 gallons of groundwater are anticipated to be required by the treatability testing laboratory. The final volume of water required will be determined after reviewing the analytical results from Phase 1 sampling and testing. The sample containers will be shipped under chain-of-custody control to the treatability testing laboratory (SiREM in Knoxville, Tennessee).

3.3 Health and Safety Planning

The groundwater samples for treatability testing will be collected in accordance with the OU-1/OU-2 PDIWP, the Uniform Federal Policy Quality Assurance Project Plan (UFP-QAPP), and the project health and safety plan.

3.4 Treatability Testing and Analysis Program

The following bench-scale treatability tests will be conducted using the groundwater samples from T-11:

- A batch isotherm test

- A rapid small-scale column test

A description of each test is provided in the following subsections. Prior to conducting the test, the provided T-11 groundwater will be filtered through a 10-µm polypropylene bag filter in the lab. This filtration will mimic the anticipated full-scale system where a 10-µm bag filter will be installed upstream of the LGAC vessels.

3.4.1 Sorption Isotherm Batch Test

A sorption isotherm batch test will be performed to evaluate how effective two different GAC types are at removing PCBs and to determine the equilibrium partitioning relationship between dissolved and adsorbed PCBs. The two types of GAC to be tested will be coconut-shell-based 8x30 mesh size (the same carbon used in the OU-3 carbon treatment systems) and bituminous-coal-based 8x30 mesh size, which has been reported to outperform coconut-shell-based GAC for removing compounds with a high molecular weight (McNamara et al. 2018).

Five different quantities of both GAC types will be weighed into separate bottles, and 1.5 liters of T-11 groundwater will be distributed into each of the bottles. Two untreated control bottles containing no GAC will also be prepared for sampling at the beginning and end of the reaction period. The bottles will be sealed and tumbled for 20 to 24 hours. The bottles will then be removed from the tumbler, and the GAC will be allowed to settle for approximately 0.5 hours. After the GAC has settled, the pH of each sample will be measured and recorded because GAC adsorption capacity has been shown to increase with decreasing pH in the range of 8.5 to 5 (Hong 1998, Semmens et al. 1986).

Following pH testing, the sample supernatants (i.e., the groundwater within the bottles) from all 12 bottles will be collected and shipped to a third-party laboratory for PCB homologs analysis per the UFP-QAPP. The data will be reviewed to determine which GAC type was most efficient (i.e., adsorbs the highest concentration of PCB with the least amount of GAC) and cost effective for long-term use. The selected GAC material will be used for further testing in the rapid small-scale column test (RSSCT) described below.

3.4.2 Rapid Small-Scale Column Test

The RSSCT is used to determine the volume of groundwater at a specific flow rate that can be treated before the GAC is saturated and PCB breakthrough occurs. The column test is scaled to simulate the performance of a full-scale system thereby replicating mass transfer and hydrodynamic properties, which allows for accurate estimations of full-scale GAC usage and operating time to breakthrough. To simulate the full-scale system performance, the ratio of the anticipated full-scale system GAC vessel diameter to the carbon particle size will be maintained in the laboratory columns by reducing the particle size of the carbon used in the RSSCT. Maintaining this ratio will ensure that the RSSCT recreates similar contact between the groundwater and carbon particles in the anticipated full-scale treatment system.

The column test will be conducted in a small-diameter stainless steel column (approximately 0.5-inches in diameter by 4 inches in height) packed with a measured volume of powdered carbon of a specific mesh size, as indicated by ASTM International (ASTM) D 6586-03, and a size required

to maintain the vessel diameter-to-particle size ratio. For example, the anticipated full-scale 24-inch (610 millimeters [mm]) GAC vessel filled with 12x40-mesh size carbon (with an approximate particle size of 1.7 mm) would have a vessel diameter-to-particle size ratio of 359. The RSSCT conducted in a 0.5-inch (12.7 mm) diameter lab column would thus require a carbon particle size of 0.035 mm to maintain the diameter-to-particle size ratio. The 0.035-mm carbon particle size to be used in the RSSCT column would be achieved by grinding the 12x40-mesh size carbon and sieving through a 400-mesh sieve. Actual RSSCT testing parameters will be determined in part based on the results of the sorption isotherm tests.

The column will be sealed at both ends using threaded fittings connected to stainless steel tubing to allow T-11 groundwater to be delivered to the column. The column will be positioned vertically and connected to a peristaltic pump using flexible vinyl tubing. Deionized water will be pumped into the column in an upflow manner to flood the column and expel air. The deionized water will then be allowed to gravity drain, and the quantity of water collected is the column void volume, or pore volume. Deionized water will be replaced with the prefiltered T-11 groundwater and passed through the column in an upflow manner as further described below.

Based on the carbon vendor's (Evoqua Water Technologies LLC) preliminary modeling estimates of GAC performance, it is anticipated that 30,000 empty bed volumes (EBVs; 1 EBV for the RSSCT equates to approximately 13 milliliters) need to be passed through the lab column to achieve PCB breakthrough. The column will be continuously operated at a flow rate between 30 and 65 milliliters per minute, as dictated by maximum allowable backpressure, 40 pounds per square inch, monitored at column influent. It is anticipated that PCB breakthrough will occur between approximately 4 and 8 days of column operation.

Effluent from the GAC column will be collected and labeled as to the EBV or range of EBVs represented by the sample. The sample volume necessary for analysis of PCBs, total organic carbon, and pH is 1.5 liters, which equates to approximately 115 EBVs. It is anticipated that aliquot effluent samples will be collected as practicable, based on laboratory staff availability, continuously throughout the duration of the experiment. At a minimum, samples will be collected at intervals of 1,000 to 2,000 EBVs, with a maximum of 30 effluent samples collected during the operation of the column test. A total of 15 effluent samples are anticipated to be analyzed to determine PCB breakthrough in the column. An additional 4 samples will be collected from the column influent, at intervals of approximately 7,500 EBVs, to test for groundwater stability throughout the test. In total, 19 samples will be analyzed for PCBs, TOC, and pH during the RSSCT test.

3.5 Residuals Management

A variety of residuals might be generated from the treatability testing. Examples of these residuals are as follows:

- Materials obtained but not used for testing
- Tested materials and treatment residuals
- Used containers, other field disposables, and personal protective equipment

If these materials are generated at the Site, they will be managed using P/S's in-place system to containerize and dispose of PCB-containing wastes and nonhazardous waste. If the materials are generated away from the Site, P/S will work with the laboratory either to use laboratory disposal protocols or to return the materials to the Site for treatment under the P/S system.

3.6 Data Management and Quality

Data management and quality are important components of the TS process and ultimately provide the basis of data usability. Field sampling will be conducted in accordance with this Groundwater TSWP, the OU-1/OU-2 PDIWP, and the standard operating procedures provided in the UFP-QAPP.

During the field sampling and the treatability testing, detailed notes and digital photographs will be collected to document the activities completed. Any variations from the procedures outlined herein will be documented in the TS data summary report.

4. TREATABILITY STUDY REPORTING AND SCHEDULE

This section describes the TS data evaluation and the deliverables to be prepared to support remedial design.

4.1 Data Evaluation and Interpretation

The treatability testing data will be analyzed and evaluated during the development of the remedial design. Data will be specifically evaluated and interpreted considering the DQOs identified in Section 2. Relevant field information and reports, analytical data, data reviews, and other documentation will be reviewed and evaluated. The TS results will be used to support the basis of design and data inputs to evaluate the GAC components of the groundwater remediation design, including the GAC media, the optimum number and size of GAC vessels, and the estimated GAC changeout frequency.

4.2 Reporting

The results of the treatability testing will be presented in a data summary report. That report will summarize the field sampling, treatability testing activities, results, and conclusions. The data summary report will include tabulated data from the treatability testing along with relevant field information and reports, analytical data, photographs, and other related documentation.

4.3 Schedule

The treatability testing discussed in Section 3 will be conducted using samples collected from monitoring well T-11 generated during the aquifer pump testing activities of the PDI, which is planned as the third and final phase of the PDI for this area. Consequently, the schedule for the TS will be tied to the start and duration of PDI field sampling. It is anticipated that the treatability testing and associated analyses will require approximately 3 months to complete once the samples are received at the testing laboratory. Completion of the data summary report is anticipated approximately 2 months following receipt of the final laboratory testing results.

5. REFERENCES

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APPENDIX C
Treatability Study Work Plan for Sediment

SEDIMENT TREATABILITY STUDY WORK PLAN

Operable Unit 1/Operable Unit 2 Portion of the Anniston PCB Site

Submitted by

Pharmacia LLC/Solutia Inc.

June 2022

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Table 3-2: Geotechnical Characterization Analyses

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Attachment 1: Standard Operating Procedure: Drainage Study

Attachment 2: Standard Operating Procedure: Stabilization/Solidification Tests

ACRONYMS AND ABBREVIATIONS

ASTM	ASTM International
DQO	data quality objective
EGL	environmental geochemistry laboratory
EGT	Excel Geotechnical Testing, Inc.
mg/kg	milligrams per kilogram
OU	operable unit
PCB	polychlorinated biphenyl
PDI	predesign investigation
PDIWP	predesign investigation work plan
P/S	Pharmacia LLC/Solutia Inc.
ROD	Record of Decision
SOP	standard operating procedure
UFP-QAPP	Unified Federal Policy Quality Assurance Project Plan
TS	treatability study
TSWP	treatability study work plan
USEPA	United States Environmental Protection Agency

1. INTRODUCTION

This sediment treatability study work plan (Sediment TSWP), prepared on behalf of Pharmacia LLC/Solutia Inc. (P/S), presents the approach to conduct bench-scale treatability testing and data analysis to support the design of the sediment remedy component for the Operable Unit 1/Operable Unit 2 (OU-1/OU-2) portion of the Anniston polychlorinated biphenyl (PCB) Site (the Site). It is a supporting document to the predesign investigation work plan for OU-1/OU-2 (OU-1/OU-2 PDIWP) and a deliverable under the OU-1/OU-2 *Remedial Design/Remedial Action Consent Decree* (United States Environmental Protection Agency [USEPA] 2021).

This Sediment TSWP provides the objectives for the treatability study (TS) and describes the planned TS activities that will be conducted to support the development of the remedial design for Snow Creek sediment. The TS activities described herein are based on the current conceptual approach to the sediment remediation. The treatability testing outlined in this work plan may be modified in the future, and additional treatability testing may be identified based on the results of the predesign investigation (PDI) or as the remedial design is advanced. If necessary, modifications to the TS program will be documented in an addendum to this work plan.

1.1 Background Information

As described in the Record of Decision (ROD), the sediment remedy for OU-1/OU-2 requires excavating contaminated sediment in Snow Creek and disposing of the excavated sediment at an appropriate off-site disposal facility (USEPA 2017). The primary constituent of concern in sediment is PCB, and two previously identified sediment deposits contained one or more metals (i.e., chromium, lead, manganese, and nickel) at concentrations above the remedial goals.

The existing sediment sampling data indicate that the highest total PCB concentrations (60 milligrams per kilogram [mg/kg]) in Snow Creek sediment are generally found in sediment from the upstream portions of the creek between the 11th Street Ditch and Highway 202, including the culverts that convey flow under the highway. The data also indicate that there is a small portion of Snow Creek with a mean sediment PCB concentration of 12 mg/kg for sediment deposits near a railroad bridge just downstream of Highway 202. The sediment in the downstream OU-1/OU-2 portion of Snow Creek between the railroad bridge and Highway 78 had an average PCB concentration of approximately 1.9 mg/kg.

Approximately 2,500 cubic yards of sediment with constituent concentrations above the remedial goals established in the ROD were identified in the OU-1/OU-2 portion of Snow Creek (Ramboll Environ 2016). These estimates do not include any sediment volume in the channelized culvert beneath Quintard Mall. The actual volume of sediment in the OU-1/OU-2 portion of Snow Creek that will be targeted for removal will be determined during the remedial design and will be based on the data from the sediment PDI.

It is anticipated that sediment with PCB concentrations exceeding the remedial criteria will be removed from Snow Creek using mechanical methods and transported to one or more sediment management areas for dewatering (by gravity) or stabilization (by adding an amendment to condition the sediment). Once the sediment has been dewatered or stabilized it will be transported to an approved off-site landfill facility for disposal. Supernatant water (i.e., water removed from

the sediment as part of the dewatering process) will either be transported for off-site treatment or treated at the on-site and discharged.

At this early stage of the remedial design, the specific means and methods for implementing the sediment remedial action have not been finalized. The specific remedial process will be selected and refined during the design phase, which will include reviewing anticipated equipment, performance requirements, and cost effectiveness. The TS results from the testing outlined in this plan will be used to evaluate methods for implementing the sediment remedial action.

1.2 Existing Data to Support Treatability Studies

Data generated during prior investigations in OU-1/OU-2 and data collected as part of the planned PDI will provide data and information on conditions within the OU-1/OU-2 section of Snow Creek. The existing information and data will be supplemented with the TS data to support the remedial design.

Most sediment deposits identified in the OU-1/OU-2 portion of Snow Creek during prior investigations contained fine to coarse sands and gravel. Silts or clays were identified in a smaller subset of the sediment deposits predominately at the upstream and downstream portions of Snow Creek. The abundant sands and gravel present in the sediment deposits are expected to allow for efficient dewatering; however, the presence of fine particles (i.e., silts and clays) in portions of the creek warrants treatability testing to evaluate the potential dewatering characteristics of the sediment that will be targeted for removal.

1.3 Work Plan Organization

The remainder of this Sediment TSWP is organized as follows:

- Section 2 details the data quality objectives (DQOs) for the sediment TS.
- Section 3 describes the field sampling and treatability testing.
- Section 4 presents the treatability testing reporting and schedule.
- Section 5 lists the references cited in this document.

2. TREATABILITY STUDY DATA QUALITY OBJECTIVE

The overall objective and purpose of the TS is to generate data to use in the design and evaluation of sediment removal, transport, processing, and disposal. Accordingly, the following TS DQO has been established as discussed in the PDIWP:

- SED-DQO 3: Determine efficient and cost-effective methods for dewatering and stabilizing sediment excavated from Snow Creek.
- SED-DQO 4: Determine what treatment technologies might be necessary to meet the requirements for discharging the supernatant water that drains from sediment excavated from the creek.

The sediment to be excavated from Snow Creek might require processing, treatment, or conditioning prior to transport to allow it to be loaded into trucks, to meet regulatory requirements for transportation on public roads (i.e., no free liquids permitted), and to strengthen the sediment to the requirements of the selected off-site landfill. During the remediation, it is anticipated that the full extent of sediment deposits that exceed the remedial criteria of 3 mg/kg will be removed. The current thinking is that excavated sediment from a nearby geographic portion of the creek would be managed together within the creek or at a temporary staging area even if the materials contain a mixture of fine- and coarse-grained sediments. Blending coarse-grained sediments (if available) with fine-grained sediments to achieve transport and disposal requirements can also be employed. The remedial action may also include a combination of dewatering methods for the excavated sediment to meet transportation and facility requirements (e.g., gravity dewatering and stabilization/solidification), which is common on sediment remediation projects. The need for combining process options will depend on the nature of the sediment excavated, the results of treatability testing, and the means, methods, and schedule of the remedial contractor.

The data gathered as part of the PDI and the results of the TS will provide information on the nature of the sediment that will be targeted for removal and will provide information that will be used to evaluate potential processing and dewatering options as part of the design phase. The PDI and treatability testing data will also be provided to the remedial contractor to determine the means and methods for the remediation.

Bulk sediment samples for the bench-scale treatability evaluations will be collected from targeted remedial areas that will be identified based on the results of the PDI sampling (Section 3.2). The physical (geotechnical) properties of the sediment collected will also be tested as part of the TS.

Bench-scale treatability tests will include a drainage study and stabilization/solidification testing. In addition, geotechnical analyses will be conducted on treated material. Bulk sediment samples collected from target remedial areas in conjunction with the PDI sampling will be submitted for laboratory characterization and treatability testing.

Sediment excavated from water bodies contain excess water (referred to as supernatant) that will be removed during dewatering prior to transport for disposal. The supernatant will likely require treatment prior to discharge. The bench-scale treatability testing will provide data on the potential concentrations of parameters within the supernatant for review as part of the remedial design to evaluate potential treatment requirements. During design, data from analytical testing of the

supernatant will be compared to the discharge requirements to determine treatment necessary prior to discharge.

3. FIELD SAMPLING AND TREATABILITY TESTING

This section describes the roles and responsibilities for the TS, the field sampling, and the treatability testing and analytical program.

3.1 Roles and Responsibilities

P/S is the primary responsible party and, therefore, has overall responsibility for managing the TS. Agency communications will be through P/S, unless otherwise directed by P/S to other members of the team. USEPA is the lead agency on the Anniston PCB Site project. P/S has retained a technical team to support the design efforts. As part of the technical team, Anchor QEA, LLC, (Anchor QEA) will have the technical responsibility for completing the TS activities described herein.

3.2 Field Sampling Plan

Bulk sediment samples for treatability testing will be collected from Snow Creek in conjunction with the PDI sampling program.

As described in the OU-1/OU-2 PDIWP, the sediment PDI program will follow a phased approach. Initially, Phase 1, which includes probing, survey, and visual observations, will be conducted along the OU-1/OU-2 portion of Snow Creek to map the current sediment deposit locations, estimate sediment volumes, and determine where sediment sampling will be performed to supplement existing sediment characterization data. Phase 2 of the sediment PDI will involve collecting sediment samples for analytical testing to evaluate and refine the extent of sediment in the OU-1/OU-2 portion of Snow Creek that has PCB concentrations above the remedial criteria. The locations where bulk sediment samples will be collected for the TS will be determined after the data collected from the Phase 1 sediment mapping program and the Phase 2 sediment sampling have been reviewed.

Three bulk sediment samples will be collected for treatability testing from areas within Snow Creek that will be targeted for removal. The bulk sediment sampling locations will be selected based upon the following criteria:

- To provide a range in sediment types (i.e., fine material vs. coarse material) based on historical data and data collected during Phase 1 and Phase 2 of the sediment PDI
- To focus on areas with the largest expected volume of sediment to be removed
- To be spatially representative of the sediment removal areas within Snow Creek

The bulk sediment samples will be composited from sediment samples collected in the target remedial areas. It is anticipated that the full extent of sediment deposits that exceed the remedial criteria of 3 mg/kg will be removed and that the sediments removed from deposits located in a nearby geographic portion of the creek would be managed together. As such, the bulk material samples collected for the treatability testing will be collected from the full-depth samples of the sediment deposits at up to five locations and then composited together. It is anticipated that the TS bulk sampling will target a range of sediment types from coarse-grained sediment deposits to fine-

grained sediment deposits, to the extent the materials represent a significant volume of sediment targeted for removal.

It is anticipated that, at a minimum, one bulk sediment sample will be composited from samples collected upstream, within, and immediately downstream of the Highway 202 culverts, which are anticipated to be handled together as part of the remediation. Other sampling locations will be determined after the PDI survey and sampling data has been reviewed. Due to the specific hazards represented by the permit-required confined spaces, bulk sediment samples will be collected from the Highway 202 culverts and the Quintard Mall culvert in conjunction with the confined space entry at those locations during the Phase 2 sediment sampling. The bulk sediment samples will be held for potential treatability testing.

The bulk sediment samples will be collected and contained in sealable 5-gallon buckets. At least 15 gallons of sediment will be collected for each bulk sample collected at each location. In addition, 10 gallons of water from Snow Creek will be collected to mix with the sediment to obtain sediment/water mixes whose properties are representative of the material properties that may occur during sediment removal.

The bulk sediment samples will be sent to Anchor QEA's environmental geochemistry laboratory (EGL) in Portland, Oregon, for treatability testing. Subsamples of sediment from each bulk sample will also be submitted to Excel Geotechnical Testing, Inc., (EGT) in Roswell, Georgia, for the following analyses:

- Moisture Content (ASTM International [ASTM] D2216)
- Particle Size (ASTM D6913/D7928)
- Atterberg (Liquid and Plastic) Limits (ASTM D4318)
- Dry Density (ASTM D2937)
- Organic Content (ASTM D2974)
- Unconfined Compressive Strength (ASTM D2166)
- Paint Filter Test (USEPA Method 9095B)

Following these initial analyses, treatability testing will be performed by Anchor QEA's Portland laboratory as described in Section 3.4.

In addition, Snow Creek bulk sediment samples will be shipped to EGT for post-testing geotechnical analysis as described in Section 3.4.2.

3.3 Health and Safety Planning

The sediment samples for treatability testing will be collected in accordance with the OU-1/OU-2 PDIWP, the Unified Federal Policy Quality Assurance Project Plan (UFP-QAPP; Appendix A of the PDIWP), the OU-1/OU-2 Remedial Design/Remedial Action Health and Safety Plan, and the confined space entry plans (Appendices D and E of the PDIWP), as applicable.

3.4 Testing and Analysis Program

The following bench-scale treatability tests will be performed for each bulk sediment sample collected:

- A drainage study
- Stabilization/solidification testing

A description of each test is provided in the following subsections.

3.4.1 Drainage Study

A drainage study will be performed by the Anchor QEA EGL to evaluate the ability of dredged sediment to passively dewater via gravity drainage. The purpose of this study will be to estimate the water content before and after gravity drainage to evaluate whether the dredged material will need additional treatment to meet transportation and disposal facility requirements. The drainage study will be performed on one homogenized sample from each of the bulk sediment sampling locations.

The drainage study will involve placing sediment samples within a screen and allowing the sample to gravity drain for 72 hours. Samples will be collected at multiple time intervals throughout the drainage study for moisture content and paint filter testing. Water that drains from the samples during the drainage study will be collected, measured, and tested for total suspended solids, PCBs, metals (barium, chromium, cobalt, lead, manganese, mercury, nickel, and vanadium), and oil and grease.

The drainage study will be conducted in accordance with the standard operating procedure (SOP) provided in Attachment 1.

3.4.2 Stabilization/Solidification Testing

Stabilization of samples will be investigated using commercially available reagents at various dosages with the goal of determining effective and cost-efficient reagent doses to meet transportation and disposal facility requirements for paint filter testing and minimum structural strength. The stabilization/solidification testing will be performed regardless of the results for the drainage study results.

Three reagents will be evaluated at a minimum of three mix ratios per reagent by the EGL (Table 3-1). Mix ratios will be evaluated through paint filter testing and visual observations of physical characteristics at cure periods of 12, 24, 48, and 72 hours. Mix ratios will also be monitored for weight change.

The following solidification agents will be used in the testing:

- Type I Portland cement
- Cement kiln dust
- Calciment
- Lime

The treatability testing will be performed using these commercially available amendments at mix ratios of 2.5%, 5%, and 7.5% by weight. If adequate testing results are not obtained using the initial ratios, additional mix ratios will be evaluated. The goal of the testing is to determine effective and cost-efficient combinations of reagent, mix ratio, and cure time to allow the solidified end-product to pass the paint filter test and achieve the minimum structural strength required by the disposal facility. The amendments will be provided by the manufacturer, and the vendor-provided specification sheets for each amendment will be included in the data summary report.

The stabilization and solidification testing will be performed in accordance with the SOP provided in Attachment 2.

For the initial screening phase, mix ratios will be evaluated through paint filter testing, percent solids analysis, moisture content, weight change, and visual observations at the previously specified cure periods. A pocket penetrometer will also be used to evaluate the strength of the admixtures. A second round of solidification testing may be employed using a smaller range of reagent blend to determine the optimum dose and cure time for solidification, as appropriate.

After optimal reagents, mix ratios, and cure times are established through testing at the Anchor QEA EGL, geotechnical testing will be performed by EGT on treated sediment. The geotechnical laboratory will prepare a single sample using the selected amendment and optimum dosage based on the results of the treatability testing. Following the proper cure time, the geotechnical tests detailed in Table 3-2 will be performed to determine the strength and physical properties of the sediment and amendment mixture.

3.5 Residuals Management

A variety of residuals may be generated as a result of the treatability testing. Examples of these residuals are as follows:

- Materials obtained but not used for testing
- Tested materials and treatment residuals
- Used containers, other field disposables, and personal protective equipment

If these materials are generated at the Site, they will be managed using P/S's in-place system to containerize and dispose of PCB-containing wastes and nonhazardous waste. If the materials are generated away from the Site, P/S will work with the laboratory either to use laboratory disposal protocols or to return the materials to the Site for treatment under the P/S system.

3.6 Data Management and Quality

Data management and quality is an important component of the TS process and ultimately provides the basis of data usability. The field sampling activities will be conducted in accordance with this work plan, the OU-1/OU-2 PDIWP, and the SOPs provided in the UFP-QAPP.

During the field sampling and the treatability testing, the field crew will take detailed notes and digital photographs to document the activities completed. Any variations from the procedures outlined herein will be documented in the TS data summary report.

4. TREATABILITY STUDY REPORTING AND SCHEDULE

This section describes the TS data evaluation and the deliverables to be prepared to support the remedial design.

4.1 Data Evaluation and Interpretation

The treatability testing data will be analyzed and evaluated during the development of the remedial design. Data will be specifically evaluated and interpreted considering the DQOs identified in Section 2. Relevant field information and reports, analytical data, data reviews, and other documentation will be reviewed and evaluated. The TS results will be used to support the basis of design and data inputs to evaluate the primary components of the sediment remediation design, including removal, transport, dewatering, processing, and disposal.

4.2 Reporting

The results of the treatability testing will be presented in a data summary report. That report will summarize the field sampling and the treatability testing activities, results, and conclusions. The data summary report will include tabulated data from the treatability testing along with relevant field information and reports, analytical data, photographs, and other related documentation.

4.3 Schedule

The treatability testing will be conducted using samples collected from areas within Snow Creek that will be targeted for removal. Consequently, the treatability testing will be performed after data from Phase 1 and Phase 2 of the sediment PDI program are reviewed.

The schedule for the TS will be tied to the start and duration of the PDI field sampling activities. It is anticipated that the treatability testing and associated geotechnical analytical activities will require approximately 3 months to complete once the samples are received at the testing laboratory.

5. REFERENCES

Ramboll Environ. 2016. *OU-1/OU-2 Feasibility Study Report, Anniston PCB Site.* March.

USEPA. 2017. *Record of Decision, Operable Unit 1/Operable Unit 2, Anniston PCB Site, Anniston, Calhoun County, Alabama.* November.

USEPA. 2021. *Remedial Design/Remedial Action Consent Decree*. United States of America v. Pharmacia LLC, and Solutia, Inc. Civil Action No. 1:02-CV-0749-KOB. U.S. District Court Northern District of Alabama Eastern Division. Effective date: March 26.

TABLES

Table 3-1. Stabilization/Solidification Amendments and Design Mix Ratios
OU-1/OU-2 Sediment Treatability Study Work Plan
Anniston PCB Site, Anniston, Alabama

Solidification Reagent	Design Mix (Percent Additive)[a]
Portland Cement (Type I)	2.5
	5
	7.5
Cement Kiln Dust	2.5
	5
	7.5
Calciment	2.5
	5
	7.5
High-Calcium Pebble Lime	2.5
	5
	7.5

a. The percent additive is based on the weight of the sample.

Table 3-2. Geotechnical Testing
OU-1/OU-2 Sediment Treatability Study Work Plan
Anniston PCB Site, Anniston, Alabama

Parameter	Method[a]
Moisture Content	ASTM D2216
Grain Size Distribution	ASTM D6913/D7928
Liquid Limit, Plastic Limit, and Plasticity Index (Atterberg Limits)	D4318
Hydraulic Conductivity	ASTM D5856, D2434, or D5084
Standard Procter	ASTM D698
Unconfined Compressive Strength	ASTM D2166
Undrained Shear Strength	ASTM D2573, D4648, and D2850
Triaxial Undrained Shear Strength	ASTM 4746

a. Test methods used must be appropriate for the material tested (i.e., cohesive soils vs. non-cohesive soils).

ATTACHMENT 1
STANDARD OPERATING PROCEDURE: DRAINAGE STUDY

Standard Operating Procedure

Drainage Study

1. Scope and Application

This standard operating procedure (SOP) describes the procedures for conducting a bench-scale Drainage Study as part of the treatability study program. The purpose of this study is to determine the water content of a composite sample at different time points of gravity drainage.

2. Health and Safety

All laboratory work will be performed in accordance with the Anchor QEA Environmental Geochemistry Laboratory (EGL) Chemical Hygiene Plan (CHP) by approved staff. Approval to work in the EGL requires orientation to laboratory safety procedures and potential hazards under the guidance of the Laboratory Manager, as specified in the CHP.

3. Equipment List

The following materials, as required, will be available during this procedure:

- Personal protective equipment (PPE)
- Laboratory fume hood
- Analytical balance
- No. 200 standard sieve
- Conical paint filters (60 x 60 mesh or 60 x 48 standard mesh)
- Sampling spoon
- Laboratory stands
- Funnels
- Graduated cylinders
- Graduated beaker
- Laboratory notebook

4. Drainage Study Procedure

In this test, samples representative of sediment proposed for dredging are placed atop a screen and allowed to gravity drain for 72 hours. For each bulk sediment sample, the procedures are as follows:

1. Don appropriate PPE.
2. Homogenize the samples (ensure that water content is representative of anticipated site conditions).
3. Collect a subsample for determination of total solids.

4. Collect a subsample for paint filter testing in accordance with Method 9095B, hold until the final sampling point.
5. Collect a 1-liter subsample of each sample.
6. Place the sediment material in a conical configuration in the center of a No. 200 standard sieve. Set up the apparatus to collect any water that drains from the sample during the testing in a graduated beaker.
7. Place each screen and sample in a non-drafty location or place a non-airtight cover over the sieve.
8. After drainage durations of 6, 24, 48, and 72 hours, remove a 25-gram sample from the center of the cone and determine the total solids. Measure the amount of water that drains from the sample at each time intervals.
9. Homogenize the remaining material.
10. Conduct paint filter testing on time 0- and 72-hour drainage samples.
11. Collect the water that drains from the sample for potential laboratory analytical testing for chemical parameters in accordance with the project work plan.
12. For each sample, plot the solids concentration vs time

5. References

United States Environmental Protection Agency (USEPA), 2004. SW-846 Method 9095B – Paint Filter Liquids Test. Revision 2. November 2004.

ATTACHMENT 2
STANDARD OPERATING PROCEDURE: STABILIZATION/SOLIDIFICATION TESTS

ANCHOR QEA

Standard Operating Procedure

Stabilization/Solidification Tests

1. Scope and Application

This standard operating procedure (SOP) describes the procedures for conducting bench-scale Stabilization/Solidification (S/S) Tests. These tests will evaluate effective and cost-efficient reagent doses to meet transportation and disposal landfill facility requirements for sediment to be dredged from the water body.

2. Health and Safety

All laboratory work will be performed in accordance with the EGL Chemical Hygiene Plan (CHP) by approved staff. Approval to work in the EGL requires orientation to laboratory safety procedures and potential hazards under the guidance of the Laboratory Manager, as specified in the CHP.

3. Equipment List

The following materials, as required, will be available during this procedure:

- Personal protective equipment (PPE)
- Stainless Steel mixing bowl
- Soil mixing apparatus
- Sample containers
- Paint Filter testing equipment described in Method 9095B
- Laboratory notebook

4. Test Procedure for Stabilization/Solidification

The S/S test method described below will be conducted in a laboratory bench-scale setup using sediment collected from potential dredging locations. Samples used in the test will be approximately 1 kg in weight. The tests will be conducted under three different S/S reagent percentages as described in the project work plan.

Upon receipt of the Site sediments, conduct the sediment stabilization bench test study using the following laboratory procedure:

1. Don appropriate PPE.
2. Homogenize the sediment in each container.
3. Determine percent moisture and total solids for each sample.
4. Divide each composite sample into 9 subsamples.
5. Weigh each subsample and record the mass.
6. Record any visual observations.

7. Photograph the samples.
8. Label each of the 9 subsamples as follows to indicate that amendment type and dose:
    a. Three subsamples subject to the addition of Portland cement (PC) are to be labeled with the suffixes "-PC (2.5%)", "-PC (5%)", and "-PC (7.5%)".
    b. Three subsamples subject to the addition of Cement Kiln Dust (CKD) are to be labeled with the suffixes "-CKD (2.5%)", "-CKD (5%)", and "-CKD (7.5%)".
    c. Three subsamples subject to the addition of Calciment (Calc) are to be labeled with the suffixes "-Calc (2.5%)", "-Calc (5%)", and "-Calc (7.5%)".
    d. Three subsamples subject to the addition of lime (CaO) are to be labeled with the suffixes "-CaO (2.5%)", "-CaO (5%)", and "-CaO (7.5%)".
9. Add the stabilization agents (by weight) listed in Step 8 to correspond to the sample labels specified in Step 8. Thoroughly mix the stabilization agent into the sample. For example, add 5% PC by weight to the sample labeled PC (5%).
10. Let the samples cure for a period of 12 hours, weigh each sample and then analyze each of the subsamples for percent solids (Standard Method 2540G), moisture content (ASTM - D2216), paint-filter test (USEPA Method 9095B), and strength using a pocket penetrometer.
11. Repeat the procedure listed in Step 10 after letting the samples cure for 24, 48, and 72 hours.

5. References

United States Environmental Protection Agency (USEPA), 2004. SW-846 Method 9095B – Paint Filter Liquids Test. Revision 2. November 2004.

APPENDIX D
Highway 202 Culverts Confined Space Entry Plan

HIGHWAY 202 CULVERTS CONFINED SPACE ENTRY PLAN

Operable Unit 1/Operable Unit 2 Portion of the Anniston PCB Site

Submitted by

Pharmacia LLC/Solutia Inc.

June 2022

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Table 4-1: Action Levels for Air

LIST OF ATTACHMENTS

Attachment 1: Permit-Required Confined Space Entry Permit Form

Attachment 2: Confined Space Hazard Assessment Form

Attachment 3: JSA: Working in Confined Spaces

1. INTRODUCTION

This confined space entry plan and its accompanying job safety analysis (JSA) describe potential hazards and safety procedures associated with accessing the culverts through which Snow Creek flows under Highway 202 in Anniston, Alabama. Requirements and procedures for working in confined spaces are discussed below and in the project-specific health and safety plan (HASP). This confined space entry plan for the Highway 202 culverts must be followed in conjunction with the project-specific HASP and the associated JSAs when entering any of the culverts.

Five separate concrete box culverts, each approximately 10 feet wide by 10 feet tall, pass beneath the width of Highway 202. The culverts are open at both ends and contain varying amounts of accumulated sediment. Considerable amounts of sediment have also been deposited at both the upstream and downstream ends outside the culverts. The Highway 202 culverts are considered a permit-required confined space and are not entered regularly. The activities anticipated to be conducted within this confined space include surveying, visual observation, sediment probing, and sediment sampling.

2. CONFINED SPACE DEFINITION AND REQUIREMENTS

A confined space is defined as a space that is large enough and configured so that a person can enter the space and perform assigned work, has limited means for entry or exit, and is not designed for continuous employee occupancy. A confined space is considered to have been entered as soon as any part of the entrant's body breaks the plane of an opening into the space. Confined space entry may pose the possibility of engulfment. Potentially contaminated confined spaces may pose additional hazards such as air contamination, flammable or explosive atmosphere, and oxygen deficiency.

The Site Safety Officer is responsible for identifying confined spaces into which personnel will enter. All confined spaces will be initially considered to require a permit.

Personnel must be properly trained to supervise and participate in confined space entry procedures or to serve as standby attendants. Personnel entering confined spaces or conducting activities near confined spaces must meet the regulatory requirements outlined in the Code of Federal Regulations (CFR) at 29 CFR 1910.146: Permit-required confined spaces. Each contractor participating in the confined space entry will provide qualified, competent, and trained personnel as defined in 29 CFR 1910.146, who will be responsible and have authority to perform confined space entry activities.

3. SPECIFIC MEASURES: HIGHWAY 202 CULVERTS CONFINED SPACE ENTRY

Entry into the Highway 202 culverts requires a confined space entry permit. At least two entrants and one trained attendant will be involved in the confined space entry. All entrants shall wear (in the breathing zone) a properly calibrated and bump-tested MicroRAE gas monitor (or equivalent). In addition, the attendant will monitor the air at the entry location with a MultiRAE (or equivalent) monitor. The entrants and the attendant will be equipped with radios for communication during the entry. All personnel shall wear personal protective equipment (PPE) in accordance with the project-specific HASP and anticipated hazards. At a minimum, Level D PPE shall be worn, including a hard hat, safety glasses, reflective vests, and steel-toed shoes.

Confined space entry into the Highway 202 culverts will require the following specific measures:

- Confined space entry permit (Attachment 1)
- Hazard assessment form (Attachment 2)
- JSA review (Attachment 3)
- Pre-entry briefing
- Continuous air monitoring by attendant and entrants
- Lifelines attached to a body harness on each entrant
- Radio communications, with back up air horns
- Minimal creek flow and no significant rain in the daily forecast
- Prior notification of the Anniston Fire Department and agreement that they will respond in the event of an emergency, that they are able to meet the response requirements described in 29 CFR 1926.1211, and that they are familiar with the exact location and hazards

4. AIR MONITORING REQUIREMENTS

Air monitoring prior to entry and continuous monitoring of the space during entry are required. Entrants will wear (in the breathing zone) a properly calibrated and bump-tested MicroRAE gas monitor (or equivalent), and the attendant will perform air monitoring at the entry location with a MultiRAE (or equivalent). All air monitoring instruments shall be equipped with a photoionization detector (PID), oxygen, lower explosive limit (LEL), carbon monoxide, and hydrogen sulfide sensors. The air monitoring results will be compared with the action levels and response actions listed in Table 4-1.

Table 4-1. Action Levels for Air

Parameter	Reading	Action
Volatile Organics	0 to 5 ppm	Normal operations with continuous monitoring. If getting consistent detectable readings, try to identify the source. Consider adjusting the action levels depending on possible source materials.
	5 to 20 ppm	Must investigate cause of readings to determine source
	> 20 ppm	Stop work, and contact Site Safety Officer
Hydrogen Sulfide	0 to < 5 ppm	Normal operations
	> 5 ppm	Stop work, evacuate confined spaces/work area, investigate cause of reading, and ventilate area
Oxygen	< 19.5%	Stop work, evacuate confined spaces/work area, investigate cause of reading, and ventilate area
	> 19.5% to < 23.5%	Normal operations
	> 23.5%	Stop work, evacuate confined spaces/work area, investigate cause of reading, and ventilate area
Flammable Vapors (lower explosive limit [LEL])	< 10% LEL	Normal operations
	> 10% LEL	Stop work, ventilate area, investigate source of vapors
Carbon Monoxide	0 to 20 ppm	Normal operations
	> 20 ppm	Stop work, evacuate confined spaces/work area, investigate cause of reading, and ventilate area

ppm: parts per million

5. ROLES AND RESPONSIBILITIES

The roles and responsibilities for personnel involved in the confined space entry are specified in the following subsections.

5.1 Entry Supervisor

The role and responsibilities of the entry supervisor are as follows:

- Know the hazards that may be faced during entry and the mode, signs or symptoms, and consequences of exposure to those hazards.
- Check that the entry permit has been properly prepared, all tests specified by the permit have been conducted, and all procedures and equipment specified by the permit are in place before endorsing the permit and allowing entry to begin.
- Terminate the entry and cancel the permit as required.
- Remove unauthorized individuals who enter or attempt to enter the confined space during entry operations.
- Determine that entry operations remain consistent with the terms of the entry permit and that acceptable entry conditions are maintained.
- Document on the entry permit any incidents or circumstances requiring review of the confined space entry program, including the following:
  - Unauthorized entry
  - The detection of a condition or hazard not authorized by the permit
  - The occurrence of an injury or near miss during entry
  - A change in the use or configuration of the space
  - Personnel complaints about the program
- Prescribe procedures for coordinating entry when personnel from multiple employers are working simultaneously in or around the confined space.

5.2 Authorized Entrants

The role and responsibilities of the authorized entrants include the following:

- Know the hazards that may be faced during entry and the mode, signs or symptoms, and consequences of exposure to those hazards.
- Communicate with the attendant as necessary to enable the attendant to monitor entrant status and to alert entrants of the need to evacuate the space.
- Alert the attendant whenever either of the following situations occurs:
  - The entrant recognizes warning signs or symptoms of exposure to a dangerous situation.
  - The entrant detects a prohibited condition.

- Exit the permit-required confined space as quickly as possible whenever one of the following situations occurs:
    - An order to evacuate is given by the attendant or the entry supervisor.
    - The entrant recognizes warning signs or symptoms of exposure to a dangerous situation.
    - The entrant detects a prohibited condition.
    - An evacuation alarm is activated.

5.3 Attendant(s)

The role and responsibilities of the attendants include the following:

- Know the hazards that may be faced during entry and the mode, signs or symptoms, and consequences of exposure to those hazards.
- Be aware of possible behavioral effects of hazard exposure in authorized entrants.
- Maintain an accurate count of authorized entrants in the confined space.
- Accurately identify who is in the permit space by tagging each individual's lifeline with the entrant's name and recording the names of the entrants.
- Remain outside the confined space during entry operations until relieved by another attendant.
- Communicate with the authorized entrants as necessary to monitor entrant status and to alert entrants of any important information (e.g., the need to evacuate the space).
- Monitor activities inside and outside the space to determine whether it is safe for entrants to remain in the space.
- Order the authorized entrants to evacuate the confined space immediately under any of the following conditions:
    - The attendant detects a prohibited condition.
    - The attendant detects the behavioral effects of hazard exposure in an authorized entrant.
    - The attendant detects a situation outside the space that could endanger the authorized entrants.
    - The attendant cannot effectively and safely perform all their duties.
- Summon rescue and other emergency services as soon as the attendant determines that authorized entrants may need assistance to escape from confined space hazards. Must have a positive verified means of communication to alert the Anniston Fire Department in the event of an emergency.
- Take the following actions when unauthorized persons approach or enter a confined space while entry is underway:
    - Warn the unauthorized persons that they must stay away from the confined space.

    - Advise the unauthorized persons that they must exit immediately if they have entered the permit space.
    - Inform the authorized entrants and the entry supervisor if unauthorized persons have entered the permit space.
- Perform non-entry rescues.
- Do nothing that might interfere with the attendant's primary duty to monitor and protect the authorized entrants.

6. ENTRY PROCEDURES

The following procedures are to be followed when entering the Highway 202 culverts:

1. Verify whether entry into the confined space is necessary to perform work.
2. Review creek flow conditions and schedule confined space entry for when there is no significant rain in the daily forecast.
3. Have the following minimum-required equipment on hand:
    - Ventilation (if determined necessary based on air monitoring results)
    - Barrier (tape or hard barricade) and warning signs
    - Air monitor(s) capable of measuring, at a minimum, concentrations of oxygen, flammable gases, hydrogen sulfide, and carbon monoxide
4. Inspect the confined space from the access location and eliminate any unsafe conditions prior to setting up for entry.
5. Install a barricade or tape and signs to prevent unauthorized persons from accidentally entering the space.
6. Conduct a hazard assessment that includes the following activities:
    - Test the real or potential atmospheric hazards, as follows:
        - Oxygen content: less than 19.5% or greater than 23.5%
        - Flammable gases and vapors: greater than 10% of the LEL
        - Hydrogen sulfide concentrations: 5 parts per million (ppm) or greater
        - Carbon monoxide concentrations: greater than 20 ppm
        - Volatile organic compound concentrations greater than 20 ppm.
    - Review the space for other observable serious safety and health hazards, including the following:
        - Mechanical
        - Electrical
        - Burn
        - Engulfment
        - Entrapment hazards
    - Consider the following environmental hazards:
        - Heat stress
        - Cold stress
        - Inclement weather
        - Insects and spiders

- Bees and wasps
- Ticks
- Mosquitoes
- Poisonous snakes
- Alligators
- Water-borne pathogens
- Feral dogs
- Bears
- Wolves and coyotes
- Other wildlife
- Poisonous plants
- Public at large

7. If any hazardous atmosphere exists, take the following actions:
    - If possible, determine and eliminate the source of the atmospheric hazards (e.g., carbon monoxide from a nearby truck or gas-powered generator).
    - If the atmosphere contains toxins or flammables, ventilate the space by drawing air out until the air has been changed over several times.
    - If oxygen is deficient, ventilate by pushing air into the space until the air has been changed over several times followed by monitoring to verify acceptable oxygen levels.
    - Verify the hazardous atmosphere has been eliminated by testing the air. Air monitoring results must be within acceptable limits without the use of ventilation equipment prior to entry.

8. Take the following pre-entry precautions:
    - Determine start and stop times for authorized entry.
    - Assign roles and responsibilities to entrant(s), attendant(s), and field lead(s).
    - Notify the Anniston Fire Department prior to entry and after work in the confined space is concluded. The entry must not begin and it must be terminated if the Anniston Fire Department is unable to respond at any time.
    - Notify anyone who could be affected by the entry.
    - Do not allow entry to occur or continue until any sources of energy (e.g., steam, electric, mechanical) that pose a risk to entry workers are controlled.
    - Wear appropriate PPE.
    - Provide entrants and attendants with two-way radios of the appropriate National Electrical Code rating for continuous communications.

- Provide portable air horns for use by all entrants and attendants. Horn signals will be developed and documented on the entry permit.
- Check communication devices to confirm they are functioning properly.
- Have lights available that meet the following criteria:
  - Lighting and electrical are of the appropriate National Electrical Code rating.
  - Rating should be Class I, Division I unless the space specifically meets other rating requirements at the time of entry.
- Set up non-entry rescue equipment consisting of independent lifelines attached to a harness on each entrant.
  - Lifelines must be attached to a mechanical device outside the space so that a rescue can begin as soon as the rescuer becomes aware that a rescue is necessary.
  - Lifelines at least 0.5 inches in diameter and 2,000 pounds test must be attached to a harness worn by the entrant. Wristlets may be used only when a harness presents a greater hazard to the worker and wristlets are the safest, most-effective alternative.
  - All lifelines will be secured to a fixed point outside the confined space.
- Coordinate with other contractors, as applicable.
- Have appropriate safety data sheets.
- Conduct continuous air monitoring.

9. Conduct pre-entry briefings with all personnel involved in the confined space entry. Do the following at each briefing:
   - Review the planned work scope.
   - Review confined space entry hazards, procedures, and precautions.
   - Review the confined space entry permit.
   - Review the hazard assessment.
   - Review this plan and the applicable JSAs.
   - Ensure the attendant and all entrants have signed the acknowledgement.
10. Before entry is authorized, the entry supervisor will verify and make any necessary updates to the Hazard Assessment Form.
11. Use protective barriers to protect entrants from external pedestrian, vehicle, or equipment hazards.
12. Before entry is authorized, the entry supervisor will complete and sign the entry permit according to the written confined space program and document that all pre-entry requirements have been met and that acceptable entry conditions exist. Each entry permit must be completely executed and include all required information.
13. The completed permit will be posted at the primary entrance to the permit space.

14. Entry permits are valid for a maximum of one work shift and will be canceled by the entry supervisor when the shift ends, when confined space operations are complete, or whenever a prohibited condition arises in or near the space.
15. Under the following conditions, personnel must exit the space, reevaluate hazards, and modify entry procedures:
    - Any hazardous atmosphere is detected after entry.
    - Any unanticipated health or safety hazard develops.
    - An attendant cannot effectively perform their duties.
    - Personnel in the confined space are experiencing symptoms of heat stress or overexposure to atmospheric hazards.
16. When work is completed, exit the space and close out the permit.

SPECIFIC CONFINED SPACE ENTRY PLAN ACKNOWLEDGEMENT

My signature below certifies that I have read and understand the policies and procedures specified in this Plan.

Date	Name (print)	Signature	Company

Attachment 1

Permit-Required Confined Space Entry Permit Form

Attachment 1: PRCS Entry Permit Form

1. Permit space to be entered: _____ Project Name & No.: _____
2. Purpose of entry: _____
3. Good on this date only: _____ From: _____ AM/PM _____ To: _____ AM/PM

Authorized Entrants:	Authorized Attendants:	Entry Supervisor:
_____	_____	_____
_____	_____	_____
_____	_____	_____

4. Hazards within the permit space: _____

5. Permit Space Preparation

	Yes	No
1. All energy sources locked/tagged out?	Yes	No
2. All input lines capped/blinded?	Yes	No
3. Permit Space contents drained/flushed/neutralized?	Yes	No
4. Permit Space cleaned/purged?	Yes	No
5. Ventilation provided 30 minutes before entry?	Yes	No

6. Initial atmospheric testing.

	Reading	Time	Acceptable level
Oxygen%	_____	_____	_____
LEL	_____	_____	_____
Other Contaminants	_____	_____	_____
Other Contaminants	_____	_____	_____
Other Contaminants	_____	_____	_____

Test (s) To Be Taken	Permissible Entry Levels	Test 1	Test 2	Test 3	Test 4
A. Percent of Oxygen	19.5% to 23.5%				
B. Explosivity	<10% LEL				
C. Carbon Monoxide	< 20 ppm				
D. Hydrogen Sulfide	< 5 ppm				
E. MISC.					
Name of Tester					
Test Times					

7. Rescue Services (circle one) On-Site Off-Site

Phone # for Rescue Services or means of summoning:
_____

9. Communication devices and procedures to be used during entry: _____
_____

10. Safety equipment required for entry:

PPE	N/A ( )	YES ( )	Specify_____
Testing Equipment	N/A ( )	YES ( )	Specify_____
Alarm System	N/A ( )	YES ( )	Specify_____
Rescue Equipment	N/A ( )	YES ( )	Specify_____
Other	N/A ( )	YES ( )	Specify_____

11. Additional Permits Required:

Hot work: _____ Yes _____ No
Other: _____ Yes _____ No

Attachment 1: PRCS Entry Permit Form

I certify that I have inspected the work area for safety and reviewed all safety precautions recorded on this permit.

Permit Authorization by entry Supervisor (Signature):_____ Date/Time_____

13. Permit Conditions Verification:

Physical conditions at confined space checked and verified to be in accordance with the permit Yes No
If no; please record the deviation observed and corrective action taken:

_____
_____

Verified by: _____ _____ _____
(Entry Supervisor) Date Time

14. Review & Close-Out:

Please list problems encountered during entry:

_____
_____

Corrective / Preventive Action Taken:

_____
_____

Additional Precautions / Recommendation for Future Entry:

_____
_____

Based on this review, this confined space shall be considered:

Permit Required Confined Space Yes No
Non-Permit Required Confined Space Yes No

Reviewed By: _____ _____ _____
(Entry Supervisor) Date Time

Attachment 2
Confined Space Hazard Assessment Form

Attachment 2

Confined Space Hazard Assessment Form

Part 1: Confined Space Identification

Confined space name: Highway 202 Culverts

Dimensions: Openings are approximately 10 ft x 10 ft

Description of space: The space consists of 5 separate culverts that have openings approximately 10 ft X 10 ft and traverse the width of Highway 202. The separate culverts are open on both sides. There are varying amounts of sediment buildup in each of the separate culverts.
This Hazard Assessment needs to be verified and updated as necessary in the field prior to entry.

Is this space entered on a routine basis? ☐ Yes ☒ No

Describe tasks and frequency: This is anticipated to be a one time task for sediment sampling

Part II: Nature of the Hazards Assumptions. Tanks are empty and clean, all energy sources identified and isolated, and no other hazards are introduced into the spaces. A more formal hazard assessment must be done at the time of entry.

Potential Atmospheric Hazards

- ☒ O_2 Deficient/Enriched
- ☒ Combustibles/Flammables
- ☒ CO
- ☒ H_2S
- ☒ Other Toxics

Potential Nonatmospheric Hazards

- ☐ Contains Material That Could Engulf Entrant? _____
- ☐ Internal Config. Could Trap Entrant? _____
- ☐ Electrical (live circuits)? _____
- ☐ Mechanical (pipes, linkages)? _____
- ☒ Slick/Residue Covered Surfaces? _____
- ☐ Equipment Preventing Safe Exit? _____
- ☐ Low/Inadequate Lighting? _____
- ☐ Hazardous Chemicals Present? _____
- ☐ Fall Potential? _____
- ☐ Potential for Dropped Objects? _____
- ☐ Multiple Work Groups/Nature of Work _____
- ☒ Other potential for insects and wildlife _____

Photo of Space Here



Upstream Side of Highway 202 Culvert



Downstream Side of Highway 202 Culvert

Attachment 3
JSA – Working in Confined Spaces

Job Safety Analysis

Working in Confined Spaces

Project Name: Highway 202 Culverts	**Project Number:** 171718-01.33	**JSA Number:** 001	**Issue Date:** 10/7/2021
Location: Anniston, Alabama	**Contractor:** Anchor QEA, LLC	**Analysis by:** Tim Shaner	**Analysis Date:** 10/7/2021
Work Operation: Working in Confined Spaces	**Superintendent/Competent Person:** TBD	**Revised by:**	**Revised Date:**
Required Personal Protective Equipment (PPE): • Modified Level D—Safety glasses, hard hat, safety glasses, long pants, long sleeves, and/or Tyvek coveralls if handling potentially contaminated media, and steel-toed footwear conforming to ASTM International (ASTM) F2412-05/ASTM F2413-05 • Depending on activity, the following PPE may also be required: splash goggles, nitrile outer gloves and latex inner gloves		**Reviewed by:** Christopher Yates	**Reviewed Date:** 10/12/2021
		Approved by: Chuck Guest	**Approved Date:** 10/12/2021

PLAYING IT SAFE

Job Safety Analysis

Working in Confined Spaces

Work Activity	Potential Hazards	Preventive or Corrective Measures	Inspection Requirements
Working in confined spaces	• Lack of Oxygen • Flammable or toxic atmosphere • Animals • Insects • Slipping and tripping • Temperature extremes • Flowing water	• Personnel entering the confined space will be required to complete an approved confined space training class prior to entry. • Coordinate with the site owner prior to entry. • Follow procedures to test the atmosphere for lack of oxygen, or flammable or toxic atmosphere. • Personnel should be aware of animals and pests lurking in and around the confined space. • Inspect entrances for signs of animal habitation prior to entry. • Personnel should note all areas where there is a potential for falls and take proper measures to prevent. • Personnel should take appropriate actions in areas where debris or slick surfaces are present to prevent tripping and slipping accidents. • Temperatures may vary dramatically from the outside atmosphere as there could be limited air movement. Personnel must dress accordingly for these extremes, including both cold and hot, and take other appropriate actions as necessary. • Only perform entry when water levels are low, and it has been several days since the last rain event.	• A complete understanding of all hazards • Review of entry procedures, entry plan, permit, and hazard assessment • Bump test and calibration of monitoring equipment • Verify stream flow and water depth

For more information: https://www.mvs.usace.army.mil/Portals/54/docs/expertise/Ordnance_Technical/Confined_Space_Entry.pdf

Job Safety Analysis

Working in Confined Spaces

Training Requirements:

- All staff entering confined spaces must complete approved confined space training.
- All personnel working on hazardous waste sites must receive appropriate training as required by 29 Code of Federal Regulations (CFR) 1910.120(e), including but not limited to initial 40-hour, 8-hour supervisor, and annual 8-hour refresher trainings.
- Medical clearance must be received on an annual basis as required by 29 CFR 1910.120(f).
- All assigned employees are required to familiarize themselves with the contents of this JSA, the confined space entry plan, hazard assessment, and project specific HASP before starting a work activity and review it with their supervisor during their daily safety meeting.

APPENDIX E
Quintard Mall Confined Space Entry Plan

QUINTARD MALL CULVERT: CONFINED SPACE ENTRY PLAN

Operable Unit 1/Operable Unit 2 Portion of the Anniston PCB Site

Submitted by

Pharmacia LLC/Solutia Inc.

June 2022

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LIST OF TABLES

Table 4-1: Action Levels for Air

LIST OF ATTACHMENTS

Attachment 1: Permit-Required Confined Space Entry Permit Form

Attachment 2: Confined Space Hazard Assessment Form

Attachment 3: JSA: Working in Confined Spaces

Attachment 4: Culvert Design Drawings

1. INTRODUCTION

This confined space entry plan and its accompanying job safety analysis (JSA) describe potential hazards and safety procedures associated with accessing the culvert through which Snow Creek flows under the Quintard Mall in Oxford, Alabama. Requirements and procedures for working in confined spaces are discussed below and in the project-specific health and safety plan (HASP). This confined space entry plan must be followed in conjunction with the project-specific HASP and associated JSAs when entering the culvert.

The Quintard Mall culvert conveys Snow Creek stream flow for approximately 0.3 miles beneath a portion of the parking lot and mall structure. The culvert is considered a permit-required confined space with unique hazards. There are only two entry points to the culvert, at the upstream and downstream ends. This confined space is not entered regularly. The activities anticipated to be conducted within this confined space include surveying, sediment probing, and sediment sampling.

The Quintard Mall culvert is accessible during periods of low or normal stream flow. At a minimum, access to the culvert can be made by walking into the culvert from the downstream end. Details for access from the upstream end of the culvert will be confirmed during predesign investigation (PDI) preparation tasks. Based on culvert foundation plan design drawings, the Quintard Mall culvert is trapezoidal with an upper width of approximately 100 feet. The height of the culvert varies and is estimated to be between 9 and 11 feet tall. Dimensions of the culvert will be confirmed during the PDI.

2. CONFINED SPACE DEFINITION AND REQUIREMENTS

A confined space is defined as a space large enough and configured so that a person can enter the space and perform assigned work, has limited means for entry or exit, and is not designed for continuous employee occupancy. A confined space is considered to have been entered as soon as any part of the entrant's body breaks the plane of an opening into the space. Confined space entry may pose the possibility of engulfment. Potentially contaminated confined spaces may pose additional hazards such as air contamination, flammable or explosive atmosphere, and oxygen deficiency.

The Site Safety Officer is responsible for identifying confined spaces into which personnel will enter. All confined spaces will be initially considered to require a permit.

Personnel must be properly trained to supervise and participate in confined space entry procedures or to serve as standby attendants. Personnel entering confined spaces or conducting activities near confined spaces must meet the regulatory requirements outlined in the Code of Federal Regulations (CFR) at 29 CFR 1910.146: Permit-required confined spaces. Each contractor participating in the confined space entry will provide qualified, competent, and trained personnel as defined in 29 CFR 1910.146, who will be responsible and have authority to perform confined space entry activities.

3. SPECIFIC MEASURES: QUINTARD MALL CONFINED SPACE ENTRY

Due to the potentially unique hazards of this confined space, a sewer inspection contractor, or the equivalent, with remotely operated video surveying equipment is to be used. The confined space will initially be surveyed remotely prior to allowing entrants into the space. Depending on the results of the remote survey, the Site Safety Officer, Entry Supervisor, and a Structural Engineer will jointly determine whether it is acceptable for entry to be made. To the extent possible, work in the space should be limited or performed remotely.

Entry into this confined space will require a confined space entry permit, at least two entrants, and at least two trained attendants (one at the upstream end of the culvert and one at the downstream end). All entrants shall wear (in the breathing zone) a properly calibrated and bump-tested MicroRAE gas monitor (or equivalent). In addition, the attendants will monitor air at the entry location with a MultiRAE (or equivalent) monitor. The entrants and attendants will be equipped with radios for communication during the entry. All personnel shall wear personal protective equipment (PPE) in accordance with the project-specific HASP and anticipated hazards. At a minimum, Level D PPE shall be worn, including a hard hat, safety glasses, steel-toed shoes, reflective vests, and a minimum of a 10-minute Immediately Dangerous to Life or Health (IDLH) emergency escape respirator. Entrants are to travel only halfway into the space, then return to the entry point. The other half of the confined space will be entered from the opposite side, again not traveling more than halfway into the culvert.

Confined space entry into the Quintard Mall culvert will require the following specific measures:

- Confined-space entry permit (Attachment 1)
- Hazard assessment form (Attachment 2)
- JSA review (Attachment 3)
- Culvert design drawings (Attachment 4)
- Pre-entry briefing
- Continuous air monitoring by attendants and entrants
- Emergency escape respirator
- Lifelines attached to a body harness on each entrant
    - Minimum of 1,800 feet in length and marked every 10 feet for the first 100 feet, then every 50 feet for the remaining length, so the attendant can track and call out how far the entrants are into the space
- Radio communications with back-up air horns
- Minimal creek flow and no significant rain in the daily forecast
- Prior notification of the Oxford Fire Department and agreement that they will respond in the event of an emergency, that they are able to meet the response requirements described in 29 CFR 1926.1211, and that they are familiar with the exact location and hazards.

4. AIR MONITORING REQUIREMENTS

Air monitoring prior to entry and continuous monitoring of the space during entry are required. Entrants will wear (in the breathing zone) a properly calibrated and bump-tested MicroRAE gas monitor (or equivalent), and the attendant will monitor air at the entry location with a MultiRAE (or equivalent) monitor. Air monitoring instruments shall be equipped with a photoionization detector (PID), oxygen, lower explosive limit (LEL), carbon monoxide, and hydrogen sulfide sensors. The air monitoring results will be compared with the action levels and response actions listed in Table 4-1. If at any time air monitoring results trigger an action to stop work, the entrants will don their escape respirator and exit the space.

Table 4-1. Action Levels for Air

Parameter	Reading	Action
Volatile Organics	0 to 5 ppm	Normal operations with continuous monitoring. If getting consistent detectable readings, try to identify the source. Consider adjusting the action levels depending on possible source materials.
	5 to 20 ppm	Must investigate cause of readings to determine source
	> 20 ppm	Stop work, and contact the Site Safety Officer
Hydrogen Sulfide	0 to < 5 ppm	Normal operations
	> 5 ppm	Stop work, evacuate confined spaces/work area, investigate cause of reading, and ventilate area
Oxygen	< 19.5%	Stop work, evacuate confined spaces/work area, investigate cause of reading, and ventilate area
	> 19.5% to < 23.5%	Normal operations
	> 23.5%	Stop work, evacuate confined spaces/work area, investigate cause of reading, and ventilate area
Flammable Vapors (LEL)	< 10% LEL	Normal operations
	> 10% LEL	Stop work, ventilate area, investigate source of vapors

Table 4-1. Action Levels for Air

Parameter	Reading	Action
Carbon Monoxide	0 to ≤ 20 ppm	Normal operations
	> 20 ppm	Stop work, evacuate confined spaces/work area, investigate cause of reading, and ventilate area

LEL: lower explosive limit
ppm: parts per million

5. ROLES AND RESPONSIBILITIES

The roles and responsibilities for personnel involved in the confined space entry are specified in the following subsections.

5.1 Entry Supervisor

The role and responsibilities of the entry supervisor are as follows:

- Know the hazards that may be faced during entry and the mode, signs or symptoms, and consequences of exposure to those hazards.
- Check that the entry permit has been properly prepared, all tests specified by the permit have been conducted, and all procedures and equipment specified by the permit are in place before endorsing the permit and allowing entry to begin.
- Terminate the entry and cancel the permit as required.
- Remove unauthorized individuals who enter or attempt to enter the confined space during entry operations.
- Determine that entry operations remain consistent with the terms of the entry permit and that acceptable entry conditions are maintained.
- Document on the entry permit any incidents or circumstances requiring review of the confined space entry program, which include the following:
  - Unauthorized entry
  - The detection of a condition of hazard not authorized by the permit
  - The occurrence of an injury or near miss during entry
  - A change in the use or configuration of the space
  - Personnel complaints about the program
- Prescribe procedures for coordinating entry when personnel from multiple employers are working simultaneously in or around the confined space.

5.2 Authorized Entrants

The role and responsibilities of the authorized entrants include the following:

- Know the hazards that may be faced during entry and the mode, signs or symptoms, and consequences of exposure to those hazards.
- Communicate with the attendant as necessary to enable the attendant to monitor entrant status and to alert entrants of the need to evacuate the space.
- Alert the attendant whenever either of the following situations occurs:
  - The entrant recognizes warning signs or symptoms of exposure to a dangerous situation.
  - The entrant detects a prohibited condition.

- Exit from the permit-required confined space as quickly as possible whenever one of the following situations occurs:
  - An order to evacuate is given by the attendant or the entry supervisor.
  - The entrant recognizes warning signs or symptoms of exposure to a dangerous situation.
  - The entrant detects a prohibited condition.
  - An evacuation alarm is activated.

5.3 Attendant(s)

The role and responsibilities of the attendants include the following:

- Know the hazards that may be faced during entry and the mode, signs or symptoms, and consequences of exposure to those hazards.
- Be aware of possible behavioral effects of hazard exposure in authorized entrants.
- Maintain an accurate count of authorized entrants in the confined space.
- Accurately identify who is in the permit space by tagging each individual's lifeline with the entrant's name and recording the names of the entrants.
- Remain outside the permit space during entry operations until relieved by another attendant.
- Communicate with authorized entrants as necessary to monitor entrant status and to alert entrants of any important information (e.g., the need to evacuate the space).
- Monitor activities inside and outside the space to determine whether it is safe for entrants to remain in the space.
- Order the authorized entrants to evacuate the permit space immediately under any of the following conditions:
  - The attendant detects a prohibited condition.
  - The attendant detects the behavioral effects of hazard exposure in an authorized entrant.
  - The attendant detects a situation outside the space that could endanger the authorized entrants.
  - The attendant cannot effectively and safely perform all their duties.
- Summon rescue and other emergency services as soon as the attendant determines that authorized entrants may need assistance to escape from confined space hazards. Must have a positive verified means of communication to alert the Oxford Fire Department in the event of an emergency
- Take the following actions when unauthorized persons approach or enter a confined space while entry is underway:
  - Warn the unauthorized persons that they must stay away from the confined space.

    - Advise the unauthorized persons that they must exit immediately if they have entered the permit space.
    - Inform the authorized entrants and the entry supervisor if unauthorized persons have entered the permit space.

- Perform non-entry rescues.
- Do nothing that might interfere with the attendant's primary duty to monitor and protect the authorized entrants.

6. ENTRY PROCEDURES

The following procedures are to be followed when entering the Quintard Mall culvert:

1. Verify whether entry into the confined space is necessary to perform work.
2. Review creek flow conditions and schedule confined space entry for when there is no significant rain in the daily forecast.
3. Have the following minimum-required equipment on hand:
    - 10-minute IDLH emergency escape respirators
    - Ventilation (if determined necessary based on air monitoring results)
    - Barrier (tape or hard barricade) and warning signs
    - Air monitor(s) capable of measuring, at a minimum, concentrations of oxygen, flammable gases, hydrogen sulfide and carbon monoxide.
4. Inspect the confined space and eliminate any unsafe conditions prior to setting up for entry.
5. Install a barricade or tape and signs to prevent people from accidentally entering the space.
6. Conduct a hazard assessment that includes the following activities:
    - Test the real or potential atmospheric hazards, as follows:
        - Oxygen content: less than 19.5% or greater than 23.5%
        - Flammable gases and vapors: greater than 10% of the LEL
        - Hydrogen sulfide concentrations: 5 parts per million (ppm) or greater
        - Carbon monoxide concentrations: greater than 20 ppm
        - Volatile organic compound concentrations greater than 20 ppm.
    - Review the space for other observable serious safety and health hazards, including the following:
        - Mechanical
        - Electrical
        - Burn
        - Engulfment
        - Entrapment hazards

- Consider the following environmental hazards:
    - Heat stress
    - Cold stress
    - Inclement weather
    - Insects and spiders

- Bees and wasps
- Ticks
- Mosquitoes
- Poisonous snakes
- Alligators
- Water-borne pathogens
- Feral dogs
- Bears
- Wolves and coyotes
- Other wildlife
- Poisonous plants
- Public at large

7. If any hazardous atmosphere exists, take the following actions:
   - If possible, determine and eliminate the source of the atmospheric hazards (e.g., carbon monoxide from a nearby truck or gas-powered generator).
   - If the atmosphere contains toxins or flammables, ventilate the space by drawing air out until the air has been changed over several times.
   - If oxygen deficient, ventilate by pushing air into the space until the air has been changed over several times followed by monitoring to verify acceptable oxygen levels.
   - Verify the hazardous atmosphere has been eliminated by testing the air. Air monitoring results must be within acceptable limits without the use of ventilation equipment prior to entry.

8. Take the following pre-entry precautions:
   - Determine start and end times for authorized entry.
   - Assign roles and responsibilities as entrant(s), attendant(s), field lead(s).
   - Notify the Oxford Fire Department prior to entry and when work in the confined space is concluded. The entry must not begin and it must be terminated if the Oxford Fire Department is unable to respond at any time.
   - Notify anyone who could be affected by the entry.
   - Do not allow entry to occur or continue until any sources of energy (e.g., steam, electric, mechanical) that pose a risk to entry workers are controlled.
   - Wear appropriate PPE.
   - Provide entrants and attendants with two-way radios of the appropriate National Electrical Code rating for continuous communications.

- Provide portable air horns for use by all entrants and attendants. Horn signals will be developed and documented on the entry permit.
- Check communication devices to confirm they are functioning properly.
- Have hand and head lights required for entry into the confined space that meet the following criteria:
  - Lighting and electrical are of the appropriate National Electrical Code rating.
  - Rating should be Class I, Division I unless the space specifically meets other rating requirements at the time of entry.
- Set up non-entry rescue equipment consisting of independent lifelines attached to a harness on each entrant.
  - Lifelines must be attached to a mechanical device outside the space such that a rescue can begin as soon as the rescuer becomes aware that a rescue is necessary.
  - Lifelines at least 0.5 inches in diameter and 2,000 pounds test must be attached to a harness worn by the entrant. Wristlets may be used only when a harness presents a greater hazard to the worker and wristlets are the safest, most-effective alternative.
  - All lifelines will be secured to a fixed point outside the confined space.
- Coordinate with other contractors, as applicable.
- Have appropriate safety data sheets.
- Continuous air monitoring will be conducted.

9. Conduct pre-entry briefing with all personnel involved in the confined space entry. Do the following at each briefing:
   - Review the planned work scope.
   - Review confined space entry hazards, procedures, and precautions.
   - Review the confined space entry permit.
   - Review the hazard assessment.
   - Review this plan and applicable JSAs.
   - Ensure the attendant and all entrants have signed the acknowledgement.
10. Before entry is authorized, the entry supervisor will verify and make any necessary updates to the Hazard Assessment Form.
11. Use protective barriers to protect entrants from external pedestrian, vehicle, or equipment hazards.
12. Before entry is authorized, the entry supervisor will complete and sign the entry permit according to the written confined space program and document that all pre-entry requirements have been met and that acceptable entry conditions exist. Each entry permit must be completely executed and include all required information.

13. The completed permit will be posted at the primary entrance to the permit space.
14. Entry permits are valid for a maximum of one work shift and will be canceled by the entry supervisor when the shift ends, when confined space operations are complete, or whenever a prohibited condition arises in or near the space.
15. Under the following conditions, personnel must exit the space, reevaluate hazards, and modify entry procedures:
    - Any hazardous atmosphere is detected after entry.
    - Any unanticipated health or safety hazard develops.
    - An attendant cannot effectively perform their duties.
    - Personnel in confined space are experiencing symptoms of heat stress or overexposure to atmospheric hazards.
16. When work is completed, exit the space and close out the permit.

SPECIFIC CONFINED SPACE ENTRY PLAN ACKNOWLEDGEMENT

My signature below certifies that I have read and understand the policies and procedures specified in this Plan.

Date	Name (print)	Signature	Company

Attachment 1

Permit-Required Confined Space Entry Permit Form

Attachment 1: PRCS Entry Permit Form

1. Permit space to be entered: _____ Project Name & No.: _____
2. Purpose of entry: _____
3. Good on this date only: _____ From: _____ AM/PM _____ To: _____ AM/PM

Authorized Entrants:	Authorized Attendants:	Entry Supervisor:
_____	_____	_____
_____	_____	_____
_____	_____	_____

4. Hazards within the permit space: _____

5. Permit Space Preparation

	Yes	No
1. All energy sources locked/tagged out?	Yes	No
2. All input lines capped/blinded?	Yes	No
3. Permit Space contents drained/flushed/neutralized?	Yes	No
4. Permit Space cleaned/purged?	Yes	No
5. Ventilation provided 30 minutes before entry?	Yes	No

6. Initial atmospheric testing.

	Reading	Time	Acceptable level
Oxygen%	_____	_____	_____
LEL	_____	_____	_____
Other Contaminants	_____	_____	_____
Other Contaminants	_____	_____	_____
Other Contaminants	_____	_____	_____

Test (s) To Be Taken	Permissible Entry Levels	Test 1	Test 2	Test 3	Test 4
A. Percent of Oxygen	19.5% to 23.5%				
B. Explosivity	<10% LEL				
C. Carbon Monoxide	< 20 ppm				
D. Hydrogen Sulfide	< 5 ppm				
E. MISC.					
Name of Tester					
Test Times					

7. Rescue Services (circle one) On-Site Off-Site

Phone # for Rescue Services or means of summoning:
_____

9. Communication devices and procedures to be used during entry: _____
_____

10. Safety equipment required for entry:

PPE	N/A ( )	YES ( )	Specify_____
Testing Equipment	N/A ( )	YES ( )	Specify_____
Alarm System	N/A ( )	YES ( )	Specify_____
Rescue Equipment	N/A ( )	YES ( )	Specify_____
Other	N/A ( )	YES ( )	Specify_____

11. Additional Permits Required:

Hot work: _____ Yes _____ No
Other: _____ Yes _____ No

Attachment 1: PRCS Entry Permit Form

I certify that I have inspected the work area for safety and reviewed all safety precautions recorded on this permit.

Permit Authorization by entry Supervisor (Signature):_____ Date/Time_____

13. Permit Conditions Verification:

Physical conditions at confined space checked and verified to be in accordance with the permit Yes No
If no; please record the deviation observed and corrective action taken:

_____
_____

Verified by: _____ _____ _____
(Entry Supervisor) Date Time

14. Review & Close-Out:

Please list problems encountered during entry:

_____
_____

Corrective / Preventive Action Taken:

_____
_____

Additional Precautions / Recommendation for Future Entry:

_____
_____

Based on this review, this confined space shall be considered:

Permit Required Confined Space Yes No
Non-Permit Required Confined Space Yes No

Reviewed By: _____ _____ _____
(Entry Supervisor) Date Time

Attachment 2
Confined Space Hazard Assessment Form

Attachment 2

Confined Space Hazard Assessment Form

Part 1: Confined Space Identification

Confined space name: Quintard Mall Culvert

Dimensions: The bridge traverses approximately 0.3 miles

Description of space: The space is under a bridge that traverses approximately 0.3 miles under portions of the Quintard Mall structure and parking area. Conditions under most of the bridge are unknown. This Hazard Assessment needs to be verified and updated as necessary in the field prior to entry.

Is this space entered on a routine basis? ☐ Yes ☒ No

Describe tasks and frequency: This is anticipated to be a one time task for sediment sampling

Part II: Nature of the Hazards Assumptions. Tanks are empty and clean, all energy sources identified and isolated, and no other hazards are introduced into the spaces. A more formal hazard assessment must be done at the time of entry.

Potential Atmospheric Hazards

- ☒ O_2 Deficient/Enriched
- ☒ Combustibles/Flammables
- ☒ CO
- ☒ H_2S
- ☒ Other Toxics

Potential Nonatmospheric Hazards

- ☒ Contains Material That Could Engulf Entrant? Unknown
- ☒ Internal Config. Could Trap Entrant? Unknown
- ☐ Electrical (live circuits)? _____
- ☐ Mechanical (pipes, linkages)? _____
- ☒ Slick/Residue Covered Surfaces? _____
- ☐ Equipment Preventing Safe Exit? _____
- ☒ Low/Inadequate Lighting? Only natural light at entry locations
- ☐ Hazardous Chemicals Present? _____
- ☒ Fall Potential? Unknown
- ☒ Potential for Dropped Objects? Unknown
- ☒ Multiple Work Groups/Nature of Work Inspection contractor
- ☒ Other potential for insects and wildlife

Photo of Space Here







Attachment 3
JSA – Working in Confined Spaces

Job Safety Analysis

Working in Confined Spaces

Project Name: Quintard Mall Bridge	**Project Number:** 171718-01.33	**JSA Number:** 001	**Issue Date:** 10/7/2021
Location: Oxford, Alabama	**Contractor:** Anchor QEA, LLC	**Analysis by:** Tim Shaner	**Analysis Date:** 10/7/2021
Work Operation: Working in Confined Spaces	**Superintendent/Competent Person:** TBD	**Revised by:**	**Revised Date:**
Required Personal Protective Equipment (PPE): • Modified Level D—Safety glasses, hard hat, safety glasses, long pants, long sleeves, and/or Tyvek coveralls if handling potentially contaminated media, steel-toed footwear conforming to ASTM International (ASTM) F2412-05/ASTM F2413-05, at a 10-minute IDLH emergency escape respirator (at a minimum). • Depending on activity, the following PPE may also be required: splash goggles, nitrile outer gloves and latex inner gloves		**Reviewed by:** Christopher Yates	**Reviewed Date:** 10/12/2021
		Approved by: Charles Guest	**Approved Date:** 10/12/2021

PLAYING IT SAFE

Job Safety Analysis

Working in Confined Spaces

Work Activity	Potential Hazards	Preventive or Corrective Measures	Inspection Requirements
Working in confined spaces	• Lack of Oxygen • Flammable or toxic atmosphere • Animals • Insects • Slipping and tripping • Temperature extremes • Flowing water	• Personnel entering the confined space will be required to complete an approved confined space training class prior to entry. • Coordinate with the site owner prior to entry. • Follow procedures to test the atmosphere for lack of oxygen, or flammable or toxic atmosphere. • Personnel should be aware of animals and pests lurking in and around the confined space. • Inspect entrances for signs of animal habitation prior to entry. • Personnel should note all areas where there is a potential for falls and take proper measures to prevent. • Personnel should take appropriate actions in areas where debris or slick surfaces are present to prevent tripping and slipping accidents. • Temperatures may vary dramatically from the outside atmosphere as there could be limited air movement. Personnel must dress accordingly for these extremes, including both cold and hot, and take other appropriate actions as necessary. • Only perform entry when water levels are low, and it has been several days since the last rain event.	• A complete understanding of all hazards • Review of entry procedures, entry plan, permit, and hazard assessment • Bump test and calibration of monitoring equipment • Verify stream flow and water depth

For more information: https://www.mvs.usace.army.mil/Portals/54/docs/expertise/Ordnance_Technical/Confined_Space_Entry.pdf

Job Safety Analysis

Working in Confined Spaces

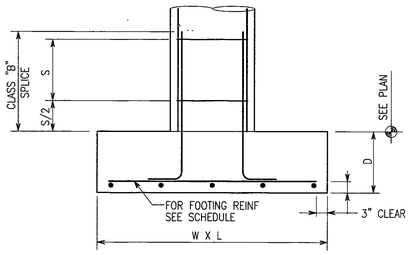
Training Requirements:

- All staff entering confined spaces must complete approved confined space training.
- All personnel must be trained on how to properly use the IDLH emergency escape respirator.
- All personnel working on hazardous waste sites must receive appropriate training as required by 29 Code of Federal Regulations (CFR) 1910.120(e), including but not limited to initial 40-hour, 8-hour supervisor, and annual 8-hour refresher trainings.
- Medical clearance must be received on an annual basis as required by 29 CFR 1910.120(f).
- All assigned employees are required to familiarize themselves with the contents of this JSA, the confined space entry plan, hazard assessment, and project specific HASP before starting a work activity and review it with their supervisor during their daily safety meeting.

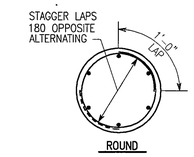
Attachment 4
Culvert Design Drawings

ABBREVIATIONS

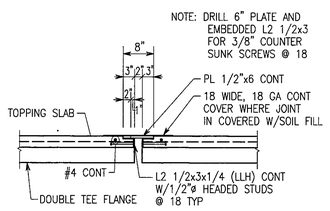
ABOVE FINISH FLOOR	- AFF	KIPS (1000 LBS)	- K
ADDITIONAL	- ADDN	KIPS PER LINEAL FOOT	- KLF
ADJACENT	- ADJ	KIPS PER SQUARE INCH	- KSI
AIR CONDITIONER	- A/C	KIPS PER SQUARE FOOT	- KSF
AIR HANDLING UNIT	- AHU		
ALTERNATE	- ALT	LIGHTWEIGHT CONCRETE	- LWT CONC
ANCHOR	- ANC	LIVE LOAD	- LL
ANCHOR BOLT	- AB	LONGITUDINAL	- LONG.
AND	- &	LONG LEG HORIZONTAL	- LLH
ANGLE	- L	LONG LEG VERTICAL	- LLV
APPROVED	- APPRV		
APPROXIMATE	- APPROX	MANUFACTURE(R)	- MFR
ARCHITECTURAL	- ARCH.	MASONRY	- MAS
AT (WHEN INDICATING SPACING ONLY)	- @	MASONRY OPENING	- MO
		MATERIAL	- MATL
		MAXIMUM	- MAX
BACK TO BACK	- B TO B	MECHANICAL	- MECH
BALANCE	- BAL	MEZZANINE	- MEZZ
BASEMENT	- BSMT	MIDDLE	- MID
BASE PLATE	- BSPL	MINIMUM	- MIN
BEAM	- BM	MISCELLANEOUS	- MISC
BEARING	- BRG	MOMENT	- M
BELOW FINISH FLOOR	- BFF	MOMENT CONNECTION(S)	- MC
BETWEEN	- BTWN		
BLOCK	- BLK	NEAR FACE	- NF
BLOCKING	- BLKG	NEAR SIDE	- NS
BOTTOM	- BOT	NOMINAL	- NOM
BOTTOM CHORD EXTENSION	- BCX	NOT IN CONTRACT	- NIC
BRICK	- BRK	NOT TO SCALE	- NTS
BRIDGING	- BRDG	NUMBER	- NO. or #
BUILDING	- BLDG		
		ON CENTER	- OC
CENTER	- CTR	OPENING(S)	- OPNG(S)
CENTERLINE	- CL	OPPOSITE	- OPP
CENTER TO CENTER	- C TO C	OUTSIDE FACE	- O.F.
CHANNEL	- C	OUTSIDE DIAMETER	- OD
CLEAR OR CLEARANCE	- CLR	OUTSTANDING LEG	- OSL
COLUMN	- COL		
COMPLETE JOINT PENETRATION	- CJP	PARALLEL	- PAR.
COMPRESSION	- COMP	PARTITION(S)	- PARTN(S)
CONCRETE	- CONC	PENETRATION	- PEN
CONCRETE MASONRY UNIT	- CMU	PERMANENT	- PERM
CONNECTION(S)	- CONN(S)	PERPENDICULAR	- PERP
CONTINUOUS	- CONT	PLATE	- PL
CONTRACTOR	- CONTR	PLUMBING	- PLBG
CONSTRUCTION	- CONST	PNEUMATIC	- PNEU
CONTROL JOINT	- CJ	POST-TENSION	- PT
CORNER	- COR	POUNDS	- LBS
COVER PLATE	- COV PL	POUNDS PER LINEAL FOOT	- PLF
		POUNDS PER SQUARE INCH	- PSI
DEGREE	- DEG OR °	POUNDS PER SQUARE FT	- PSF
DETAIL	- DET	POUNDS PER CUBIC YARD	- PCY
DEAD LOAD	- DL	PRECAST CONCRETE	- PC
DIAGONAL	- DIAG	PREFABRICATED	- PREFAB
DIAMETER	- DIA or ø	PRELIMINARY	- PRELIM
DIMENSION(S)	- DIM(S)	PRESSURE INJECTED FOOTING	- PIF
DOVETAIL	- DVTL	PROJECTION	- PROJ
DRAWING(S)	- DWG(S)		
DOUBLE	- DBL	RADIUS	- RAD
DOUBLE EXTRA STRONG	- XXS	REFERENCE	- REF
DOWEL(S)	- DWL(S)	REINFORCED CONC PIPE	- RCP
DOWN	- DN	REINFORCING	- REINF
		REQUIRED	- REQD
EACH	- EA	RISER	- RIS
EACH FACE	- EF	ROOF	- RF
EACH WAY	- EW	ROOF DRAIN	- RD
ELECTRICAL	- ELEC	ROOF TOP UNIT	- RTU
ELEVATION	- EL	ROOM	- RM
ELEVATOR	- ELEV	ROUND	- RND
ENGINEER	- ENGR		
EQUAL	- EQ	SCHEDULE	- SCHED
EQUIPMENT	- EQUIP.	SECTION	- SECT
EXISTING	- EXIST.	SHEAR	- V
EXPANSION	- EXP	SHEET	- SHT
EXPANSION ANCHOR	- EXP ANC	SIMILAR	- SIM
EXPANSION JOINT	- EJ	SPACE	- SP
EXTENSION	- EXTN	SPECIFICATION(S)	- SPEC(S)
EXTERIOR	- EXT	SPECIFIED	- SPECD
EXTRA STRONG	- XS	SQUARE	- SQ
		STANDARD	- STD
FABRICATOR	- FABR	STEEL	- STL
FACE TO FACE	- F TO F	STIFFENER	- STIFF.
FAR SIDE	- FS	STRAIGHT	- STR
FASTENER	- FAS	STIRRUPS	- STIR.
FIELD VERIFY	- FV	STRUCTURE OR STRUCT'L	- STRUCT
FINISH(ED)	- FIN.	SYMMETRICAL	- SYM
FLANGE	- FLG	SUPPORT(S)	- SUPT(S)
FLOOR	- FL		
FLOOR DRAIN	- FD	TEMPERATURE	- TEMP
FOOT	- FT	TENSION	- T
FOOTING	- FTG	THICK	- THK
FOUNDATION	- FDN	TONGUE AND GROOVE	- T&G
FRAMING	- FRMG	TOP AND BOTTOM	- T&B
		TOP CHORD EXTENSION	- TCX
GAGE OR GAUGE	- GA	TOP OF BEAM	- TOB
GALVANIZED	- GALV	TOP OF FOOTING	- TOF
GENERAL	- GEN	TOP OF JOIST	- TOJ
GOVERNMENT	- GOVT	TOP OF PIER	- T. O. P.
GRADE	- GR	TOP OF PILE CAP	- TOPC
GRADE BEAM	- GR BM	TOP OF STEEL	- TOS
GROUND	- GRD	TOP OF WALL	- TOW
		TREAD	- TR
HARD ROCK	- HD RK	TYPICAL	- TYP
HEADED STUD(S)	- H STUD(S)		
HEIGHT	- HT	UNLESS NOTED	- U. N.
HIGH STRENGTH	- HS		
HOOK	- HK	VERTICAL	- VERT
HORIZONTAL	- HORZ		
		WATERPROOFING	- WPFG
INFORMATION	- INFO	WATERSTOP	- WS
INSIDE DIAMETER	- ID	WELDED WIRE FABRIC	- WWF
INSIDE FACE	- I. F.	WIND LOAD	- WL
INTERIOR	- INT	WINDOW	- WDW
INTERMEDIATE	- INTM	WITH	- W/
		WITHOUT	- W/O
JOINT	- JT	WOOD	- WD
JOIST(S)	- JST(S)	WORK POINT	- WP
JOIST GIRDER	- JG	WEIGHT	- WT



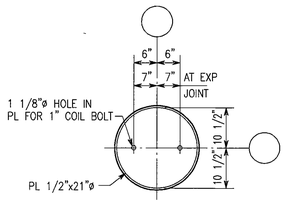
FOOTING DETAIL
TYPICAL



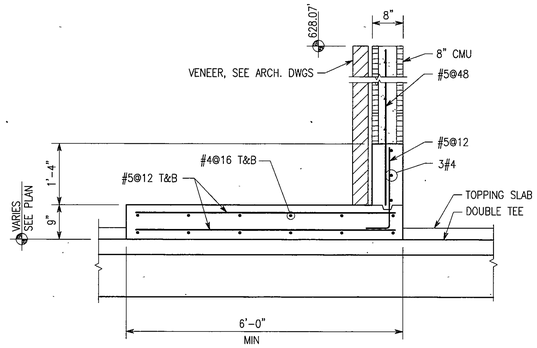
COLUMN TIE DETAIL
TYPICAL



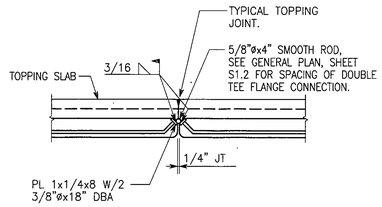
EXPANSION JOINT COVER
TYPICAL



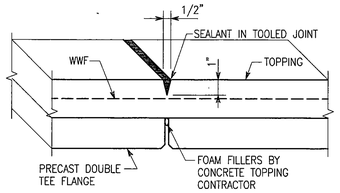
BEARING PLATE ON 22" DIAMETER COLUMN
TYPICAL



SCREEN WALL DETAIL AT SERVICE AREAS
TYPICAL
(FOR LOCATION SEE CIVIL DRAWINGS)

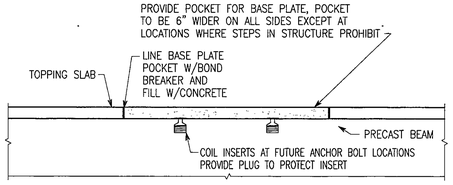


DOUBLE TEE FLANGE CONNECTION DETAIL 1 1/2"=1'-0"
TYPICAL



CONTROL JOINT DETAIL 3"=1'-0"
TYPICAL

CONTROL JOINT NOTES:
1. JOINT IS TOOLED IN TOPPING WITH A GOLDBLATT GROOVER 06-314-M7 IN THE PLANE WHERE TEES ABUT, OVER THE END OF EVERY TEE AND OVER THE ENDS OF BEAMS.
2. ALL JOINTS ARE TO BE DRESSED WITH THE ISO-FLEX VEE ABRASIVE BLADE TO REMOVE LAITANCE, CURING COMPOUNDS AND DRESS THE JOINT PRIOR TO SEALING.
3. JOINTS ARE TO BE PRIMED IN ACCORDANCE WITH MANUFACTURERS INSTRUCTIONS AND SEALED WITH ISO-FLEX 880 G.B. URETHANE SEALANT OR APPROVED EQUAL.
4. JOINTS PROVIDED BY SAWING ARE AN UNACCEPTABLE ALTERNATIVE DUE TO PROBLEMS RELATED TO: A) EDGE SPALLING, B) LACK OF CONTROL WHEN SAWING IS DONE, C) LOCATION DIFFICULTIES, D) CUTTING OF STEEL MESH.



TOPPING SLAB POCKET AT FUTURE MALL COLUMN
TYPICAL

CLASS B TENSION LAP SPLICE LENGTHS

CONCRETE f'c	BAR SIZE						
	#5	#6	#7	#8	#9	#10	#11
3000	27	32	38	43	48	58	71
4000	23	28	33	37	42	50	62
5000	21	25	29	33	38	45	55
6000	19	23	27	32	41	51	63
8000	19	23	27	30	34	39	44

COLUMN SCHEDULE

DESIGNATION	TYPICAL	COLUMN AT RIP RAP ALTERNATE
SIZE	22"ø	22"ø
VERTICAL REINF	5#8	6#8
TIE SPACINGS	#3@16	#3@16

GENERAL NOTES

1. GENERAL

1.1 DESIGN CRITERIA:

A. CODES AND SPECIFICATIONS:

1. BRIDGE SPECIFICATION FOR EXTERIOR AREAS: AMERICAN ASSOCIATION OF STATE HIGHWAY AND TRANSPORTATION OFFICIALS STANDARD SPECIFICATIONS FOR HIGHWAY BRIDGES, 1989.
2. GENERAL BUILDING CODE FOR COVERED MALL: STANDARD BUILDING CODE, 1991.
3. CONCRETE: BUILDING CODE REQUIREMENTS FOR REINFORCED CONCRETE (ACI 318-89 REVISED 1992).
4. STRUCTURAL STEEL: SPECIFICATION FOR STRUCTURAL STEEL BUILDINGS, ALLOWABLE STRESS DESIGN AND PLASTIC DESIGN, JUNE 1, 1989, AMERICAN INSTITUTE OF STEEL CONSTRUCTION.

B. DESIGN LOADS (PSF):

1. DEAD LOADS: SELF WEIGHT OF PRECAST CONCRETE FRAMING PLUS TOPPING SLAB. ANY CHANGES IN CONSTRUCTION MATERIALS FROM THOSE SHOWN ON THE STRUCTURAL DRAWINGS SHALL BE REPORTED BY THE GENERAL CONTRACTOR TO THE STRUCTURAL ENGINEER FOR VERIFICATION OF LOAD CARRYING CAPACITY OF THE STRUCTURE.
2. SUPERIMPOSED DEAD AND LIVE LOADS: SUPERIMPOSED LOADS VARY ALONG LENGTH OF THE BRIDGE. FOR LOADING TYPE, LOCATION, AND EXTENT, SEE SHEET S1.2.

1.2 SUBMIT ALL SHOP DRAWINGS ON ONE REPRODUCIBLE PRINT (SEPIA) AND ONE BLUE LINE PRINT ONLY. THE REPRODUCIBLE PRINT WILL BE RETURNED. ALL BLUE LINE PRINTS REQUIRED BY THE CONTRACTOR ARE THE RESPONSIBILITY OF THE CONTRACTOR AND SHOULD BE MADE AFTER THE REPRODUCIBLE PRINT IS RETURNED. IF ADDITIONAL BLUE LINES ARE SUBMITTED, THEY WILL BE RETURNED UNMARKED.

1.3 ALL DETAILS SHOWN ARE TYPICAL. SIMILAR DETAILS APPLY TO SIMILAR CONDITIONS, UNLESS NOTED.

1.4 CONTRACTOR SHALL VERIFY ALL DIMENSIONS AND SITE CONDITIONS PRIOR TO FABRICATION/CONSTRUCTION. NOTIFY STRUCTURAL ENGINEER OF ANY DISCREPANCIES PRIOR TO FABRICATION/CONSTRUCTION.

2. FOUNDATION

2.1 GEOTECHNICAL REPORT: FOUNDATION DESIGN IS BASED ON THE GEOTECHNICAL REPORT BY GALLET & ASSOCIATES, INC., ENTITLED "GEOTECHNICAL INVESTIGATION, QUINTARD MALL EXPANSION, OXFORD, ALABAMA DATED OCTOBER 4, 1994 PROJECT NO. 94-G-07750.A, AND ADDENDA LETTERS DATED DECEMBER 21, 1994, APRIL 5, 1995, AND MAY 2, 1995". THE GENERAL CONTRACTOR SHALL OBTAIN A COPY OF THE GEOTECHNICAL REPORT AND ADDENDA LETTERS FROM THE OWNER AND FOLLOW ALL REQUIREMENTS WITHIN THE RECOMMENDATION SECTIONS.

2.2 MAXIMUM ROCK BEARING PRESSURE: 5000 PSF.

2.3 ALL FOUNDATION BEARING SURFACES SHALL BE REVIEWED BY THE GEOTECHNICAL ENGINEER PRIOR TO PLACING CONCRETE TO INSURE THEIR COMPLIANCE WITH PRESSURES NOTED. ALL BOTTOM ELEVATIONS ARE ESTIMATED AND MAY BE ADJUSTED IN THE FIELD BY THE GEOTECHNICAL ENGINEER.

3. CONCRETE

3.1 CONCRETE COMPRESSIVE STRENGTH AT 28 DAYS:
3000 PSI ----- NORMAL WT. ----- FOOTINGS.
4000 PSI ----- NORMAL WT. ----- COLUMNS, TOPPING SLAB.
4000 PSI ----- NORMAL WT. ----- SLAB AND BEAM AT TRANSITION.

3.2 REINFORCING BARS: ASTM A615 GRADE 60.

3.3 WELDED WIRE FABRIC (WWF): ASTM A185. MINIMUM LAP AND EMBEDMENT TO BE THE GREATER OF ONE CROSS WIRE SPACING PLUS 2 INCHES OR 6 INCHES.

3.4 REINFORCING STEEL SHOWN IN SECTIONS OF BEAMS AND COLUMNS IS A SCHEMATIC INDICATION THAT REINFORCING EXISTS. SEE SCHEDULES, SECTION NOTES AND GENERAL NOTES FOR ACTUAL REINFORCING REQUIRED.

3.5 REINFORCING BAR PLACING ACCESSORIES IN ACCORDANCE WITH AASHTO AND ACI MANUAL OF STANDARD PRACTICE.

3.6 DETAIL REINFORCEMENT IN ACCORDANCE WITH AASHTO AND ACI 315. REINFORCEMENT SHALL NOT BE WELDED UNLESS NOTED OR APPROVED BY THE ENGINEER.

3.7 ALL SPLICES SHALL BE CLASS "B" TENSION LAP SPLICE, UNLESS NOTED.

3.8 ALL REINFORCING MARKED CONTINUOUS SHALL BE SPLICED WITH CLASS "B" TENSION LAP SPLICE, UNLESS NOTED.

3.9 CONCRETE COVERAGE OF REINFORCEMENT:

FOOTINGS----------3" BOTTOM & SIDES
COLUMNS----------2" CLEAR OF TIES
BEAMS----------2" CLEAR OF STIRRUPS
TOPPING SLABS----------1 1/2"

3.10 COLUMN REINFORCING: DOWEL TO FOUNDATION WITH HOOKED BARS OF SAME SIZE AND SPACING AS VERTICAL REINFORCING.

3.12 CONCRETING OPERATIONS SHALL COMPLY WITH ACI STANDARDS.

4. PRECAST CONCRETE

4.1 PRECAST MANUFACTURER IS TO BE RESPONSIBLE FOR THE DESIGN OF ALL PRECAST MEMBERS AND THEIR CONNECTIONS TO THE STRUCTURE. CALCULATIONS AND SHOP DRAWINGS SHALL BE SUBMITTED BEARING THE SEAL OF A PROFESSIONAL ENGINEER REGISTERED IN THE STATE OF ALABAMA.

4.2 ANY CONNECTIONS SHOWN ON CONTRACT DRAWINGS ARE SHOWN FOR GENERAL ARRANGEMENT ONLY. THE CONTRACTOR SHALL COORDINATE ALL PRECAST CONNECTIONS AND EMBEDDED ITEMS WITH THE PRECAST MANUFACTURER.

4.3 ERECTOR SHALL BE RESPONSIBLE FOR PROVIDING ALL TEMPORARY BRACING UNTIL ALL CONNECTIONS HAVE BEEN MADE AND TOPPING HAS BEEN CAST.

4.4 PRECAST MANUFACTURER SHALL PROVIDE STABILIZING ANGLES, AS REQUIRED, IN ALL PRECAST WORK.

4.5 PRECAST MANUFACTURER TO VERIFY THAT THE DOUBLE TEE CAMBER DOES NOT INTERFERE WITH THE 3" MINIMUM TOPPING AND SHALL MAKE ALLOWANCES FOR ADDITIONAL WEIGHT WHERE TOPPING EXCEEDS 3".

4.6 ALL EXPOSED STEEL CONNECTIONS AND SUPPORT ANGLES, PLATES, BARS AND BOLTS IN CONJUNCTION WITH ALL PRECAST CONCRETE SHALL BE HOT-DIP GALVANIZED AFTER FABRICATION AND FIELD TOUCHED UP WITH ZINC RICH PAINT.

4.7 TOPPING SLAB: 3" THICK UNLESS NOTED, REINFORCE WITH 6X6 W1.9XW2.9 WWF IN FLAT SHEETS AT MID-DEPTH OF TOPPING.

4.8 TOOLED JOINTS IN CONCRETE TOPPING OVER PRESTRESSED TEES ARE TO BE LOCATED WHERE EVERY TEE ABUTS, ABOVE THE END OF EVERY TEE AND AT THE ENDS OF BEAMS. BEAMS AND TEES MAY NOT ALIGN.

5. STRUCTURAL STEEL

5.1 STRUCTURAL STEEL: ASTM A36.

5.2 WELDED CONNECTIONS: E70XX ELECTRODES. MINIMUM FILLET WELD 3/16".

5.3 HEADED STUDS: ASTM A108, GRADE 1015 OR 1020, COLD FINISHED CARBON STEEL WITH DIMENSIONS COMPLYING WITH THE AMERICAN INSTITUTE OF STEEL CONSTRUCTION.

5.4 GALVANIZING: ALL STRUCTURAL STEEL SHALL BE HOT DIP GALVANIZED IN ACCORDANCE WITH ASTM A123.

FUTURE MALL COLUMN LOAD SCHEDULE

COLUMN DESIGNATION	M1	M2	M3	M4	M5	M6	M7	M8	M9	M10	M11	M12
FUTURE TOTAL ROOF LOAD	42	42	42	83	83	83	95	95	100	100	100	100
FUTURE EXTERIOR WALL WEIGHT	100	100	100	100	100	100	---	---	---	---	---	---

FUTURE MALL COLUMN LOAD SCHEDULE

COLUMN DESIGNATION	M13	M14	M15	M16	M17	M18	M19	M20	M21	M22	M23, M34	M24
FUTURE TOTAL ROOF LOAD	100	120	120	120	120	120	100	100	100	100	100	50
FUTURE EXTERIOR WALL WEIGHT	---	---	---	---	---	---	---	---	---	---	---	52

FUTURE MALL COLUMN LOAD SCHEDULE

COLUMN DESIGNATION	M25	M26	M27	M28	M29	M30, M33	M31, M35	M32, M36 M37				
FUTURE TOTAL ROOF LOAD	50	50	50	45	45	45	23	45				
FUTURE EXTERIOR WALL WEIGHT	62	54	60	50	50	50	55	100				

FOOTING SCHEDULE

FOOTING DESIGNATION		F1	F2	F3	F4	F5	F6	F7	F8	F9			
FOOTING	SIZE	5'-0"x5'-0"	5'-6"x5'-6"	6'-0"x6'-0"	6'-6"x6'-6"	7'-0"x7'-0"	9'-0"x9'-0"	8'-0"x8'-0"	11'-6"x11'-6"	9'-0"x9'-0"			
	DEPTH	1'-6"	1'-6"	1'-6"	1'-6"	1'-6"	2'-0"	1'-9"	2'-8"	2'-0"			
	REINF EW	5#5	5#6	6#6	7#6	7#7	11#7	9#7	11#9	11#7			
	NOTES						1		1				

NOTES: 1. PROVIDE SCHEDULED REINFORCING TOP AND BOTTOM.

QUINTARD MALL EXPANSION
SNOW CREEK BRIDGE

ALABAMA REGISTERED No. 16034 PROFESSIONAL ENGINEER 12-20-96

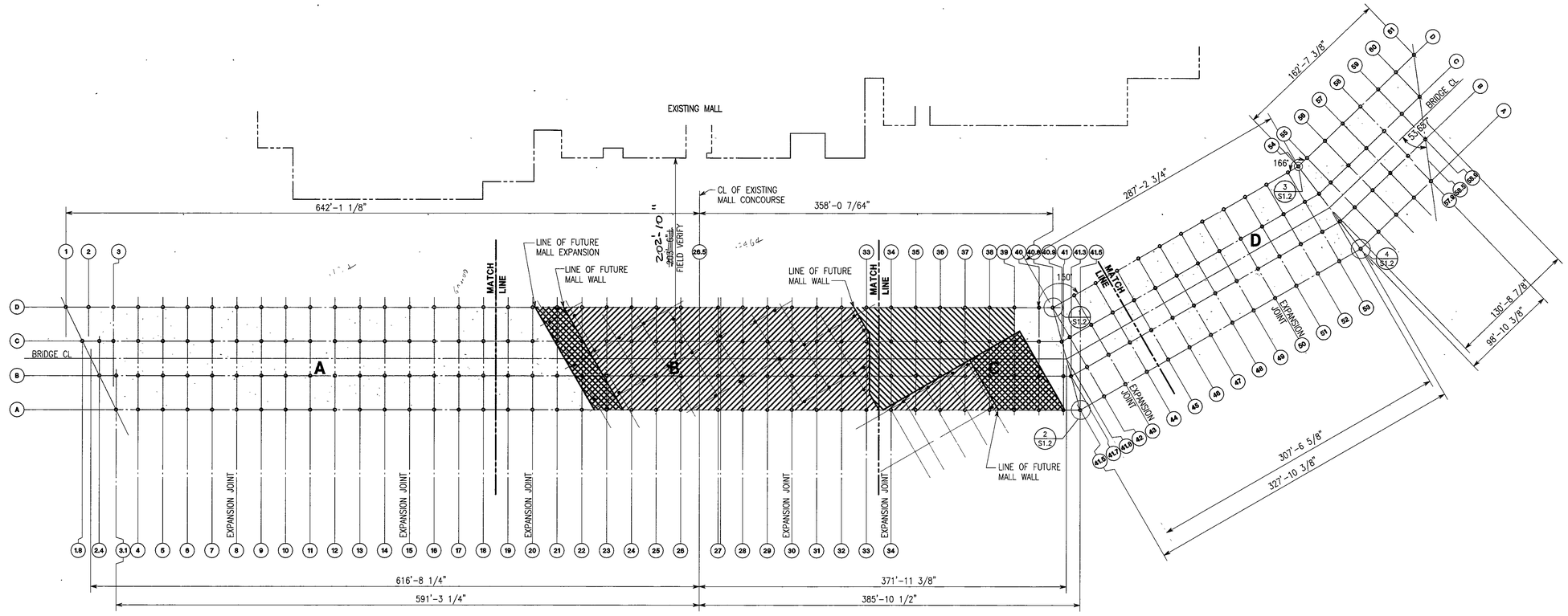
REVISIONS

LaneBishopYorkDelahayInc.
Consulting Structural Engineers
716 South 30th Street Birmingham Alabama 35233
(205)251-4500

LEVD

SHEET TITLE
GENERAL NOTES AND ABBREVIATIONS

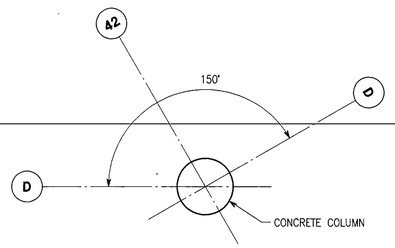
DATE 12-20-96
DRAWN JJA
CHECKED DMT
APPROVED DDY
JOB 4676
DRAWING S1.1
SHEET 1 OF 10



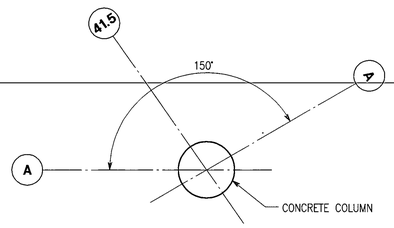
PROJECT NORTH

GENERAL PLAN 1"=50'-0"

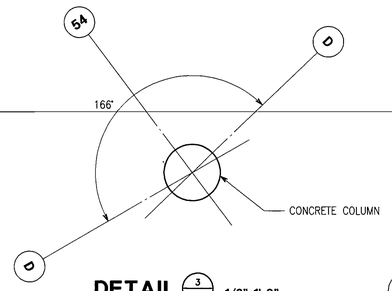
- DESIGN PRECAST DOUBLE TEES AND BEAMS FOR A SUPERIMPOSED HS20 LIVE LOAD (DOUBLE TEE FLANGE CONNECTION SPACING – 10'–0").
- DESIGN PRECAST DOUBLE TEES AND BEAMS FOR A SUPERIMPOSED 170 PSF DEAD LOAD AND 100 PSF LIVE LOAD (DOUBLE TEE FLANGE CONNECTION SPACING – 10'–0").
- DESIGN PRECAST DOUBLE TEES AND BEAMS FOR A SUPERIMPOSED HS20 LIVE LOAD (DOUBLE TEE FLANGE JONT SPACING – 5'–0").
- DESIGN PRECAST DOUBLE TEES AND BEAMS FOR A SUPERIMPOSED HS20 LIVE LOAD (DOUBLE TEE FLANGE CONNECTION SPACING – 10'–0"). (NOTE THIS AREA TO BE PART OF FUTURE MALL EXPANSION.)



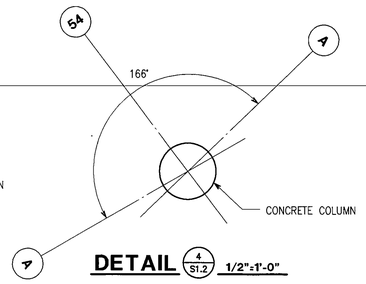
DETAIL (1 / S1.2) 1/2"=1'-0"



DETAIL (2 / S1.2) 1/2"=1'-0"



DETAIL (3 / S1.2) 1/2"=1'-0"



DETAIL (4 / S1.2) 1/2"=1'-0"

QUINTARD MALL EXPANSION

SNOW CREEK BRIDGE

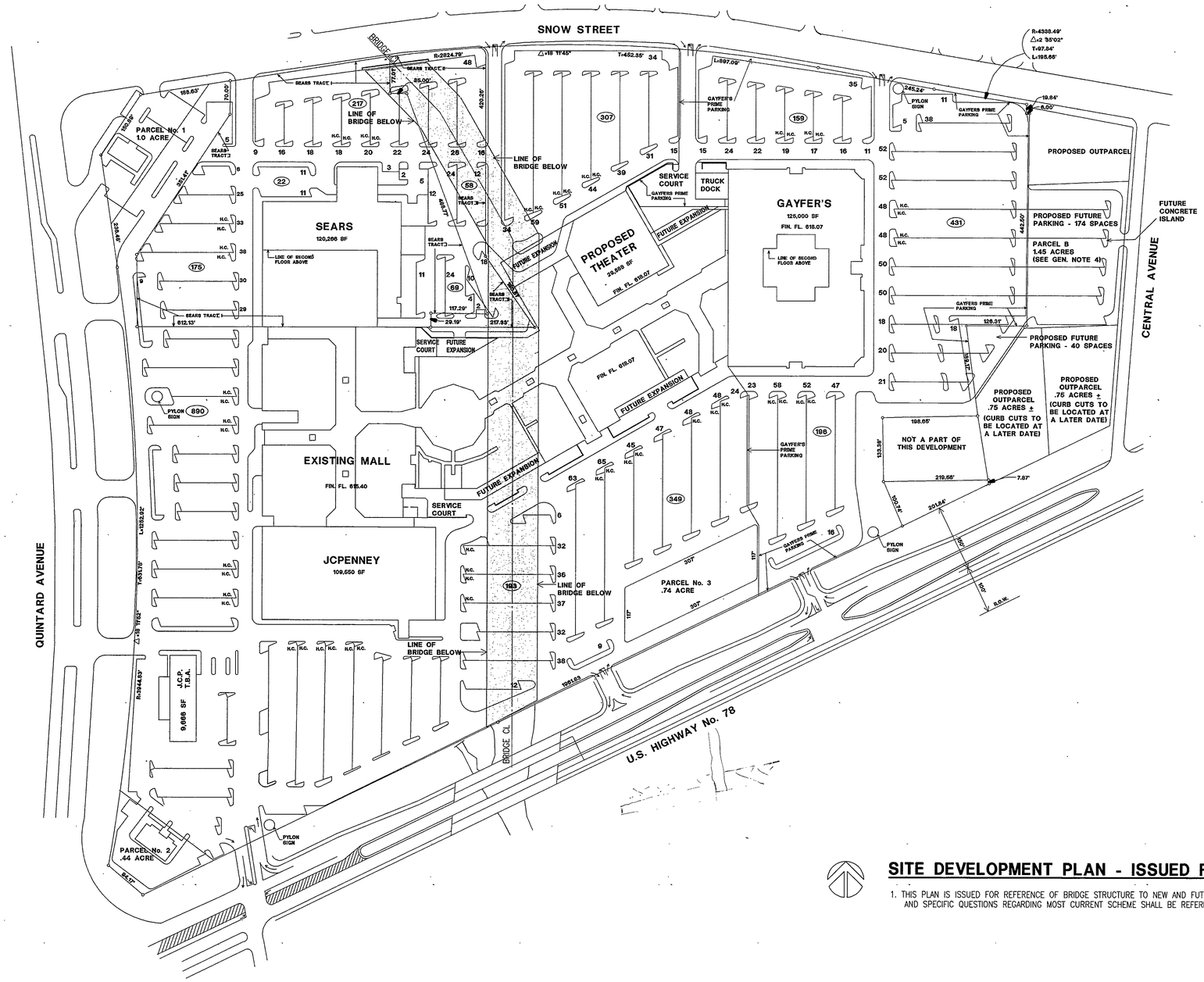
REVISIONS

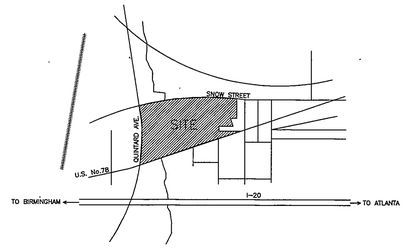
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Alabama 35233
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SHEET TITLE: OVERALL PLAN

DATE	12-20-96
DRAWN	JJA
CHECKED	GBJ
APPROVED	DDY
JOB	4676
DRAWING	S1.2
SHEET	2 OF 10

Qm-1-37





VICINITY MAP
NOT TO SCALE

SITE DEVELOPMENT PLAN - ISSUED FOR REFERENCE ONLY 1"=100'-0"

1. THIS PLAN IS ISSUED FOR REFERENCE OF BRIDGE STRUCTURE TO NEW AND FUTURE CONSTRUCTION. SITE DEVELOPMENT IS SUBJECT TO CHANGE AND SPECIFIC QUESTIONS REGARDING MOST CURRENT SCHEME SHALL BE REFERRED TO THE OWNER.

REVISIONS

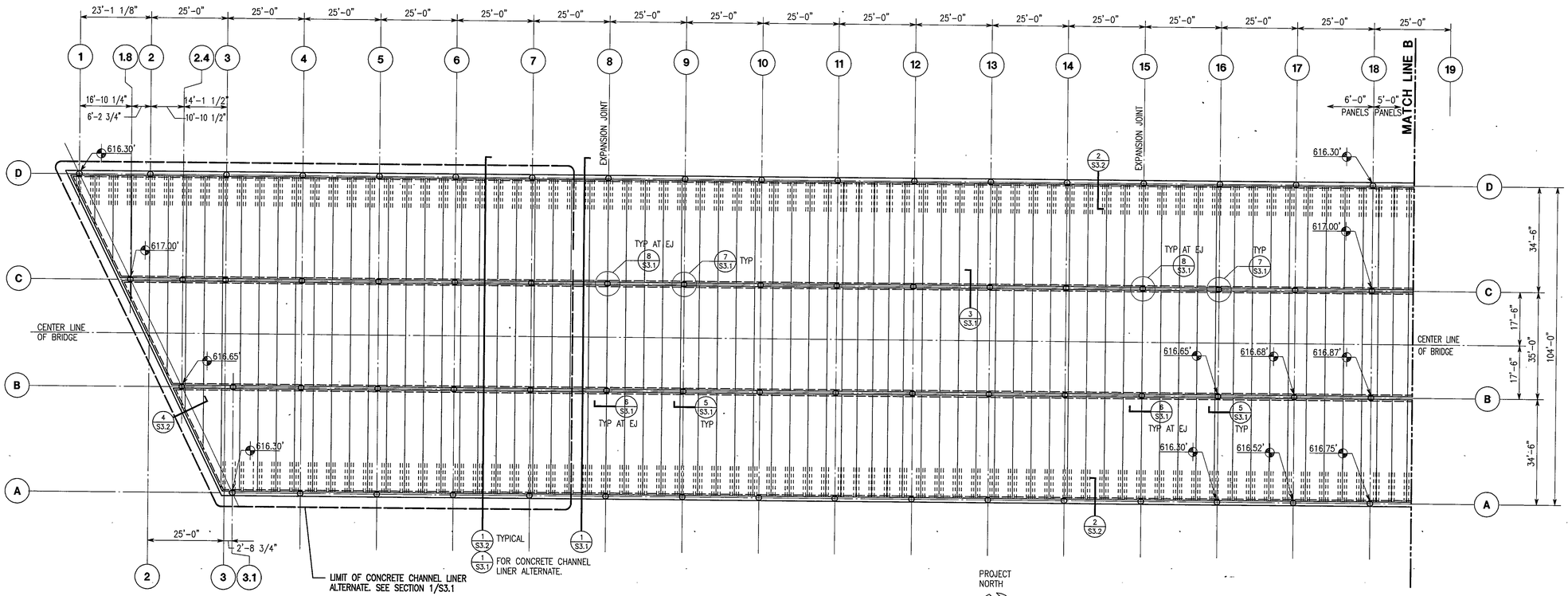
QUINTARD MALL EXPANSION
SNOW CREEK BRIDGE

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SHEET TITLE
SITE DEVELOPMENT PLAN

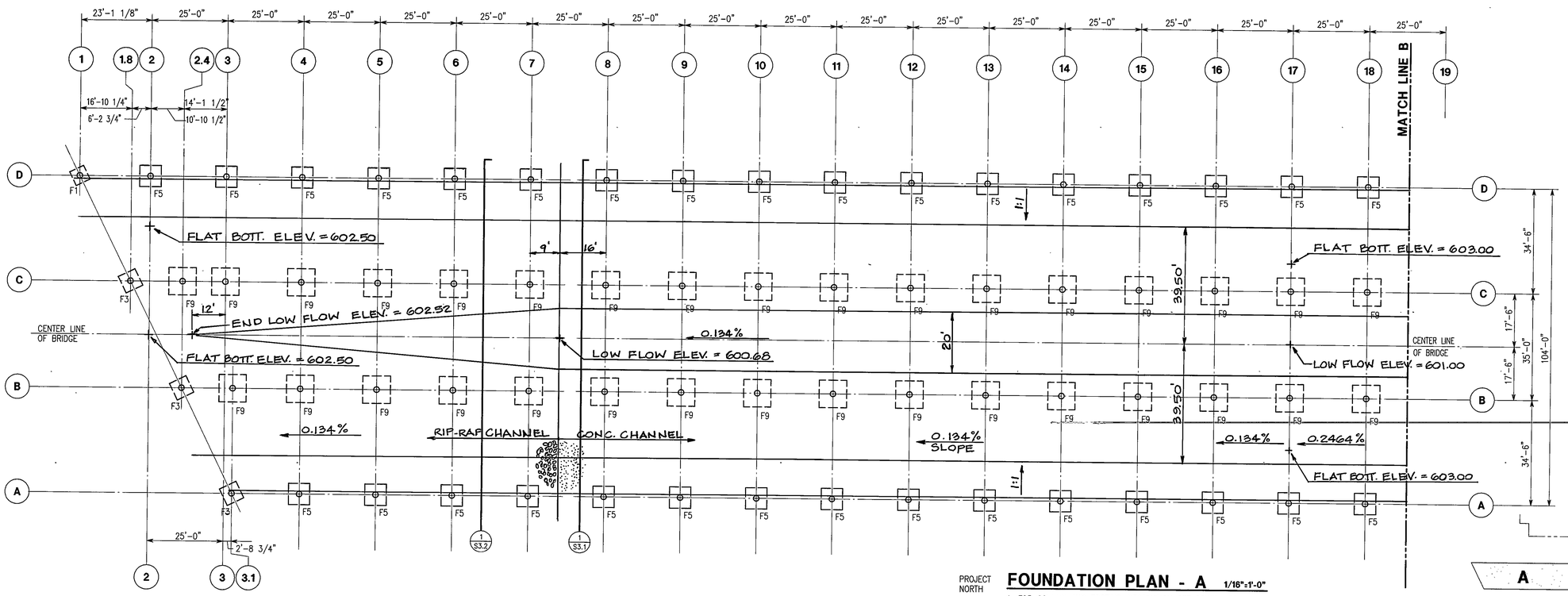
DATE 12-20-96
DRAWN HKW/JJA
CHECKED GBJ
APPROVED DDY
JOB 4676
DRAWING S1.3
SHEET 3 OF 10

QM-1-37



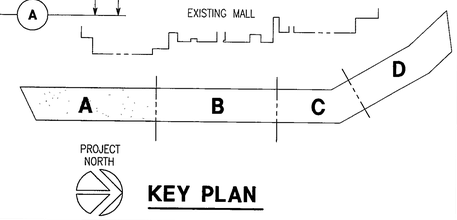
FRAMING PLAN - A 1/16"=1'-0"

1. TOP OF TOPPING SLAB AT CENTERLINE OF COLUMN LINE OF COLUMN NOTED ⊕ XXX.XX'
2. TOP OF TOPPING SLAB IS EITHER LEVEL OR SLOPING UNIFORMLY BETWEEN NOTED ELEVATIONS.
3. FOR BIDDING PURPOSES ONLY, TOP OF COLUMNS IS 3.25' BELOW TOP OF TOPPING SLAB.



FOUNDATION PLAN - A 1/16"=1'-0"

1. FOR COLUMN AND FOOTING INFORMATION SEE SCHEDULES.
2. TOP OF FOOTING ELEVATIONS UNLESS NOTED,
   GRIDS 1-7 598.50'
   GRIDS 1-7 ALTERNATE 601.00'
   GRIDS 8-16 601.25'
   GRIDS 17-18 601.50'



KEY PLAN

ALABAMA REGISTERED No. 16034 PROFESSIONAL G. E. ENGINEERS
12-20-96

REVISIONS

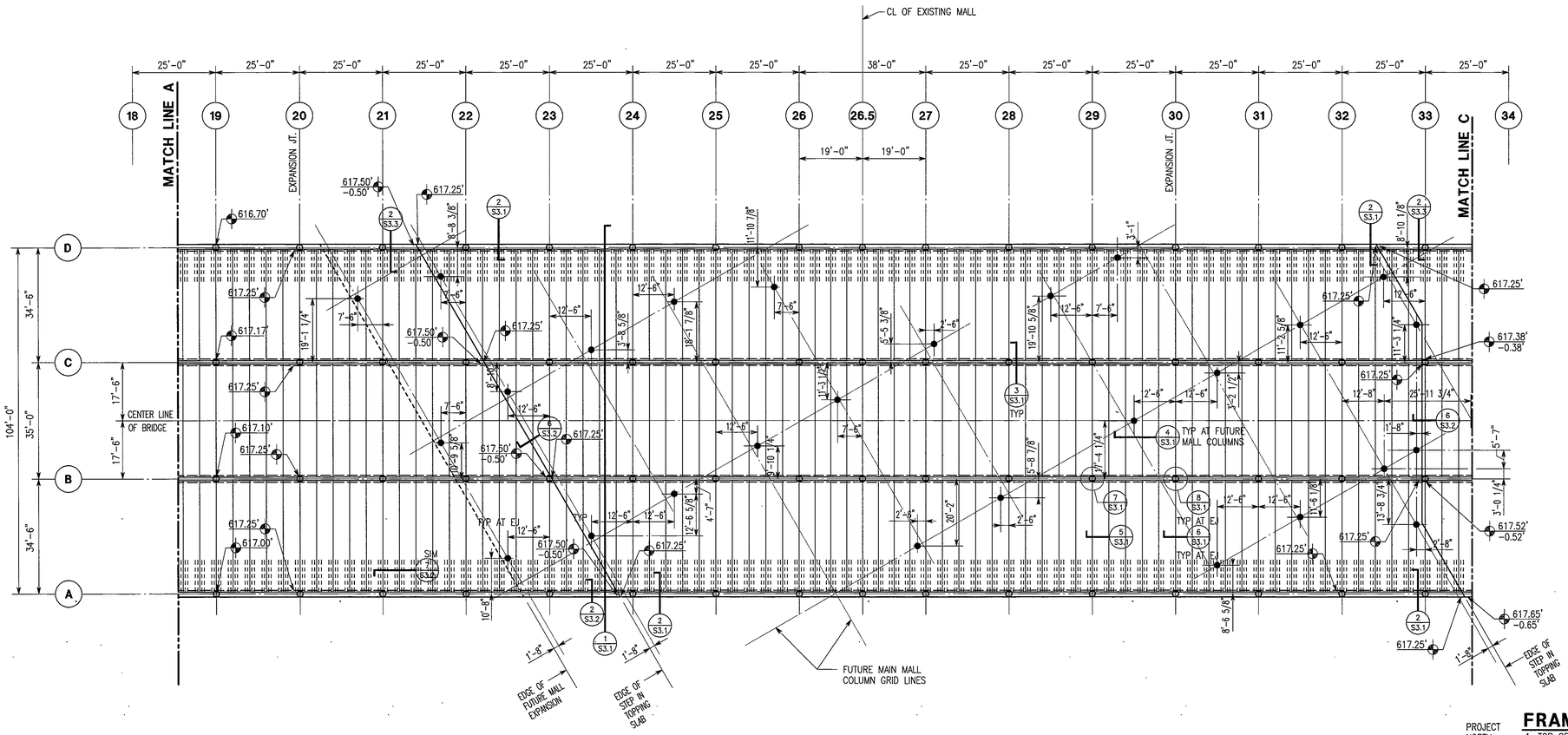
QUINTARD MALL EXPANSION
SNOW CREEK BRIDGE

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Consulting Structural Engineers
716 South 30th Street Birmingham
Alabama 35233
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LEMD

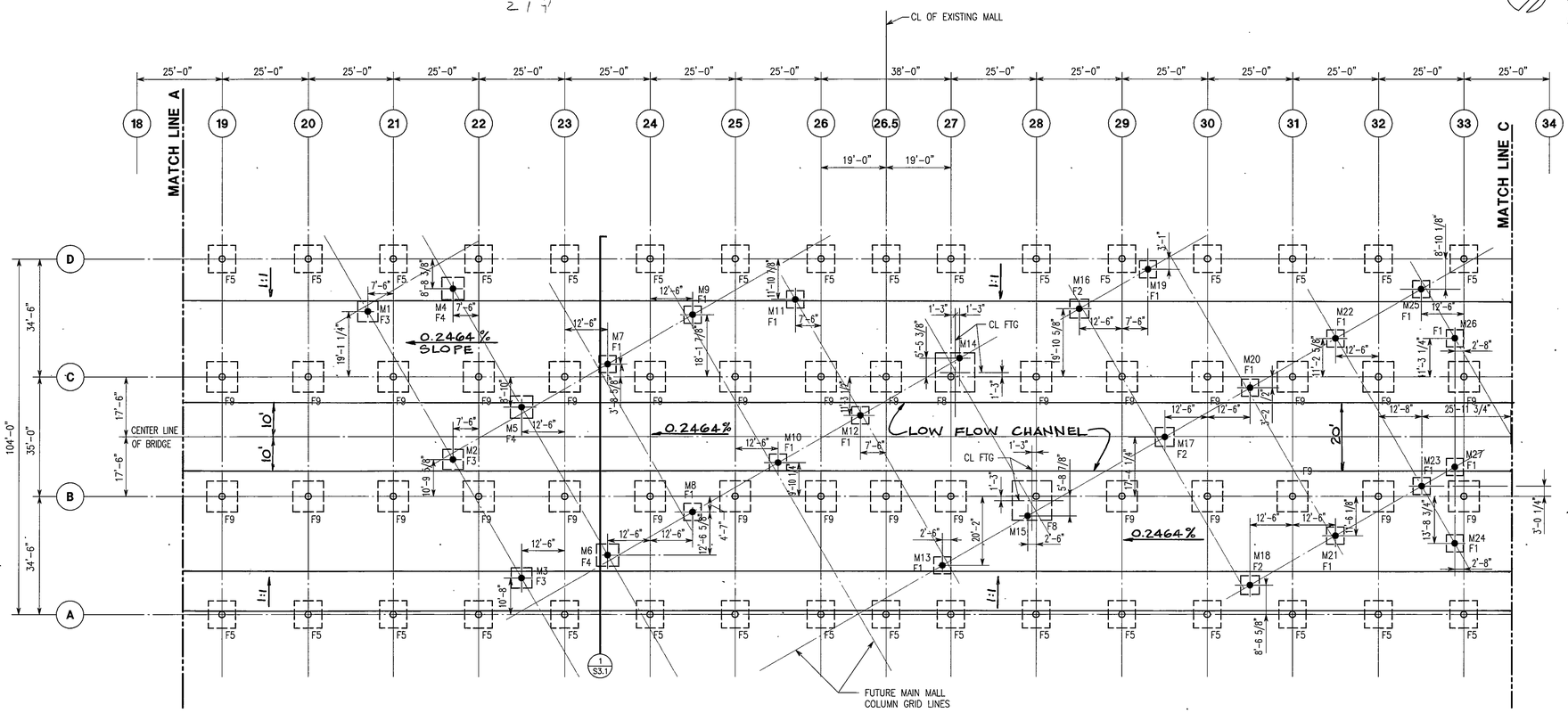
SHEET TITLE
FOUNDATION AND FLOOR PLAN PART "A"

DATE	12-20-96
DRAWN	JJA
CHECKED	GBJ
APPROVED	DDY
JOB	4676
DRAWING	S2.1
SHEET	4 OF 10



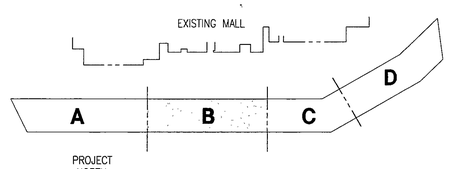
FRAMING PLAN - B 1/16"=1'-0"

1. TOP OF TOPPING SLAB AT CENTERLINE OF COLUMN LINE OF COLUMN NOTED ⌖ XXX.XX'.
2. TOP OF TOPPING SLAB IS EITHER LEVEL OR SLOPING UNIFORMLY BETWEEN NOTED ELEVATIONS.
3. TOP OF PRECAST DOUBLE TEE IS 0.25' BELOW TOP OF TOPPING SLAB UNLESS NOTED ⌖ -0.XX'.
4. COLUMNS SUPPORTING FUTURE MALL COLUMNS ABOVE NOTED ●. FOR ALLOWABLE FUTURE LOADS SEE SCHEDULE. FOR TOPPING SLAB POCKET AT FUTURE COLUMNS, SEE TYPICAL DETAIL.
5. FOR BIDDING PURPOSES ONLY, TOP OF COLUMNS IS 3.25' BELOW TOP OF TOPPING SLAB.



FOUNDATION PLAN - B 1/16"=1'-0"

1. FOR COLUMN AND FOOTING INFORMATION SEE SCHEDULES.
2. TOP OF FOOTING ELEVATION UNLESS NOTED.
   GRIDS 19–24 601.50'
   GRIDS 25–33 602.00'
3. COLUMNS SUPPORTING FUTURE MALL COLUMNS ABOVE NOTED ● MX. FOR ALLOWABLE FUTURE LOADS SEE SCHEDULE.



KEY PLAN

QUINTARD MALL EXPANSION

SNOW CREEK BRIDGE

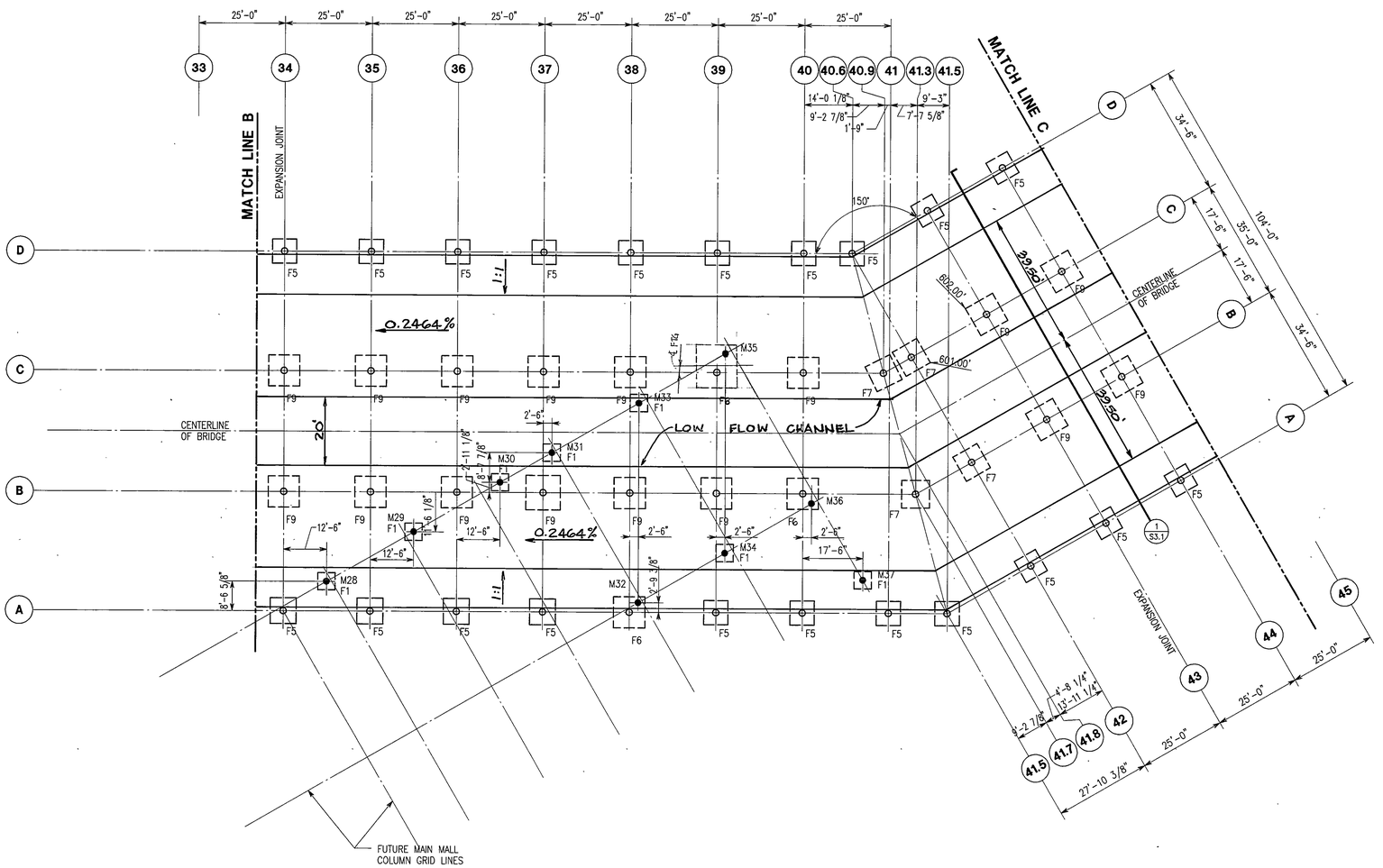
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SHEET TITLE
FOUNDATION AND FLOOR PLAN PART "B"

DATE	12-20-96
DRAWN	JJA
CHECKED	GBJ
APPROVED	DDY
JOB	4676
DRAWING	S2.2
SHEET	5 OF 10

REVISIONS

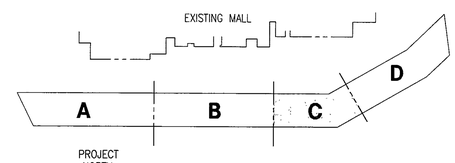
QM 1-37



PROJECT NORTH

FOUNDATION PLAN - C 1/16"-1'-0"

1. FOR COLUMN AND FOOTING INFORMATION SEE SCHEDULES.
2. TOP OF FOOTING ELEVATION UNLESS NOTED.
   GRIDS 34–41 602.50'
   GRIDS 42–44 603.00'
3. COLUMNS SUPPORTING FUTURE MALL COLUMNS ABOVE NOTED ●. FOR ALLOWABLE FUTURE LOADS SEE SCHEDULE.



PROJECT NORTH

KEY PLAN

QUINTARD MALL EXPANSION

SNOW CREEK BRIDGE

ALABAMA REGISTERED No. 16034 PROFESSIONAL ENGINEER GREGORY B. JONES 12/20/96

REVISIONS

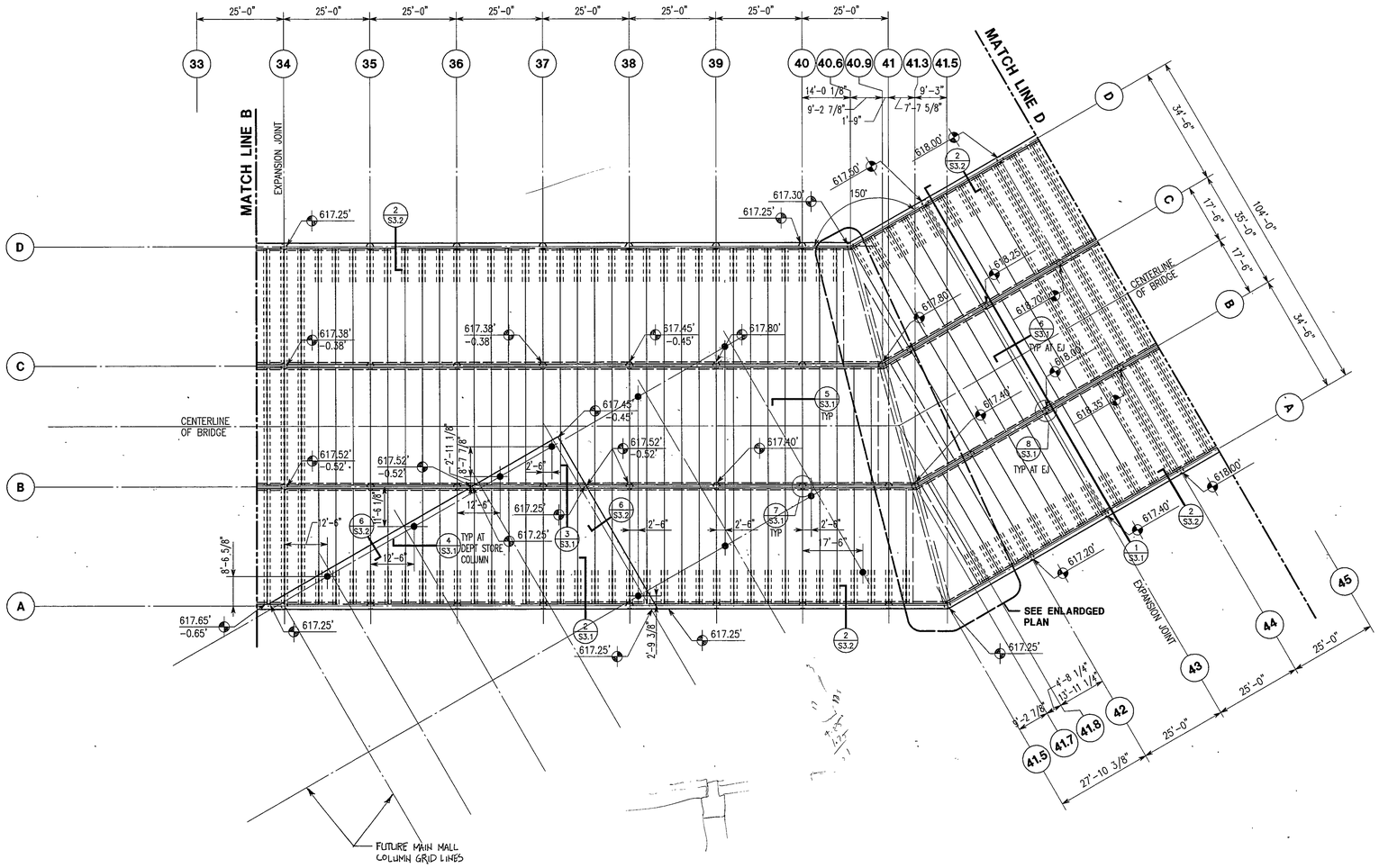
LaneBishopYorkDelahayInc.
Consulting Structural Engineers
716 South 30th Street Birmingham
Alabama 35233 (205)251-4500

LBYD

SHEET TITLE
FOUNDATION PART "C"

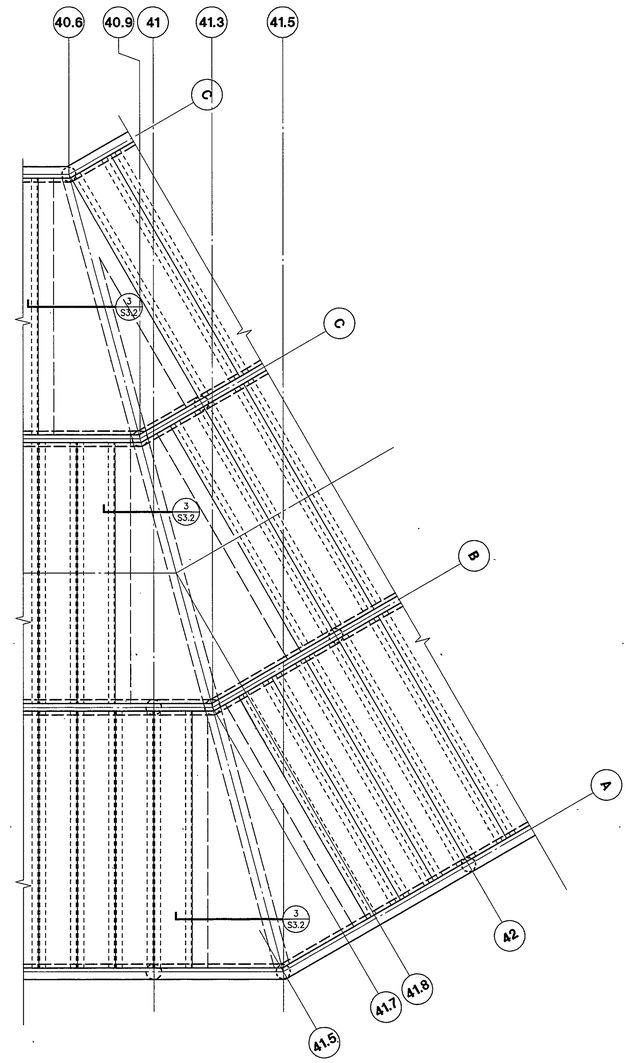
DATE	12-20-96
DRAWN	JJA
CHECKED	GBJ
APPROVED	DDY
JOB	4676
DRAWING	S2.3
SHEET	6 OF 10

QM1-37

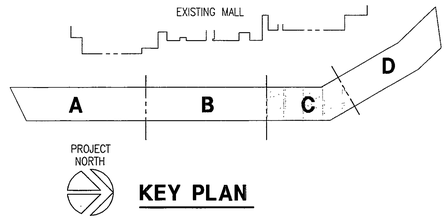


FRAMING PLAN - C 1/16"=1'-0"

1. TOP OF TOPPING SLAB AT CENTERLINE OF COLUMN LINE OF COLUMN NOTED ⊕ XXX.XX
2. TOP OF TOPPING SLAB IS EITHER LEVEL OR SLOPING UNIFORMLY BETWEEN NOTED ELEVATIONS.
3. TOP OF PRECAST DOUBLE TEE IS 0.25" BELOW TOP OF TOPPING SLAB UNLESS NOTED ⊕ -0.XX .
4. COLUMNS SUPPORTING FUTURE MALL COLUMNS ABOVE NOTED ●. FOR ALLOWABLE FUTURE LOADS SEE SCHEDULE. FOR TOPPING SLAB POCKET AT FUTURE COLUMNS, SEE TYPICAL DETAIL.
5. FOR BIDDING PURPOSES ONLY, TOP OF COLUMNS IS 3.25' BELOW TOP OF TOPPING SLAB.



ENLARGED PLAN AT TRANSITION 1/8"=1'-0"



KEY PLAN

QUINTARD MALL EXPANSION

SNOW CREEK BRIDGE

REVISIONS

ALABAMA REGISTERED No. 16034 PROFESSIONAL ENGINEER GARY B. JONES 12-20-96

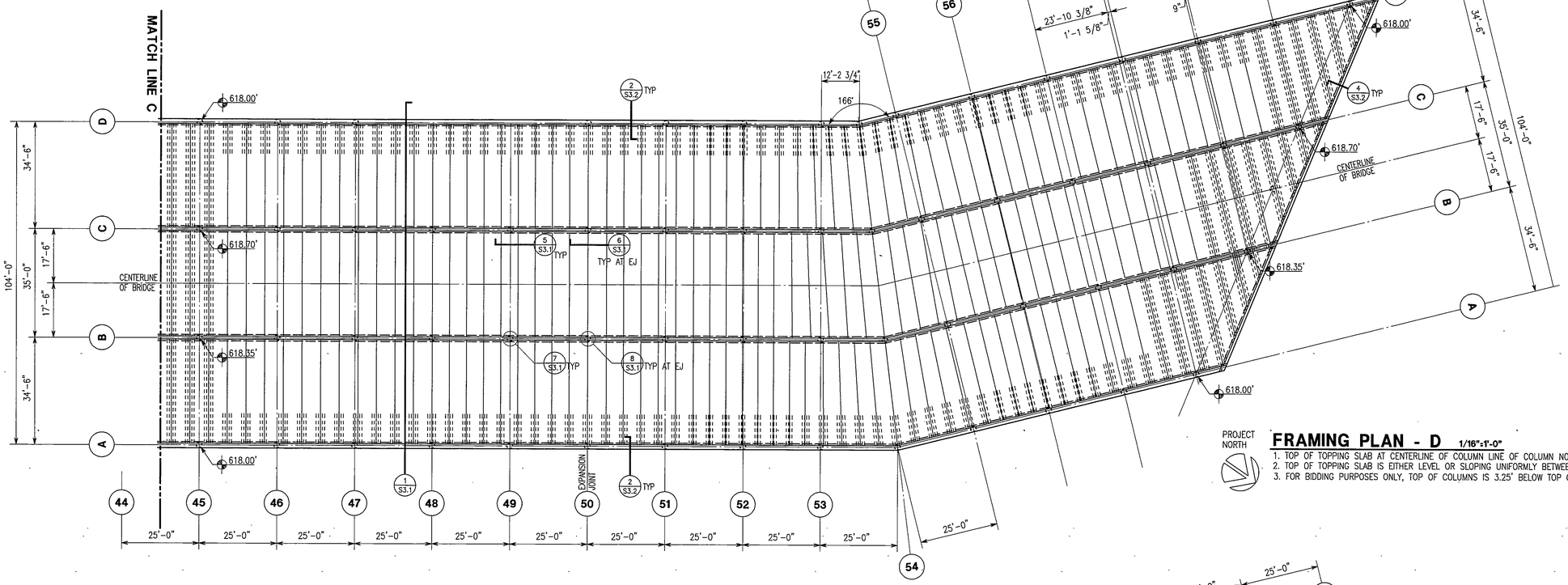
LaneBishopYorkDelahayInc.
Consulting Structural Engineers
716 South 30th Street Birmingham
Alabama 35233
(205)251-4500

SHEET TITLE

FLOOR PLAN PART "C"

DATE	12-20-96
DRAWN	JJA
CHECKED	GBJ
APPROVED	DDY
JOB	4676
DRAWING	S2.4
SHEET	7 OF 10

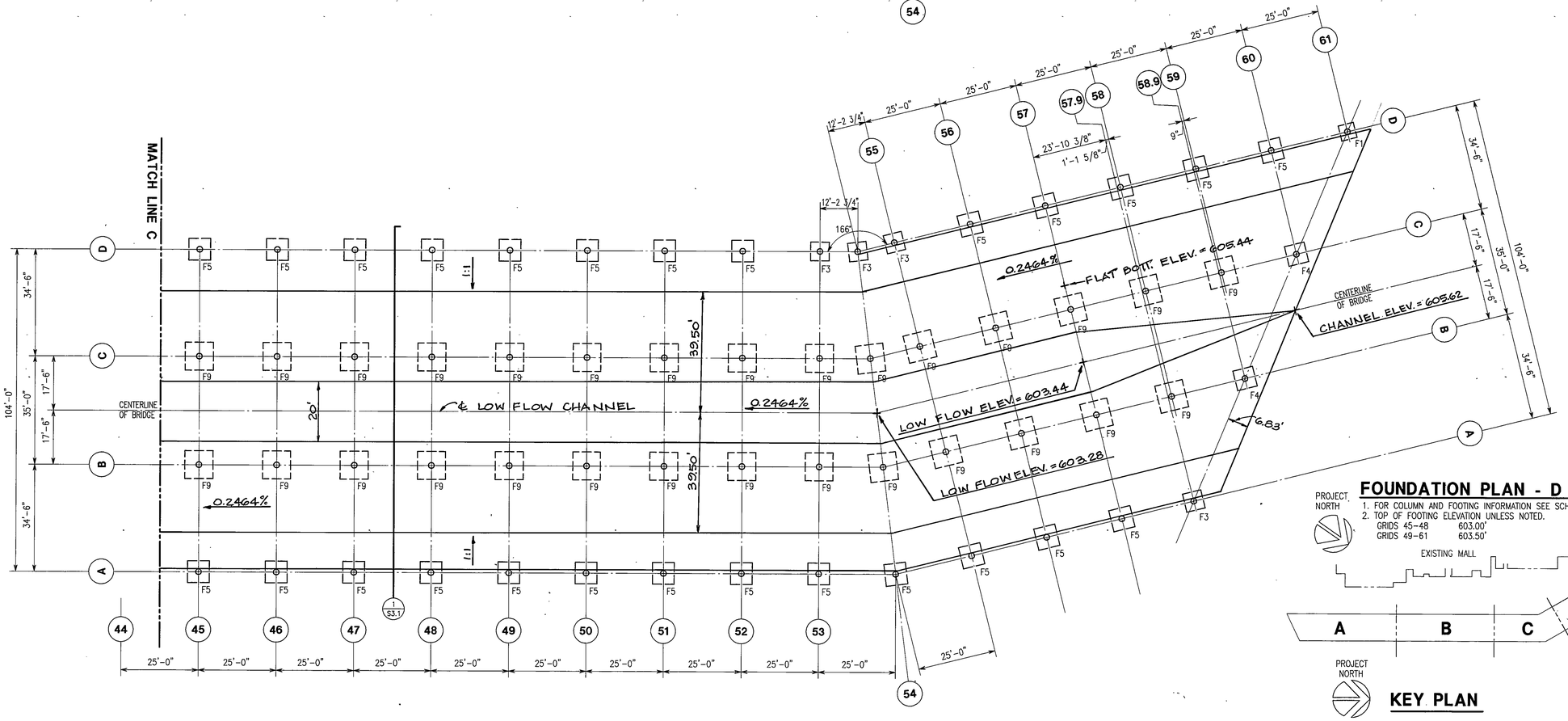
Qm1-37



PROJECT NORTH

FRAMING PLAN - D 1/16"=1'-0"

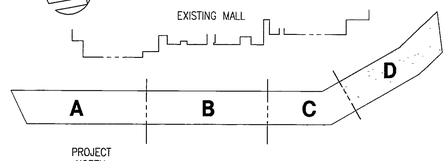
1. TOP OF TOPPING SLAB AT CENTERLINE OF COLUMN LINE OF COLUMN NOTED ⊕ XXX.XX'.
2. TOP OF TOPPING SLAB IS EITHER LEVEL OR SLOPING UNIFORMLY BETWEEN NOTED ELEVATIONS.
3. FOR BIDDING PURPOSES ONLY, TOP OF COLUMNS IS 3.25' BELOW TOP OF TOPPING SLAB.



PROJECT NORTH

FOUNDATION PLAN - D 1/16"=1'-0"

1. FOR COLUMN AND FOOTING INFORMATION SEE SCHEDULES.
2. TOP OF FOOTING ELEVATION UNLESS NOTED.
   GRIDS 45–48 603.00'
   GRIDS 49–61 603.50'



PROJECT NORTH

KEY PLAN

QUINTARD MALL EXPANSION

SNOW CREEK BRIDGE

REVISIONS

ALABAMA REGISTERED No. 16034 PROFESSIONAL ENGINEER GARY J. JONES 12-20-96

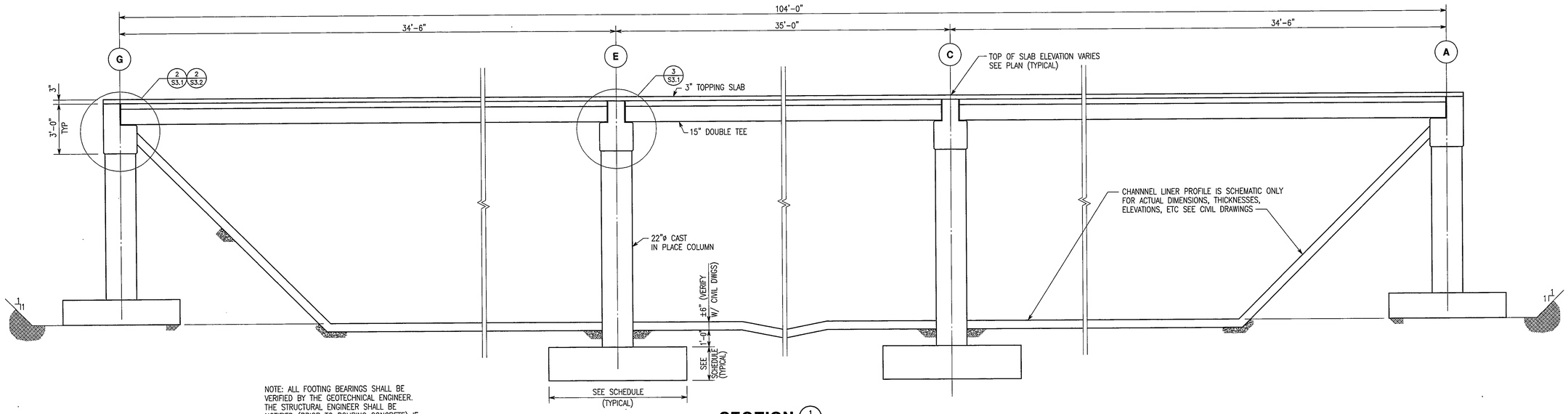
LaneBishopYorkDelahayInc.
Consulting Structural Engineers
716 South 30th Street Birmingham
Alabama 35233
(205)251-4500

LBYD

SHEET TITLE
FOUNDATION AND FLOOR PLAN PART "D"

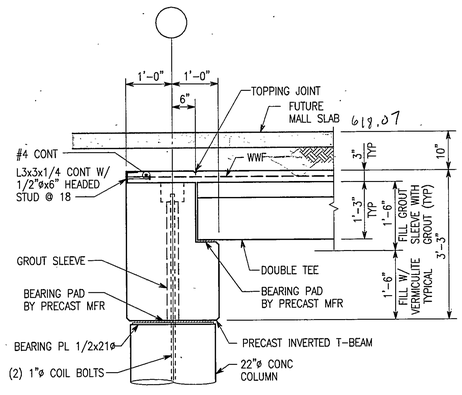
DATE	12-20-96
DRAWN	JJA
CHECKED	GBJ
APPROVED	DDY
JOB	4676
DRAWING	S2.5
SHEET	8 OF 10

QM-1-37

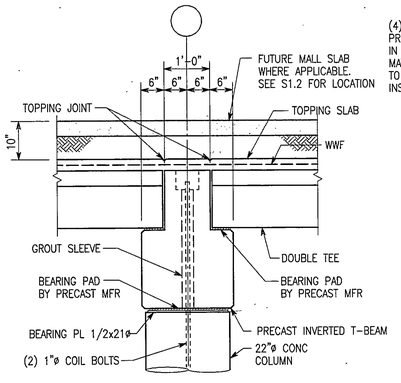


NOTE: ALL FOOTING BEARINGS SHALL BE VERIFIED BY THE GEOTECHNICAL ENGINEER. THE STRUCTURAL ENGINEER SHALL BE NOTIFIED (PRIOR TO POURING CONCRETE) IF BEARING ELEVATION IS ADJUSTED BY MORE THAN 1'-0".

SECTION (1 / S3.1) 3/8"=1'-0"

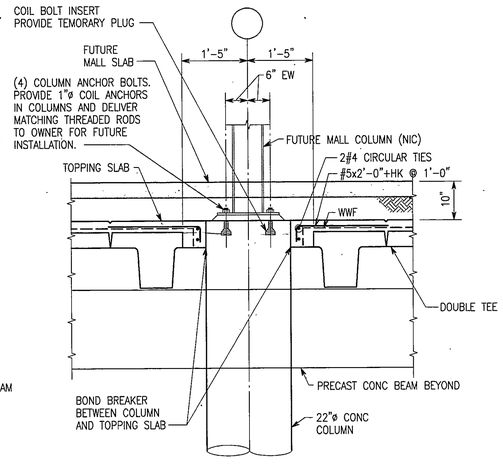


SECTION (2 / S3.1) 3/4"=1'-0"



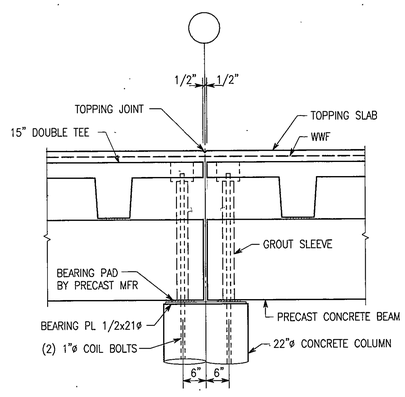
SECTION (3 / S3.1) 3/4"=1'-0"

NOTE: FOR GROUT SLEEVE FILL, SEE SECTION 2/S3.1



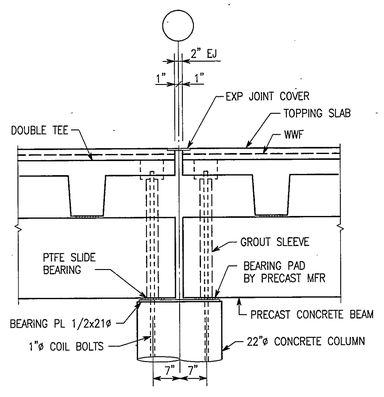
SECTION (4 / S3.1) 3/4"=1'-0"

NOTE: FOR GROUT SLEEVE FILL, SEE SECTION 2/S3.1



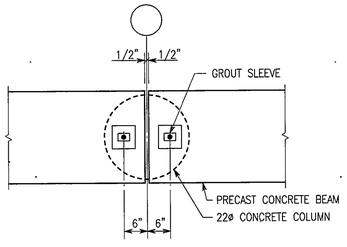
SECTION (5 / S3.1) 3/4"=1'-0"

NOTE: FOR GROUT SLEEVE FILL, SEE SECTION 2/S3.1



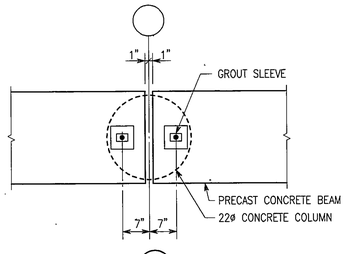
SECTION (6 / S3.1) 3/4"=1'-0"

NOTE: FOR GROUT SLEEVE FILL, SEE SECTION 2/S3.1



DETAIL (7 / S3.1) 3/4"=1'-0"

NOTE: FOR GROUT SLEEVE FILL, SEE SECTION 2/S3.1



DETAIL (8 / S3.1) 3/4"=1'-0"

NOTE: FOR GROUT SLEEVE FILL, SEE SECTION 2/S3.1

ALABAMA REGISTERED No. 16034 PROFESSIONAL STRUCTURAL ENGINEER
12-20-96

REVISIONS

QUINTARD MALL EXPANSION

SNOW CREEK BRIDGE

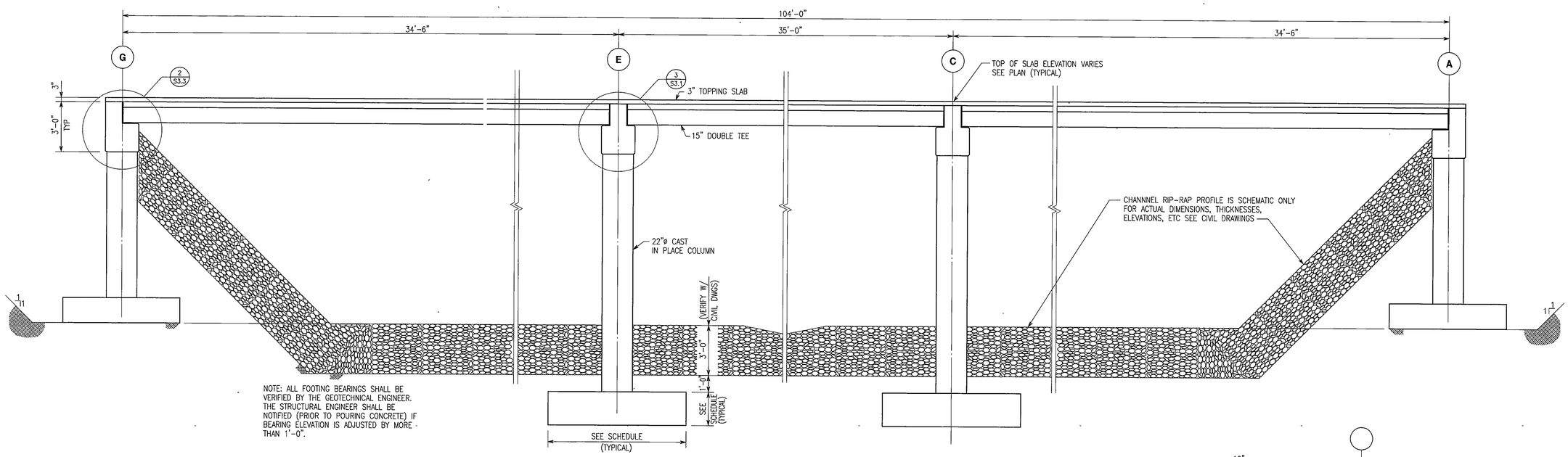
LaneBishopYorkDelahayInc.
Consulting Structural Engineers
716 South 30th Street Birmingham
Alabama 35233
(205)251-4500

LBYD

SHEET TITLE
SECTIONS

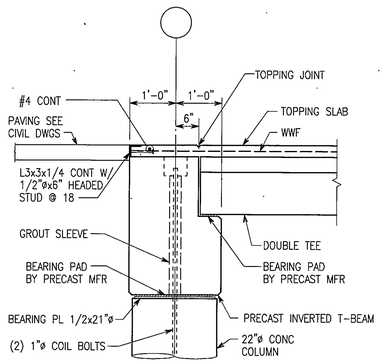
DATE	12-20-96
DRAWN	JJA
CHECKED	GBJ
APPROVED	DDY
JOB	4676
DRAWING	S3.1
SHEET	9 OF 10

QM-1-37



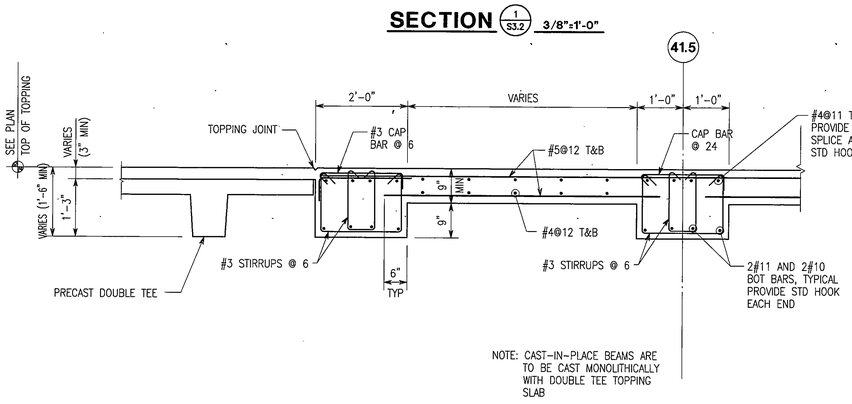
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SECTION (1 / S3.2) 3/8"=1'-0"



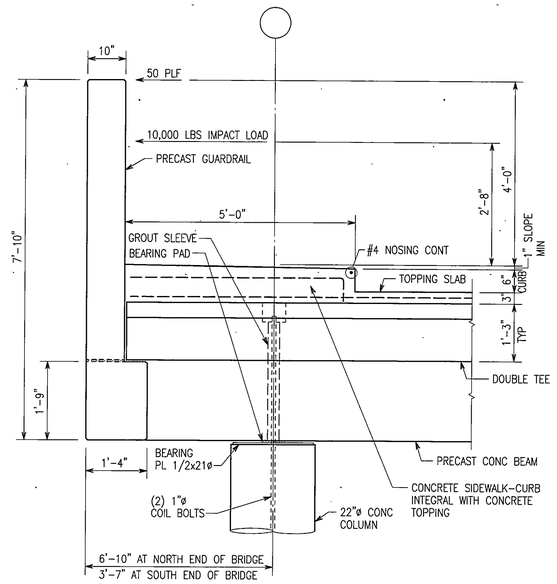
SECTION (2 / S3.2) 3/4"=1'-0"

NOTE: FOR GROUT SLEEVE FILL, SEE SECTION 2/S3.1



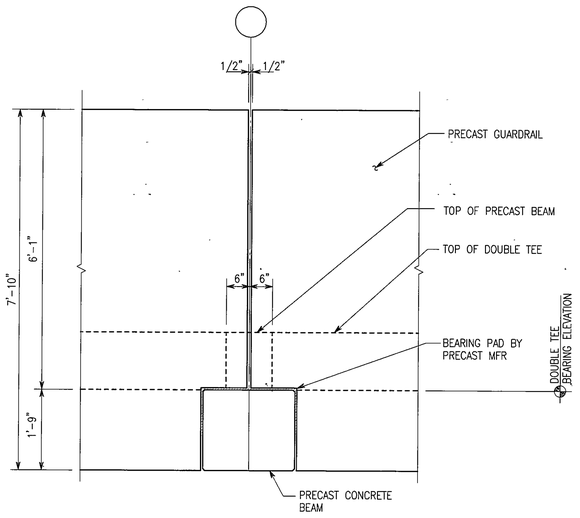
NOTE: CAST-IN-PLACE BEAMS ARE TO BE CAST MONOLITHICALLY WITH DOUBLE TEE TOPPING SLAB

SECTION (3 / S3.2) 3/4"=1'-0"

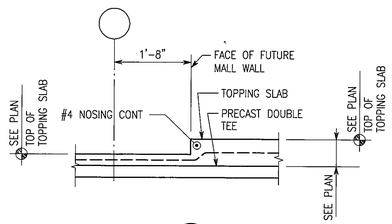


SECTION (4 / S3.2) 3/4"=1'-0"

NOTE: FOR GROUT SLEEVE FILL, SEE SECTION 2/S3.1



ELEVATION (5 / S3.2) 3/4"=1'-0"



SECTION (6 / S3.2) 3/4"=1'-0"

QUINTARD MALL EXPANSION

SNOW CREEK BRIDGE

ALABAMA REGISTERED No. 16034 PROFESSIONAL ENGINEER GREGORY B. JONES 12-20-96

REVISIONS

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SHEET TITLE
SECTIONS

DATE 12-20-96
DRAWN JJA
CHECKED GBJ
APPROVED DDY
JOB 4676
DRAWING S3.2
SHEET 10
OF 10