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April 5, 2022

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RE: Butte Priority Soils Operable Unit (BPSOU) 2022 Draft Final Unreclaimed Areas Quality Assurance Project Plan (QAPP)

Agency Representatives:

I am writing to you on behalf of Atlantic Richfield Company to submit the Butte Priority Soils Operable Unit (BPSOU) 2022 Draft Final Unreclaimed Areas Quality Assurance Project Plan (QAPP) for your review and approval. Once your comments have been received and incorporated, Atlantic Richfield will submit a final version of the QAPP for approval. The report may be downloaded at the following link:

https://pioneertechnicalservices.sharepoint.com/:f:/s/submitted/EttOmUAW4SpPmVKaHWHh-KoB6oZDC4T1PhWuM4uCvgsqRQ.

If you have any questions or comments, please call me at (907) 355-3914.

Sincerely,

Mike McAnulty

Mike Mc Anulty
Liability Manager & Global Risk Champion
Remediation Management Services Company
An affiliate of **Atlantic Richfield Company**

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SILVER BOW CREEK/BUTTE AREA NPL SITE
BUTTE PRIORITY SOILS OPERABLE UNIT

2022

Draft Final

Unreclaimed Sites
Quality Assurance Project Plan (QAPP)

Atlantic Richfield Company

April 2022

SILVER BOW CREEK/BUTTE AREA NPL SITE
BUTTE PRIORITY SOILS OPERABLE UNIT

2022

Draft Final

Unreclaimed Sites
Quality Assurance Project Plan (QAPP)

Prepared for:

Atlantic Richfield Company
317 Anaconda Road
Butte, Montana 59701

Prepared by:

Pioneer Technical Services, Inc.
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April 2022

APPROVAL PAGE

Silver Bow Creek/Butte Area NPL Site
Butte Priority Soils Operable Unit Unreclaimed Sites Quality Assurance Project Plan

Approved: _____ Date: _____
Nikia Greene, Site Project Manager, EPA, Region 8

Approved: _____ Date: _____
Daryl Reed, Project Officer, Montana DEQ

Approved: _____ Date: _____
Mike Mc Anulty, Liability Manager
Atlantic Richfield Company

Approved: _____ Date: _____
David Gratson, Quality Assurance Manager
Environmental Standards Inc.

2022
Plan is effective on date of last signature above.

DOCUMENT REVISION SUMMARY

Revision No.	Author	Description	Date
	Pioneer Technical Services, Inc.	Annual Update	June 2021
	Pioneer Technical Services, Inc.	Annual Update	April 2022

DISTRIBUTION LIST

Silver Bow Creek/Butte Area NPL Site Butte Priority Soils Operable Unit Soils
Unreclaimed Sites Quality Assurance Project Plan (QAPP)
Butte, Silver Bow County, Montana

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LIST OF ACRONYMS

Acronym	Definition	Acronym	Definition
BPSOU	Butte Priority Soils Operable Unit	**mm**	millimeter
BSB	Butte-Silver Bow	**MS**	Matrix Spike
CD	Consent Decree	**NFG**	National Functional Guidelines
CFRSSI	Clark Fork River Superfund Site Investigation	**NPL**	National Priority List
COC	Contaminant of Concern	**NRDP**	Natural Resource Damage Program
CPM	Contractor Project Manager	**PARCC**	Precision, Accuracy, Representativeness, Comparability, and Completeness
DEQ	(Montana) Department of Environmental Quality	**PDF**	Portable Document Format
DM/DV	Data Management/Data Validation	**PPE**	personal protection equipment
DOJ	Department of Justice	**PRR**	Poore, Roth and Robinson
DQA	Data Quality Assessment	**QA**	Quality assurance
DQO	Data Quality Objective	**QAM**	Quality Assurance Manager
DSR	Data Summary Report	**QAO**	Quality Assurance Officer
FSP	Field Sampling Plan	**QAPP**	Quality Assurance Project Plan
EDD	Electronic Data Deliverable	**QC**	Quality control
EPA	U.S. Environmental Protection Agency	**RCRA**	Resource Conservation and Recovery Act
GPS	Global Positioning System	**RL**	Reporting Limit
HAZWOPER	Hazardous Waste Operations and Emergency Response	**ROD**	Record of Decision
HSSE	Health Safety Security and Environment	**RPD**	Relative Percent Difference
ICP-AES	Inductively Coupled Plasma Atomic Emission Spectroscopy	**RSD**	Relative Standard Deviation
IM	Integrity Management	**SOP**	Standard Operating Procedure
LCS	laboratory control sample	**SRM**	Standard reference material
LCSD	Laboratory control sample duplicate	**SSHASP**	Site-Specific Health and Safety Plan
MBMG	Montana Bureau of Mines and Geology	**USGS**	U.S. Geological Survey
mg/kg	milligrams per kilogram	**XRF**	X-ray fluorescence

1.0 INTRODUCTION AND PURPOSE

Unreclaimed sites exist within the Butte Priority Soils Operable Unit (BPSOU) that could pose a threat to human health or surface water quality due to the presence of historical mine waste. Although many source areas have been previously reclaimed, areas still exist in which soils have not yet been evaluated; such sites may provide a pathway for human exposure or impact surface water quality via storm water runoff. These unreclaimed sites will be evaluated in accordance with Appendix D, Attachment C, Section 8.0 of the BPSOU Consent Decree (CD) (EPA, 2020a).

This Quality Assurance Project Plan (QAPP) describes the activities necessary to conduct soil sampling and characterization activities on unreclaimed sites. It also describes the quality assurance/quality control (QA/QC) policies and procedures to be used during collection and analysis. This QAPP is intended to standardize the sampling process to provide accurate and defensible testing results necessary to make a final site declaration. A Field Sampling Plan (FSP) will be produced to outline the site-specific activities to be performed at each unique site. Supplemental information mentioned throughout the document is included in the appendices below:

Appendix A Reference Documents
Appendix B Standard Operating Procedures
Appendix C Forms
Appendix D Summary of Revisions

A map in Appendix A shows the BPSOU area. Individual site figures will be provided for site-specific FSPs. Data unique to each site will be provided in a data summary report (DSR), in addition to historical data. A separate report will be prepared for each site that will include the declaration as to whether reclamation is required (as described further in Section 2.0).

This QAPP was prepared in a manner consistent with the EPA *Requirements for Quality Assurance Project Plans (EPA QA/R-5)* (EPA, 2001) and the BPSOU *Quality Management Plan* (Atlantic Richfield, 2016) and includes the following:

- Project management and objectives.
- Measurement and data acquisition.
- Assessment and oversight.
- Data review.

The sections below provide the basic plan elements and describe the appropriate content required for planning soil sampling and analysis activities at unreclaimed sites within the BPSOU. This QAPP expands or references information from other site-wide documents to comply with the EPA Requirements for QAPPs (EPA, 2001) and to present project-specific requirements.

2.0 PROJECT MANAGEMENT

This section addresses project administrative functions, project concerns, and goals and approaches to be followed during characterization sampling activities on the specific site.

2.1 Project Organization and Responsibilities

An example chart showing the overall organization of the project team is provided in Appendix A. Responsibilities of key individuals comprising a project team are described below.

Liability Manager – Mike Mc Anulty (Atlantic Richfield Company)

The Liability Manager monitors the performance of the contractor(s), consults with the QAM, Contractor Project Manager (CPM) and Contractor Quality Assurance Officer (QAO) on deficiencies, and helps finalize resolution actions.

Quality Assurance Manager (QAM) – David Gratson (Environmental Standards, Inc)

The QAM interfaces with the Liability Manager on company policies regarding quality and has the authority and responsibility to approve specific QA documents including this QAPP.

Contractor

Atlantic Richfield Company (Atlantic Richfield) assign a Contractor to be responsible for completing individual site investigations.

Contractor Project Manager (CPM)

The CPM is responsible for scheduling all sampling work to be completed and ensuring that the work is performed in accordance with the requirements contained herein. The CPM is also responsible for consulting with the specific project QA personnel regarding any deficiencies and finalizing resolution actions. The CPM for each project will be listed in the supporting documents for each project area under this QAPP.

Field Team Leader

The Field Team Leader ensures that the QAPP for each project area has been reviewed by all members of the field team and that the QAPP is properly followed during field activities. The Field Team Leader will conduct daily safety meetings, assist in field activities, and document activities in the logbook.

The Field Team Leader is responsible for equipment, problem solving and decision making in the field, and for addressing technical aspects of the project. The Field Team Leader will provide "on-the-ground" overviews of project implementation by observing site activities to ensure compliance with technical project requirements, Health Safety Security and Environment (HSSE) requirements, and the Site-Specific Health and Safety Plan (SSHASP). Finally, the Field Team Leader is responsible for identifying potential Integrity Management (IM) issues, as appropriate, and preparing required project documentation.

Contractor Quality Assurance Officer (QAO)

The Contractor QAO is responsible for verifying effective implementation of QAPP requirements and procedures. This includes reviewing field and laboratory data and evaluating data quality. The Contractor QAO for each project will be listed in the supporting documents created for each project area under this QAPP and will be independent from the unit generating the data.

Safety and Health Manager

Where applicable the Safety and Health Manager is responsible for developing the SSHASP and reviewing it with all members of the field team. The Safety and Health Manager will lead applicable Task Risk Assessments and conduct the initial safety meeting prior to starting fieldwork. The Safety and Health Manager will ensure that work crews comply with all site safety and health requirements and will revise the SSHASP, if necessary.

Contract Laboratory

The Contract Laboratory will ensure that the laboratory QA personnel are familiar with the QAPP and are available to perform the work as specified. Contract Laboratory personnel are responsible for reviewing final analytical reports produced by the laboratory, scheduling laboratory analyses, and supervising in-house chain of custody procedures. The Contract Laboratory will be an Atlantic Richfield-approved laboratory.

All samples for analyses will be sent to the analytical laboratory listed below, or equivalent:

Pace Analytical Services, LLC
Pace Environmental Sciences
1700 Elm Street SE
Minneapolis, MN 55414

2.2 Problem Definition and Background

As stated previously, unreclaimed sites exist within the BPSOU that could pose a threat to human health or surface water quality due to the presence of historical mine waste. Although many source areas have been previously reclaimed, areas still exist in which soils have not yet been evaluated; such sites may provide a pathway for human exposure or impact surface water quality via storm water runoff. The list of known unreclaimed sites is identified in Appendix D, Attachment C, Section 8.0 of the BPSOU CD (EPA, 2020a). Additional unreclaimed sites may be identified as remedial actions are implemented within BPSOU. If so, the newly identified sites will be evaluated in accordance with this QAPP.

This QAPP will function as a general QA document for all soil sampling activities at unreclaimed sites within the BPSOU. Individual figures, historical data (where applicable), and other supporting documents will be included in the site-specific FSPs.

2.3 Project/Task Description

Soil sampling will be performed to provide contaminant of concern (COC) concentrations and pH at each site in accordance with this QAPP and site-specific FSPs. These concentrations, as well as other site characteristics, will support making a declaration as to whether site-specific response actions are necessary. The objectives of the QAPP are as follows:

1. Provide consistent results in identifying the specific types and quality of data needed to support decisions regarding each site as a result of the investigation.
2. Describe specific requirements for collecting and analyzing samples.

Below is a summary of project tasks to be completed under this QAPP at each unreclaimed area.

Sampling: Field samples will consist of 3-point composites. Soil samples will be collected as described in standard operating procedure (SOP) Surface Soil Sampling General (SOP-S-01) and Subsurface Soil Sampling (SOP-S-02) included in Appendix B. The location and number of samples collected will be detailed in the documents specific to each site. The location and number of samples collected will be based on individual site parameters as determined by experienced personnel familiar with the local area.

Analyses: All samples will be analyzed using the Thermo Fisher Scientific Niton Analyzer XL3 X-Ray Fluorescence (XRF) Analyzer (Niton XL3), or equivalent, per Operating XL3 X-Ray Fluorescence Analyzer General SOP (SOP-SFM-02), and for pH per the Field Measurement of pH in Soil SOP (SOP-SFM-01). Confirmation laboratory samples will be analyzed for arsenic, cadmium, copper, lead, and zinc according to the laboratory Metals Analysis by Inductively Coupled Plasma Optical Emission Spectrometry (ICP-OES) – Method 6010 and 200.7 (ENV-SOP-MIN4-0052 Rev.07); ICP-OES sample preparation will be per laboratory ENV-SOP-MIN4-0056. Confirmation laboratory samples will be analyzed for mercury by cold vapor atomic absorption (CVAA) according to Mercury in Liquid and Solid/Semi-Solid Waste by 7470A, 7471, 7471B, and 245.1 (ENV-SOP-MIN4-0054). Appendix B contains the SOPs referenced above. Field XRF confirmation samples will be submitted to the laboratory at a rate of 1 per 10 samples, with additional samples sent to the laboratory for samples with field concentrations of any one analyte at ± 35% of BPSOU Soil Action Levels for Human Health (Table 1) or with field concentrations of one or more analytes within ± 35% action level and two or more within ± 35% or exceeding 35% of BPSOU Soil Screening Criteria for Storm Water COCs (Table 2). The 35% criteria may be adjusted based on the statistical analysis of the confirmation sample results. When confirmation analyses are performed by laboratory methods, these results will supersede XRF data to determine final concentrations detected in the sample.

Documentation and Records: The field team will ensure that all samples collected have a corresponding Global Positioning System (GPS) location, XRF measurement, and that each sample is appropriately logged and documented (refer to Section 2.6 and Section 3.0).

Site Declaration: For each site, the CPM will complete a site declaration provided within the Site-Specific Evaluation Summary Report stating whether the site is at or above human

health action levels and/or storm water waste identification criteria listed Table 1 and Table 2, based on land use determination; whether the site is contributing metals-impacted sediment to existing or planned wet weather control features; and whether historical mine waste at the site is contributing to the degradation of surface water quality.

Data Verification, Validation, and Reporting: Once site investigation activities and data validation and review are completed, the CPM will develop a site-specific evaluation summary report containing a DSR and data validation report (DVR) specific to each designated unreclaimed site. The Evaluation Summary Report will contain the results of sedimentation analysis and the site declaration (described above) and will include the following information: historical site information and sample data (if available) and a summary of all newly collected data . A draft version of the report will be submitted to the Agencies for review and a final version will be submitted for approval. When finalized, the Site-Specific Evaluation Summary Report including the DSR and DVR will be archived in the project geodatabase.

2.4 Data Quality Objectives and Criteria

The EPA Data Quality Objective (DQO) process (EPA, 2006a) is used to establish performance or acceptance criteria that serve as the basis for designing a plan to collect data of sufficient quality and quantity to support the goals of a study. Each step of the DQO process defines criteria that will be used to establish the final data collection designs. This QAPP followed the EPA process to develop criteria for each site. The process consists of seven steps as follows:

Step 1: State the Problem.
Step 2: Identify the Goals of the Study.
Step 3: Identify Information Inputs.
Step 4: Define the Boundaries of the Study.
Step 5: Develop the Analytical Approach.
Step 6: Specify Performance and Acceptance Criteria.
Step 7: Develop the Plan for Obtaining Data.

These DQOs (detailed below) will be used to guide the data collection and analysis activities.

Step 1: State the Problem.

The purpose of this step is to describe the problem to be studied so that the focus of the investigation will not be ambiguous.

Unreclaimed sites are identified as areas that could negatively impact human health and/or potentially degrade surface water quality. Site evaluations will determine which, if any, COCs are present within the soil, if concentrations are above action/screening levels listed in Table 1 (on page 7) and Table 2 (on page 8) and support future remedial action efforts within the BPSOU area.

Step 2: Identify the Goals of the Study.

This step identifies the principal question the study will attempt to resolve and what actions may result.

Specific to each unreclaimed site, key questions would be:

- Are contaminants, if present on site, the result of historical mining operations or related activities?
- Are the residual concentrations of arsenic, lead, or mercury present and above the human health action levels shown on Table 1 (on page 7)?
- Are the residual concentrations of cadmium, copper, zinc, arsenic, lead, or mercury present and above the storm water screening criteria shown on Table 2 (on page 8)?

Resulting alternative actions addressing the principal question regarding COC levels include the following:

- Perform additional remedy in the area if COC concentrations exceed action levels.
- Perform additional site-specific analyses if COCs exceed storm water screening criteria.
- If acceptable levels of COCs are met, take no action. (See Unreclaimed Area Decision Logic diagram in Appendix A.)

Step 3: Identify Information Inputs.

The purpose of this step is to identify the informational variables that will be required to resolve the decision statements and determine which variables require environmental measurements.

For each individual site, the following information is required to satisfy or resolve the decision statements:

- Existing data (described in Section 3.3.6 of the BPSOU Data Management Plan [Atlantic Richfield, 2020]) from the individual project area or a similar area to provide preliminary information on variability in sample measurements across the site. This will be important when designing the sampling strategy.
- Arsenic, cadmium, copper, lead, mercury, and zinc results from soil samples that are representative of metals concentrations within the individual project sites.
- BPSOU EPA-developed risk-based action levels for arsenic, mercury, and lead that will dictate remedial action, according to land zoning; and will lead to a resolution of the decision statement.
- BPSOU EPA-developed risk-based screening levels for cadmium, copper, and zinc that will dictate the screening level and inform possible remediation efforts.

Step 4: Define the Study Boundaries.

The purpose of this step is to define the spatial and temporal boundaries of the problem.

For each identified unreclaimed area, the site and sample locations will be delineated on a drawing and submitted with supporting documents to the Agencies for review and comment. Samples will be collected at each site to determine if the COC concentrations are above action/screening levels (Table 1 on page 7 and Table 2 on page 8. Each site is within the BPSOU boundary and, generally, the sites are connected by the main drainages at the base of the contributing areas. The work will focus on quantifying the presence of COCs and identifying potential pathways to surface water at each individual site.

Potential constraints that could delay fieldwork include adverse weather conditions or the inability to obtain property access. Major project delays resulting from these constraints will be recorded in the field logbooks and reported to the Agencies. Individual site sampling efforts are expected to take one to two days to complete. Sampling will be performed as weather conditions permit but most of the effort will be completed from June through October until all sites have been characterized.

Step 5: Develop the Analytical Approach.

The purpose of this step is to define the parameters of interest, specify action levels, and integrate any previous DQO inputs into a single statement.

For the BPSOU area, the EPA developed specific risk-based screening levels for human health COCs (arsenic, mercury. and lead) based on land-use exposure scenarios. Professional judgment by the Field Team Leader, informed by current county zoning, current land use, end land use, and guidance from the EPA Record of Decision (ROD) (EPA, 2006) will inform individual site action levels. The screening levels for cadmium, copper, and zinc will inform possible future remediation efforts. Field samples will be tested for pH at a minimum rate of 1 per 200-foot x 200-foot area. The action/screening levels are in Table 1 and Table 2:

Table 1. BPSOU Soil Action Levels for Human Health

Analyte	Solid Media	Action Levels
Lead[1]	Non-Residential/Residential	2,300 mg/kg/1,200 mg/kg
Arsenic[1]	Recreational/Commercial/Residential	1,000 mg/kg/500 mg/kg/250 mg/kg
Mercury[2]	Residential	147 mg/kg

1. From EPA Record of Decision Amendment (RODA) BPSOU, Table 2-1 (EPA, 2020a).
2. From Exhibit 1, Section 2.1.5, UAO Amendment (EPA, 2020a).
mg/kg: milligrams per kilogram.

Table 2. BPSOU Soil Screening Criteria for Storm Water COCs

Analyte	Action/Screening Levels
Cadmium [1,2]	20 mg/kg
Copper [1,2]	1,000 mg/kg
Zinc [1,2]	1,000 mg/kg
Lead[1,2]	1,000 mg/kg
Arsenic[1,2]	200 mg/kg
Mercury[1,2]	10 mg/kg
Any single analyte above 5,000 mg/kg	

1. From BPSOU CD, Appendix D Table 1, Waste Identification Criteria (EPA, 2020).
2. Screening levels to determine possible remediation efforts.
mg/kg: milligrams per kilogram.

Elevated levels of arsenic, cadmium, copper, mercury, lead, and zinc may have negative impacts on human health and surface water quality. If 3 or more contaminant screening level criteria listed in Table 2 are exceeded, or if 1 or more of the contaminant criteria exceeds 5,000 milligrams per kilogram (mg/kg), the site will be further analyzed to determine the materiality of the load to the degradation of surface water.

If results from any of the project site samples exceed BPSOU Soil Action Levels for Human Health, the site will be addressed in future remediation efforts. If screening criteria are exceeded for analytes listed in Table 2, additional analysis will be performed to determine the materiality of the load to the degradation of surface water.

All analytical data will be evaluated and validated consistent with the procedures described within this document and will determine data usability.

Step 6: Specify Performance and Acceptance Criteria

The purpose of this step is to specify the decision maker's tolerable limits on decision errors, which are used to establish performance goals for the data collection design.

There are limitations in extrapolating data over a given area and inherent variability of the matrix being sampled. Measurement error, occurring from variability of collection, preparation, and analyses of environmental samples will be minimized by implementation of this QAPP and individual site FSPs. This QAPP specifies processes to obtain the necessary data to estimate COC concentrations within the site while minimizing variability of the matrix, collection, preparation, and analyses, as feasible. Sampling design and measurement errors will be minimized by following the procedures outlined in this QAPP and the SOPs in Appendix B. Following these processes, utilizing only data which meet specified acceptance criteria for screening or enforcement quality, will ensure that an adequate quantity of information will be collected to determine COC concentrations across the site, and that the data usability will be assigned based on QA/QC criteria outlined in this QAPP.

Step 7: Develop the Plan for Obtaining Data.

The purpose of this step is to identify a resource-effective data collection design to generate data that satisfies the DQOs.

The data acquisition plan detailed in Section 3.0 is designed to ensure that data will be of sufficient quality and quantity to determine COC concentrations at each unreclaimed site and help determine if remedial action is needed. Any site-specific instructions or conditions will be detailed in the supporting documents for each site. The site-specific FSPs will present data collected under previous sampling efforts (related and current investigations), enabling comparison of existing data to newly collected data. Agency representatives are encouraged to participate in field activities and provide input on specific sample locations.

Evaluation of unreclaimed sites will include the following tasks and follow the specific measurement performance criteria listed in Section 2.4.1 and will allow the data gathered to be used in future remediation efforts.

- Complete a site inspection, including identification of physical hazards, such as subsidence areas, where present.
- Determine any rill depths and adjust sampling depths as needed if rill depths exceed stated sampling depths.
- Conduct the soil sampling activities.
- Capture pertinent data with daily logs and photographs.
- Develop draft and final data summary documents.

2.4.1 Measurement Performance Criteria for Data

Specific data validation processes ensure that analytical results are within acceptable limits. All information and data gathered according to this QAPP for each unreclaimed site will be checked to ensure they are usable for their intended purposes. An evaluation of analytical control limits and of the precision, accuracy, representativeness, comparability, and completeness (PARCC) parameters will be performed. If significant issues with the data are found, results will be discussed with the EPA and Montana Department of Environmental Quality (DEQ) project managers. The EPA, in consultation with DEQ, will then decide if the total study error could cause an incorrect decision. Using this approach, the probability of making an incorrect decision (i.e., either a false negative or positive) based on the information collected is considered small. Precision, accuracy, and completeness calculations formulas are presented in Appendix A. Quality control acceptance criteria for field and analytical data validation are provided in Table 6.

The definitions of the PARCC parameters are provided below along with the acceptance criteria for data collected.

Precision

Data precision is assessed by determining the agreement between replicate measurements of the same sample and/or measurements of duplicate samples. The overall random error component of precision is a function of sampling. The analytical precision is determined by the analyses of field duplicates and by replicate analyses of the same sample. An analytical duplicate is the preferred measure of analytical method precision. When analytes are present in samples at concentrations below or near the quantitation limit, precision may be evaluated using duplicate analyses of laboratory-prepared samples such as laboratory control sample (LCS) duplicates (LCSD) and laboratory matrix spike (MS) duplicate (MSD) samples. Precision can be measured as relative percent difference (RPD) or as relative standard deviation (RSD, also known as a coefficient of variation). See Precision Calculations in Appendix A.

For this QAPP, precision will be determined by the analyses of field duplicates, field replicates, laboratory (analytical) duplicates, confirmation samples, and the evaluation of the RPD or RSD for these various paired measurements. Information related to specific sites will be included in the individual site FSP or remedial action work plan. The RPD precision goal for soil duplicates will be 35% for sample pairs with both sample results being greater than 5 times the reporting limit (RL). For duplicate pairs with 1 or both sample results being less than 5 times the RL, a difference of less than or equal to 2 times the RL (difference $\leq$ 2xRL) will be used as the precision goal.

Accuracy/Bias

Accuracy of sample analysis is controlled primarily by the laboratory and is reported as bias. Accuracy is the degree of difference between the measured or calculated value and the true value. It is a measure of the bias or systematic error of the entire data collection process. Potential sources of systematic errors include the following:

- Sample collection methods.
- Physical or chemical instability of the samples.
- Interference effects during sample analysis.
- Calibration of the measurement system.
- Contamination.

Field and laboratory field blanks will be analyzed to assess artifacts introduced during sampling, transport, and/or analyses that may affect the accuracy of the data. The XRF blank and field check sample are used to assess accuracy of field analysis. The LCS and MS are used to measure accuracy, based on the percent recovery of the MS and LCS. Acceptable ranges for MS and LCS percent recovery are presented in Table 6. Additional laboratory QC samples may be used to assess accuracy as appropriate to the analytical method. Proposed minimum detection limits and reporting limits for the specific laboratory analytes are listed in Table 3. Accuracy in the field is assessed through the adherence to all sample handling, preservation, and holding times.

Table 3. Proposed Minimum Detection Limits and Reporting Limits for Specific Analytes

Analyte	Proposed Minimum Detection Limits (mg/kg)	Reporting Limit (mg/kg)
Arsenic[1]	0.153	1.00
Cadmium[1]	0.0341	0.15
Copper[1]	0.0731	0.50
Lead[1]	0.103	0.50
Zinc[1]	0.223	1.00
Mercury[2]	0.00868	0.02

1. EPA Method 6010 (EPA, 2014).
2. EPA Method 7471B (EPA, 2007).
Mg/kg: milligrams per kilogram.

Representativeness
Data representativeness is defined as the degree to which data accurately and precisely represent a characteristic of a population, parameter variations at a sampling point, or environmental conditions. Representativeness is a qualitative parameter that is most concerned with the proper design of the sampling program. Representativeness will be achieved through judicious selection of sampling locations and methods. This QAPP has been designed to ensure that the sample locations selected are representative of the medium being sampled and that there are a sufficient number of samples to meet the project DQOs and to satisfy the project remedial action design elements. Sample representativeness may also be evaluated using the RPD values for field duplicate results.

Comparability
Data comparability is defined as the measure of the confidence with which one data set can be compared to another. Comparability is a qualitative parameter but must be considered in the design of the sampling plan and selection of analytical methods, QC protocols and data reporting requirements. Comparability will be ensured by analyzing samples obtained in accordance with this QAPP as well as the appropriate SOPs, which are comparable to the sampling methods used during previous investigations at similar sites. All data will be reported in units consistent with standard reporting procedures so that the results of the analyses can be compared with results from previous investigations. Soil will be reported in units of milligrams per kilogram (mg/kg).

Completeness
Completeness refers to the amount of usable data produced during a sampling and analysis program relative to project objectives. The procedures established in this QAPP are designed to ensure, to the extent possible, that data will be valid and usable. To achieve this objective, every effort will be made to collect each required sample and to avoid sample loss.

2.5 Special Training/Certification

All field personnel conducting site investigations will be trained to collect samples and will review the requirements of this QAPP in a project meeting held prior to fieldwork. Hazardous Waste Operations and Emergency Response (HAZWOPER) training will be required for field

sampling personnel. All field personnel will read the QAPP document prior to the start of fieldwork and will acknowledge that they have read and understand the document at the time of the project meeting. Field personnel will be trained on how to use field equipment and in decontamination procedures and custody procedures in accordance with field data collection SOPs used for the sampling event (Appendix B.). This training will be documented within the appropriate section of each SOP. The CPM and Safety and Health Manager will be responsible for ensuring that training requirements are fulfilled.

Depending on individual company or agency safety policies, a review of the associated SSHASPs will be conducted with all field personnel prior to fieldwork to assess the particular hazards at the specific site and the control measurements that have been put in place to mitigate these hazards. The SSHASP review will cover all other safety aspects of working at the site including personnel responsibilities and contact information, additional site-specific safety requirements and procedures, and the emergency response plan.

Laboratories providing analytical services will have a documented QC program that complies with EPA Requirements for QAPPs (EPA, 2001). The laboratory QA personnel will be responsible for ensuring that all laboratory personnel have been properly trained and are qualified to perform assigned tasks.

2.6 Documentation and Records

This section describes procedures for documentation management and record keeping related to this QAPP and the individual site investigation reports from initial record generation through final data formatting and storage.

2.6.1 Property Access Agreements

Atlantic Richfield or BSB will request that property owners grant access to their properties for all remedial action-related activities including sampling. The CPM will manage access requests, track their status, and maintain copies of completed agreements received from property owners. Completed agreements will be photocopied and scanned with the electronic version stored on a server. Photocopied access agreements will also be copied to the project record files. Fieldwork will not proceed until access agreements have been finalized.

2.6.2 Field Logbook

All field sampling activities and field data collection will be recorded in a bound field logbook dedicated to the project or on field data sheets (XRF results) that are referenced in the logbook. All documents will follow SOP-SA-05 Project Documentation General (Appendix B). The CPM or Field Team Leader will be responsible for recording information including the sample collection date and time, weather conditions, field crew members, site visitors, samples collected, procedures used, field data collected, and deviations from the site FSP. Sufficient information should be recorded to allow the sampling event to be reconstructed without having to rely on the sampler's memory. Individual field team members may be responsible for required documentation based on specific tasks assigned by the CPM or the Field Team Leader.

Completed field data sheets and logbooks will be photocopied and scanned with the electronic version stored in the project file. Photocopied field records will also be copied to the project record files. No bound field logbooks will be destroyed or thrown away even if they are illegible or contain inaccuracies that require a replacement document.

2.6.3 Field Photographs

Field personnel will also document field-sampling activities using a digital camera, cell phone, or tablet. Documentation of all photographs taken during sampling activities will be recorded in the bound field logbook or appropriate field data sheets (refer to field SOPs for the individual site), and will specifically include the following for each photograph taken:

- The photographer's name, date, time, and the general direction faced.
- A brief description of the subject and the fieldwork portrayed in the picture.
- Sequential number of the photograph.

The digital files will be placed in project files with copies of supporting documentation from the bound field logbooks.

2.6.4 Chain of Custody Records

After samples have been collected, they will be maintained under strict chain of custody protocols in accordance with SOP-SA-04 Chain of Custody Form for Environmental Samples General (Appendix B). The field sampling personnel will complete a chain of custody form (Appendix C) for each shipping container of samples to be delivered to the laboratory for analysis. A copy of each as-transmitted chain of custody form will be scanned and stored in the project file. The chain of custody records will also be copied to the project record files.

2.6.5 Analytical Laboratory Records

Results received from the laboratory will be documented both in report form and in an electronic format. Laboratory documentation will include copies of the signed chain of custody forms, laboratory confirmation reports that include information on how samples were batched and the analyses requested, sample data packages that include the laboratory report and the electronic data deliverable (EDD), and any change requests or corrective action requests. Section 6.1.2 lists the laboratory reporting requirements in detail. The deliverable ("standard data package" or "report") issued by the laboratory will include data necessary to complete Stage 2A validation of laboratory results in accordance with specifications included in Section 6.0. Original hard copy deliverables and electronic files received from laboratory will be maintained with the project QA/QC records.

Excel spreadsheets have been developed to enable data retrieval for validation. These spreadsheets are populated during the data validation process and resubmitted to the data management team. The validated data, including associated validation qualifiers, codes, quality designation for each data point, and Level A/B status for each sample, are then uploaded to the

database. Analytical data submitted directly to the database coordinator will be uploaded to the EQuIS system once review and validation are complete. The QA/QC checks are in place to ensure that data upload is successful, and that data quality is preserved.

2.6.6 Evaluation Summary Reports

An evaluation summary report containing a DSR and DVR specific to each designated unreclaimed site will be prepared following data collection, validation, evaluation, and interpretation. The report will include figures displaying sample locations, analytical results, required declarations about the results (Section 2.3), and program records as detailed in Section 2.6.7. The summary report will be submitted to the Agencies for comment and approval.

2.6.6.1 Site Declaration

A site declaration stating whether a specific site is at or above human health action levels and/or storm water waste identification criteria listed Table 1 and Table 2, based on land use determination; whether the site is contributing metals-impacted sediment to existing or planned wet weather control features; and whether historical mine waste at the site is contributing to the degradation of surface water quality will be submitted to the Agencies for comment and approval. The site declaration will be contained in the evaluation summary report for each specific unreclaimed site.

2.6.7 Program Quality Records

Program quality records are documents that furnish objective evidence of the quality of items or services, activities affecting quality, or the completeness of data. These records will be organized and managed by the remedial action entity and will include the following, at a minimum:

- This QAPP and any approved revisions or addenda.
- Site-specific figures and supporting documentation.
- SSHASP and any addenda.
- Copies of SOPs for field data collection, with any updates or revisions or addenda to those SOPs.
- Formal incoming and outgoing project correspondence.
- Copies of completed access agreements for the individual properties sampled.
- Individual property maps including any field drawings and field photographs.
- Field documentation forms.
- Copies of all bound field logbooks.
- Copies of all field data sheets.
- Electronic field forms.
- Electronic copies of completed sample chain of custody forms.
- Copies of all laboratory agreements and amendments.
- As-received laboratory data packages (hard copy and electronic).
- Documentation of field and/or laboratory audit findings and any corrective actions.

- Draft and final delivered versions of all reports and supporting documents.

Any addendums or revisions to this QAPP, such as annual updates, will be electronically distributed to all parties identified on the distribution list by the Atlantic Richfield Liability Manager. All records will be maintained and archived electronically for future reference.

3.0 DATA ACQUISITION

This section describes the requirements to complete sampling events at a site to ensure the collection methods and handling procedures result in reliable data that can inform possible future efforts at the site.

3.1 Site Evaluation Objectives

The primary objective of preliminary site evaluations is to characterize the site to determine if sampling and testing are required due to historical mining operations. Site evaluations include visual examination of the site area to determine historical mining activity, identify presence of erosion such as gullies and/or rills, and the potential contribution to downstream contaminated sediment accumulations.

3.2 Soil Sampling Objectives

The primary objective of sampling the unreclaimed sites is to comprehensively characterize COC concentrations in the soils. Samples will be collected from multiple, hand dug test holes from possible waste sources as identified by trained professionals and outlined in the specific supporting documents for each individual site. If no potential source areas are identified, grab samples will be collected to characterize dissimilar soil types and usage areas.

For a specific site, the site layout figure and supporting documents will identify the number of potential samples to be collected, show the locations of each sample, and list any specific sample labeling requirements. Sampling will be conducted by professionals familiar with the sampling processes and the local area. If, during field activities, additional samples need to be collected to evaluate a potential source, the reason and sample collection method will be recorded in the field logbook. Field personnel and representatives from the Agencies (if present) will make the decisions regarding collection of additional “opportunistic” samples to characterize site conditions accurately.

If a site becomes inaccessible due to weather conditions, the sampling date will be adjusted as required. If access to the site is not granted (access agreement not signed by private property owner), the site will remain uncharacterized and be removed from further consideration, barring Agency intervention on the behalf of the sampling team.

To mitigate variability within soil samples, field personnel will use field XRF analysis, which provide instantaneous data that allows the field team to adjust the location and number of samples while at the site. Field XRF confirmation samples will be submitted to the laboratory for arsenic, cadmium, copper, lead, mercury, and zinc analyses at a rate of 1 per 10 samples, with

additional samples sent to the laboratory for samples with field concentrations of any one analyte at ± 35% of BPSOU Soil Action Levels for Human Health (Table 1) or with field concentrations of 1 or more analytes within ± 35% action level and 2 or more within ± 35% or exceeding 35% of BPSOU Soil Screening Criteria for Storm Water COCs (Table 2). The 35% criteria may be adjusted based on statistical analyses of the confirmation sample results.1/10 or for any sample When confirmation analyses are performed by laboratory methods, these results will supersede XRF data to determine final concentrations detected in the sample.

All sampling will be conducted as per SOPs listed in the Table 4 below. All applicable SOPs are provided in Appendix B.

Table 4. List of Applicable SOPs for Sampling

Reference Number	Title and Revision Date	Originating Organization
SOP-S-01	Surface Soil Sampling 12/11/2014	Pioneer
SOP-S-02	Subsurface Soil Sampling 11/23/2020	Pioneer
SOP-SA-01	Soil and Water Sample Packaging General 1/4/2018	Pioneer
SOP-SA-04	Chain of Custody Forms for Environmental Samples 11/12/2020	Pioneer
SOP-SA-05	Project Documentation General 1/4/2018	Pioneer
SOP-SFM-01	Field Measurement of pH in Soil 1/4/2018	Pioneer
SOP-SFM-02	Operating XL3-X-Ray Fluorescence Analyzer General 1/4/2018	Pioneer
SOP-DE-01	Personal Decontamination Procedures General 1/4/2018	Pioneer
SOP-DE-02	Equipment Decontamination 9/8/2020	Pioneer
ENV-SOP-MIN4-0052	Metals Analysis by ICP - Method 6010 and 200.7 - 6010-200.7 Rev. 07 11/3/2021	Pace
ENV-SOP-MIN4-0054	Mercury in Liquid and Solid/Semi-Solid Waste by 7470A, 7471, 7471B, and 245.1 - 7471B Rev 06, 8/10/2021	Pace
ENV-SOP-MIN4-0056	Metals Preparation of Solid Samples for Analysis by ICP and ICP-MS by 3050B - Preparation of Solid Samples Rev 04 10/6/2021	Pace

3.2.1 General Sampling Procedure

All unreclaimed site areas will be sampled according to the general procedures in this QAPP and the more detailed procedures listed in the specific site layout figure and supporting documents. Prior to soil sampling activities, a site condition inspection and identification of site physical hazards, will be completed. Sample locations identified in the site layout figure will be checked to ensure they meet the sampling objectives. Potential source areas will be sampled preferentially. Depending on XRF readings or field conditions, additional opportunity samples may be obtained to define the extent of any contaminants found. If no visually identifiable source areas are present, samples will be collected from general locations to characterize soil types and usage areas. A minimum of 3 samples will be collected per acre at each site. Each sample is composed of 3 test pits installed (typically triangular) surrounding the identified sample location. Subsamples (0-2 inch, 2-6 inch, and 6-12 inch) are composited using equal aliquots of each depth interval from the 3 discrete test pit locations. As a rule, the diagonal distance between the points will be 10 feet, depending on soil homogeneity. The diagonal distance can be adjusted in the field to account for soil differences. Materials such as plant matter, debris, and large rocks

will be removed, to a reasonable extent, prior to placing the sample in the sample container. Samplers will collect samples using the following protocol:

Collect Samples – Test Pit Method

1. Remove vegetation and debris from the surface prior to digging. Excavate each subsample pit to a minimum of 12″.
2. Don a new pair of disposable nitrile gloves.
3. Mark each sample collection interval using a tape measure or marked equivalent. Unpainted golf tees may be used to mark the holes at 12″, 6″, and 2″.
4. Use a new disposable plastic scoop for each sample.
5. Use the disposable plastic scoop to scrape the wall of the pit to expose a fresh surface for sampling.
6. Collect composite samples from bottom to top, avoiding cross contamination (12-6″, 6-2″, and 0-2″) composed of equal aliquots from subsample test pit intervals. Excessive vegetation, tree roots, hard rock areas, and other sampling obstacles may cause problems with planned sample locations. If obstacles are encountered during sampling, choose a new subsample location within 10 feet of the original location.
7. If a vegetative mat is present, separate it from the soil surface by shaking (or with the plastic scoop) over the 0-2″ sample collection bag, catching as much loose material as possible.
8. Use the disposable plastic scoop to scrape the wall of the pit to expose a fresh surface for sampling.
9. Collect a sample from the freshly exposed surface with the plastic scoop by scraping from the bottom to the top of the specified interval, removing material evenly from all around the pit in accordance with Surface Soil Sampling General (SOP-S-01) and Subsurface Soil Sampling (SOP-S-02) included in Appendix B.
10. Screen the soils with a stainless steel #10 (2-millimeter [mm]) screen or disposable sieve into an appropriately labeled resealable plastic bag. If stainless steel screens are utilized, field blank/equipment rinsate blank samples must be collected to verify field decontamination procedures.
11. If debris is identified in the screen, remove the debris and make a note in the field logbook.
12. Record sample identification, collection information, and pertinent information in the field logbook or on the field data sheet.

Collect Samples – Stainless Steel Probe Method

1. Define the composite sampling interval and test locations.
2. Insert probe to the sampling depth.
3. Remove and composite proper depth profile (12-6″, 6-2″, and 0-2″).
4. Sieve the sample if gravelly as described in step 9 under **Collect Samples – Test Pit Method** (listed previously).

5. Record appropriate data in the field logbook.
6. If stainless steel probe is utilized, field blank/equipment rinsate blank samples must be collected to verify field decontamination procedures.

Field personnel will analyze samples in the field using a Niton XL3 XRF, or equivalent. This will allow the field team to adjust the location and number of samples to sufficiently characterize each site. Prior to field XRF analysis, the sampler will follow the general procedures below. Specific details are included in SOP-SFM-02 (Appendix B).

XRF Analysis

1. Thoroughly homogenize the pre-sieved sample by kneading the soil.
2. Place sample aliquot into a pre-labeled 1-quart resealable plastic bag so that is fits in the analyzer measurement stand.
3. Compact the material so that there is a flat surface on the area to be analyzed and visually inspect this area to ensure that only fines will be present in the XRF aperture.
4. Place the sample bag on the measurement stand and take the measurement.
5. Record the results for the selected metals on the XRF field data sheet (Appendix C).
6. Complete duplicate and replicate XRF analyses on at least 5% of the samples analyzed with the XRF unit.

3.2.2 Sample Identification

The sampler will identify each sample and mark the sample bags as follows: operable unit, area, sample site, month, day, year, sample interval, and unique number. For example, BPSOU-XXXXZZZZ-MMDDYY-# where:

- BPSOU denotes Butte Priority Soils Operable Unit.
- XXXX denotes the specific area (i.e., UR32 for UR-32).
- ZZZZ denotes the sample site within the specific area (i.e., SS01 for SS-01).
- MM denotes the month in which the sample was collected (07 for July, 08 for August, etc.).
- DD denotes the day of the month on which the sample was collected (01, 02, etc.).
- YY denotes the year in which the sample was collected (18 for 2018).
- # denotes sample interval where:

  1 = 0-2 inches,
  2 = 2-6 inches,
  3 = 6-12 inches.

A sample marked as BPSOU-UR32SS01-091218-2 means the sample was collected in the BPSOU UR-32 area at sample site SS-01 on September 12, 2018, at the 2-6-inch level.
For field duplicates, "-FD" will be added to the end of the primary sample. For example, a field duplicate of BPSOU-UR32SS01-091218-2 would be BPSOU-UR32SS01-091218-2-FD.

For XRF duplicate, "-D" will be added to the end of the primary sample. For example, an XRF duplicate of BPSOU-UR32SS01-091218-2 would be BPSOU-UR32SS01-091218-2-D.
For XRF replicates, "-R" will be added to the end of the primary sample. For example, an XRF replicate of BPSOU-UR32SS01-091218-2 would be BPSOU-UR32SS01-091218-2-R.

3.2.3 Sampling Equipment

Resources and field equipment used for the soil sampling will include the following (at a minimum):

- Copy of the current approved QAPP.
- Field notebook, pens, camera, batteries, and cell phone/tablet.
- Maps of sample locations.
- GPS unit.
- Nitrile gloves.
- Assorted shovels and breaker bars.
- Soil Probe.
- Disposable plastic scoops.
- #10 (2 mm) stainless steel screens and/or disposable sieves.
- Disposable foil pans.
- 1-quart resealable plastic bags.
- Niton XL3 XRF Analyzer, or equivalent.
- Hanna Instruments, HI 99121 Soil pH Meter, or equivalent.
- Equipment and deionized water for decontamination.
- Sample coolers, ice, and tape.
- Required Level D Personal Protective Equipment (PPE) as detailed in the SSHASP.

Any problems due to equipment failures will be addressed by the Field Team Leader and resolved in a timely and orderly fashion. All actions will be documented in the field logbook.

3.2.4 Decontamination Procedures

Field personnel will decontaminate all non-disposable sampling equipment after use at each sampling location according to SOP-DE-02, Equipment Decontamination General (Appendix B). Disposable equipment and PPE intended for one-time use will not be decontaminated but will be packaged for appropriate disposal as a solid waste in the local landfill. Soil removed from holes during excavation will be returned to the sample holes.

Field personnel will decontaminate reusable sampling equipment within the site boundaries at a centralized location. Sampling equipment will be decontaminated using the procedure below. All equipment will also be decontaminated before leaving the site to prevent off-site transport of contaminants (refer to SOP-DE-02, Equipment Decontamination General).

- Rinse with water.
- Wash with non-phosphate detergent.

- Rinse three times with deionized water.
- Air dry.

For safety, all personnel will undergo decontamination procedures when leaving a contaminated area. Personnel decontamination includes routine practices as well as emergency decontamination. All personnel will follow SOP-DE-01, Personnel Decontamination Procedures General (Appendix B) protocols and take every measure possible to prevent the spread of potentially contaminated materials to clean areas.

3.2.5 Sample Containers and Handling

Soil samples will be collected in a labeled plastic bag, mixed, and analyzed using the field XRF. Individual soil samples will be placed in a cooler as soon as possible after sample collection and XRF analysis. If the laboratory requires different sample containers, the laboratory will provide the container and field personnel will handle the containers in such a way as to prevent accidental contamination. Field personnel will wear a new pair of nitrile gloves when transferring samples from the bag used for XRF analysis to the laboratory sample container.

Samples will be stored in insulated coolers with double-bagged ice as necessary to maintain a temperature of at less than 6 degrees Celsius (ºC) and then transported to the laboratory. Table 5 lists the required sample preservation, containers, and holding times. Sample holding times are established to minimize chemical changes in a sample prior to analysis or extraction. A holding time is defined as the allowable time between sample collection and analysis recommended to ensure accuracy and representativeness of analysis results, based on the nature of the analytes of interest and chemical stability factors.

Table 5. Required Sample Preservation, Containers, and Holding Times

Media	Parameter	Analytical Method	Preservation[2]	Holding Time	Sample Size	Sample Container
Solid	Total Metals*	EPA 6010, 7471B[1]	≤ 6ºC (but not frozen)	180 days (28 days for mercury)	4 ounces	Resealable plastic bag

* Arsenic, cadmium, copper, lead, mercury, and zinc.
[1]. EPA Method 6010 (EPA, 2014) and EPA Method 7471B (EPA, 2007) for mercury.
[2] Temperature requirement is for mercury analysis only (EPA, 2020b).
°C: degrees Celsius.

3.2.6 Sample Custody Protocols

Once the samples are collected, they will be maintained under strict protocols in accordance with SOP-SA-04, Chain of Custody Forms for Environmental Samples General (Appendix B). Field personnel will complete a chain of custody form (Appendix C) for each shipping container (e.g., cooler, ice chest, or other container) to be delivered to the laboratory. The sampler will be responsible for initiating and filling out the chain of custody form. The chain of custody form for a shipping container will list only the samples in that shipping container. Information contained on the form will include the following:

- Project name and identification number.
- Sampler's signature and affiliation.
- Date and time of collection.
- Sample identification number and matrix.
- Analyses requested.
- Remarks or additional notes to laboratory personnel (e.g., do not use for QC).
- Signature of persons relinquishing custody, dates, and times.
- Signature of persons accepting custody, dates, and times.

The sampler will cross out any blank spaces on the chain of custody form below the last sample number listed. Any documentation, including chain of custody forms, placed inside the cooler during sample shipment should be placed inside a resealable plastic bag.

The sampling person whose signature appears on the chain of custody form is responsible for the custody of the samples from the time of sample collection until custody is transferred to a designated laboratory, a courier, or another project employee for the purpose of transporting the samples to the designated laboratory. The sample is considered to be *in custody* when the sample is:

- in the responsible individual's physical possession;
- in the responsible individual's visual range after having taken possession;
- secured by the responsible individual so that no tampering can occur;
- secured or locked by the responsible individual in an area in which access is restricted to authorized personnel; or
- transferred to authorized personnel.

A completed chain of custody form will be placed in a sealed zip lock bag and taped to the inside of the cooler lid. Custody seals will be attached to each cooler and samples will be delivered to the laboratory for analysis within the holding times specified for the test requested (Table 5).

The field sampler will file one copy of each chain of custody form with the project files as a temporary record of sample transfer. The original form will accompany the samples and be returned to the contractor as part of the laboratory QA/QC requirements. The original form will be filed as part of the project's permanent records.

3.2.7 Laboratory Sample Handling and Storage

When the laboratory receives the shipment, laboratory personnel will review the chain of custody form to verify it is complete and then the designated technician will sign and date it. Any broken custody seals, damaged sample containers, sample labeling discrepancies between container labels and the chain of custody form, or analytical request discrepancies will be noted on the chain of custody form. If any of these conditions exist, the laboratory will notify the Field Team Leader and CPM. The Field Team Leader and CPM will resolve discrepancies or non-conformance issues before the samples are analyzed. The laboratory will provide the Field Team Leader and CPM with a copy of the chain of custody form and the associated sample receipt information. The typical sample receipt information provided includes sample receipt date, sample identifications transcribed from the chain of custody forms, sample matrix type, and the list of analyses to be performed for each sample. The laboratory will be responsible for following their internal custody procedures from the time of sample receipt until sample disposal.

3.3 Analytical Methods

The anticipated field and laboratory analytical methods to be used are detailed below.

3.3.1 Field Analysis

Field personnel will use a Niton XL3 XRF, or equivalent, for the XRF field analysis. A sample stand, which allows the samples to be analyzed in plastic bags, will be used during analysis to ensure consistent exposure times and position of the XRF aperture for each sample. Results for the analytes (listed in Table 1) will be recorded on the field data sheets. Samples will be tested for pH in the field using the Hanna Instruments, HI 99121 Soil pH Meter, or equivalent.

3.3.2 Sedimentation Analysis

The CPM will determine whether the site contributes metals-impacted sediment to waterways or existing infrastructure and rate the site impacts as marginal (little to no sediment impacts), moderate (some impacts that may need maintenance efforts), or major (remediation necessary). Each site will be rated on the following criteria:

1. Presence of rills. If present, determine the amount of soil lost.
2. Concentrated outflow. Check outflow for soil loss.
3. Sediment in downstream infrastructure. Determine the amount of soil in the infrastructure and the last maintenance operation. If maintained, determine the amounts of material removed.
4. Determination as to whether the infrastructure is part of Superfund or Reclaimed areas. If Superfund, maintenance will be performed under an Operations and Maintenance Plan; if Reclaimed, opportunistic maintenance will be performed per a reclaimed area Monitoring and Maintenance Plan.
5. Condition of downstream infrastructure. Determine if flow rates are impeded by poor condition.

6. Sediment loading contributions. Check for contributing sediment loading above the site in question.
7. Linkage to Silver Bow Creek. Determine if the drainage links to Silver Bow Creek.

Information on each of the above criteria will be documented with photographs.

3.3.3 Laboratory Analysis

Confirmation samples will be submitted to the laboratory for analysis per criteria outlined in Section 2.3 and Section 3.2. The actual number of sample locations will be evaluated in the field based on environmental conditions of the site and after consultation with the Agencies. Samples will be prepared for metals analysis in accordance with Table 5. Sample turnaround time will be standard (10 days from date of sample receipt), unless rush confirmation is deemed necessary by CPM. Laboratory analysis will be performed in accordance with EPA Test Methods for Evaluating Solid Waste, Physical/Chemical Methods (SW-846) 6010D Inductively Coupled Plasma Optical Emission Spectrometry (ICP-AES) (EPA, 2014) and 7471B Mercury in Solid or Semi-Solid Waste (Manual Cold-Vapor Technique) (EPA, 2007).

3.3.4 Laboratory Audit

The laboratory QA manager will conduct internal laboratory audits to evaluate compliance with the project requirements and this document. The laboratory will be responsible for verifying that QC procedures are followed and that the results of QC analyses are within the specified acceptance criteria, as well as for implementing corrective action if the QC acceptance criteria are not met.

3.3.5 Sample Disposal

Laboratory samples will be disposed of by the laboratory after all analyses have been completed. Field samples will be archived until confirmations have been completed and approved.

3.4 Quality Assurance/Quality Control

3.4.1 Field QC Samples

Field QC samples are used to identify any biases from transportation, storage, and field handling processes during sample collection and to determine sampling precision. All field QC samples will be delivered with field samples to the laboratory. This section includes brief descriptions of the QC samples to be collected during sampling activities along with frequency, collection, and analytical instructions. The measured values of a standard will be compared to the expected results and if a measured value falls outside this range, then the check sample will be reanalyzed. If the value continues to fall outside the acceptance range, the sampler will note this information on the XRF log. If any of the check sample results indicate that the XRF is not analyzing accurately, the XRF will be cleaned, turned off, and the energy calibration rerun. This information will be noted in the logbook and on the XRF field data sheet. The batch of samples analyzed prior to the unacceptable calibration verification check samples will be reanalyzed.

3.4.1.1 Equipment Rinsate Blanks/Field Blanks

Field personnel will analyze equipment rinsate blanks to assess the efficiency of field equipment decontamination procedures in preventing cross contamination of samples, at a rate of 1 per 20 samples, only if reusable equipment is utilized. If an equipment rinsate blank is collected, a field blank will also be collected simultaneously. Equipment rinsate blanks will be created by pouring certified distilled or deionized water over or through decontaminated (clean) sampling equipment that has been used to collect investigative samples, and subsequently collecting this (poured) water in prepared sampling containers. Field blanks will be created by pouring certified distilled or deionized water directly into sampling containers. The blank samples will be shipped with the associated field samples and submitted for the same analyses as the associated samples. All blanks submitted to the laboratory will be designated as not for use in preparation of MS samples or analytical duplicate samples.

3.4.1.2 Field Duplicate

A field duplicate consists of 1 well-mixed and homogenized sample that is split in the field into 2 samples and placed in different sample containers for separate analyses. Each split will have its own sample number. Both split samples will be analyzed for identical chemical parameters. The results of the field duplicate will be compared to determine laboratory and sampling precision. Field duplicate samples will be collected at a frequency of 1 per 20 samples or once per sampling event, whichever is more frequent.

3.4.1.3 pH Calibration Check

The pH calibration check is performed immediately after calibration of the pH probe and should be within 0.10 pH units. If the acceptance criterion is not met, field personnel will terminate analysis, correct the problem, recalibrate the unit, and attempt a new pH calibration check.

3.4.2 Field XRF Quality Control Samples

3.4.2.1 Energy Calibration Check

Field personnel will run a preprogrammed energy calibration check on the equipment at the beginning of each working day. If the individual believes that drift is occurring during analysis, that individual will run the energy calibration check. The energy calibration check determines whether the characteristic X-ray lines are shifting, which would indicate drift within the instrument.

3.4.2.2 Blank Samples

The silicon dioxide sample, as provided by Niton, is a “clean” quartz or silicon dioxide matrix that contains concentrations of selected analytes near or below the XL3 XRF machine lower limit of detection. These samples are used to monitor for cross contamination. Field personnel will analyze this sample at the beginning of each day, once per every 20 samples, and at the end

of each day's analysis. The sample information will be recorded as "SIO_2" on the XRF field data sheets. This sample will also be analyzed whenever field personnel suspect contamination of the XRF aperture. Any elements with concentrations above the established lower limit of detection will be evaluated for potential contamination. If it is determined that the concentration is higher than that recorded at the start of the day, the probe window and the silicon dioxide sample will be checked for contamination. If it is determined that contamination is not a problem, and the concentration is significantly above the limit of detection, sample results will be qualified by the XRF operator as 'J' estimated, and the problem recorded on the XRF field data sheet and in the logbook. If the problem persists, the XRF will be returned to Niton for calibration.

3.4.2.3 Calibration Verification Check Samples

Calibration verification check samples help check the accuracy of the XL3 and assess the stability and consistency of the analysis for the analytes of interest. A check sample will be analyzed as one of the initial samples, once per every 20 samples and as the last analysis. Results for the check sample (standard reference material [SRM]) will be recorded on the individual site XRF field data sheets and identified as a check sample. There will be 3 Niton-provided SRM check samples for the project: NIST 2709a- Joaquin Soil, USGS SdAR-M2 (an SRM created by the U.S. Geological Survey [USGS]), and a Resource Conservation and Recovery Act (RCRA) sample. There will also be Niton-provided machine-specific expected results for several elements for the check samples. Pioneer has further refined the range of expected results for each SRM standard for each of the field XRFs in use. The measured values of a standard will be compared to the expected results and if a measured value falls outside this range, then the check sample will be reanalyzed. If the value continues to fall outside the acceptance range, this information will be noted on the XRF log. If any of the check sample results indicate that the XRF is not analyzing accurately, the XRF will be cleaned, turned off, and the energy calibration rerun. This information will be noted in the logbook and on the XRF field data sheet. The batch of samples analyzed prior to the unacceptable calibration verification check samples will be reanalyzed.

3.4.2.4 Duplicate Samples

The XRF duplicate samples will be analyzed to assess reproducibility of field procedures and soil heterogeneity. To run a duplicate sample on the Niton XL3, field personnel will remove the sample bag from the analytical stand, knead it once or twice, and replace it in the stand to be analyzed a second time. Duplicate samples will be recorded on the XRF field data form with a D designator in the sample identification number. One duplicate sample will be analyzed at the rate of 1 per 20 natural samples.

3.4.2.5 Replicate Samples

Field personnel will analyze a replicate sample at the rate of 1 per 20 natural XRF samples. To run a replicate sample on the Niton XL3, once the primary sample analysis has been completed, requires restarting the XRF to analyze the same sample a second time with the same soil in the XRF aperture. Replicate samples help in assessing the stability and consistency of the XRF analysis. Replicate sample results will be recorded on the XRF field data form and designated with an R in the sample identification number.

3.4.2.6 Confirmatory Samples

The comparability of the field XRF analysis with laboratory samples will be determined by submitting field XRF-analyzed samples for analysis to the laboratory. The confirmatory analyses can be used to verify the quality of the field XRF data. All samples submitted to the laboratory will be analyzed using the field XRF prior to submittal. The samples analyzed by field XRF will be submitted to the laboratory for metals testing (Table 1) and the results will be used to verify field XRF results and to develop a statistical relationship to the laboratory XRF results.

3.4.3 Laboratory Quality Control Samples

Laboratory QC samples are introduced into the measurement process to evaluate laboratory performance and sample measurement bias. Laboratory QC samples may be prepared from environmental samples or generated from standard materials in the laboratory per the internal laboratory SOPs.

3.4.3.1 Laboratory Blanks

Method blanks will be used to monitor laboratory processes and performance. A method blank is a volume of deionized water or a specified weight of inert material for solid samples that is carried through the entire sample preparation and analyses procedures. The method blank volume or weight will be approximately equal to the sample volumes or sample weights being processed. Method blanks are used to monitor interference caused by constituents in solvents and reagents and on glassware and other sampling equipment. Blank results outside of specified control limits will be re-run and/or flagged by the laboratory per the QC requirements of the analytical method.

3.4.3.2 Laboratory Control Samples

An LCS, or a blank spike, control sample of known composition that is analyzed using the same sample preparation, reagents, and analytical methods employed for the project samples. The LCS is obtained from an outside source or is prepared in the laboratory by spiking reagent water or a clean solid matrix from a stock solution that is different from that used for the calibration standards. The LCS is the primary indicator of process control used to demonstrate whether the sample preparation and analytical steps are in control, apart from sample matrix effects. If the LCS recovery falls outside the specified control limits, the samples will be re-run and/or flagged by the laboratory per the QC requirements of the analytical method.

LCS analyses will be performed every 20 samples; failure will trigger corrective action and reanalysis of non-detect samples per laboratory method (Appendix B).

3.4.3.3 Analytical Duplicates

Analytical duplicates are samples that are split in the laboratory at some step in the measurement process and then carried through the remaining steps of the process. Duplicate analyses provide

information on the precision of the operations involved. As the analytical duplicates are a pair of subsamples from a field sample taken through the entire preparation and analyses procedure, any difference between the results indicates the precision of the entire method in the given matrix. Analyses of analytical duplicates and MS duplicates monitor the precision of the analytical process. The frequency of analyses, precision goals, and corrective action information pertaining to analytical duplicates are included in example SOPs included in Appendix B. Information related to specific sites will be included in the individual site documents. If the analytical duplicate precision falls outside the specified control limits, the samples will be re-run and/or flagged by the laboratory per the QC requirements of the analytical method.

3.4.3.4 Matrix Spikes

Laboratory MS samples are used to evaluate potential sample matrix effects on the accurate quantitation of an analyte using the prescribed analytical method. The MS and MS duplicates are prepared by adding an analyte to a subsample of a field sample before sample preparation and analyses. A percent recovery is calculated from the concentrations of the analyte in the spiked and unspiked samples. Control limits vary based on laboratory method. MS and/or MSD failure will trigger corrective action including, for some analyses, performing a post digestion spike.

3.4.3.5 Post Digestion Spike

Post digestion spikes (PDS) will be prepared and analyzed based on laboratory method and as corrective action in the event of MS and/or MSD failure. Control limits also depend on the method and are contained in the applicable laboratory method and SOP included in Appendix B.

3.5 Instrument Testing, Inspection, and Maintenance

3.5.1 Field Equipment

The Field Team Leader or designee will examine field equipment to certify that it is in proper operating order prior to its first use and at intermittent intervals during the day. Equipment, instruments, tools, and other items requiring preventative maintenance will be serviced in accordance with the manufacturer's specified recommendations. Any routine maintenance recommended by the equipment manufacturer will also be performed and documented in field logbooks or appropriate data sheets. Equipment will be inspected and the calibration checked, if applicable, before it is used. Should equipment deficiencies be found, including calibration failures, the equipment will be immediately removed from service and repaired. Specialized repair parts will be purchased from the manufacturer. Once equipment failure has been resolved and testing/calibration demonstrates proper equipment function, the particular piece of equipment will be returned to service. The Field Team Leader, or designee, will be responsible for field equipment checks and maintaining the Equipment Log.

3.5.2 Laboratory Equipment

Instruments used by the laboratory will be maintained in accordance with each laboratory's QA plan and analytical method requirements. All analytical measurement instruments and equipment

used by the laboratory will be controlled by a formal calibration and preventive maintenance program. Required equipment for XRF analysis of soil samples is a drying oven, sieves, a grinder, and an x-ray fluorescence analyzer.

The laboratory will keep maintenance records and make them available for review, if requested, during laboratory audits. Laboratory preventive maintenance will include routine equipment inspection and calibration at the beginning of each day or each analytical batch, per the laboratory internal SOPs and method requirements.

3.6 Inspection/Acceptance for Supplies and Consumables

All supplies and consumables received for the project (e.g., sampling equipment, XRF blanks and SRMs, etc.) will be checked for damage and other deficiencies that would affect their performance. The types of equipment that will be needed to complete sampling activities are described in the relevant SOPs. The Field Team Leader or designee will inspect field supplies.

Per laboratory QA procedures, laboratory personnel will be responsible for inspecting laboratory supplies.

4.0 DATA MANAGEMENT

The Contractor will maintain all project records, either electronic or hard copy, to include the following:

- Individual site maps (hard copy or scanned field drawings and electronic files).
- Project documents, with any approved modifications.
- Field documentation.
- Chain of custody forms.
- Laboratory documentation (results received from the laboratory will be documented both in report form and in an electronic format).
- Data summary reports.

Contractor will maintain the project field and laboratory records at a location in Butte, Montana. The CPM will be responsible for managing the project documents. The original field and laboratory documents will be filed chronologically and scanned into a Portable Document Format (PDF) file for future reference. The electronic versions of these records will be maintained on a central server system that is backed up daily.

4.1 Field Data Management

Field data provides information on conditions at the time of sampling and a permanent record of field activities. Field data may be categorized as spatial and non-spatial data.

Spatial Data
Spatial data includes features (such as point, lines, or polygons) geographically referenced (georeferenced) to a known coordinate system. Spatial data produced for the unreclaimed sites

will be provided in Montana State Plane North American Datum of 1983 (NAD83) format. Site photographs will also be georeferenced to include photograph location and direction of picture. To produce spatial data features, GPS capable devices may be used. Photographs will also include a timestamp feature to include the time and date that photograph was taken, and a general description of the photograph will be logged.

Spatial data will transfer to the BSB reclamation database upon finalization and completion of the project for long-term maintenance and archival by BSB.

Non-spatial Data
Non-spatial field data may consist of project notes and observations collected in a project logbook, preliminary field sampling results, and documentation obtained during collection of samples such as field forms and/or chain of custody forms.

A dedicated project field logbook will be used to record pertinent field notes for each sampling effort throughout the project. The project logbook will be maintained by the Field Team Leader for each sampling event. Pages from the project logbook may be transferred to an electronic format and saved with project data. No pages will be removed from the logbook. When logbooks are filled, a new project logbook will be initiated to continue with project documentation. Each logbook will have a unique sequential project number. Upon completion of the project all logbooks will be retained with project files and archived in accordance with the Superfund project archival requirements.

Electronic or hard copy field data forms (refer to individual SOPs) may be used as appropriate for each field sampling occurrence. Each form will have a unique document control number. Once completed, the forms will be checked for accuracy and completeness and saved. The Field Team Leader will maintain field data throughout the project phases. Hard copy field data will be maintained in the project's central data file, where original field and laboratory documents will be filed chronologically for future reference. The electronic versions of these records will be maintained on a central server system that is backed up on a daily basis.

Final data will be transferred to BSB personnel and uploaded to the BPSOU Soils database.

4.2 Laboratory Data Management

Analytical data received from the laboratory in an electronic data package with the results in Microsoft Excel format. The CPM imports the raw data results into the project database for long-term data retention. Additionally, data are imported to a centralized validation database to complete data validation and reporting. Preliminary data are used for performance monitoring reporting. The CPM is responsible for ensuring electronic data are imported into the project database.

4.2.1 Laboratory Electronic Data Deliverable

Each electronic data package, as described in the previous section, will be accompanied by an EDD prepared by the laboratory. Additional laboratory QC data can be included in the EDD.

At a minimum, the data packages from the laboratory will contain the following information:

- A narrative addressing any anomalies encountered during sample analysis, and a discussion of any exceedances in the laboratory QC sample results.
- Analytical method references.
- Definition of any data flags or qualifiers used.
- Chain of custody documentation signed and dated by the laboratory to indicate sample receipt.
- Method detection limits and reporting limits.
- Analytical results for each field sample.
- Blank and QC sample results (as applicable).

5.0 ASSESSMENTS AND RESPONSE ACTIONS

Assessment and oversight of data collection and reporting activities are designed to verify that sampling and analyses are performed in accordance with the procedures established in this QAPP. The audits of field and laboratory activities include two independent parts: internal and external audits. Internal audits will be performed by the Contractor QAO and/or Atlantic Richfield QAM as necessary. External audits will be performed by the Agencies as necessary.

5.1 Corrective Actions

Assessment of sampling data will be performed during fieldwork on a daily basis. Any equipment malfunctions and data outliers will be reviewed by field technicians and reported to the CPM. All activities will be documented within the project logs. Equipment malfunctions will be remedied by following manufacturers' recommendations. Corrective actions during fieldwork will include replacing/repairing defective equipment and resampling to verify or negate original results. All field personnel and the CPM will have the authority to stop work until any issues are remedied.

Laboratory assessments and corrective actions will follow established procedures and published performance criteria common to accredited facilities and will be documented and reported by the laboratory to the CPM. If a performance criteria issue is unresolved by established laboratory procedures, the CPM, in consultation with the Agencies, will resolve the issue by reanalyzing or resampling. Any actions outside the scope of this QAPP will be reviewed and approved by the Agencies prior to work being completed.

5.2 Corrective Action during Data Assessment

The Contractor QAO may identify the need for corrective action during data assessment. Potential types of corrective actions could include resampling of an area by field personnel, reanalysis of samples by the laboratory, or resubmission of data packages with corrected clerical errors. The appropriate and feasible corrective actions will depend on the ability to mobilize

field personnel and whether the data to be collected are necessary to meet the required QA objectives (e.g., the holding time for samples is not exceeded, etc.). Any corrective actions outside the scope of the project documents will be performed after consultation with the Agencies. Corrective actions of this type will be documented by the Contractor QAO and will be included in any subsequent reports.

5.3 Quality Assurance Reports to Management

The information to be reported to and retained by the CPM includes the following:

- Description of field activities.
- Physical characteristics of the study area.
- Field documentation.
- Field measurements/analyses.
- Equipment calibration and preventive maintenance activities.
- Results of data precision and accuracy calculations.
- Evaluation of data completeness and contract compliance.
- Field and laboratory QA deficiencies and recommended or implemented corrective actions.
- Data validation reports and laboratory data reports.
- Deviations to the approved QAPP including an explanation for the deviation and the effect on data quality and usability, if any.

This information will be included in the DSR at the completion of each project. A draft version of the report will be submitted to the Agencies for review and a final version will be submitted for approval. The CPM will be responsible for preparing the report

6.0 DATA VALIDATION AND USABILITY

Data validation and usability elements determine if the data meet project DQOs described in Section 2.4 and evaluate the data against the method, procedure, or contractual requirements. Assessments related to data verification, validation, and usability will be completed as summarized:

- Review of field data and comparison of data to anticipated range(s).
- Secondary review of field data entered into electronic device(s) to identify obvious anomalies.
- Screening level review of preliminary results from the laboratory.
- Data validation by qualified, independent data validation personnel who are not associated with data collection or sampling responsibilities and who have applicable training.
- Assessment of data by the project team for usability as described below.

Laboratory analytical and field XRF data collected under this QAPP will undergo data verification and validation following the requirements defined in EPA *Guidance for Labeling Externally Validated Laboratory Analytical Data for Superfund Use* (EPA, 2009). Stage 2A data verification and validation will be performed on all field and laboratory metals analyses. Laboratory analytical data validation will include Stage 2A verification and validation of standard data packages provided. Comparison of data against historical values will be performed and anomalous data, including significantly greater or lower than historical values, will be presented in the DSR.

6.1 Data Review, Verification, and Validation

Data review, verification, and validation will be performed in general accordance with EPA *Guidance on Environmental Data Verification and Data Validation (QA/G-8)* (EPA, 2002), but aligned with method-specific criteria.

6.1.1 Data Review Requirements

The data producer will review the data to ensure that the data have been recorded, transmitted, and processed correctly.

6.1.1.1 Internal Field Data Review

Field data review will include verification that any QC checks and calibrations, if necessary, were performed and were recorded properly in the field logbook and that any necessary actions were implemented and recorded. Calibration data for applicable field instruments must be recorded in the field logbook at the time calibrations were completed. Any errors recorded in the logbook must be legibly crossed out with a single line strikeout and contain the initials of the field member, the date, and the correction in a space adjacent to the original (erroneous) entry. The Field Team Leader will review the field logs to determine whether any transcription errors have been made. In the event of errors, the Field Team Leader and field crew will address the errors to provide resolution. The Field Team Leader will review all field data for accuracy and completeness before the information is entered into the electronic database. The electronic data will be maintained as part of the project's quality records.

6.1.1.2 Internal Laboratory Data Review

The laboratory will perform initial internal data reduction as described in the individual laboratory's quality management plan. At a minimum, records will be maintained by the analysts to document sample identification number and the sample tag number with sample results and other details, such as the analytical method used (e.g., method SOP #), name of analyst, the date of analysis, matrix sampled, raw data, and flag unacceptable data. These records will be signed and dated by the analyst. A secondary review will be completed by the Laboratory Supervisor,

Laboratory Project Manager, or designated alternate. Hard copy records or PDF files will be maintained to document completion of data reduction.

Records will also be maintained internally by laboratory personnel to include records of instrument calibrations, results, and maintenance activities, as described within the laboratory's internal QA manual.

6.1.2 Data Verification Requirements

Data verification is the process for evaluating the completeness, correctness, and conformance / compliance of a specific data set against the method, procedural, or contractual specifications.

6.1.2.1 Field Data Verification

The Level A/B review, as described in the Clark Fork River Superfund Site Investigation (CFRSSI) Data Management/Data Validation (DM/DV) Plan (ARCO, 1992a) and the CFRSSI DM/DV Plan Addendum (AERL, 2000), will be used in the verification process for field documentation related to samples collected for laboratory analysis.

The Level A criteria are:

- Sampling date.
- Sample team and/or leader.
- Physical description of sample location.
- Sample depth (soil).
- Sample collection technique.
- Field preservation technique.
- Sample preservation technique.
- Sample shipping records.

The Level B criteria are:

- Field instrumentation methods and standardization complete.
- Sample containers preparations.
- Collection of field duplicates.
- Proper and decontaminated sampling equipment.
- Field custody documentation.
- Shipping custody documentation.
- Traceable sample designation number.
- Field notebook(s), custody records in secure repository.
- Complete field forms.

6.1.2.2 Laboratory Data Verification

The sample data package provided by the laboratory typically includes a PDF laboratory report and an EDD. The information provided in the PDF laboratory report and EDD will depend on the level of data validation required for each project. At a minimum, the data packages from the laboratory will contain the following information:

- A narrative addressing any anomalies encountered during sample analysis, and a discussion of any exceedances in the laboratory QC sample results.
- Analytical method references.
- Definition of any data flags or qualifiers used.
- Chain of custody documentation signed and dated by the laboratory to indicate sample receipt.
- Method detection limits and reporting limits.
- Analytical results for each field sample.
- Blank and QC sample results (as applicable).

6.1.2.3 Resolution of Deficiencies

Any deficiencies found during the verification process will be discussed with the data producer and may be resolved with a revised data package.

6.1.3 Data Validation Requirements

Data validation is the process of ensuring data are correct and useful. Data validation will be performed by qualified, independent data validation personnel who are not associated with data collection or sampling responsibilities and who have applicable training. The QC criteria used during the data validation process will follow the National Functional Guidelines (NFG) for Inorganic Superfund Methods Data Review (EPA, 2020b), except when superseded by the CFRSSI QAPP (ARCO, 1992b), the CFRSSI DM/DV Plan (ARCO, 1992a), the CFRSSI DM/DV Plan Addendum (AERL, 2000), laboratory-specific QC criteria, and/or method-specific criteria where applicable. Other methods are listed below.

- Methods for Chemical Analysis of Water and Wastes, EPA-600/4-79-020 (EPA, 1983).
- EPA Method 6200 Field Portable XRF Spectrometry for the Determination of Elemental Concentrations in Soil and Sediment (EPA, 2007).

6.2 Verification and Validation Methods

The Level A/B checklist included in Appendix C is based on the CFRSSI DM/DV Plan Addendum (AERL, 2000) guidance.

Stage 2A verification and validation checks include an evaluation of the following, as applicable for each analytical method:

- Completeness of laboratory data package.
- Requested analytical methods performed.
- Holding times.
- Reported detection limits.
- Dilution factors.
- Method blanks.
- LCS and LCSD samples.
- MS and MS duplicate samples.
- Post-digestion spikes (per corrective action based on laboratory method only)
- Laboratory duplicate samples.
- Field blanks.
- Field duplicates.

Data qualifiers will follow those used in the NFG (EPA, 2020b) and outlined in Table 6 on the next page. Data validation for each laboratory data package will be documented on the data validation checklists in Appendix C.

Table 6 also lists the QC criteria to be used during the data validation process to evaluate the laboratory and field QC samples.

The data validator will be responsible for reviewing field documentation associated with sample collection, conducting the verification and validation of laboratory-produced data, and completing a DVR, which will be reviewed by the project manager.

Table 6. Data Validation QC Criteria

XRF							
Quality Control	**Frequency**	**Acceptance Criteria**	**Criteria**	**Action**			**Reference**
				Associated Sample Result Detected	**Associated Sample Result Non-Detected**	**Reason Code**	
System Check	Performed daily, prior to sample analysis	Performed daily, prior to sample analysis	System Check not performed	Professional Judgment J/R	Professional Judgment UJ/R	CX	SOP-SFM-02
		Resolution < 195	Resolution ≥ 195	Professional Judgment J/R	Professional Judgment UJ/R	SC	
SiO_2 Standard	Performed daily, prior to sample analysis, at least 1 for every 20 sample analyses, and at end of each day of analysis	Performed daily, prior to sample analysis, at least 1 for every 20 sample analyses, and at end of each day of analysis	Frequency criteria not met	J	UJ	CX	SOP-SFM-02 Niton XL3 Soil QC Sheet
		Arsenic ≤10 mg/kg Cadmium ≤50 mg/kg Copper ≤20 mg/kg Lead ≤10 mg/kg Mercury ≤10 mg/kg Zinc ≤10 mg/kg	>10 mg/kg >50 mg/kg >20 mg/kg >10 mg/kg >10 mg/kg >10 mg/kg	Results < 10x the SiO2 result - J+	No Qualification	B	
Calibration Check Samples	Performed daily, prior to sample analysis, at least 1 for every 20 sample analyses, and at end of each day of analysis	Performed daily, prior to sample analysis, at least 1 for every 20 sample analyses, and at end of each day of analysis	Frequency criteria not met	J	UJ	CX	SOP-SFM-02 Niton XL3 Soil QC Sheet
		NIST Standard: Arsenic 0 - 35 mg/kg; Cadmium 0 - 60 mg/kg; Copper 0 - 60 mg/kg; Lead 0 - 35 mg/kg; Mercury 0 - 12 mg/kg; Zinc 50 - 160 mg/kg	< Lower Control Limit	J-	UJ	CSS	
		RCRA Standard: Arsenic 400 - 600 mg/kg; Cadmium 400 - 600 mg/kg; Lead 400 - 600 mg/kg	> Upper Control Limit	J+	No Qualification		
XRF Duplicate	1 per 20 samples	RPD ≤ 35% for detected results	Frequency criteria not met	J	UJ	DX	SOP-SFM-02 UR QAPP
			RPD ≤ 35%	No Qualification	No Qualification	D%	
			RPD > 35%	J	UJ		
XRF Replicate	1 per 20 samples	RPD ≤ 35% for detected results	Frequency criteria not met	J	UJ	RX	SOP-SFM-02 UR QAPP
			RPD ≤ 35%	No Qualification	No Qualification	R%	
			RPD > 35%	J	UJ		
Field Duplicate	1 per 20 samples	RPD ≤ 35% for detected results	Frequency criteria not met	J	UJ	FDX	UR QAPP
			RPD ≤ 35%	No Qualification	No Qualification	FD	
			RPD > 35%	J	UJ		

Table 6. Data Validation QC Criteria

Laboratory							
Quality Control	**Frequency**	**Acceptance Criteria**	**Criteria**	**Data Validation Action**			**Reference**
				Associated Sample Result -Detected	**Associated Sample Result - Non-Detected**	**Reason Code**	
Laboratory Quality Control Samples							
Holding Time	Every Sample	EPA 6010D (metals/metalloids)	≤ 6 months	J-	Professional Judgement UJ or R	H	NFG
		EPA 7471B (mercury)	≤ 28 days	J-	Professional Judgement UJ or R		
Preservation	Every Sample	EPA 6010D (metals/metalloids)	N/A (solids)	No Qualification	No Qualification	Pres	NFG
		EPA 7471B (mercury)	≤ 6 ºC	No Qualification	No Qualification		
			≥ 6 ºC but ≤ 10 ºC	Professional Judgement J	Professional Judgement UJ		
			> 10 ºC	J-	Professional Judgement UJ or R		
Method Blank (MB)	One per batch of up to 20 samples.	≤ 1/2 RL (6010D) ≤ Absolute Value of RL (7471B)	≤ 1/2 RL (6010D) or Absolute Value of RL (7471B)	No Qualification	No Qualification	MB	CFRSSI QAPP Pace SOP
			> 1/2 RL (6010D) or Absolute Value of RL (7471B)	sample result < 10x blank detection: U	No Qualification		
Laboratory Control Sample (LCS)	One per batch of up to 20 samples.	%R 80-120% (all methods)	%R < 40%	J-	R	L%	CFRSSI QAPP NFG Pace SOP
			%R 40-79%	J-	UJ		
			%R 80-120%	No Qualification	No Qualification		
			%R > 120%	J+	No Qualification		
			%R > 150%	R	No Qualification		
Laboratory Duplicate Sample (LDS)[3]	One per batch of up to 20 samples.	All methods: 1. If both original sample and duplicate sample results are ≥ 5x the RL, then RPD ≤ 20% (LCSD/MSD), RPD ≤35% (soil); 2. If original sample or duplicate sample result < 5x the RL, then absolute difference between sample and duplicate ≤ 2x RL (soils)	Both original and duplicate sample results are ≥ 5x the RL and RPD ≤ 20% (LCSD/MSD), RPD ≤35% (soil).	No Qualification	No Qualification	D%	CFRSSI QAPP NFG Pace SOP
			Both original and duplicate sample results are ≥ 5x the RL and RPD is > 20% (LCSD/MSD), > 35% (soil).	J	UJ		
			RPD > 100%	Professional Judgement	Professional Judgement		
			Original sample or duplicate sample result < 5x the RL, and absolute difference between sample and duplicate ≤ 2x RL (soils)	No Qualification	No Qualification		
			Original sample or duplicate sample result is < 5x the RL and absolute difference between the sample and duplicate > 2x RL (soil).	J	UJ		
Laboratory Matrix Spike (LMS)	One per batch of up to 20 samples.	6010D - %R 75-125% 7471B - %R 80-120% if sample analyte concentration < 4x spike concentration	%R < 30%	J-	R	S%	CFRSSI QAPP NFG Pace SOP
			%R 30-74% (6010D) %R 30-79% (7471B)	J-	UJ		
			%R 75-125% (6010D) %R 80-120% (7471B)	No Qualification	No Qualification		
			%R >125% (6010D) %R >120% (7471B)	J+	No Qualification		
			sample analyte concentration ≥ 4x spike concentration	No Qualification	No Qualification		
Field Quality Control Samples							
Field Duplicate Sample	One per 20 samples collected.	All methods: 1. If both original sample and duplicate sample results are ≥ 5x the RL, RPD ≤ 35% (soil); 2. If original sample or duplicate sample result < 5x the RL, then absolute difference between sample and duplicate ≤ 2x RL (soils)	Both original and duplicate sample results are ≥ 5x the RL and RPD RPD ≤35% (soil).	No Qualification	No Qualification	FD	CFRSSI QAPP NFG
			Both original and duplicate sample results are ≥ 5x the RL and RPD is > 35% (soil).	J	UJ		
			RPD > 100%	Professional Judgement	Professional Judgement		
			Original sample or duplicate sample result < 5x the RL, and absolute difference between sample and duplicate ≤ RL (soils)	No Qualification	No Qualification		
			Original sample or duplicate sample result is < 5x the RL and absolute difference between the sample and duplicate > RL (soil).	J	UJ		

Notes:

1. Associated sample results:
   - For Field Blank results that do not meet technical criteria, apply action to all samples in the SDG.
   - For Field Duplicate results that do not meet technical criteria, apply action to field duplicate pair and any samples from the same sample location in the SDG.
   - For MB and LCS results that do not meet technical criteria, apply action to all samples in the analytical batch.
   - For LDS or LMS/MSD results that do not meet technical criteria, apply action to the parent sample and, per the NFG, "apply the action to all samples of the same matrix if the samples are considered sufficiently similar."
   - For holding time and preservation that do not meet technical criteria, apply action to sample.
2. For consistency in validations between validators, if a sample result is reported as non-detect, the MDL is used for the duplicate absolute difference calculations.
3. An LCS, an LMS, or an original sample may all be used to perform a laboratory duplicate. If a LCS Duplicate or LMS Duplicate is used, the QC sample must also meet the applicable %R technical criteria.

Qualifications:

U - Non-detect
UJ - Estimated non-detect
J - Estimated
J+ - Estimated high
J- - Estimated low
R - Rejected

Abbreviations:

MDL - method detection limit
RL - reporting limit
%R - percent recovery
RPD - relative percent difference

References:

CFRSSI QAPP - ARCO, 1992. Clark Fork River Superfund Site Investigations (CFRSSI) Quality Assurance Project Plan (QAPP). Prepared for ARCO by PTI Environmental Services, Bellevue, Washington. May 1992.

NFG - EPA, 2020. National Functional Guidelines for Inorganic Superfund Methods Data Review. November 2020.

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UR QAPP - Silver Bow Creek/Butte Area NPL Site Butte Priority Soils Operable Unit 2022 Final Unrelaimed Sites Quality Assurance Project Plan (QAPP). Prepared for Atlantic Richfield Company by Pioneer Technical Services, Inc, Butte, Montana. June 2021.

Niton XL3 Soil QC Sheet - Niton XL3 Soil QC Certificate of Calibration. Thermo Fisher Scientific. June 2014.

Pace SOP -

EPA 6010D - ENV-SOP-MIN4-0052: Metals Analysis by ICP - Method 6010 and 200.7

EPA 7471B - ENV-SOP-MIN4-0054: Mercury in Liquid and Solid/Semi-Solid Waste by 7470A, 7471, 7471B, and 245.1

6.3 Reconciliation and User Requirements

The Data Quality Assessment (DQA) process described in the CFRSSI DM/DV Plan Addendum (AERL, 2000) and the guidance for data quality assessment EPA QA/G-9 (EPA, 2006b) will be performed to determine whether project-specific DQOs have been satisfied. The DQA process consists of five steps that relate the quality of the results to the intended use of the data:

Step 1: Review DQOs and sampling design.
Step 2: Conduct preliminary data review.
Step 3: There are no statistical tests planned in the interpretation of the results; laboratory results will be compared directly to action limits defined in the DQOs (Section 2.4).
Step 4: Verify assumptions.
Step 5: Draw conclusions about the quality of the data (data report will not include interpretation of results but will state conclusions regarding the quality of the results).

If, as a result of the DQA process, it is determined that data do not satisfy all DQOs, then corrective action(s) should be recommended. Corrective actions include, but are not limited to, revision of the DQOs based on the results of the investigation, or collection of more information or data. It may be determined that corrective actions are not required, or the decision process may continue with the existing data, with recognition of the limitations of the data.

The PARCC data quality indicators (Section 2.4.1) will be used when conducting the DQA. If the PARCC indicator results satisfy the project DQOs, then usability of the data will follow the enforcement/screening/unusable data categories as described in the CFRSSI DV/DM Plan (ARCO, 1992a):

1. Enforcement Quality (Unrestricted Use) Data

   Enforcement quality data may be used for all purposes under the Superfund program including the following: site characterization, health and safety, Engineering Evaluation/Cost Analyses, remedial investigations/feasibility studies, evaluation of alternatives, conformational purposes, risk assessments, and engineering design.

2. Screening Quality (Restricted Use) Data

   Potential uses of screening quality data, depending upon their quality, include site characterization, determining the presence or absence of contaminants, developing or refining sampling and analysis techniques, determining relative concentrations, scoping and planning for future studies, engineering studies and engineering design, and monitoring during implementation of the response action.

3. Unusable Data

   These data are not useable for Superfund-related activities.

Data that meet the Level A and Level B criteria and are not qualified as estimated or rejected during the data validation process are assessed as enforcement quality data and can be used for all Superfund purposes and activities. Data that meet only the Level A criteria and are not rejected during the data validation process can be assessed as screening quality data. Screening quality data can be used only for certain activities, which include engineering studies and design. Data that do not meet the Level A and/or B criteria and/or are rejected during the data validation process are designated as unusable. The data are assigned one of the following qualifiers:

E = Enforcement quality. No qualifiers or U qualifier and meets Level A and B criteria.
S = Screening quality. J, J+, J-, or UJ qualifier and/or meets only Level A criteria.
R = Unusable. R qualifier and/or does not meet Level A or B requirements.

The list below identifies the qualifies.

Enforcement/Screening Designation:

	Meets Level A and B	**Meets Level A**	**Does not meet Level A or B**
No qualifier, A, U, or laboratory results reported between the method detection limit and reporting limit (RL) with a J qualifier	E	S	R
J, J+, J-, or UJ	S	S	R
R	R	R	R

Note: It is appropriate to note that sample results qualified as estimated "J" by the laboratory, because the reported result was between the method detection limit and RL values, are considered enforcement quality data if no other qualifiers were required during validation.

Results of the QA review and/or validation will be included in any subsequent report, which will provide a basis for meaningful interpretation of the data quality and evaluate the need for corrective actions.

6.3.1 Specific Quality Control/Assessment Procedures

The accuracy, precision, completeness, and representativeness of analytical data will be described relative to the project's control limits through a process of field and laboratory data quality review. Results from these reviews will be documented in the site-specific DSRs. Any qualification of the data resulting from that review will also be incorporated into the project's electronic database (Section 4.0) so that all data users are aware of any uncertainties.

A DQA will be performed to determine whether the project-specific DQOs have been satisfied. The DQA consists of five steps that relate the quality of the results to the intended use of the data:

Step 1: Review DQOs and sampling design.

Step 2: Conduct preliminary data review.

Step 3: Apply statistical test(s) as described in this QAPP to the data set.

Step 4: Verify assumptions.

Step 5: Draw conclusions about the quality of the data (data report will not include interpretation of results but will state conclusions regarding the quality of the results).

During the DQA process, if it is determined that data do not satisfy all DQOs then corrective action(s) will be recommended and documented in the data reporting. Corrective actions include, but are not limited to, revision of the DQOs based on the results of the investigation or collection of more information. The review may also determine that corrective actions are not required, or the decision process may continue with the existing data with recognition of the data limitations.

Results of the QA review and/or validation will be included in any subsequent report, which will provide a basis for meaningful interpretation of the data quality and evaluate the need for corrective actions.

7.0 REFERENCES

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EPA, 2020a. Consent Decree for the Butte Priority Soils Operable Unit. Partial Remedial Design/Remedial Action and Operation and Maintenance. U.S. Environmental Protection Agency. February 13, 2020. Available at https://www.co.silverbow.mt.us/2161/ButtePriority-Soils-Operable-Unit-Conse. Appendix A to the Consent Decree is the 2006 Record of Decision, Butte Priority Soils Operable Unit Silver Bow Creek/Butte Area NPL Site.

EPA, 2020b. U.S. Environmental Protection Agency National Functional Guidelines for Inorganic Superfund Methods Data Review (SFAM01.1), November 2020. Available at https://www.epa.gov/clp/national-functional-guidelines-inorganic-superfund-methodsdata- review-sfam011.

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Appendix A
Reference Documents

Appendix A.1 BPSOU Area

Appendix A.2 Program Organizational Chart

Appendix A.3 Unreclaimed Area Decision Logic

Appendix A.4 Precision, Accuracy, and Completeness Calculations

LEGEND

BPSOU BOUNDARY

N

DISPLAYED AS:
PROJECTION/ZONE: MSP
DATUM: NAD 83
UNITS: INTN'L FT
SOURCE: PIONEER/ESRI

0 1,500 3,000 6,000
Feet

FIGURE 1

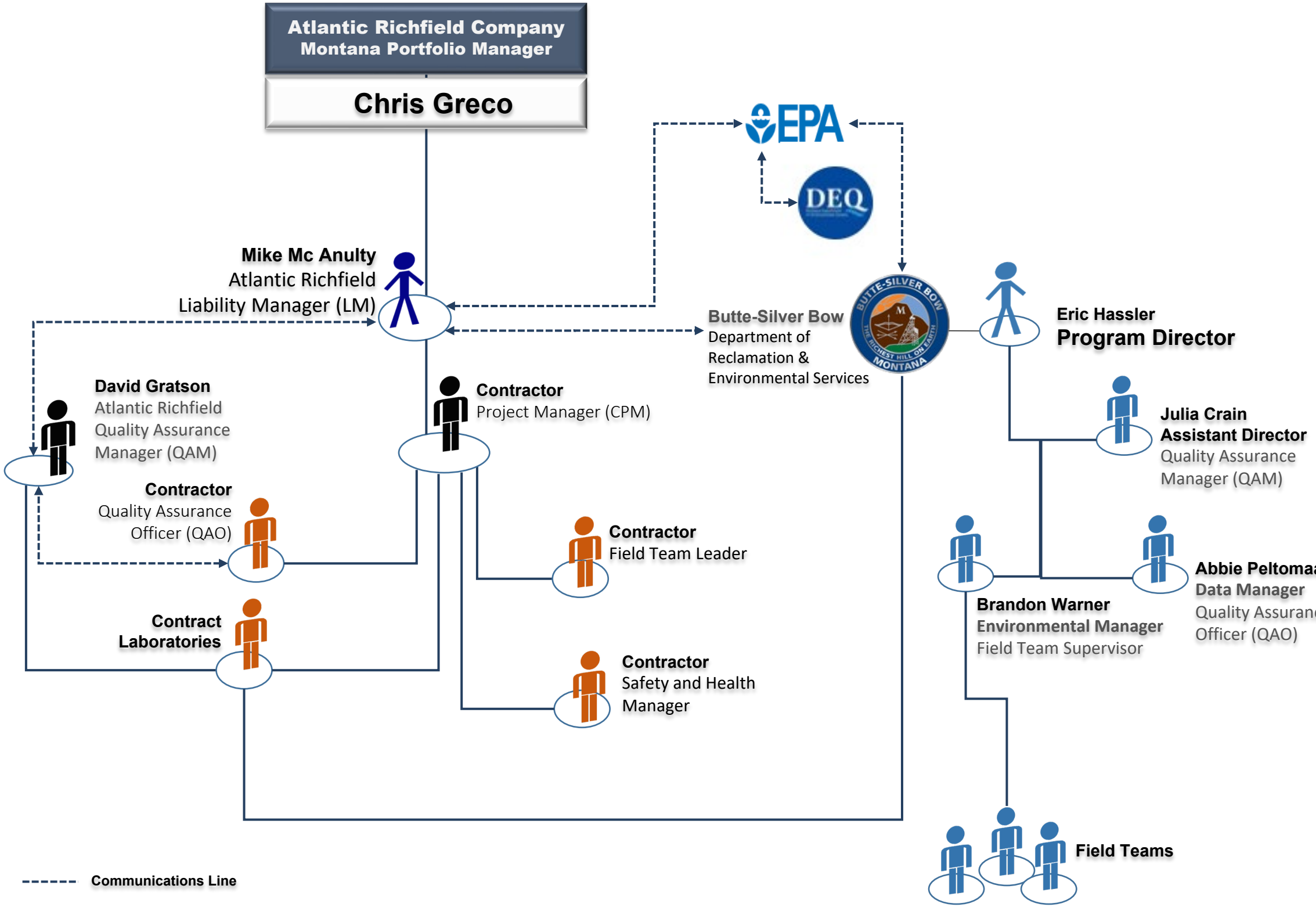
BPSOU AREA

PIONEER TECHNICAL SERVICES, INC.

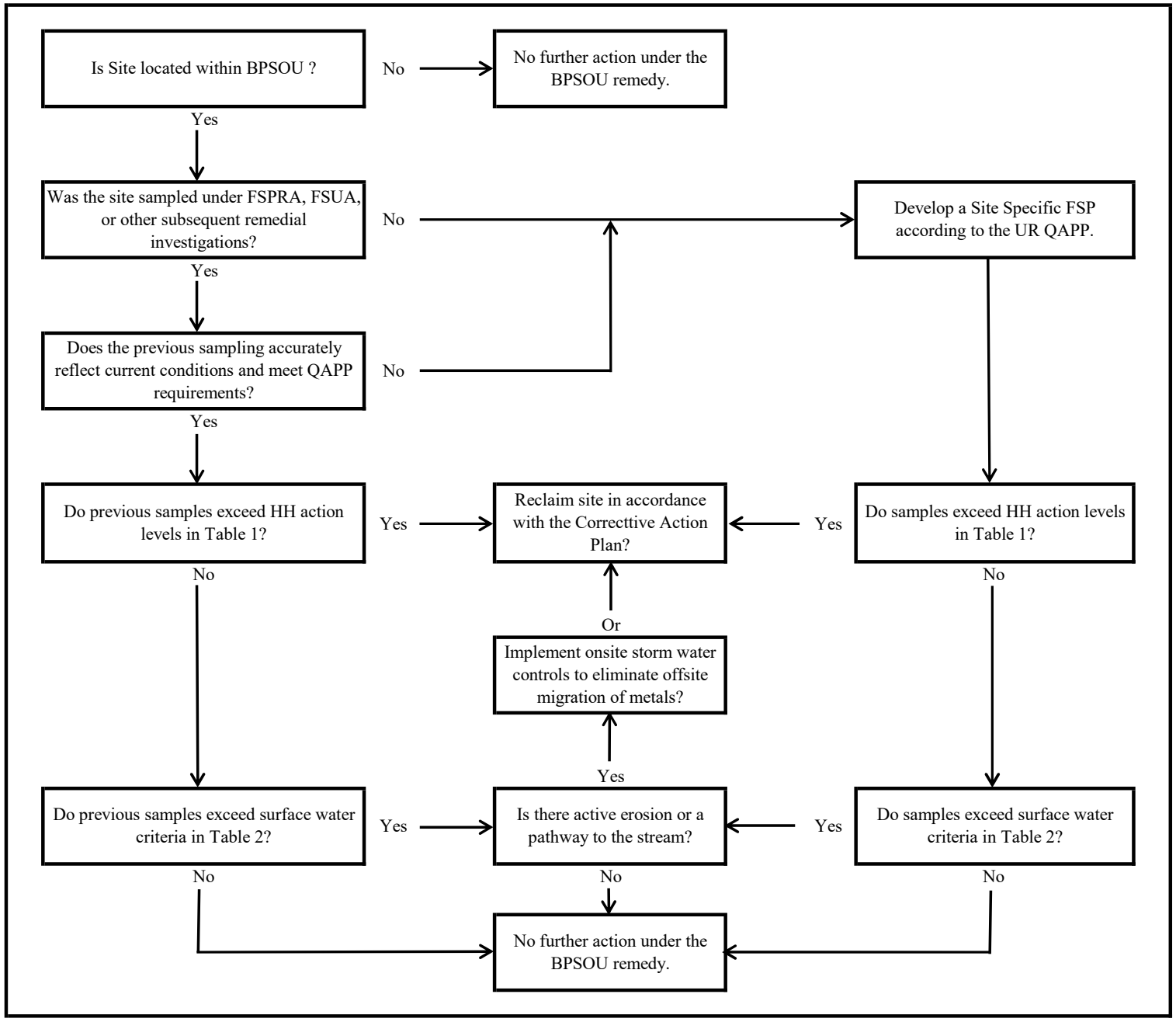
DATE: 1/6/2021

Path: Z:\Shared\Active Projects\ARCO\BPSOU_Misc\GIS\BPSOU_Bndry.mxd

Appendix A2. Program Organizational Chart



Appendix A.3:
Unreclaimed Area Decision Logic



Appendix A.4: Precision, Accuracy, and Completeness Calculations

Characteristic	Formula	Symbols
Precision (as relative percent difference, RPD)	$RPD = \frac{\lvert(x_i - x_j)\rvert}{\left(\frac{x_i + x_j}{2}\right)} \times 100$	x_i, x_j: replicate values of x
Precision (as relative standard deviation, RSD, otherwise known as coefficient of variation)	$RSD = \frac{\sigma}{\bar{x}} \times 100$	σ: sample standard deviation $\bar{x}$: sample mean
Accuracy (as percent recovery, R, for samples without a background level of the analyte, such as reference materials, laboratory control samples and performance evaluation samples)	$R = \frac{x}{t} \times 100$	x: sample value t: true or assumed value
Completeness (as a percentage, C)	$C = \frac{n}{N} \times 100$	n: number of valid data points produced N: total number of samples taken

Appendix B
Standard Operating Procedures

Appendix B.1 SOP-S-01 Surface Soil Sampling General

Appendix B.2 SOP-S-02 Subsurface Soil Sampling 11/23/2020

Appendix B.3 SOP-SA-01 Soil and Water Sample Packaging General

Appendix B.4 SOP-SA-04 Chain of Custody Forms for Environmental Samples General

Appendix B.5 SOP-SA-05 Project Documentation General

Appendix B.6 SOP-SFM-01 Field Measurement of pH in Soil

Appendix B.7 SOP-SFM-02 Operating XL3-X-Ray Fluorescence Analyzer General

Appendix B.8 SOP-DE-01 Personal Decontamination Procedures General

Appendix B.9 SOP-DE-02 Equipment Decontamination General

Appendix B.10 ENV-SOP-MIN4-0052 Metals Analysis by ICP – Method 6010 and 200.7

Appendix B.11 ENV-SOP-MIN4-0054 Mercury in Liquid and Solid/Semi-Solid Waste by 7470A, 7471, 7471B, and 245.1

Appendix B.12 ENV-SOP-MIN4-0056 Metals Preparation of Solid Samples for Analysis by ICP and ICP-MS by 3050B – Preparation of Solid Samples

SOP-S-01.
SURFACE SOIL SAMPLING-GENERAL

PURPOSE	To provide standard instructions for surface soil sampling for unreclaimed sites in the BPSOU area.
SCOPE	Work described in this procedure includes visual assessment and site documentation, sample collection and handling, and chain of custody protocol required to complete routine soil sampling tasks.
DEFINITIONS	Surface Sample: a surface sample is defined as a mineral soil sample collected from immediately beneath the vegetative mat. It generally includes some interval from the upper six inches of soil. Surface sampling under biased conditions may be selected after considering factors such as type of contaminant, length of time the area has been contaminated, the type of soil, and the past use of the area.

WORK INSTRUCTIONS

The following instructions are intended to provide sufficient guidance to perform the task in a safe, accurate, and reliable manner. Should these instructions present information that is inaccurate or unsafe, operations personnel must bring the issue to the attention of the Project Manager and the appropriate revisions made.

TASK	INSTRUCTIONS
Grab/Opportunistic Sample	
Visual Inspection and map	1. Verify utility locates have been performed and adjust sampling sites to avoid conflicts. 2. Inspect the area for possible hazards prior to sampling. 3. Visually inspect the site to determine the number test areas for composite sampling 4. Photograph and document the existing site conditions. 5. Draw a scaled map of the site if a pre-sampling map hasn't been completed
	Note: Sample collection devices include stainless steel scoops or trowels, stainless steel probes, and disposable Teflon trowels. For inorganic contaminants, disposable plastic scoops will be used. These procedures may be modified in the field based on field and site conditions after appropriate annotations have been made in the field log book.
	Identify site-specific hazards and verify utility locates. 1. Perform utility locates or verify utility locates have been performed. 2. Walk through the site and determine any site-specific hazards associated with the sampling area. Discuss findings with sampling crew and note in the field logbook. 3. Verify the utility locate information by identifying where natural gas pipes or other utilities enter any structures on the property or if yard lights or street lights are present with no overhead lines. Determine if an underground sprinkling system is present, where applicable. If sample locations have not been assigned in the Sampling Analysis Plan (SAP), note the already marked and/or probable locations of underground utilities and try to avoid those areas when choosing sample locations. Also, note the location of overhead lines and overhead hazards and avoid those areas, if possible. 4. If sample locations are identified in the SAP, use the appropriate survey method to locate and mark the sample locations.

Test Pit Sampling	
1. Dig a 6 to 12-inch square pit.	Dig a 6 to 12-inch square pit to a depth of approximately 6 inches. The size and depth of the sample pit required depends on the amount of material needed for sample analysis and the interval to be sampled. If a sod mat is present, separate the sod mat from the mineral soil surface with the chosen sampling tool. Shake and scrape the removed sod mat over the sample collection bowl to dislodge any mineral soil particles. Place all dislodged particles in the sample. If the surface material is coarse-grained material, free of intermixed materials (i.e., graveled driveway), collect the sample from the appropriate layer below the protective barrier. However, if the graveled driveway, alley or lot contains soil/dust material on the surface, collect the sample from the appropriate interval. If the sample area is unvegetated, collect the sample material from the designated interval inches below ground surface.
2. Measure and mark the interval to be sampled.	Measure the interval to be sampled (e.g., 0-2 inches or 0-6 inches) with a stainless steel tape measure or a ruler and mark the appropriate interval.
3. Scrape the walls of the sample pit.	Scrape the walls of the sample pit within the marked interval with a decontaminated stainless steel trowel or scoop, a Teflon scoop, or a disposable plastic scoop to expose a clean surface.
4. Collect the sample.	Once the wall of the test pit has been cleaned, collect the sample by scraping the appropriate interval on the cleaned face of the pit with the sampling tool and placing the material in a decontaminated stainless steel bowl, or a new cleaned foil pan.
5. Remove coarse fragments from the bowl.	Remove all coarse fragments greater than 0.5 inches from the bowl. Mix the remaining material in the bowl with the sampling tool.
6. Pack the samples.	Transfer the soil sample directly into the appropriate sample container according to SOP-SA-01 Soil and Water Sample Packaging and Shipping and store in a cooler at 4°C or less. Any remaining sample material will be returned to the sample holes. A sufficient quantity of soil will be collected in each sample container to provide for analysis with additional soil left over to be archived.
7. Record sampling information.	Record appropriate information about the sample collection in the field logbook.
8. Return all the removed dirt into the hole.	Return all the removed dirt into the hole and return the sample area to pre-sampling conditions.
9. Decontaminate the equipment.	Decontaminate sampling tools according to procedures outlined in SOP-DE-02 Equipment Decontamination.

Stainless steel probe opportunistic sampling	
1. Collect the sample	Collect sample as per probe manufacturers instructions
2. Pack the sample	Transfer the soil sample directly into the appropriate sample container according to SOP-SA-01 Soil and Water Sample Packaging and Shipping, label the samples, and store in a cooler at 4°C or less.
3. Record the sample	Record appropriate information about the sample collection in the field logbook.
4. Decontaminate sampling equipment	Decontaminate sampling tools according to procedures outlined in SOP-DE-02 Equipment Decontamination.
Composite Sampling/ Test Pits	
Note	In many situations, a composite sample is more appropriate for sample collection than a grab sample. Several types of composite samples can be collected. A sampler can collect a biased composite sample by identifying specific spots within the sample area that appear to be contaminated or not contaminated and digging sample pits in those locations. Composite samples can also be collected randomly as defined in a SAP. Sub samples shall be collected in a three-point (triangular) pattern. At each point, a subsample of predetermined depth is collected. The diagonal distance between the points is commonly ten feet, depending on the area of soil homogeneity. The precise method for compositing the sample will be discussed in the SAP. Each sub sample test hole will be prepared and sampled in the manner discussed above under the Grab Sample section.
1. Collect composite samples.	Composite samples will consist of discrete aliquots of equal amounts of soil from each subsample location. The soil aliquots will be collected into a stainless steel bowl and thoroughly mixed. The sampler may also "eyeball" an equal amount of sample material from each hole into a resealable plastic bag (i.e., Ziploc®). The sample material would be thoroughly mixed between each sub sample pit and prior to placing in the appropriate sample containers.
2. Remove coarse fragments.	Remove all coarse fragments greater than 0.5 inches from the bowl. Mix the remaining material in the bowl with the sampling tool.
3. Pack the samples.	Transfer the soil sample directly into the appropriate sample container according to SOP-SA-01 Soil and Water Sample Packaging and Shipping, label the samples, and store in a cooler at 4°C or less. Any remaining sample material will be returned to the sample holes. A sufficient quantity of soil will be collected in each sample container to provide for analysis with additional soil left over to be archived.
4. Record sampling information.	Record appropriate information about the sample collection in the field logbook.

5. Return all the removed dirt into the hole.	Return all the removed dirt into the hole and return the sample area to pre-sampling conditions.
6. Decontaminate the equipment.	Decontaminate sampling tools according to procedures outlined in SOP-DE-02 Equipment Decontamination.
Composite Sampling Stainless Steel Probe	
1. Collect composite samples	Collect in the same triangular pattern and mix as described above. Collect samples as per probe manufacturers instructions
1. Pack the samples	Transfer the soil sample directly into the appropriate sample container according to SOP-SA-01 Soil and Water Sample Packaging and Shipping, label the samples, and store in a cooler at 4°C or less.
2. Record sampling information	Record appropriate information about the sample collection in the field logbook.
3. Decontaminate the equipment	Decontaminate sampling tools according to procedures outlined in SOP-DE-02 Equipment Decontamination.

ADDITIONAL HSSE CONSIDERATIONS This section to be completed with concurrence from the Safety and Health Manager.	
Required PPE	**Personnel Protection Equipment** (PPE): Hard hat, safety glasses, high-visibility work shirt or vest, long pants, work boots, nitrile gloves, and leather gloves.
Applicable SDS	**Safety Data Sheets** (SDSs) will be maintained based on-site characterization and contaminants.
Required Permits/Forms	Per site/project requirements.
Additional Training	Per site/project requirements.

DRAWINGS, DOCUMENTS, AND TOOLS/EQUIPMENT The following documents should be referenced to assist in completing the associated task.	
Drawings	Map with site location and sample locations.
Related SOPs/ Procedures/ Work Plans	SOP-SA-01 Soil and Water Sample Packaging and Shipping and SOP-DE-02 Equipment Decontamination.
Tools	Sampling tools: stainless steel scoops or trowels, stainless steel probes, disposable Teflon trowels, disposable plastic scoops (for inorganic contaminants), stainless steel tape measure or a ruler, decontaminated stainless steel bowl or cleaned foil pan, one-quart plastic bag, sampling containers, and cooler. Field logbook.
Forms/Checklists	

PURPOSE	To provide standard instructions for collecting subsurface soil samples.
SCOPE	Pioneer Technical Services, Inc. (Pioneer) prepared this practice for the workforce and this Standard Operating Procedure (SOP) applies to all work performed by and on behalf of Pioneer. All members of the Pioneer workforce who conduct the work shall be trained and competent (as defined by OSHA) in the risk-assessed procedure described below before performing the work.
DEFINITIONS	**Subsurface Soil Sample** is defined as a mineral soil sample collected below 6 inches below ground surface (bgs). Sampling of subsurface soil should be evaluated by considering factors such as the precipitation, the type of soil, and the length of time the site has been contaminated. If precipitation has moved contaminants into lower soil horizons, subsurface sampling may be appropriate. Several techniques can be used to collect samples from 6 inches to 4 or 5 feet bgs. A shovel and pry bar can be used to collect samples from 6 inches to 2 feet bgs. A hand auger may be used to collect subsurface samples up to 4 or 5 feet in depth. Because the auger is twisted into the soil, the soil's cohesive structure and stratigraphic character are destroyed. An *in-situ* soil recovery auger may also be used to collect subsurface samples up to 5 feet. The auger accommodates a liner and provides fast cutting of the soil with very little soil disturbance. In particularly rocky or hard soil, a backhoe may be needed to excavate even shallow test pits. It is important to evaluate site conditions prior to choosing a subsurface sampling method. Each method of sampling is discussed below.

WORK INSTRUCTIONS

The following instructions provide guidance to perform the task in a safe, accurate, and reliable manner. If these instructions present information that is inaccurate or unsafe, personnel must notify the Project Manager, Safety Manager, and the SOP Technical Author to initiate appropriate revisions. Personnel will perform all work under this SOP in a manner that is consistent with procedures and policies described in the appropriate Operation, Maintenance, and Monitoring (O&M) Plan (where applicable), appropriate Site-Specific Health and Safety Plans (SSHASP), and Pioneer Corporate Health and Safety Plan (HASP).

TASK	INSTRUCTIONS
Hand-Dug Test Pits for Inorganic and Non-Volatile Organic Samples	
1. Identify potential sample sites and mark for utility locates.	Locate potential sample sites as directed in the appropriate Sampling and Analysis Plan (SAP) or Work Plan (WP). Use an appropriate survey method to locate and mark the sample locations if required. If sample locations are not identified in the SAP, follow the guidance in the SAP and chose and mark sample locations.
2. Coordinate utility locates.	Call in for utility locates a minimum of 48 business hours prior to conducting the sampling activities. If needed, work with the locator to adjust sample locations based on identified utility locations. Refer to the Trenching, Excavation, and Ground Disturbance Program information in Pioneer's Corporate HASP to identify safe distances for digging when adjacent to specific buried utilities.

3. Conduct site walk.	Conduct a site walk-through and determine any site-specific hazards associated with the sampling area. Discuss these with the sampling crew and note the hazards in the field logbook. As part of the site hazard assessment, identify possible locations that could contain unidentified, privately installed underground utilities. For example, identify where natural gas pipes enter any structures on the property and confirm that gas lines from the street/alley have been marked. Check yard lights or streetlights that are present with no overhead lines, underground wiring from a residence to outbuildings, or a possible gas line to a grill or outdoor kitchen. Adjust sample locations based on this information.
4. Dig test pit.	Dig a 6- to 12-inch square pit to the depth specified in the SAP plus an additional 3 to 4 inches. Place any removed material on a piece of plastic to prevent potential contamination from buried soil on the surface and to aid in returning the soil to the hole when sampling is complete.
5. Identify sample intervals.	Measure and mark the intervals to be sampled with a stainless-steel tape measure or a ruler.
6. Prepare sample location.	Scrape the walls of the sample pit within the **deepest** marked interval with a decontaminated stainless-steel trowel or scoop, a Teflon scoop, or a disposable plastic scoop to expose a clean surface.
7. Collect sample.	Place a stainless-steel bowl or a clean decontaminated disposable foil pan adjacent to or in the sample pit and collect the **deepest** interval to be sampled by scraping the interval on the cleaned face of the pit with the sampling tool. Make sure that the collection container is compatible (will not affect) with any analytes for which the sample will be analyzed. Collect soil from all the way around the sample pit at that interval. If the sample is a composite sample, collect soil from the deepest interval in the remaining sample holes and add it to the stainless-steel bowl or disposable foil pan. The same sampling tool can be used to sample all holes in the composite sample.
8. Remove unnecessary material from sample.	Remove all coarse fragments greater than 0.5 inches from the bowl. Mix the remaining material in the bowl with the sampling tool.
9. Label and transfer sample to sample container.	Label all sample containers following the requirements in the associated SAP/WP. Using the sampling tool, fill all required sample containers. Place a sufficient quantity of soil in each sample container to provide for analysis with additional soil left over to be archived (any remaining soil will be returned to the sample holes per Step 12). Immediately place the soil samples directly into the designated storage container (generally a cooler). If samples are required to be stored at 4 degrees Celsius (°C) or less by the SAP/WP or analytical method, add ice to the cooler. Samples should be kept under chain of custody protocols until transport to the laboratory as described in SOP-SA-01 Soil and Water Sample Packaging and Shipping.

10. Sample remaining depth intervals.	If soil needs to be collected from additional intervals, complete Steps 6-9 for each interval, working from the bottom or deepest interval to the top interval.
11. Document sample information.	Record appropriate information about the sample collection (sample numbers and associated depth interval, time, date, sample containers, etc.) in the field logbook as discussed in SOP-SA-05 Project Documentation. Record additional information such as soil type or rock content if required by the SAP/WP.
12. Return all the removed soil into the hole.	Return all remaining removed soil to the sample hole(s) and return the sample area to pre-sampling conditions.
13. Decontam-inate sampling tools.	Decontaminate sampling tools according to procedures outlined in SOP-DE-02 Equipment Decontamination.
Volatile Organic Sampling	
1. Identify site-specific hazards and verify utility locates.	Follow guidance in Steps 1-3 under **Hand-Dug Test Pits for Inorganic and Non-Volatile Organic Samples** to prepare the site for sampling.
2. Prepare and label the sample containers.	Based on information provided in the SAP/WP, prepare and label the appropriate sample containers. If organic samples are required, sample intervals may have been assigned in the SAP/WP, or samples may be collected based on the photoionization detector (PID), headspace readings or the presence of odor or staining. The sampler must understand sample collection protocol before digging. This is particularly important in collecting samples to be analyzed for Volatile Organic Compounds (VOCs), volatile petroleum hydrocarbon (VPH), and/or extractable petroleum hydrocarbon (EPH). Prior to starting to dig, make sure all the required sampling supplies are close at hand.
3. Dig a square pit.	Dig a 6- to 12-inch square pit to the depth of the **first** sample interval required. The size and depth of the sample pit required will depend on the amount of soil needed for analysis and the interval being sampled.
4. Conduct PID readings if required.	**All VOC and VPH samples need to be collected as quickly as possible after exposing the soil to the air.** If specified in the SAP/WP, use a PID to take readings of the sample area. Refer to SOP-FM-01 Field Headspace Analysis and VOC Measurements with PID.
5. Measure and mark the interval to be sampled.	Measure the top interval to be sampled (e.g., 0-2 inches or 0-6 inches) with a stainless-steel tape measure or a ruler and mark the appropriate interval. As discussed above, collecting samples for VOCs and VPH analysis must be accomplished quickly once soil

	is exposed to air. If collecting samples for VOCs, VPH, EPH, or other easily volatilized compounds, collect the sample for each depth interval as it is uncovered during digging. Additional information on collecting the surface sample can be found in SOP-S-01 Surface Soil Sampling.
6. Scrape the walls of the sample pit.	Scrape the walls of the sample pit within the marked interval with a decontaminated stainless-steel trowel or scoop, a Teflon scoop, or a disposable plastic scoop to expose a clean surface.
7. Collect soil samples for VOC/VPH/ EPH analysis.	Collect the required samples directly from the pit wall using a stainless-steel trowel, a new plastic disposable scoop, gloved hand, or screwdriver. **Sampling for non-organic constituents can be completed later**. Place the soil directly into the sample container and fill the jar to the top allowing no head space (or as the laboratory directs). Pack the material as tightly as feasible and try to avoid getting large particles in the jar. Place the lid on the container as soon as the jar is full. Be aware of the potential for cross contamination and if needed change gloves or scoops between intervals. Immediately place the sample containers in a cooler with ice. Keep samples at 4 ºC or less and under chain of custody protocols until they can be transported to the laboratory for analysis, as described in SOP-SA-01 Soil and Water Sample Packaging and Shipping.
8. Record PID readings and sample information in logbook.	If PID screening is conducted, record results of the screening in the project field logbook or field data sheets and include the highest reading from the sample interval. Record the sample information for each sample in the logbook and include sample number, associated depth interval, time, date, and type of containers collected. Further information on documentation is provided in SOP-SA-05 Project Documentation.
9. Collect soil samples for VOC/VPH/ EPH analysis from remaining depth intervals.	For additional sample intervals, continue to dig to the required depth and screen the newly uncovered surface with the PID (if required) and collect the appropriate sample. Complete Steps 4-8 in this section for each sample interval.
10. Collect the inorganic sample material.	Once the VOC/VPH/EPH samples have been collected, the remaining non-organic sample material can be collected for the grab or composite samples as discussed under **Hand-Dug Test Pits for Inorganic and Non-Volatile Organic Samples** Steps 5 thru 11. For inorganic and non-volatile organics, samples should be collected in the proper order starting with the deepest interval.
11. Record sampling information.	Record appropriate information about the sample collection (sample number and associated depth interval, time, date, sample containers, etc.) in the field logbook as discussed in SOP-SA-05 Project Documentation. Record additional information such as soil type and rock content if required by the SAP/WP.

12. Return all the removed soil into the hole.	Return all the remaining removed soil to the sample hole(s) and return the sample area to pre-sampling conditions.
13. Decontam-inate the equipment.	Decontaminate sampling tools according to procedures outlined in SOP-DE-02 Equipment Decontamination.
Hand Auger Sampling for Inorganic and Non-Volatile Organic Compounds	
1. Identify potential sample locations and mark for utility locates.	Locate potential sample locations as directed in the appropriate SAP or WP. Use an appropriate survey method to locate and mark the sample locations if required. If sample locations are not identified in the SAP, follow the guidance in the SAP to choose and mark sample locations.
2. Coordinate utility locates.	Call in for utility locates a minimum of 48 business hours prior to conducting the sampling activities. If needed, work with the locator to adjust sample locations based on identified utility locations. Refer to the Trenching, Excavation, and Ground Disturbance Program information in Pioneer's Corporate HASP to identify safe distances for digging when adjacent to specific buried utilities.
3. Conduct site walk.	Conduct a site walk-through and determine any site-specific hazards associated with the sampling area. Discuss these with the sampling crew and note the hazards in the field logbook. As part of the site hazard assessment, identify possible locations that could contain unidentified, privately installed underground utilities. For example, identify where natural gas pipes enter any structures on the property and confirm that gas lines from the street/alley have been marked. Check yard lights or streetlights that are present with no overhead lines, underground wiring from a residence to outbuildings, or a possible gas line to a grill or outdoor kitchen. Adjust sample locations based on this information.
4. Dig the sample hole with an auger.	Place a large piece of plastic adjacent to the sample location. Choose the appropriate auger head for the soil type at the sample site (i.e., sand, mud, loam). Measure the length of the auger head to determine the advancement depth for each full auger. Place the auger at the sample location and begin turning, when the head is full, remove the auger from the hole and empty the soil on the head onto the plastic. Measure the hole depth to determine the number of auger heads needed to reach the sample interval. Keep auguring and emptying the soil onto the plastic sheet until the top of the sampling interval is reached. Place the soil on the sheet in the order of removal so a general soil profile can be documented and photographed if required.
5. Collect sample.	Once the first sample interval is reached, place a stainless-steel bowl or a clean decontaminated disposable foil pan near the sample pit (preferably on a clean portion of the plastic) and collect the sample by emptying soil from the auger head onto the plastic adjacent to the bowl or pan. Making sure that no slough from shallower intervals is being sampled and using a stainless-steel trowel or scoop or a new disposable plastic

	scoop, place a representative aliquot of the soil from that pile into the bowl or pan. Continue auguring and collecting representative aliquots throughout the entire sampling interval.
6. Remove unnecessary material from sample.	Remove all coarse fragments greater than 0.5 inches from the mixing container. Mix the soil thoroughly with the sampling tool.
7. Label and transfer sample to sample container.	Label all sample containers following the requirements in the associated SAP/WP. Using the sampling tool, fill all required sample containers. Place a sufficient quantity of soil in each sample container to provide for analysis with additional soil left over to be archived (any remaining sample material will be returned to the sample holes per Step 10 below). Immediately place the soil samples directly into the designated storage container (generally a cooler). If required by the SAP/WP or analytical method, maintain the cooler at 4 °C or less using ice. Samples should be kept under chain of custody protocols until transport to the laboratory as described in SOP-SA-01 Soil and Water Sample Packaging and Shipping.
8. Sample remaining depth intervals.	If soil needs to be collected from additional intervals, change the auger head for each new sample interval to prevent potential cross contamination between layers. Then complete Steps 4-7, above, for each sample interval.
9. Document sample information.	Record appropriate information about the sample collection (sample numbers and associated depth interval, time, date, sample containers, etc.) in the field logbook as discussed in SOP-SA-05 Project Documentation. Record additional information such as soil type and rock content if required by the SAP/WP.
10. Return all the removed soil into the hole.	After filling sample containers for all intervals, return all the remaining removed soil into the sample hole(s) and return the sample area to pre-sampling conditions.
11. Decontam-inate sample tools.	Decontaminate sampling tools according to procedures outlined in SOP-DE-02 Equipment Decontamination.

Hand Auger Sampling for Volatile Organic Compounds	
1. Identify site-specific hazards, verify utility locates, and dig the hole with the auger.	Follow Steps 1-4 under **Hand Auger Sampling for Inorganic and Non-Volatile Organic Compounds** to prepare the site for sampling and dig the hole with the auger. The sampler must understand sample collection protocol before digging. This is particularly important in collecting samples to be analyzed for VOCs, VPH, and/or EPH. Before starting to dig, make sure all the required sampling supplies are close at hand.
2. Prepare the sample containers.	Based on information provided in the SAP/WP, prepare and label the appropriate sample containers. If organic samples are required, sample intervals may have been assigned in the SAP/WP, or samples may be collected based on PID or headspace readings or the presence of odor or staining.
3. Pull up soil from auger.	Once the first sample interval is reached, advance the auger into the sample interval. When the auger is full, remove the head from the hole and empty the soil on the head onto the plastic. Once the soil is on the plastic, immediately begin PID screening (Step 4).
4. Conduct PID readings, if required.	**All VOC, VPH, or EPH samples need to be collected as quickly as possible after exposing the soil to the air.** If specified in the SAP/WP, use a PID to take readings of the sample area, refer to SOP-FM-01 Field Headspace Analysis and VOC Measurements with PID.
5. Collect soil samples for VOC/VPH/ EPH analysis.	Collect the sample to be analyzed for VOC, VPH, and EPH using a stainless-steel trowel or scoop or a new disposable plastic scoop. If the entire sample interval is represented in the auger head place a representative aliquot of the soil from the auger head directly into the sample container, being careful not to include slough from shallower intervals. Fill the jar to the top allowing no head space (or as the laboratory directs). Pack the material as tightly as feasible and try to avoid getting large particles in the jar. Place the lid on the container as soon as the jar is full. If additional soil is required from the sample interval, fill the jar to a representative level, cap it, and place it in the shade. Continue using the auger to collect soil from the interval, complete PID screening, and add the soil to the appropriate jar. Upon completion, the sample containers should be full to the top with no head space (or as the laboratory directs). Immediately place the filled sample containers in a cooler with ice. Keep samples at 4 ºC or less and under chain of custody protocols until they can be transported to the laboratory for analysis as described in SOP-SA-01 Soil and Water Sample Packaging and Shipping. **Sampling for non-organic constituents can be completed later once VOC sampling is completed.**

6. Record PID readings and sample information in logbook.	If PID screening is conducted, record results of the screening in the project field logbook or field data sheets and include the highest reading from the sample interval. Record the sample information in the logbook and include sample number, associated depth interval, time, date, and type of containers collected as discussed in SOP-SA-05 Project Documentation.
7. Sample remaining depth intervals.	If soil needs to be collected from additional intervals, change the auger head for each new sample interval to prevent potential cross contamination between layers. Then screen the newly uncovered soil with the PID (if required) and collect the appropriate samples for analysis. Complete Steps 3-6 in this section for each sample interval. Place soil from each sample interval in separate area on the plastic to aid in identifying the soil for the inorganic/non-volatile organic sample collection.
8. Collect the inorganic sample material.	Once the samples have been collected for VOC, VPH, and EPH analysis, the non-organic sample material can be collected. Place a stainless-steel bowl or a clean, decontaminated disposable foil on the plastic near the appropriate sample interval (preferably on a clean portion of the plastic) and collect the sample using a stainless-steel trowel or scoop, a new disposable plastic scoop, or a clean glove. Place a representative aliquot of the soil from each sample interval in the mixing container. The sample intervals should be easily identifiable as should have been placed separate areas on the plastic as discussed previously. Take care when transferring soil to the pan to not contaminate other sample interval areas. Keep in mind the potential for cross contamination when sampling new intervals and change gloves and sampling tools between depth intervals. Complete sampling as discussed in **Hand Auger Sampling for Inorganic and Non-Volatile Organic Compounds** Steps 6 and 7.
9. Record sampling information.	Record appropriate information about the sample collection (sample number and associated depth interval, time, date, sample containers, etc.) in the field logbook as discussed in SOP-SA-05 Project Documentation. Record additional information such as soil type and rock content if required by the SAP/WP.
10. Return all the removed soil into the hole.	Return all the remaining removed soil to the sample hole(s) and return the sample area to pre-sampling conditions.
11. Decontam-inate sample tools.	Decontaminate sampling tools according to procedures outlined in SOP-DE-02 Equipment Decontamination.

Soil Recovery at Depths Greater than Five Feet
For soil recovery at depths greater than 5 feet, a direct-push soil recovery rig mounted on a truck or trailer (SOP-S-12), a traditional or sonic drill rig (SOP-S-13), or mechanically dug test pits (backhoe, excavator) (SOP-S-06) are the most common recommended methods. Depth of sample intervals will determine the most appropriate recovery method.

HEALTH SAFETY SECURITY ENVIRONMENT (HSSE) CONSIDERATIONS
This section to be completed with concurrence from the Safety and Health Manager.

SOURCE	*HAZARDS*	*WHERE*	*HOW, WHEN, RESULT*	*CONTROLS*
CHEMICAL	Potential contact with contaminated soil.	Sampling sites.	Inadvertent exposure to contaminated soil could lead to adverse health effects.	Personnel will practice proper personal hygiene – wash hands prior to eating and when leaving the site. Work will be suspended during high wind conditions that may produce large amounts of visible dust. Personnel will wear nitrile gloves and safety glasses when sampling and handling soil.
NOISE	Not applicable.			
ELECTRICAL	Contact with underground utilities.	Sampling sites.	Serious injury could result from contact with a live buried utility.	Established ground disturbance procedures, as outlined in the Pioneer Corporate HASP will be followed.
	Contact with overhead utilities.	Sampling sites.	Walking near low hanging overhead utilities and generators on site could result in electrocution, shock, and burn due to contact or flashover.	Visually inspect the sample location prior to accessing. If overhead hazards are present, established overhead utility procedures will be followed. When possible, personnel will avoid areas with overhead hazards.
BODY MECHANICS	Bending, squatting and kneeling.	During sample collection.	Bending, squatting and kneeling during sample collection and handling could result in muscle/back strains or other injuries. Kneeling on gravel can result in bruises and knee injuries.	Personnel should stretch prior to starting work and they will take breaks when necessary. Personnel will use a foam pad or knee pads, if necessary.

HEALTH SAFETY SECURITY ENVIRONMENT (HSSE) CONSIDERATIONS
This section to be completed with concurrence from the Safety and Health Manager.

SOURCE	*HAZARDS*	*WHERE*	*HOW, WHEN, RESULT*	*CONTROLS*
	Lifting and carrying tools, equipment, and/or samples.	Sampling sites.	Improperly lifting and carrying tools, equipment, and/or samples could result in back injuries and muscle/back strains.	Personnel will use proper lifting techniques – get a good grip, keep the load close to the body, lift with legs and not with back, and avoid lifting loads above shoulders height. Two people will lift, if necessary.
GRAVITY	Falls from slips and trips.	Uneven terrain, slick surfaces and steep slopes.	Personnel could get injured if they fall causing bruises, scrapes, or broken bones.	Personnel will wear work boots with good traction and ankle support. Personnel will plan their path and walk cautiously. Access areas will be established, if necessary.
WEATHER	Cold/heat stress.	Sampling sites.	Exposure to cold temperatures may result in cold burns, frostbite, and hypothermia. Exposure to high temperatures may result in heat cramps, heat exhaustion, or heat stroke.	Training on signs and symptoms of cold/heat stress. Personnel will wear appropriate clothing when working outdoors. Personnel will remain hydrated and will have sufficient caloric intakes during the day. Personnel will follow procedures outlined in applicable SSHASP and/or Pioneer corporate HASP.
	Lightning.	Sampling sites.	Electrocution, injury, death, or equipment damage could be caused by lightning strike.	Personnel will follow the 30/30 rule during lightning storms.
RADIATION	Ultraviolet (UV) radiation.	Outdoors.	Personnel could be exposed to UV radiation causing sun burns, skin damage, and eye damage.	Personnel will wear safety glasses with tinted lenses, long-sleeve work shirts, and long pants. Personnel should wear sunscreen, if necessary.

HEALTH SAFETY SECURITY ENVIRONMENT (HSSE) CONSIDERATIONS
This section to be completed with concurrence from the Safety and Health Manager.

SOURCE	*HAZARDS*	*WHERE*	*HOW, WHEN, RESULT*	*CONTROLS*
BIOLOGICAL	Plants, insects, and animals.	Sampling sites.	Exposure to plants, insects, and/or animals may cause rashes, blisters, redness, and swelling.	Training on the signs and symptoms of exposure to plants, insects, and animals is required. Avoid contact with plants, insects, and animals. First-aid kits will be available on site. Personnel with allergies will notify their supervisor.
MECHANICAL	Hand injuries.	Test pits.	Personnel could cut their fingers if debris (e.g., glass, steel) is present in test pits. Personal injury to the hands could occur when using excavation tools.	Personnel will wear nitrile gloves when sampling and handling soil. Personnel will wear leather gloves while using excavation tools.
	Struck by shovel or auger.	Carrying tools.	Personnel can strike other workers or objects when carrying shovels and augers to/from sampling stations resulting in bodily injuries and/or property damage.	Personnel will be aware of their surroundings and, if needed, use a spotter. When carrying tools, maintain a safe distance (e.g., 4 feet or more depending on side of tool) from other personnel.
PRESSURE	Not applicable.			
THERMAL	Not applicable.			

HEALTH SAFETY SECURITY ENVIRONMENT (HSSE) CONSIDERATIONS This section to be completed with concurrence from the Safety and Health Manager.				
SOURCE	***HAZARDS***	***WHERE***	***HOW, WHEN, RESULT***	***CONTROLS***
HUMAN FACTORS	Inexperienced and improperly trained personnel.	Sampling sites.	Inexperienced personnel and improper training could cause incidents resulting in adverse health effects and/or property damage.	Personnel will be properly trained in this procedure and other applicable procedures. Personnel will implement stop work procedures, if necessary.
	Public entering the work area.	Sampling sites.	Third-party members of the public could enter the work area resulting in an unsafe work environment.	Stop work if members of the public enter the work area.
SIMOPS (Simultaneous Operations)	Not applicable.			

ADDITIONAL HSSE CONSIDERATIONS This section to be completed with concurrence from the Safety and Health Manager.	
REQUIRED PPE	**Personal Protection Equipment** (PPE): Hard hat, safety glasses, high-visibility work shirt or vest, long pants, work boots, nitrile gloves, and leather gloves.
APPLICABLE SDSs	**Safety Data Sheets** (SDSs) will be maintained based on site characterization and contaminants. Safety Data Sheets are available to Pioneer personnel on the internal website under Safety.
REQUIRED PERMITS/ FORMS	Per site/project requirements.
ADDITIONAL TRAINING	Per site/project requirements.

DRAWINGS, DOCUMENTS, AND TOOLS/EQUIPMENT The following documents should be referenced to assist in completing the associated task.	
DRAWINGS	Map with site location and sample locations.
RELATED SOPs/ PROCEDURES/ WORK PLANS	SOP-DE-02 Equipment Decontamination SOP-FM-01 Field Headspace Analysis and VOC Measurements with PID SOP-S-01 Surface Soil Sampling SOP-S-06 Test Pit Sampling SOP-S-12 Sampling Soil from a Geoprobe Liner SOP-S-13 Sampling Core from Sonic Drill SOP-SA-01 Soil and Water Sample Packaging and Shipping SOP-SA-05 Project Documentation
TOOLS/ EQUIPMENT	Sampling tools (e.g., shovel, breaker bar, stainless-steel tape, ruler, hand auger, plastic sheeting, trowels/scoops, screwdriver, sample containers, stainless-steel bowls or disposal foil pans, and camera), cooler with ice (if needed), and PID (if required).
FORMS/ CHECKLIST	Field logbook and field data sheets.

APPROVALS/CONCURRENCE By signing this document, all parties acknowledge the completeness and applicability of this SOP for its intended purpose. Also, by signing this document, it serves as acknowledgement that I have received training on the procedure and associated competency testing.	
SOP TECHNICAL AUTHOR	**DATE**
Julie Flammang **Julie Flammang**	**11/23/2020**
SAFETY AND HEALTH MANAGER	**DATE**
Tara Schleeman **Tara Schleeman**	**11/23/2020**

PURPOSE	To provide standard instructions for soil and water sample packaging and shipping for unreclaimed sites in the BPSOU area.
SCOPE	Work described in this procedure includes instruction on the correct methods to package, ship and Chain of Custody documentation.

WORK INSTRUCTIONS

The following instructions are intended to provide sufficient guidance to perform the task in a safe, accurate, and reliable manner. Should these instructions present information that is inaccurate or unsafe, operations personnel must bring the issue to the attention of the Project Manager and the appropriate revisions made.

TASK	INSTRUCTIONS
1. Place the sample containers in Ziploc bags.	Based on the analytes requested (e.g., low level mercury, low level chromium, etc.), it may be necessary to place each filled sample container in separate Ziploc bags to prevent cross contamination; keep the container clean, dry, and isolated; and protect the sample label. In most cases, all sample containers collected from a specific sample location are placed in a large Ziploc bag and shipped together.
2. Package the samples.	Place samples in a cooler, which has been previously lined with a plastic bag. Surround the samples with non-contaminating packaging materials to reduce movement and absorb any leakage. Double bag the ice and place it in the cooler. Seal the plastic bag in the cooler to contain the samples, packing material, and ice.
3. Review and sign Chain of Custody forms.	The Field Team Leader or their designated representative will double check the Chain-of-Custody (CoC) forms to assure those samples recorded on the CoC forms are in the cooler. The Field Team Leader or the designated representative will then sign the CoC form to relinquish custody. One copy of the signed CoC form will remain with the Field Team Leader. Make a photocopy of the completed forms, if there are no carbon copies available.
4. Tape paperwork to cooler.	Place paperwork in a sealed Ziploc bag and tape it to the inside of the cooler lid.
5. Bag samples for separate analytical batches.	If the shipping cooler contains more samples than can be analyzed in one analytical batch, the laboratory may request that the samples in the cooler be bagged for separate analytical batches. This may be necessary so that the appropriate Quality Control/ Quality Assurance samples are included in each analytical batch. In this case, fill out separate COC forms for each batch and include the forms in the appropriate plastic bags. Place the COC forms for each batch in a sealed Ziploc bag. The COC forms for each batch should be placed at the top of the plastic bag so that they are clearly visible to laboratory personnel when they open the plastic bags.
6. Label the cooler.	Label the cooler with the appropriate labels to describe the content of the cooler (e.g., NOS, flammable liquids, flammable solids, this side up, fragile, etc.). Close the cooler and place the appropriate shipping labels (e.g., overnight shipping from Federal Express, UPS, or the U.S. Postal Service or equivalent) on the lid of the cooler.
7. Sign CoC seals.	The Field Team Leader or the designated representative will sign CoC seals and place the signed seals over the opening edge of the cooler.

8. Tape the cooler.	Place tape over the custody seals and around the cooler.
9. Transport the cooler.	Transport the cooler(s) to a secure storage, to the shipping agent, or directly to the laboratory. If shipping the cooler, follow established federal and state regulations depending on cooler content.
Note:	Bagging of samples and lining of coolers is not necessary, if samplers transport the samples directly to the laboratory.

DRAWINGS, DOCUMENTS, AND TOOLS/EQUIPMENT **The following documents should be referenced to assist in completing the associated task**.	
P&IDS	
Drawings	
Related SOPs/ Procedures/ Work Plans	As per individual site SAPs.
Tools	Plastic bags, Ziploc bags, non-contaminating packaging materials, tape, COC seals, ice, and cooler
Forms/Checklist	Chain of Custody forms.

PURPOSE	This Standard Operating Procedure (SOP) establishes the requirements for documenting and maintaining environmental sample chain of custody from point of origin to receipt of sample at the analytical laboratory. This procedure will apply to all types of air, soil, water, sediment, biological, and/or core samples collected in environmental investigations by Pioneer Technical Services, Inc. (Pioneer). It is applicable from the time of sample acquisition until custody of the sample is transferred to an analytical laboratory.
SCOPE	Pioneer prepared this practice for the workforce and this SOP applies to all work performed by and on behalf of Pioneer. All members of the Pioneer workforce who conduct the work shall be trained and competent (as defined by OSHA) in the risk-assessed procedure described below before performing the work.
DEFINITIONS	**Chain of custody** is an unbroken trail of accountability that ensures the physical security of samples, data, and records. Custody refers to the physical responsibility for sample integrity, handling, and/or transportation. Custody responsibilities are effectively met, if the samples are: • In the responsible individual's physical possession; • In the responsible individual's visual range after having taken possession; • Secured by the responsible individual so that no tampering can occur (usually for shipping); or • Secured or locked by the responsible individual in an area in which access is restricted to authorized personnel only.

WORK INSTRUCTIONS

The following instructions provide guidance to perform the task in a safe, accurate, and reliable manner. If these instructions present information that is inaccurate or unsafe, personnel must notify the Project Manager, Safety Manager, and the SOP Technical Author to initiate appropriate revisions. Personnel will perform all work under this SOP in a manner that is consistent with procedures and policies described in the appropriate Operation, Maintenance, and Monitoring (O&M) Plan (where applicable), appropriate Site-Specific Health and Safety Plans (SSHASP), and Pioneer Corporate Health and Safety Plan (HASP).

TASK	INSTRUCTIONS
Project Manager's Responsibilities	The Project Manager is responsible for overall management of environmental sampling activities, designating sampling responsibilities to qualified personnel, and reviewing any changes to the sampling plan.
Field Team Leader's Responsibilities	The Project Manager may act as the Field Team Leader or may choose to appoint a Field Team Leader. The Field Team Leader is responsible for general supervision of field sampling activities and ensuring proper storage/transportation of samples from the field to the analytical laboratory. The Field Team Leader is also responsible for maintaining sample custody as defined above until the sample has been properly relinquished as documented on the chain of custody form.

	The Field Team Leader will review chain of custody forms for accuracy and completeness to preserve sample integrity from collection to receipt by an analytical laboratory. The review of chain of custody forms may be delegated to qualified personnel.
Field Sampler's Responsibilities	The Field Sampler is responsible for sample acquisition in compliance with technical procedures, initiating the chain of custody, and checking sample integrity and documentation prior to transfer. Field samplers are also responsible for initial transfer of samples consisting of physical transfer of samples directly to the internal laboratory or transferred to a shipping carrier, (e.g., United Parcel Service or Federal Express) for delivery.
Laboratory Technician's Responsibilities	The receiving Laboratory Technician is responsible for inspecting transferred samples to ensure proper labeling and satisfactory sample condition. Unacceptable samples will be identified and segregated. The Laboratory Project Manager will be notified. The Laboratory Technician will review the chain of custody for completeness and file as part of the project's permanent record.
Fill out Chain of Custody Forms	The Field Team Leader or designated Field Sampler will initiate the chain of custody form for the initial transfer of samples. A chain of custody form will be completed and accompany every sample set. Only those samples included in the shipping container (cooler or box) should be listed on the chain of custody form included in the container. All chain of custody forms must be completed and include the following information: • Project code. • Project name. • Sampler's signature. • Sample identification. • Date sampled. • Time sampled. • Analysis requested. • Remarks column should contain information about a sample that the laboratory might need. Examples of remarks that should be included: ▪ If samples could have very high or low expected concentrations (outside of normal instrument calibration range). ▪ DO NOT USE FOR QA/QC (quality assurance/quality control) should be indicated for field blanks, bottle blanks, or equipment rinsate blanks. ▪ If a sample should be held for later analysis (i.e., if sample being analyzed requires results from another sample to determine analysis status).

	▪ The sample should be archived after initial analysis by the laboratory for potential additional analysis in the future. ▪ Requires filtering (if not completed in the field). ▪ Requires preservation (if not completed in the field). ▪ Any other sample specific information that will aid the laboratory in completing the appropriate analysis. • Relinquishing signature, data, and time. • Receiving signature, date, and time. Laboratory-provided chain of custody forms should be used if provided, and all required fields should be filled out. Pioneer also has generic chain of custody forms that can be used if no laboratory forms are available. Make sure that the above required information is on the form and include the laboratory name and address to which the samples are being shipped. The Field Sampler relinquishing custody and the responsible individual accepting custody will sign, date, and note the time of transfer on the chain of custody form. Note: if the transporter is not an employee of Pioneer, the Field Sampler may identify the carrier and reference the bill of lading number in lieu of the transporter's signature. One copy of the chain of custody form will be filed as a temporary record of sample transfer by the Field Sampler. The original form will accompany the sample set and will be returned to Pioneer as part of the contracted laboratory QA/QC requirements. The original form and the transporter's receipt will be filed as part of the project's permanent records. The Project Manager (or designee) will track the chain of custody to ensure timely receipt of samples by an analytical laboratory. Shipping information, including date shipped, laboratory shipped to, transporter's identity (i.e., Federal Express), and tracking number should be recorded in the field logbook. If more than one sample shipment occurs during a project, the associated samples per shipment should be referenced (sample numbers or samples collected on these dates).
Sample Handling.	All samples will be collected and handled in accordance with SOP-SA-01 Soil and Water Sample Packaging and Shipping and SOP-SA-02 Sample Preservation and Containerization for Aqueous Samples, or methods described in the Sampling and Analysis Plan (SAP) or Work Plan (WP). Samples will be transported in insulated coolers with ice as necessary to maintain a temperature of 4 degrees Celsius (°C) plus or minus 2 °C until receipt by the analytical laboratory. Alternate shipping containers can be used if the analytical method, SAP, or WP does not have temperature requirements for the samples.

HEALTH SAFETY SECURITY ENVIRONMENT (HSSE) CONSIDERATIONS

This section to be completed with concurrence from the Safety and Health Manager.

SOURCE	*HAZARDS*	*WHERE*	*HOW, WHEN, RESULT*	*CONTROLS*
CHEMICAL	Potential contact with contaminated water/soil samples.	Outside of bottles.	Inadvertent exposure to contaminated water/soil samples could lead to adverse health effects.	Personnel will practice proper personal hygiene – wash hands prior to eating/drinking and when leaving the site. Personnel will wear nitrile gloves and safety glasses when handling sample containers.
	Preservatives (HCL, HNO_3, H_2SO_4, Zinc, Acetate, and NaOH).	Outside of bottles.	Inadvertent exposure to preservatives could lead to adverse health effects.	Safety Data Sheets for each preservative chemical are available to all Personnel on the Pioneer company web site. Personnel will wear nitrile gloves and safety glasses when handling the bottles. Refer to the Chemical Flushing Guidelines available inside vehicle's first aid kit for first-aid procedures in case of contact with preservatives.
NOISE	Not applicable.			
ELECTRICAL	Not applicable.			
BODY MECHANICS	Improper lifting.	Sites.	Back injuries and muscle/back strains could result when using improper techniques to lift and carry packaged samples and coolers.	Personnel will use proper lifting techniques – get a good grip, keep the load close to the body, lift with legs and not with back, and avoid lifting loads above shoulder's height. Two workers will lift/carry packaged samples and coolers, if needed.

HEALTH SAFETY SECURITY ENVIRONMENT (HSSE) CONSIDERATIONS

This section to be completed with concurrence from the Safety and Health Manager.

SOURCE	*HAZARDS*	*WHERE*	*HOW, WHEN, RESULT*	*CONTROLS*
GRAVITY	Falls from slips and trips.	Uneven terrain, slick/muddy/wet surfaces and steep slopes.	Walking/working on slick/muddy/ wet and uneven terrain could cause slips and trips resulting in falls and injuries.	Personnel will wear work boots with good traction and ankle support. Personnel will be aware of working/walking surfaces and choose a path to avoid hazards. Keep work areas as dry as possible.
WEATHER	Not applicable.			
RADIATION	Not applicable.			
BIOLOGICAL	Not applicable.			
MECHANICAL	Not applicable.			
PRESSURE	Not applicable.			
THERMAL	Not applicable.			
HUMAN FACTORS	Inexperienced and improperly trained personnel.	Sites.	Inexperienced personnel and improper training could cause incidents resulting in adverse health effects and/or property damage.	Personnel will be properly trained in this procedure and other applicable procedures. Personnel will implement stop work procedures, if necessary.
SIMOPS (Simultaneous Operations)	Not applicable.			

ADDITIONAL HSSE CONSIDERATIONS This section to be completed with concurrence from the Safety and Health Manager.	
REQUIRED PPE	**Personal Protection Equipment** (PPE): Safety glasses, high-visibility work shirt or vest, long pants, work boots, and nitrile gloves.
APPLICABLE SDSs	**Safety Data Sheets** (SDSs): HCL, HNO_3, H_2SO_4, Zinc, Acetate, and NaOH. Safety Data Sheets are available to Pioneer employees at the link below: https://pioneertechnicalservices.sharepoint.com/Safety/SafetyDataSheets
REQUIRED PERMITS/ FORMS	Per site/project requirements.
ADDITIONAL TRAINING	Per site/project requirements.

DRAWINGS, DOCUMENTS, AND TOOLS/EQUIPMENT The following documents should be referenced to assist in completing the associated task.	
DRAWINGS	
RELATED SOPs/ PROCEDURES/ WORK PLANS	SOP-SA-01 Soil and Water Sample Packaging and Shipping and SOP-SA-02 Sample Preservation and Containerization for Aqueous Samples.
TOOLS/ EQUIPMENT	Seals and labels, chain of custody forms, chain of custody seals (provided by contracted laboratory), packing and shipping materials, cooler, and ice.
FORMS/ CHECKLIST	Chain of custody forms.

APPROVALS/CONCURRENCE

By signing this document, all parties acknowledge the completeness and applicability of this SOP for its intended purpose. Also, by signing this document, it serves as acknowledgement that I have received training on the procedure and associated competency testing.

SOP TECHNICAL AUTHOR	DATE
Julie Flammang **Julie Flammang**	**11/12/2020**
SAFETY AND HEALTH MANAGER	**DATE**
Tara Schleeman **Tara Schleeman**	**11/12/2020**

PURPOSE	This SOP establishes the requirements for documenting and maintaining field logbooks and photographs. These procedures apply from the time field work begins until site activities are completed.
SCOPE	This practice has been prepared as a basic guide for project documentation.

WORK INSTRUCTIONS

The following instructions are intended to provide sufficient guidance to perform the task in a safe, accurate, and reliable manner. Should these instructions present information that is inaccurate or unsafe, operations personnel must bring the issue to the attention of the Project Manager and the appropriate revisions made.

TASK	INSTRUCTIONS
Logbooks	A designated field logbook will be used for each field project. If requested by the Project Manager, use a separate field logbook for each field task within a larger project. Label each logbook with the project name, dates that it covers, and logbook number. Use a waterproof marker, such as a Sharpie©, to write down the information. The logbooks will be bound and have consecutively numbered pages. The information recorded in these logbooks shall be written in ink. Begin a new page for each days notes. Write on every line of the logbook. If a blank space is necessary for clarity, such as a change of subject, skip one line before beginning the new subject. Do not skip any pages or parts of pages unless a day's activity ends in the middle of a page. Draw a diagonal line on any blank spaces of four lines or more to prevent unauthorized entries. The author will initial and date entries at the end of each day. All corrections will consist of a single line-out deletion in ink, followed by the author's initials and the date. Information not related to the project should not be entered in the logbook. The language used in the logbook should be factual and objective. These bound logbooks shall include the following entries: 1. A description of the field task. 2. Time and date fieldwork started. 3. Location and/or a description of the work areas including sketches, if needed, any maps or references needed to identify locations, and sketches of construction activities. If the location has been documented in the logbook during/prior visits, only changes in conditions should be noted. 4. Names and company affiliations of field personnel. 5. Name, company affiliation or address, and phone number of any field contacts or official site visitors. 6. Meteorological conditions at the beginning of fieldwork and any ensuing changes in these conditions. 7. Details of the fieldwork performed and reference to field data sheets, if used. 8. Deviation from the task-specific Sampling and Analysis Plan (SAP), Work Plan (WP), or Standard Operating Procedures (SOP). 9. All field measurements made.

	10. Any field laboratory analytical results. 11. Personnel and equipment decontamination procedures, if appropriate. For any field sampling work, the following entries should be made: 1. Sample location and number. 2. Sample type and amount collected. 3. Date and time of sample collection. 4. Type of sample preservation. 5. Split samples taken by other parties. Note the type of sample, sample location, time/date, name of person for whom the split was collected, that person's company, and any other pertinent information. 6. Sampling method, particularly any deviations from the SOP. 7. Documentation or reference of preparation procedures for reagents or supplies that will become an integral part of the sample, if available. This information may not be available for water or soil sampling bottles that come preserved from the laboratory or for preservatives provided by the laboratory. Bottle blanks will need to be used to evaluate the provided reagents. 8. The laboratory where the samples will be sent. No bound field logbooks will be destroyed or thrown away even if they are illegible or contain inaccuracies that require a replacement document.
Photographs	Take photographs of field activities using a digital camera. Photographs should include a scale in the picture when practical. Telephoto or wide-angle shots will not be used, since they cannot be used in enforcement meetings. The following items shall be recorded in the bound field logbook or on a field data sheet for each photograph taken: 1. The photographer's name, the date, the time of the photograph, and the general direction faced. 2. A brief description of the subject and the fieldwork portrayed in the picture. 3. Sequential number of the photograph. An electronic copy and/or a hard copy of the photographs shall be placed in task files in the field office after each day of field activities. Supporting documentation from the bound field logbooks or field data sheets shall be photocopied and placed in the task files to accompany the photographs once the field activities are complete

DRAWINGS, DOCUMENTS, AND TOOLS/EQUIPMENT The following documents should be referenced to assist in completing the associated task.	
P&IDS	
Drawings	
Related SOPS/ Procedures/ Work Plans	
Tools	Field logbook, Sharpie©, black pen, digital camera, and field data sheets.
Forms/Checklist	

STANDARD OPERATING PROCEDRE

FIELD LABORATORY DETRMINATION OF SOIL PH USING HI 99121 SOIL PH METER

February 10, 2012

Field Laboratory Procedure

1. **Operation of Device**
    a. To turn the device on or off Press: On/Off.
    b. To Freeze the device Press: Set/Hold.

2. **Calibrate the PH Meter**
    a. Connect the PH probe to the meter.
    b. Hold the On/Off button until Calibration is visible on the screen.
    c. Put the probe in 7.01 calibration solution.
    d. The meter will recognize the solution and calibrate.
    e. Once the calibration is recognized and stable, press: On/Off

3. **To Take a Measurement**
    a. Connect the probe when the device is off.
    b. Remove the protective cap from the probe.
    c. Insert the probe into the sample.
    d. Wait until the "not stable" read out has turned off; and
    e. Record the measurement.

4. **Direct Ground Measurement of PH**
    a. Verify that the Meter is calibrated.
    b. Dig a small hole, discarding the top 5 centimeters (2 inches) of soil.
    c. Perforate the soil with the included soil drill to a depth of at least 20 centimeters (8 inches).
    d. If the soil is dry, moisten with a small amount of distilled water.
    e. Rinse the probe with tap water (not distilled).
    f. Insert the probe slightly into the soil, making sure that it is in contact with the soil surfaces.
    g. Once the readings have stabilized record the measurement.
    h. Remove the probe from the hole, gently clean off loose soil with your fingers (avoid using a rag or cloth) and then rinse the probe with tap water;
    i. Repeat this procedure in several locations; then
    j. Average the results.

5. **Measurement of Soil PH Solution**
    a. Verify that the Meter is calibrated.
    b. Collect a soil sample:
        i. Collect a minimum of one sample per 0.25 acres if the area is homogeneous (soil type, vegetation type, slope etc.).
            1. A minimum of 2 subsamples are recommended for each sample.
            2. If the area is considered "contaminated" collect all samples for that composite within that area.
            3. Collect a similar quantity for each subsample.
        ii. Dig a small hole, discarding the top 5 centimeters (2 inches) of soil, collect the sample from the hole. Complete this step for each subsample.
        iii. Thoroughly mix the subsamples for each sample together, discarding vegetation and aggregates.
        iv. Spread the sample on a sheet (paper, foil or aluminum pan) and allow to dry in a shaded area or place in an oven to dry. Discard sheet when done drying.
    c. Measuring PH of the Soil Sample
        i. Sift the soil sample through a clean #10 screen.
        ii. Measure 10 grams of the sample and place it in a beaker.
        iii. Measure 25 milliliters of Soil Solution HI 7051 into the beaker.

iv. Mix for 30 seconds.
v. Let the mixture sit 5 minutes.
vi. Mix again; and
vii. Place probe in mixture and wait for reading to stabilize. Record the measurement.
viii. Rinse the probe with tap water prior to next use. If needed remove any remaining soil on the probe using a finger (avoid using a rag or cloth).

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PURPOSE	To provide standard instructions for operating XL3 X-Ray Fluorescence (XRF) analyzer
SCOPE	This practice has been prepared for task trained personnel conducting work on unreclaimed sites within the BPSOU area. The tasks are general and are to be used in conjunction with published manufacturer and internal practices.

WORK INSTRUCTIONS

The following instructions are intended to provide general guidance to perform the task in a safe, accurate, and reliable manner. Should these instructions present information that is inaccurate or unsafe, operations personnel must bring the issue to the attention of the Project Manager and the appropriate revisions made. All work carried out under this SOP will be consistent with procedures and policies described within appropriate internal policies.

TASK	INSTRUCTIONS
1. Assemble XRF stand.	a. Open the case containing the stand and insert 4 legs into base of stand. b. Place stand on a solid, level surface.
2. Prep XRF sample for analysis.	a. Wearing latex or nitrile gloves, remove any large aggregate from the sample and place in a separate bag for disposal. For gravel or rocky soils, a sieve can be used to remove the large aggregates. If a sieve is used, it needs to be decontaminated between samples. Refer to SOP General Equipment Decontamination for instructions. b. Consolidate the sample into the bottom of the baggie. c. Open the lid to the XRF stand and place sample inside, making sure that sample is flush against the opening on the inside of the XRF stand. d. Close the lid to the XRF stand.
3. Turn on XRF case.	a. Open the XRF case and remove XRF gun from case. b. Slide XRF battery onto bottom of XRF gun handle. c. Press and hold power button ( ) until XRF gun turns on and wait for system to start. d. Press where it says 'press to logon.' A warning message appears asking to verify that the user is aware of the radiation source in the XRF unit. e. Press 'Yes' to continue.
4. Log in and calibrate detector.	a. Type in appropriate password when prompted. b. Click ‘E’ to log in. After logging in, a screen appears with 7 icons appears, this is the Main Menu screen. c. Tap the ‘System Check’ icon. d. Tap ‘Yes.’ e. The XRF unit will then go through an internal calibration. f. When the calibration is done, tap ‘CLOSE’ on the XRF gun to return to the Main Menu screen. **The detector should be calibrated at the start of each day of operation.**
5. Set up XRF run test.	a. Set parameters (e.g., analysis types, time, and analytes) required for the analysis as detailed in the XL3 user’s manual, Sampling and Analysis Plan (SAP), or Work Plan. b. Once logged into XRF system, tap the ‘Analyze’ icon on XRF screen. A screen appears. c. On the next screen tap ‘Soils.’ d. On the next screen tap ‘Data Entry.’ A Data Entry screen appears showing several options (Sample Name, Sampler, Date, etc.).

	e. In the upper right-hand corner, next to the 'Sample Name' icon, click the symbol that looks like a miniature keyboard to display a keyboard on the screen. f. Type in the sample name (do not press return yet). g. Insert XRF gun into the bottom of the XRF stand with the XRF gun handle pointing away from you. Be sure that the XRF gun is securely in place in the bottom of the stand. h. Press 'return' in the lower right corner of the keyboard screen. i. To activate the unit, pull the trigger on the gun handle. The analysis will take approximately 2 minutes to complete.
6. Record data.	a. After the XRF analysis is complete, results from the analysis will appear on the screen. b. Record the results and Test Number displayed on the screen; use the up and down arrows on the XRF gun to scroll through data. c. Open the lid on the XRF stand and remove the sample. d. Mark the sample baggie as "RAN" so that sample does not get analyzed twice. Place ran samples in a labeled box for storage and record keeping.
7. Run additional samples.	a. With the XRF gun still in the XRF stand, press the return button (↵) on the XRF gun. This will display the 'Data Entry' screen. b. On the Data Entry Screen, press the keyboard symbol located to the right of 'Sample Name' to display the keyboard. c. Type the next sample name (do not press return yet). d. Place the sample into the XRF stand and close the lid to the stand (as discussed in Task 2). e. Repeat the steps in Task 5 to activate the XRF unit. f. Repeat Tasks 6 and 7 until all samples are analyzed.
8. Turn off XRF.	a. After all samples have been analyzed, remove the XRF gun from the bottom of the stand (press and hold buttons on the side of the stand to allow XRF gun to be removed from stand). b. Press the return button (↵) on the XRF gun until the Main Menu screen appears. c. Press and hold the power button (⏻) until the XRF turns off. d. Remove the battery from the gun and place these items back into the appropriate case. e. Disassemble the XRF stand and place back into the appropriate case.

Quality Assurance/ Quality Control (QA/QC) Requirements.	**Required QA/QC tasks:** 1. Run the Niton-supplied XRF blanks and NIST standards at the start of each day. 2. Record the results in the field logbook or on the XRF field datasheet or equivalents. If the results are not within the ranges supplied by NITON in the user manual, initiate troubleshooting tasks on the analyzer (refer to the user's manual). 3. Run the blank and one standard QA/QC samples during sample analysis at the rate of 1 for every 20 samples analyzed. QA/QC includes analyzing a replicate sample every 20 samples and a duplicate sample (see the steps below). **Analyze a replicate sample (1 for every 20 samples analyzed)** 1. After recording the initial reading for a sample, DO NOT remove the sample from the holder. 2. Restart the XRF gun and rerun the sample. 3. Record the information on the field data form or logbook as a replicate (or R sample). Replicates samples help track the precision of the XRF.

	Analyze a duplicate sample (after every 20 samples analyzed) 1. After every 20 samples, analyze a duplicate sample by recording the results of the 20th sample. 2. Remove the sample bag from the XRF stand, remix the sample, and replace it in the XRF stand. 3. Reanalyze the sample. 4. Record the results as a duplicate (or D sample). Duplicates help to determine the precision of the XRF analysis as well as the homogeneity of the sample matrix. 5. Run a NITON-supplied blank or NIST standard after the replicate/duplicate QA/QC samples to monitor the accuracy of the XRF results.

DRAWINGS, DOCUMENTS, AND TOOLS/EQUIPMENT **The following documents should be referenced to assist in completing the associated task**.	
Drawings	
Related SOPs/ Procedures/ Work Plans	SOP-DE-02 General Equipment Decontamination.
Tools	XRF and hand tools.
Forms/Checklist	Private Property Access Agreement, if required.

APPROVALS/CONCURRENCE

By signing this document, all parties acknowledge the completeness and applicability of this SOP for its intended purpose. Also, by signing this document, it serves as an acknowledgement that I have received training on the procedure and associated competency training

Manager	**Date**
Lead Operator	**Date**
Operator	**Date**

SOP-DE-01. PERSONAL DECONTAMINATION PROCEDURES - GENERAL	REVISION: 0 PAGE 1 of 2

PURPOSE	To provide standard instructions for decontamination of all personnel leaving a contaminated area.
SCOPE	This practice has been prepared for task trained personnel conducting work on unreclaimed sites within the BPSOU area. The tasks are general and are to be used in conjunction with published manufacturer and internal practices.

WORK INSTRUCTIONS

The following instructions are intended to provide general guidance to perform the task in a safe, accurate, and reliable manner. Should these instructions present information that is inaccurate or unsafe, operations personnel must bring the issue to the attention of the Project Manager and the appropriate revisions made. All work carried out under this SOP will be consistent with procedures and policies described within appropriate internal policies.

TASK	INSTRUCTIONS
1. Wash/ Remove outer contaminated items.	Remove nitrile or latex gloves by grasping the outside of the opposite glove near the wrist. Pull and peel the glove away from the hand, turning the glove inside out with the contaminated side now on the inside. Hold the removed glove in the opposite gloved hand. Slide one or two fingers of the ungloved hand under the wrist of the remaining glove. Peel glove off from the inside, creating a bag for both gloves. If wearing protective coveralls such as Tyvec suites, brush built up material off the suit, only if in designated decontamination zone. Unzip the coverall and begin rolling that outwards, rolling it down over your shoulders. Place both hands behind your back and pull down each arm until completely removed. Sit down and remove each shoe then roll the coveralls down (ensuring the contaminated side is not touched or comes into contact with clothing) over your knees until completely removed. If there is not a designated decontamination zone, remove personal protective equipment (PPE) carefully to contain material and place it in the appropriate disposal container. For instructions to remove additional PPE not described in this document, refer to the project's HASP. Wash with soap (nonphosphate) and tap water the outer, more heavily contaminated items, such as boots. Rinse the items in tap water.
2. Wash inner contaminated items.	If necessary, wash with soap (nonphosphate) and tap water the inner, less contaminated items. Rinse the items in tap water.
3. Store/ transport items.	Store/transport contaminated items in a separate designated area to prevent cross contamination prior to disposal.
4. Dispose of contaminated items.	Dispose of contaminated clothing and equipment in accordance with site/project, client, and/or federal and state requirements.
5. Contact the Safety and Health Manager.	For contaminants other than those found typically at uncontrolled hazardous waste sites, such as asbestos, PCB, PCE, etc. see the Safety and Health Manager.

Information about Emergency Decontamination	
1. During life-saving process.	If the decontamination procedure is essential to the life-saving process, decontamination must be performed immediately.
2. During heat-related illness.	If heat-related illness develops, protective clothing should be removed as soon as possible. Wash, rinse, and/or cut off protective clothing/equipment.
3. When medical treatment is needed.	If medical treatment is required to save a life, decontamination should be delayed until the victim is stabilized. Wrap the victim to reduce contamination of others.

DRAWINGS, DOCUMENTS, AND TOOLS/EQUIPMENT The following documents should be referenced to assist in completing the associated task.	
Drawings	
Related SOPS/ Procedures/ Work Plans	
Tools	In general, the following items will be needed: soap, tap water, tarps, decontamination tubs, brushes, and sprayer. The Sampling and Analysis Plan (SAP) will describe additional items needed for decontamination, if required.
Forms/Checklist	

APPROVALS/CONCURRENCE

By signing this document, all parties acknowledge the completeness and applicability of this SOP for its intended purpose. Also, by signing this document, it serves as an acknowledgement that I have received training on the procedure and associated competency training

Manager	**Date**
Lead Operator	**Date**
Operator	**Date**

PURPOSE	To provide standard instructions for equipment decontamination.
SCOPE	Pioneer Technical Services, Inc. (Pioneer) prepared this practice for the workforce and this Standard Operating Procedure (SOP) applies to all work performed by and on behalf of Pioneer. All members of the Pioneer workforce who conduct the work shall be trained and competent (as defined by OSHA) in the risk-assessed procedure described below before performing the work.
NOTES	All equipment leaving the contaminated area of a site must be decontaminated. Decontamination methods include removal of contaminants through physical, chemical, or a combination of both methods. Decontamination procedures are to be performed at the same level of protection used in the contaminated area of a site. In some cases, decontamination personnel may be sufficiently protected by wearing one level lower protection. The information for site-specific equipment decontamination and personnel protection levels, as detailed in the Sampling and Analysis Plan (SAP), work plan (WP), and Site-Specific Health and Safety Plan (SSHASP), should be followed. The following decontamination procedures are for typical uncontrolled hazardous waste sites. For a specific or unusual contaminant, such as dioxins, see the SSHASP and consult with the Safety and Health Manager. Decontamination procedures should be used in conjunction with methods to prevent contamination of sampling and monitoring equipment. If practical, particularly with organic contaminants, one-time-use equipment should be used and disposed of in accordance with the SAP, WP, and SSHASP. This SOP covers all equipment decontamination EXCEPT for submersible pumps. Decontamination of pumps is detailed in SOP-DE-02A – Equipment Decontamination - Pumps for Well Sampling.

WORK INSTRUCTIONS

The following instructions provide guidance to perform the task in a safe, accurate, and reliable manner. If these instructions present information that is inaccurate or unsafe, personnel must notify the Project Manager, Safety Manager, and the SOP Technical Author to initiate appropriate revisions. Personnel will perform all work under this SOP in a manner that is consistent with procedures and policies described in the appropriate Operation, Maintenance, and Monitoring (O&M) Plan (where applicable), appropriate Site-Specific Health and Safety Plans (SSHASP), and Pioneer Corporate Health and Safety Plan (HASP).

TASK	INSTRUCTIONS
1. Set up decontamination station.	a. Review the SAP or WP and determine if decontamination fluids need to be contained and the need for special decontamination requirements (i.e., chemical rinse). b. If the fluids require containment, set up the decontamination station so that it is located within a small plastic swimming pool or on plastic sheeting with turned up edges to contain water that may slop over during the decontamination process.

	c. If pressurized or gravity flow water is available, attach a hose or piping to reach the decontamination area. If no water is available, bring 5-gallon containers of tap and deionized water (DI) to the decontamination area to clean the equipment. d. Label empty 5-gallon buckets: *gross wash, soap wash, DI rinse, final rinse, and chemical rinse* (if required). e. Lay out clean plastic or foil to place cleaned equipment on to allow for air drying. f. If a chemical rinse is required, fill a spray bottle with the appropriate chemical and label the spray bottle with the chemical's name. g. Pour approximately 2.5 to 3 gallons of tap water into the buckets labeled: *gross wash* and *soap wash*. h. Add a **few drops** (1-3 drops) of Liquinox© soap to the bucket marked *soap wash*. i. Pour 2.5-3 gallons of DI water into the buckets labeled: *DI rinse* and *final rinse.* If a chemical rinse is required, pour DI water into the bucket labeled: *chemical rinse.*
2. Remove gross contamination.	Remove gross contamination using pressurized or gravity flow tap water, if available. If not, manually scrub the equipment using the 5-gallon bucket of water marked *gross wash* and a stiff brush (dedicated to the gross wash step).
3. Wash equipment.	Move the equipment to the 5-gallon bucket marked *soap wash*. Wash equipment with a stiff brush (dedicated to the soap wash step).
4. Triple rinse equipment.	In the bucket marked *DI rinse*, triple rinse the equipment with DI water to remove any soap residue.
5. Second rinse with deionized water.	Using DI water, triple rinse the equipment again in the bucket marked *final rinse* if a chemical rinse is not required.
6. Rinse equipment with chemicals.	In many cases, the tap water and DI water rinses will be sufficient. However, if specified in the SAP, WP, or SSHASP, chemical rinses of the equipment may be required. For inorganic contaminants, a mixture of 10:1 nitric acid in distilled water (10 parts water to 1 part nitric acid) may be specified. A methanol rinse may be required for some organic contaminants, such as hydrocarbons. Spray bottles, clearly marked with the appropriate chemical name, are an acceptable means of rinsing most equipment. **To perform the chemical rinse:** a. Hold the equipment over a collection container (5-gallon bucket or bowl). b. Make sure that all personnel and vehicles are upwind of the spray. c. Spray the piece of equipment inside and out starting at the top and working down to the bottom. d. Dispose of the contained chemicals as described in the SAP, WP or SSHASP. The Safety and Health Manager and/or Project Manager must approve the disposal method used.

7. Rinse equipment with deionized water.	After a required chemical rinse, rinse the equipment again with the DI water in the bucket marked *chemical rinse*. This DI water will need to be retained (i.e., do not dispose of this water on the site), tested, and disposed of according to federal and state requirements for the chemical used. The Safety and Health Manager and/or Project Manager must approve the disposal method used. After the rinse in the *chemical rinse* bucket, triple rinse the equipment again in the bucket marked *final rinse.*
8. Air dry equipment.	Place equipment on plastic sheeting or foil to air dry.
9. Transport/ store equipment.	Wrap equipment in foil or plastic wrap to transport or store.
10. Clean decontamination equipment.	a. Triple rinse equipment from the *gross wash* and *soap wash* (brushes and buckets) with clean tap water, preferably with pressurized water. Soap can be used on particularly dirty equipment. b. Triple rinse all decontamination equipment with DI water, including *DI rinse* and *final rinse* buckets. c. Store decontamination equipment, labeled and in a clean location so they are used only for decontamination purposes.
11. Dispose of decontamination solutions.	Storage of contained decontamination fluids as required by the SAP, QAPP, or WP or of residue from a chemical rinse should have been arranged on site prior to sampling. Once the sampling and associated decontamination is complete, sampling of the stored fluids for hazardous waste criteria will be required. If the fluids are determined to be hazardous (e.g., meet the characteristics of a hazardous waste [ignitability, corrosivity, reactivity, or toxicity] or contain listed wastes from title 40 of the Code of Federal Regulations [CFR] in part 261.4), dispose of them according to federal and state requirements. The Safety and Health Manager and/or Project Manager must approve the disposal method used. Note: when using other than the above-mentioned solutions, check with the Safety and Health Manager and the Project Manager.
12. Measure effectiveness of procedures.	Measure the effectiveness of the decontamination procedures using field equipment rinsate blanks as discussed in the SAP, QAPP, or WP.

HEALTH SAFETY SECURITY ENVIRONMENT (HSSE) CONSIDERATIONS
This section to be completed with concurrence from the Safety and Health Manager.

SOURCE	*HAZARDS*	*WHERE*	*HOW, WHEN, RESULT*	*CONTROLS*
CHEMICAL	Potential contact with contaminated items and resulting water from decontamination procedures.	Sites.	Inadvertent exposure to contaminated items and water resulting from decontamination procedures could lead to adverse health effects.	Personnel will practice proper personal hygiene (wash hands prior to eating/drinking and when leaving the site); follow decontamination procedures as described above; and wear nitrile gloves and safety glasses when handling contaminated items.
	Chemical rinse (e.g., dilute nitric acid, methanol, and hexane).	Sites.	Personnel could be exposed to chemicals via ingestion and skin/eye contact when decontaminating equipment. Exposure could cause irritation of skin/eye and adverse health effects.	Personnel will check and follow safety procedures as outlined in the chemical-specific Safety Data Sheets. Personnel will prevent skin/eye contact with chemicals and they will wear nitrile gloves and eye protection when handling chemicals. Personnel will practice proper personal hygiene (wash hands prior to eating/drinking, after decontaminating equipment, and when leaving the site). All personnel and vehicles will stand upwind when spraying equipment with chemicals. Refer to the Chemical Flushing Guidelines available inside any Pioneer vehicle's first aid kit for first-aid procedures in case of contact with chemicals.
NOISE	Not applicable.			
ELECTRICAL	Not applicable.			

HEALTH SAFETY SECURITY ENVIRONMENT (HSSE) CONSIDERATIONS
This section to be completed with concurrence from the Safety and Health Manager.

SOURCE	*HAZARDS*	*WHERE*	*HOW, WHEN, RESULT*	*CONTROLS*
BODY MECHANICS	Improper lifting.	Sites.	Back injuries and muscle/back strains could result when using improper techniques to lift and carry 5-gallon containers.	Personnel will use proper lifting techniques: get a good grip, keep the load close to the body, lift with legs and not with back, and avoid lifting loads above shoulder's height. Two people will lift awkward/heavy tools and equipment.
GRAVITY	Falls from slips and trips.	Areas designated for decontamin-ation procedures.	Slips and falls could occur while performing decontamination procedures due to slippery surfaces resulting in bruises, scrapes, or broken bones.	Personnel will wear work boots with good traction and ankle support. Personnel will also be aware of working/ walking surfaces and choose a path to avoid hazards, keep work areas as dry as possible, and wear muck boots as necessary.
WEATHER	Cold/heat stress.	Sites.	Exposure to cold climates may result in cold burns, frostbites, and hypothermia. Exposure to high temperatures may result in heat cramps, heat exhaustion, or heat stroke.	Training on signs and symptoms of cold/heat stress is required. Personnel will wear appropriate clothing when working outdoors, remain hydrated, and have sufficient caloric intakes during the day. Personnel will also follow procedures outlined in applicable SSHASP and/or Pioneer corporate HASP.
	Hypothermia/ frostbite.	Sites where air temperature is 35.6 °F (2 °C) or less.	Personnel whose clothing becomes wet during decontamination procedures may be exposed to hypothermia and/or frostbite.	Personnel will change clothing if it becomes wet.

HEALTH SAFETY SECURITY ENVIRONMENT (HSSE) CONSIDERATIONS
This section to be completed with concurrence from the Safety and Health Manager.

SOURCE	*HAZARDS*	*WHERE*	*HOW, WHEN, RESULT*	*CONTROLS*
	Lightning.	Outdoor sites.	Electrocution, injury, death, or equipment damage could be caused by lightning strike.	Personnel will follow the 30/30 rule during lightning storms.
RADIATION	Ultraviolet (UV) radiation.	Outdoors.	Personnel could be exposed to UV radiation during summer months causing sun burns, skin damage, and eye damage.	Personnel will wear safety glasses with tinted lenses, long-sleeve work shirts, and long pants. Personnel should wear sunscreen, if necessary.
BIOLOGICAL	Plants, insects, and animals.	Sites.	Exposure to plants, insects, and/or animals may cause rashes, blisters, redness, and swelling.	Training on the signs and symptoms of exposure to plants, insects, and animals is required. Personnel will avoid contact with plants, insects, and animals. First-aid kits will be available on the site. Personnel with allergies will notify their supervisor.
MECHANICAL	Not applicable.			
PRESSURE	Not applicable.			
THERMAL	Contact with hot surfaces.	Foil and decontamination equipment.	If foil and decontamination equipment are placed directly in the sun, they could get hot. Contact with hot surfaces could result in personal injury.	Personnel will not set decontamination stations directly in the sun.

HEALTH SAFETY SECURITY ENVIRONMENT (HSSE) CONSIDERATIONS This section to be completed with concurrence from the Safety and Health Manager.				
SOURCE	***HAZARDS***	***WHERE***	***HOW, WHEN, RESULT***	***CONTROLS***
HUMAN FACTORS	Inexperienced and improperly trained personnel.	Sites.	Inexperienced personnel and improper training could cause incidents resulting in injuries and/or property damage.	Personnel will be properly trained in this procedure and other applicable procedures. Personnel will implement stop work procedures, if necessary.
SIMOPS (Simultaneous Operations)	Not applicable.			

ADDITIONAL HSSE CONSIDERATIONS This section to be completed with concurrence from the Safety and Health Manager.	
REQUIRED PPE	**Personnel Protection Equipment** (PPE)**:** Safety glasses, high-visibility work shirt or vest, long pants, work boots, and nitrile gloves.
APPLICABLE SDSs	**Safety Data Sheets** (SDSs) for corresponding chemicals used during chemical rinse will be maintained based on the site characterization and contaminants. Safety Data Sheets are available to Pioneer personnel at the link below: https://pioneertechnicalservices.sharepoint.com/Safety/SafetyDataSheets
REQUIRED PERMITS/ FORMS	Per site/project requirements.
ADDITIONAL TRAINING	Per site/project requirements.

DRAWINGS, DOCUMENTS, AND TOOLS/EQUIPMENT The following documents should be referenced to assist in completing the associated task.	
DRAWINGS	
RELATED SOPs/ PROCEDURES/ WORK PLANS	

TOOLS/ EQUIPMENT	Five empty 5-gallon buckets, tap water, stiff brushes, Liquinox soap, four 5-gallon containers of DI (or distilled water if DI water is not available), chemicals for chemical rinse (if required), small plastic swimming pool/plastic sheeting or foil, tarps, and sprayers (if available). If additional items for decontamination are needed, they will be listed on the SAP.
FORMS/ CHECKLIST	

APPROVALS/CONCURRENCE

By signing this document, all parties acknowledge the completeness and applicability of this SOP for its intended purpose. Also, by signing this document, it serves as acknowledgement that I have received training on the procedure and associated competency testing.

SOP TECHNICAL AUTHOR	**DATE**
Julie Flammang **Julie Flammang**	**09/08/2020**
SAFETY AND HEALTH MANAGER	**DATE**
Tara Schleeman **Tara Schleeman**	**09/08/2020**

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ENV-SOP-MIN4-0052

QM Approval

Name/Signature	Title	Date	Meaning/Reason
Janielle Ward (007319)	Manager - Quality	11 Oct 2021, 12:29:29 PM	Approved

Management Approval

Name/Signature	Title	Date	Meaning/Reason
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Adam Haugerud (005828)	General Manager 2	02 Nov 2021, 05:15:03 PM	Approved

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TEST METHOD STANDARD OPERATING PROCEDURE
TITLE: Metals Analysis by ICP-OES
TEST METHOD 6010B, 6010C, 6010D, and 200.7
ISSUER: Pace ENV – Minneapolis – MIN4

1.0 SCOPE AND APPLICATION

This standard operating procedure (SOP) describes the laboratory procedure for the determination of dissolved and total recoverable metals by Inductively Coupled Plasma – Optical Emission Spectrometry (ICP-OES).

1.1 Target Analyte List and Limits of Quantitation (LOQ)

The target analytes and the normal LOQ that can be achieved with this procedure are provided in Table 1, Appendix A.

LOQ are established in accordance with Pace policy and SOPs for method validation and for the determination of detection limits (DL) and quantitation limits (LOQ). DL and LOQ are routinely verified and updated when needed. The current LOQ for each target analyte that can be determined by this SOP as of the effective date of this SOP is provided in Table 1, Appendix A.

The reporting limit (RL) is the value to which analytes are reported as detected or not detected in the final report. When the RL is less than the lower limit of quantitation (LLOQ), all detects and non-detects at the RL are qualitative. The LLOQ is verified daily by running a QC solution (CRDL) at the LOQ and evaluating against method specific limits.

DL, LOQ, and RL are always adjusted to account for actual amounts used and for dilution.

1.2 Applicable Matrices

This SOP is applicable to air filters, drinking water, ground water, aqueous samples, liquid samples, leachates, industrial wastes, soils, sludges, sediments, and other solid wastes.

2.0 SUMMARY OF METHOD

Prior to analysis, samples are solubilized or digested using appropriate sample preparation methods. This method describes the determination of elements by ICP-OES. The method measures element-emitted light by optical spectrometry. Samples are nebulized and the resulting aerosol is transported to the plasma torch. Element-specific atomic-line emission spectra are produced by a radio-frequency inductively coupled plasma. The spectra are dispersed by a grating spectrometer, and the intensities of the lines are monitored by a charge coupled device detector (CCD). All data is collected by simultaneous measurement. Software is used to measure and apply corrections due to background or inter-element interferences using a variety of techniques. Alternate wavelengths are also monitored for confirmation or to use in correction equations.

3.0 INTERFERENCES

3.1 Spectral Interferences are caused by background emission from continuous or recombination phenomena, stray light from the line emission of high concentration elements, overlap of a spectral line from another element, or unresolved overlap of molecular band spectra.

3.2 Spectral overlap can be compensated by computer-correcting the raw data after monitoring and measuring the interfering element. Unresolved overlap requires selection of an alternate

wavelength. Background contribution and stray light can usually be compensated for by a background correction adjacent to the analyte line.

3.3 Physical Interferences are effects associated with the sample nebulization and transport processes. Changes in viscosity and surface tension can cause significant inaccuracies, especially in samples containing high dissolved solids or high acid concentrations. A high solids nebulizer is used on all instruments. Internal standards are also used to monitor and correct for physical effects.

3.4 Chemical interferences include molecular compound formation, ionization effects and solute vaporization effects. Normally, these effects are not significant with the ICP technique, but if observed, can be minimized by careful selection of operating conditions, use of an ionization buffer, or by matrix matching of standards and samples.

3.5 Memory interferences result when analytes in a previous sample contribute to the signals measured in the new sample. Memory effects can result from sample deposition on the uptake tubing to the nebulizer and from buildup of sample material in the plasma torch and spray chamber. Regular maintenance and awareness of samples with high concentrations minimize these interferences.

4.0 DEFINITIONS

Refer to the Laboratory Quality Manual for a glossary of common lab terms and definitions.

5.0 HEALTH AND SAFETY

The toxicity or carcinogenicity of each chemical material used in the laboratory has not been fully established. Each chemical should be regarded as a potential health hazard and exposure to these compounds should be as low as reasonably achievable.

The laboratory maintains documentation of hazard assessments and OSHA regulations regarding the safe handling of the chemicals specified in each method. Safety data sheets for all hazardous chemicals are available to all personnel. Employees must abide by the health, safety and environmental (HSE) policies and procedures specified in this SOP and in the Pace Chemical Hygiene / Safety Manual.

Personal protective equipment (PPE) such as safety glasses, gloves, and a laboratory coat must be worn in designated areas and while handling samples and chemical materials to protect against physical contact with samples that contain potentially hazardous chemicals and exposure to chemical materials used in the procedure.

Concentrated corrosives present additional hazards and are damaging to skin and mucus membranes. Use these acids in a fume hood whenever possible with additional PPE designed for handing these materials. If eye or skin contact occurs, flush with large volumes of water. When working with acids, always add acid to water to prevent violent reactions. Any processes that emit large volumes of solvents (evaporation/concentration processes) must be in a hood or apparatus that prevents employee exposure.

Contact your supervisor or local HSE coordinator with questions or concerns regarding safety protocol or safe handling procedures for this procedure.

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6.0 SAMPLE COLLECTION, PRESERVATION, HOLDING TIME, AND STORAGE

Samples should be collected in accordance with a sampling plan and procedures appropriate to achieve the regulatory, scientific, and data quality objectives for the project.

The laboratory does not perform sample collection or field measurements for this test method. To assure sample collection and field checks and treatment are performed in accordance with applicable regulations Pace project managers will inform the client of these requirements at the time of request for analytical services when the request for testing is received prior to sample collection. If samples were already collected, the laboratory will record any nonconformance to these requirements in the laboratory's sample receipt record when sufficient information about sample collection is provided with the samples.

General Requirements

Matrix	Routine Container	Minimum Sample Amount[1]	Preservation	Holding Time
Aqueous	250 mL Plastic	25 mL	Acidified[2] with nitric acid to pH<2, stored ambient	Must be analyzed within 180 days of collection.
Solid	8 oz glass jar	1 gram	<6°C, but above freezing	

[1] *Minimum amount needed for each discrete analysis.*
[2] *Samples must equilibrate for a minimum of 24 hours if acidification is performed in the lab.*

Thermal preservation is checked and recorded on receipt in the laboratory in accordance with laboratory ENV-SOP-MIN4-0008 *Sample Management* (current or equivalent replacement). Chemical preservation is checked and recorded at time of receipt or prior to sample preparation.

After receipt, samples are stored either at ambient or 6°C until sample preparation. Prepared sample digestates are stored at ambient temperatures until sample analysis.

After analysis, unless otherwise specified in the analytical services contract, samples are retained for 21 days from date of final report and then disposed of in accordance with Federal, State, and Local regulations.

7.0 EQUIPMENT AND SUPPLIES

7.1 Equipment

Equipment	Description
ICPOES (Inductively Coupled Plasma Optical Emiison Spectrometer)	Agilent 5100 or5110 ICP instrumentation equipped with an CCD Detector, full wavelength region. Each instrument has an associated auto-sampler and recirculating chiller.
Centrifuge	Thermo Sorvall Legend XT
Analytical Balance	Sartoriius or equivalent, capable of weighing to 0.01g
Mechanical pipettors	Eppendorf, Fisher brand or equivalent replacement, various sizes
Glassware	Class A or B volumetric flasks and graduated cylinders of various sizes

7.2 Supplies

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Supply	Description
Argon gas	Praxair or equivalent, High purity grade, 99.99%
Filters	Filtermate filters, 2 um PTFE, Environmental Express, SC0408
Auto-sampler tubes	Moldpro or equivalent, 15 mL metals free auto-sampler tubes
Digestion cups	Moldpro or equivalent, 50 mL disposable digestion cups
Data-Uploading Software	Pace internal software used to transfer data from the instrument to the LIMS

8.0 REAGENTS AND STANDARDS

8.1 Reagents

Reagent	Description
Reagent water	ASTM Type I – 18 megaohm
Nitric Acid (HNO_3), trace metals grade	Fisher Scientific, A-509-P212 or equivalent
Hydrochloric acid (HCl),trace metals grade	Fisher Scientific, A-508-P212 or equivalent
4% (v/v) Nitric Acid/5% (v/v) Hydrochloric Acid Solution	400 mL nitric acid (above) + 500 mL hydrochloric acid (above) to 10 liters with ASTM Type I water (18 megaohm). Used for all blanks and rinsing and preparation of standards.

8.2 Standards

Reagent	Description
Calibration Stock Standards	Custom blend of elements. See Appendix D for the standard information
Initial Calibration Verification (ICV) Stock Standard solutions	Custom blend. Must be separate stock from the calibration standards. Spex Certiprep or equivalent. See Appendix D for the standard information
Wavelength Cal Solution	Various analytes, prepared in the lab
Internal Standards	Yttrium, Agilent or equivalent

9.0 PROCEDURE

9.1 Equipment Preparation

9.1.1 **Pre-Start Checks:** Turn on the computer and load the software. Initiate appropriate operating configuration of the instrument's computer according to the instrument manufacturer's instructions. Check the following:

9.1.1.1 Verify the level of nebulizer waste and rinse waste, if more than half full, empty it into the acid waste stream

9.1.1.2 Ar/O pressure - The argon supply pressure should be set at about 80-100psi. If the supply argon pressure falls below about 80psi, a safety interlock automatically shuts off the torch.

9.1.1.3 Wash solution level - The wash solution supply is maintained in a 4-liter carboy. Ensure that there is sufficient volume present for the analytical sequence.

9.1.1.4 Peristaltic pump tubing - Change the sample and internal standard tubing, spray chamber drain tubing and the rinse station tubing as needed. Signs of degradation include flattened sections and hazy appearance. Allow at least 30 minutes for break-in period

9.1.1.4.1 Adjust the pump-tubing in such a way to ensure proper flow prior to igniting the plasma. Decrease flow to where flow of bubble actually stops or barely moves. Turn knob 2 full turns.

9.1.1.5 Ignite plasma while tubing is in a rinse solution, allow plasma to warm up at least 30 minutes and preferably 60-90 minutes.

9.1.1.6 Use the warm up time to create the sequence and pour samples. Use Horizon Uploader to copy labels into the sequence.

9.1.2 Support Equipment

Chiller temperature, pressure and water level - The temperature should be regulated at 20 ± 2ºC. Check the current temperature on the chiller to ensure it is within this range. Check the inlet cooling water pressure that must be between 45 and 55psi. Check to ensure that chiller water level is full. If it is not, fill with Polyclear 30.

9.1.3 Instrument

9.1.3.1 Routine Instrument Operating Conditions

Instrument operating conditions vary by method and by instrument. All conditions are documented with each worksheet and cannot be modified after data has been generated. Instrument conditions are stored within a worksheet template. The analyst selects the appropriate Template for analysis. The analyst does not change operating conditions. Conditions are only changed during method development.

9.2 Initial Calibration

9.2.1 Calibration Design

9.2.1.1 A calibration curve consists of a single point standard and a calibration blank.

9.2.1.2 Additional calibration procedures (where applicable) can be found in ENV-POL-CORQ-0005 *Acceptable Calibration Practices for Instrument Testing* (current or equivalent replacement).

9.2.2 Calibration Sequence

Example Analytical Sequence

CAL0
CAL1
ICV
ICB
CRDLA
ICSA
ICAB

Fe 2000 SIC
Ca 2000 SIC/LDR
Al 1000 SIC/LDR
Mg 1000 SIC/LDR
Cu 50 SICLDR
Mn 100 SIC
Ba 20 SIC/LDR
Cr 50 SIC/LDR
Co 50 SIC/LDR
CCV
CCB
V 20 SIC/LDR
Ni 50 SIC/LDR
Ti 20 SIC/LDR
Mo 10 SIC/LDR
Zr 20 SIC
Ce 10 SIC
U 20 SIC
Cd 20 SIC
Sn 20 SIC
La 20 SIC
CCV
CCB
LDR A
LDR B
LDR C
CCV
CCB
CLIENT SAMPLES
CCV
CCB

9.2.3 ICAL Evaluation

9.2.3.1 Curve Fit

With a single point calibration model, a linear regression curve is established using a calibration blank and one non-zero standard with internal standard correction referencing Yttrium.

9.2.3.2 Relative Standard Error (RSE)

With a single point calibration model using a calibration blank and one non-zero standard, relative standard error evaluation is not applicable.

9.2.3.3 Initial Calibration Verification

In addition to meeting the linearity requirement, any new calibration curve must be assessed for accuracy in the values generated. To assess the accuracy, a

single standard from a secondary source must be analyzed and the results obtained must be compared to the known value of the standard. This step is referred to as Initial Calibration Verification. The ICV, followed by an ICB, is analyzed immediately following an initial calibration curve.

9.2.4 Continuing Calibration Verification

A CCV followed immediately by a CCB must be analyzed after every 10 samples and at the end of the analytical batch to verify the system is still calibrated.

9.3 Sample Preparation

9.3.1 Label all sample tubes so that each sample can be uniquely identified on the rack.

9.3.2 If any samples in a batch need to be filtered because of suspended material, use an Environmental Express Filtermate. The Method Blank and LCS must also be filtered if any samples are. Record the ID of the Filtermates used.

9.3.3 Centrifuge soil samples to minimize need for filtering.

9.3.4 Aqueous samples are poured without initial dilution unless historical data demonstrates otherwise.

9.3.5 Use Horizon Uploader to copy labels into the sequence.

10.0 Data Analysis And Calculations

10.1 Quantitative Identification

10.1.1 Monitor all initial QC checks. One re-analysis of QC checks is allowed. If initial QC fails twice, make instrument modifications and recalibrate using a new worksheet from template.

10.1.2 During the sample analysis or after the analysis is completed, transfer valid data into LIMS system using LIMS LINK.

10.1.2.1 Export data from instrument to CSV file.

10.1.2.2 Open LIMSLINK

10.1.2.3 Click open instrument, select CSV file from list, data will import

10.1.2.4 Highlight QC + samples, select “Get LIMS Info”

10.1.2.5 Run QC will prompt for Q-Batch # plus standard selection

10.1.2.6 Sample data will prompt for SD/PDS source sample.

10.1.2.7 Right click on samples to select/de-select elements

10.1.2.8 Highlight samples to upload and select “Export Run to Epic Pro”.

Note: Be sure to make the appropriate selections in LIMSLNK rather than post-editing in EPIC. This provides for a much smoother experience and minimizes chance for error. If edits must be done in EPIC be sure to make edits prior to uploading new data from LIMSLINK, as this, again minimizes error due to confusion.

10.1.3 When Complete, select "excel bench sheet". Save the Excel Bench sheet to the instrument folder marked "LIMSLINK RAW DATA" Use convention of run date (e.g. 032917ICP5). Note discrepancies in the notes section of the run log (including dilutions, QC issues, re-runs, etc.).

10.1.4 In LIMS system make final adjustments and add any required footnotes. Complete checklist and turn data in for validation.

10.1.5 Documentation is a mix of electronic and paper files. Key data must be stored electronically so that data review may be performed from any location. Some documents are stored in the physical daily folder and archived for easy reference.

10.1.6 Label a physical file with the date. Record the file name, Q-Batch, and all prep batches on the folder for each run that day (example: 032917ICP5 and 032917ICP5B.

10.1.7 Store printed copies of batch worklist reports, the original checklist, a printed copy of the IEC Form 10-IN generated from Gandolf, and a printed copy of the run log from LIMSLINK file in this folder. If the data reviewer requests additional printed information they may print it themselves. Note, if data is validated remotely print a copy of the validation verification e-mail and include with each checklist.

10.1.8 Generate a copy of the raw data and print to the X:Drive.

10.2 Calculations

See the laboratory SOP ENV-SOP-MIN4-0171 *Laboratory Calculations* (current or equivalent replacement) for equations for common calculations.

10.2.1 Inter-element Correction Factor (IEC) = Concentration of apparent concentration (observed) in mg/L / Concentration of Interferent in mg/L.

10.2.2 The percent recovery of the spike is calculated from the following equation:

$$\%\ \text{Recovery} = \frac{(SSR-SR)\ X\ 100}{ST}$$

Where: SSR = Spiked Sample Result, ug/L or mg/kg dry
SR = Sample Result, ug/L or mg/kg dry
ST = Spike Target, ug/L or mg/kg dry

10.2.3 The relative percent difference between the MS/MSD can be calculated as follows

$$RPD = \frac{|(S-D)|\ X\ (100)}{(S+D)/2}$$

Where: RPD = Relative Percent Difference
S = Original Spiked Sample Value, ug/L or mg/kg dry
D = Second Spiked Sample Value, ug/L or mg/kg dry

10.2.4 The corrected dry weight concentration can be calculated using the following:

$$corrected\ dry\ wt\ conc = \frac{\left(c \times \frac{v_f}{wt_i}\right)}{\%\ dry\ wt}$$

Where, c = concentration on instrument, µg/L
v_f = final volume, L
wt_i = initial weight, g

$$\%Dry\ weight = \frac{Sample\ Dry\ Weight}{Sample\ Wet\ Weight} \text{x}100$$

11.0 QUALITY CONTROL AND METHOD PERFORMANCE

11.1 Quality Control

The following QC samples are prepared and analyzed with each batch of samples. Refer to Appendix B for acceptance criteria and required corrective action.

QC Item	Frequency
Method Blank (MB)	1 per batch of 20 or fewer samples.
Laboratory Control Sample (LCS)	1 per batch of 20 or fewer samples.
Laboratory Control Sample Duplicate (LCSD)	As needed
Matrix Spike (MS)	1 per batch of 20 or fewer samples for 6010B/C/D. 1 per batch of 10 or fewer samples for 200.7
Matrix Spike Duplicate (MSD)	1 per batch of 20 or fewer samples.
Sample Duplicate	Performed at client request.
Serial Dilution	1 per batch of 20 or fewer samples for 6010B/C/D.
Post Digestion Spike	1 per batch of 20 or fewer samples for method 6010B/C/D.

11.2 Instrument QC

The following Instrument QC checks are performed. Refer to Appendix B for acceptance criteria and required corrective action.

QC Item	Frequency
Initial Calibration	Daily
Initial Calibration Verification (ICV)	Immediately after each initial calibration.
Spectral Interference Check Solutions (SIC)	Immediately after initial ICSA / ICSAB
Initial Calibration Blank	Immediately after each ICV.
Continuing Calibration Verification (CCV)	Prior to the analysis of any samples and after every 10 injections thereafter. Samples must be bracketed with a closing CCV standard.
Continuing Calibration Blank	Following every CCV injection

CRDL / LLCCV verification	At the beginning of each run for 6010B/C/D/200.7 and at a minimum of once at the end of each run for 6010C.
ICSA verification	At the beginning of each sample run sequence after the CRDL.
ICSAB verification	This is analyzed following the ICSA when requested. This is required by certain clients. It is not a method requirement and need be analyzed only for clients specifying this in the QAPP.
Internal Standard	An appropriate internal standard is required.

11.3 Method Performance

11.3.1 Method Validation

11.3.1.1 Detection Limits

Detection limits (DL) and limits of quantitation (LOQ) are established at initial method setup and verified on an on-going basis thereafter. Refer to Pace ENV corporate SOP ENV-SOP-CORQ-0011 *Method Validation and Instrument Verification* (current or equivalent replacement) and to the laboratory's SOP ENV-SOP-MIN4-0163 *Determination of LOD and LOQ* (current or equivalent replacement) for these procedures.

11.3.2 Linear Dynamic Range (LDR)

Method 6010D requires that a LDR check sample be analyzed daily. Because of this requirement for 6010D, the LDR is established daily for all methods. For some elements a single element standard is used to establish the LDR while in other cases a mixed standard is used to establish the LDR. If an LDR standard is not analyzed for a particular analyte then the LDR defaults to the highest calibration point in the calibration curve. Data is reported up to 90% of the LDR. When evaluating interferences use values up to the full LDR for the interferent. The LDR may be established at higher or lower levels on a daily basis based on expected levels of samples being tested that day. The LDR may vary daily depending on slight changes in instrument performance (things like pump tubing wear, etc.). Refer to Appendix C : Linear Range Reference Table for default ranges and the typical standards used to establish them.

11.3.3 Wavelength Calibration

The recommended minimum frequency is once per month. To ensure this, a wavelength calibration and detector calibration are both performed each time the torch is changed. For the 5100 and 5110 this is every 2-3 weeks. This is documented in the respective daily maintenance logs. We make the tuning solution and document in the Standards log in the LIMS. The number is also recorded in LIMSLINK. Making the tuning solution from single stocks is a significant cost savings over purchasing the tuning solution from Agilent.

11.3.3.1 Agilent 5100 and 5110:

11.3.3.1.1 Ensure the polyboost has been on for at least 30 minutes.

11.3.3.1.2 Go to the Instrument Page. Select Calibration.

11.3.3.1.3 With the Plasma off, click detector calibration. This will complete and update the date / time. It is automatically stored.

11.3.3.1.4 Ignite the plasma and allow for 30 minute warmup. Ensure snout purge is on; this is the default in the ignition sequence.

11.3.3.1.5 Introduce the tuning solution. Click Calibrate.

11.3.3.1.6 There will be a list of analytes with red indicating failing and green indicating passing.

11.3.3.1.7 If any fail, repeat 2 more times until all are green. Wait another 30 minutes if the polyboost was just turned on 30 minutes ago, before the final attempt.

11.3.3.1.8 If after 3 attempts all are red, then a service call is required.

11.3.3.1.9 Click the axial box and repeat steps 4-7.

11.3.3.1.10 If there are failures in either radial or axial mode only then this indicates the source of the problem.

11.4 Analyst Qualifications and Training

Employees that perform any step of this procedure must have a completed Read and Acknowledgment Statement for this version of the SOP in their training record. In addition, prior to unsupervised (independent) work on any client sample, analysts that prepare or analyze samples must have successful initial demonstration of capability (IDOC) and must successfully demonstrate on-going proficiency on an annual basis. Successful means the initial and on-going DOC met criteria, documentation of the DOC is complete, and the DOC record is in the employee's training file. Refer to laboratory SOP ENV-SOP-MIN4-0165 *Orientation and Training Procedures* (current or equivalent replacement) for more information.

12.0 Data Review And Corrective Action

12.1 Data Review

Pace's data review process includes a series of checks performed at different stages of the analytical process by different people to ensure that SOPs were followed, the analytical record is complete and properly documented, proper corrective actions were taken for QC failure and other nonconformance(s), and that test results are reported with proper qualification.

The review steps and checks that occur as employee's complete tasks and review their own work is called primary review.

All data and results are also reviewed by an experienced peer or supervisor. Secondary review is performed to verify SOPs were followed, that calibration, instrument performance, and QC criteria were met and/or proper corrective actions were taken, qualitative ID and quantitative measurement is accurate, all manual integrations are justified and documented in accordance with the Pace ENV's SOP for manual integration, calculations are correct, the analytical record is complete and traceable, and that results are properly qualified.

A third-level review, called a completeness check, is performed by reporting or project management staff to verify the data report is not missing information and project specifications were met.

Refer to laboratory SOP ENV-SOP-MIN4-0092 *Data Review Process* (or equivalent replacement) for specific instructions and requirements for each step of the data review process.

12.2 Corrective Action

Corrective action is expected any time QC or sample results are not within acceptance criteria. If corrective action is not taken or was not successful, the decision/outcome must be documented in the analytical record. The primary analyst has primary responsibility for taking corrective action when QA/QC criteria are not met. Secondary data reviewers must verify that appropriate action was taken and/or that results reported with QC failure are properly qualified.

Corrective action is also required when carryover is suspected and when results are over range.

Samples analyzed after a high concentration sample must be checked for carryover and reanalyzed if carryover is suspected. Carryover is usually indicated by low concentration detects of the analyte in successive samples analyzed after the high concentration sample.

Sample results at concentrations above the upper limit of quantitation must be diluted and reanalyzed. The result in the diluted samples should be within the upper half of the calibration range. Results less than the mid-range of the calibration indicate the sample was over diluted and analysis should be repeated with a lower level of dilution. If dilution is not performed, any result reported above the upper range is considered a qualitative measurement and must be qualified as an estimated value.

Refer to Appendix B for a complete summary of QC, acceptance criteria, and recommended corrective actions for QC associated with this test method.

13.0 Pollution Prevention And Waste Management

Pace proactively seeks ways to minimize waste generated during our work processes. Some examples of pollution prevention include but are not limited to: reduced solvent extraction, solvent capture, use of reusable containers for solvent management, and real-time purchasing.

The EPA requires that laboratory waste management practice to be conducted consistent with all applicable federal and state laws and regulations. Excess reagents, samples and method process wastes must be characterized and disposed of in an acceptable manner in accordance with Pace's Chemical Hygiene Plan / Safety Manual.

14.0 Modifications

A modification is a change to a reference test method made by the laboratory. For example, changes in stoichiometry, technology, quantitation ions, reagent or solvent volumes, reducing digestion or extraction times, instrument runtimes, etc. are all examples of modifications. Refer to Pace ENV corporate SOP ENV-SOP-CORQ-0011 *Method Validation and Instrument Verification* (current or equivalent replacement) for the conditions under which the procedures in test method SOPs may be modified and for the procedure and document requirements.

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15.0 RESPONSIBILITIES

Pace ENV employees that perform any part this procedure in their work activities must have a signed Read and Acknowledgement Statement in their training file for this version of the SOP. The employee is responsible for following the procedures in this SOP and handling temporary departures from this SOP in accordance with Pace's policy for temporary departure.

Pace supervisors/managers are responsible for training employees on the procedures in this SOP and monitoring the implementation of this SOP in their work area.

16.0 ATTACHMENTS

Appendix A – Target Analyte List and Routine LOQ

Appendix B – QC Summary

Appendix C – Linear Range Reference Table

Appendix D – Standard Reference Table

Appendix E – Interference Check Standard Reference Table

17.0 REFERENCES

Pace Quality Assurance Manual- most current version.

TNI Standard, Management and Technical Requirements for Laboratories Performing Environmental Analyses, EL-V1-2009.

TNI Standard, Management and Technical Requirements for Laboratories Performing Environmental Analyses, EL-VI-2016-Rev.2.1.

Test Methods for Evaluating Water and Solid Waste, SW-846 3rd Edition, Final Update III, Revision 2, December 1996. Method 6010B.

Test Methods for Evaluating Water and Solid Waste, SW-846, Update IV, Feb. 2007. Method 6010C.

Test Methods for Evaluating Water and Solid Waste, SW-846, Update V, July 2018. Method 6010D.

Method 200.7 Revision 4.4, Determination of Metals and Trace Elements in Water and Wastes by Inductively Coupled Plasma-Atomic Emission Spectrometry, 1994.

US EPA Contract Laboratory Program Statement of Work ILM05.3, March 2004.

40 CFR Appendix B to Part 136, Definition and Procedure for the Determination of the Method Detection Limit – Rev 2, August 28, 2017.

18.0 REVISION HISTORY

This Version:

Section	Description of Change

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TEST METHOD STANDARD OPERATING PROCEDURE
TITLE: Metals Analysis by ICP-OES
TEST METHOD 6010B, 6010C, 6010D, and 200.7
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7.1	Removed reference to Agilent 720 and added reference to 5100
8.2	Removed Agilent references from table
9.1.2	Updated temperature and water pressure requirements
9.2.2	Updated calibration sequence to current sequence
11.2	Updated Spectral Interference Check Solutions (SIC) frequency information to ICSA/ICSAB
11.3.3	Added references to 5100
11.3.3.2	Removed Agilent 700Series information
Appendix A	Updated Iron, Manganese, and Zinc Soil PRL limits
Appendix C	Updated title, Ba wavelength, Cu type to SIC.LDR, Si LDR to 50, standard to LDRC, Ti Standard to Ti 20 SIC/LDR and type to SIC/LDR.
Appendix D	Updated title, updated all aliquots and final volume and stock concentrations and final concentrations as needed
Appendix E	Updated title, Aliquot volumes in Al, Ca, Fe, and Mg to 10

This document supersedes the following document(s):

Document Number	Title	Version
ENV-SOP-MIN4-0052	Metals Analysis by ICP – Method 6010 and 200.7	06

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Appendix A: Target Analyte List and Routine LOQ

Table 1: Routine Analyte List and Limits of Quantitation (LOQ)[1]

Element	Water PRL (ug/L)	Soil PRL (mg/kg)
Aluminum	200	10
Antimony	20	1.0
Arsenic	20	1.0
Barium	10	0.50
Beryllium	5.0	0.25
Boron	150	7.5
Cadmium	3.0	0.15
Calcium	500	25
Chromium	10	0.50
Cobalt	10	0.50
Copper	10	0.50
Iron	50	5
Lead	10	0.5
Magnesium	500	25
Manganese	5.0	0.5
Molybdenum	15	0.75
Nickel	20	1.0
Phosphorus	20	5
Potassium	2500	125
Selenium	20	1.0
Silicon	50	5
Silver	10	0.50
Sodium	1000	50
Strontium	5.0	0.5
Sulfur	500	25
Thallium	20	1.0
Tin	75	3.75
Titanium	25	1.25
Uranium	50	2.5
Vanadium	15	0.75
Zinc	20	2.0
Hardness	3300	N/A

[1] Values in place as of effective date of this SOP. LOQ are subject to change. For the most up to date LOQ, refer to the LIMS or contact the laboratory.

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Appendix B: QC Summary

QC Item	Frequency	Acceptance Criteria	Corrective Action	Qualification
ICAL	Daily	A calibration curve must consist of a blank and at least one calibration standard.	Identify and correct source of problem, repeat.	None. Do not proceed with analysis.
ICV	After Each ICAL	± 10% for method 6010B, 6010C and 6010D or ± 5% for method 200.7 The RSD of the standards must be below 5% for 6010B, 6010C and 6010D and below 3% for 200.7.	Identify source of problem, re-analyze. If repeat failure, repeat ICAL. Analysis may proceed if it can be demonstrated that the ICV exceedance has no impact on analytical measurements. For example, the ICV %R is high, CCV is within criteria, and the analyte is not detected in sample(s).	Qualify analytes with ICV out of criteria.
ICB	Immediately after the initial calibration verification	All elements of interest must be evaluated to a criteria of +/- ½ of the RL for method 6010D. All elements of interest must be evaluated to +/- the RL for method 6010B,6010C and 200.7. Criteria to be evaluated to method criteria unless otherwise specified by client.	Identify source of problem, re-analyze. Analysis may proceed if it can be demonstrated that the ICB exceedance has no impact on analytical measurements. For example, the ICB has detections and the analyte is not detected in sample(s).	Qualify analytes with ICB out of criteria.
CRDLA / LLCCV	The CRDLA must be analyzed at the beginning of each run for every analyte of interest. The CRDLA is analyzed at or below the RL. Additionally, the CRDLA must be analyzed after samples to bracket method 6010C samples.	± 40% (or specified by the client) For method 6010C, must be within ± 30% . For method 6010D, must be within.± 20%.	Identify source of problem, re-analyze. Analysis may proceed if it can be demonstrated that the CRDL exceedance has no impact on analytical measurements. For example, the CRDL %R is high and the analyte is not detected in sample(s). For example, the CRDL %R is high and the analyte detections exceed the continuing calibrations verification level (midpoint of the curve). If the CRDL is biased low, no data can be reported for the target elements failing criteria.	Qualify outages and explain in case narrative.
CCV	Daily, before sample analysis, after every 10, and at end of analytical window.	For method 6010B, 6010C, 6010D and 200.7, the CCV must be within ± 10% of the true value. The RSD of the CCV must be below 5% for 6010B.	Identify source of problem, re-analyze. Analysis may proceed if it can be demonstrated that the CCV exceedance has no impact on analytical measurements.	Qualify analytes with CCV out of criteria.

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QC Item	Frequency	Acceptance Criteria	Corrective Action	Qualification
			For example, the CCV %R is high, and the analyte is not detected in sample(s).	
CCB	Daily, before sample analysis, after every 10, and at end of analytical window	All elements of interest must be evaluated to a criteria of +/- the RL for 200.7, 6010B, 6010C and 6010D. Depending on the data quality objective of individual clients different criteria may apply.	Identify source of problem, re-analyze. Analysis may proceed if it can be demonstrated that the CCB exceedance has no impact on analytical measurements. For example, the CCB has detections and the analyte is not detected in sample(s).	Qualify analytes with CCB out of criteria.
Internal Standards	Every field sample, standard and QC sample	70-125% of its true concentration	Troubleshoot instrument performance. Reanalyze samples and dilute if needed.	Qualify outages and explain in case narrative.
Interference check solution (ICSA)	A mixed solution containing concentrations of Al, Ca, and Mg at 500 PPM and Fe at 200 PPM is analyzed at the beginning of each sample run sequence. In some specific client requirements the ICSA must bracket the run or the analytical batch.	Acceptance criteria for the spiked analytes are 80-120%. Unspiked analytes must have an absolute value less than the RL.	Identify and correct source of problem, repeat performance verification(s). Note: The ICSA can be re-processed after appropriate SIC solutions are analyzed and the IECs are recalculated. If ICSA passes, continue.	None. Do not proceed with analysis for elements that cannot be verified.
Interference check solution (ICSAB)	A solution containing concentrations of Al, Ca, and Mg at 500 PPM and Fe at 200 PPM with low to mid-range concentrations of target analytes as outlined in ILM5.3. This is analyzed following the ICSA when requested. This is required by certain clients. It is not a method requirement and need be analyzed only for clients specifying this in the QAPP	The acceptance criteria are 80-120% for all spiked analytes.	Identify and correct source of problem, repeat performance verification(s). Note: The ICSAB can be re-processed after appropriate SIC solutions are analyzed and the IECs are recalculated. If ICSAB passes, continue.	None. Do not proceed with analysis for elements that cannot be verified.
Spectral Interference Check Solutions (SIC)	SIC solutions are single-element solutions used to evaluate and correct IEC factors. Specific elements evaluated	Unspiked analytes must have an absolute value less than the RL.	If SIC fails, re-calculate IEC and re-process data. If a sample level exceeds an SIC level and the interfering element affects target analytes, then: a)	None. Do not proceed with analysis for elements that cannot be verified.

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QC Item	Frequency	Acceptance Criteria	Corrective Action	Qualification
	are listed in specific instrument methods.		run a higher SIC or b) dilute the sample.	
Method Blank	One per 20 samples	Method 200.7: The method blank is considered to be acceptable if it does not contain the target analytes that exceed 1/2 LLOQ or project-specific DQOs. Method 6010B, 6010C and 6010D: The method blank is considered to be acceptable if it does not contain the target analytes that exceed the LLOQ or project-specific DQOs. WIDNR and West Virginia require samples to be reported to the MDL. The blanks must be clean to the data quality objectives.	Identify source of problem, re-analyze. If reanalysis of the MB fails, all samples affected by the failing MB elements need to be re-digested and re-analyzed. If the method blank exceeds the criteria, but the associated samples are either below the reporting level or other DQOs, or detections in the sample are >10x MB detections then the sample data may be reported. J-flag qualification will be applied for blank detections between the LOQ and LOD when DQOs require evaluation to the MDL.	Qualify outages and explain in case narrative.
LCS	One per 20 samples	80-120% for 6010B,6010C and 6010D 85-115% for 200.7	Identify source of problem, re-analyze. If reanalysis of the LCS fails, all samples affected by the failing LCS elements need to be re-digested and re-analyzed. If LCS recovery is > QC limits and these compounds are non-detect in the associated samples	Qualify analytes with LCS out of criteria.
LCSD	An LCSD must be substituted in the event of insufficient sample volume for a matrix spike duplicate sample.	80-120% for 6010B,6010C and 6010D 85-115% for 200.7 %Diff ≤ 20%	Identify source of problem, re-analyze. If reanalysis of the LCS fails, all samples affected by the failing LCS elements need to be re-digested and re-analyzed. If LCS recovery is > QC limits and these compounds are non-detect in the associated samples	Qualify analytes with LCS out of criteria.
MS/MSD	One per 20 samples for 6010 / 6010C / 6010D One per 10 samples for 200.7	75-125% for 6010B, 6010C, and 6010D 70-130% for 200.7 % RPD: 20%	Perform a SD and PDS on any elements that fail to meet criteria for method 6010(C)(D).	Qualify analytes with MS out of criteria.
Sample Duplicate	Per client request	%Diff ≤ 20%	Qualify outages	Qualify outages.
Serial Dilution	One SD per batch. Method suggestion / Pace Policy, if reporting by 6010B, 6010C, or 6010D.	6010B/C: 1:5 dilution of sample, SD RPD should agree within +/- 10% of the original result when the original sample is greater than 10x the RL.	Data is qualified.	Qualify outages.

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QC Item	Frequency	Acceptance Criteria	Corrective Action	Qualification
		6010D: 1:5 Dilution of sample or MS, for concentrations 25x > LLOQ in parent sample, resultant RPD should agree within +/- 20%.		
Post Digestion Spike	Method suggestion / Pace policy if reporting by 6010B, 6010C, 6010D and MS/MSD fail outside 75-125%	80-120% for 6010C 75-125% for 6010B and 6010D.	Data is qualified.	Qualify outages.
Laboratory Filter Blank (FB)	Analyzed only with batches of lab filtered dissolved metals, one per batch of 20 or less.	All elements of interest must be evaluated to a criteria of +/- ½ the RL for method 6010D. All elements of interest must be evaluated to a criteria of +/- the RL for method 6010B,6010C and 200.7. If the FB does not contain target analytes at a level that interferes with project-specific DQOs, then the FB would be considered acceptable.	Identify source of problem, re-analyze. If reanalysis of the MB fails, all samples affected by the failing MB elements need to be re-digested and re-analyzed. If sample(s) non-detect, report the data. If sample result >10x MB detections, report the data.	Qualify outages and explain in case narrative.
Linear Dynamic Range	If a SIC/LDR standard is not analyzed for any specific element, the highest standard in the calibration becomes the linear range. See Appendix C.	The standard must recover within 10% of the true value, and if successful, establishes the linear range. In each scenario, the data reporting range is established using 90% of the highest calibration level or LDR sample.	The linear range of the instrument must be adjusted until 90% recovery of the reference standard can be achieved.	N/A

Note: In the absence of method specified recovery limits, results will be evaluated based on specifications outlined by the MPCA guidelines for Inorganic Analysis.

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Appendix C: Linear Range Reference Table

Wavelength	LDR (PPM)	Standard	Type
Ag 328	2	CAL1	LDR
Al 237	1000	Al 1000 SIC/LDR	SIC/LDR
As 188	20	LDR B	LDR
B 249	20	LDR A	LDR
Ba 233	20	Ba 20 SIC/LDR	SIC/LDR
Be 234	4	CAL1	LDR
Ca 370	2000	Ca 2000 SIC/LDR	SIC/LDR
Cd 214	20	LDR B	LDR
Co 228	50	Co 50 SIC/LDR	SIC/LDR
Cr 267	50	Cr SIC/LDR	50
Cu 327	50	Cu 50 SIC/LDR	SIC/LDR
Fe 261	200	LDR C	LDR
Fe 273*	2000	Fe 2000 SIC	SIC
K 766****	200	LDR C	LDR
Li 670	4	CAL1	LDR
Mg 383	1000	Mg 1000 SIC/LDR	SIC/LDR
Mn 257	20	LDR B	LDR
Mn 293*	100	Mn 100 SIC	SIC
Mo 204	10	Mo 10 SIC/LDR	SIC/LDR
Na 589***	200	LDR C	LDR
Ni 231	50	Ni 50 SICLDR	SIC/LDR
P 213	20	LDR B	LDR
Pb 220	100	LDR A	LDR
S 181	200	LDR C	LDR
Sb 206	20	LDR A	LDR
Se 196	20	LDR B	LDR
Si 251	50	LDR C	LDR
Sn 189	20	LDR A	LDR
Sr 421	4	CAL1	LDR
Ti 334	20	Ti 20 SIC/LDR	SIC/LDR
Tl 190	20	LDR B	LDR
U	4	CAL1	LDR
V 292	20	V 20 SIC/LDR	SIC/LDR
Zn 206	50	LDR A	LDR

*Used for Interference Correction Only
** ICP4 Only
*** ICP5 Only

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Appendix D: Standard Reference Tables

ICP Working Calibration Standard					ICP Calibration Verification Standard			
Element	Stock Conc. (mg/L)	Aliquot (mL)	Final Volume (mL)	Cal STD Final Conc. (mg/L)	Stock Conc. (mg/L)	Aliquot in (mL)	Final Volume (mL)	Final Conc. (mg/L)
Ag	100	2.0	100	2	100	1.0	100	1
Al	1000			20	1000			10
As	200			4	200			2
Ba	200			4	200			2
Be	200			4	200			2
Ca	1000			20	1000			10
Cd	200			4	200			2
Co	200			4	200			2
Cr	200			4	200			2
Cu	200			4	200			2
Fe	500			10	500			5
K	1000			20	1000			10
Mg	1000			20	1000			10
Mn	200			4	200			2
Na	1000			20	1000			10
Ni	200			4	200			2
Pb	200			4	200			2
S	1000			20	1000			10
Sb	200			4	200			2
Se	200			4	200			2
Tl	200			2	100			1
V	200			4	200			2
Zn	200			4	200			2
Mo	200			4	200			2
B	200			4	200			2
Sn	200			4	200			2
Ti	100			4	200			2
Si	500			20	500			5
Li	200			4	200			2
P	500			4	500			5
Sr	200			4	200			2
U	200			4	200			2

Appendix E: Interference Check Standard Reference Tables

ICSA				
Element	Stock Conc. (mg/L)	Aliquot in (mL)	Final Volume (mL)	Final Conc. (ug/L)
Al	5000	10	100	500000
Ca	5000	10	100	500000
Fe	2000	10	100	200000
Mg	5000	10	100	500000

ICSAB				
Element	Stock Conc. (mg/L)	Aliquot in (mL)	Final Volume (mL)	Final Conc. (ug/L)
Ag	20	1.0	100	200
Al	5000	10	100	500000
As	10	1.0	100	100
Ba	50	1.0	100	500
Be	50	1.0	100	500
Ca	5000	10	100	500000
Cd	100	1.0	100	1000
Co	50	1.0	100	500
Cr	50	1.0	100	500
Cu	50	1.0	100	500
Fe	2000	10	100	200000
Mg	5000	10	100	500000
Mn	50	1.0	100	500
Ni	100	1.0	100	1000
Pb	5	1.0	100	50
Sb	60	1.0	100	600
Se	5	1.0	100	50
Tl	10	1.0	100	100
V	50	1.0	100	500
Zn	100	1.0	100	1000

Document Information

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Document Title: Mercury in Liquid and Solid/Semi-Solid Waste by 7470A, 7471, 7471B, and 245.1

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ENV-SOP-MIN4-0054

QM Approval

Name/Signature	Title	Date	Meaning/Reason
Janielle Ward (007319)	Manager - Quality	26 Jul 2021, 02:07:12 PM	Approved

Management Approval

Name/Signature	Title	Date	Meaning/Reason
Krista Carlson (004514)	Project Manager 1	16 Jul 2021, 09:34:14 AM	Approved
Andrew Mickelson (009792)	Manager	28 Jul 2021, 01:11:02 PM	Approved
Adam Haugerud (005828)	General Manager 2	09 Aug 2021, 06:32:40 PM	Approved

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TEST METHOD STANDARD OPERATING PROCEDURE
TITLE: Mercury Analysis by CVAA
TEST METHOD 7470A, 7471A, 7471B, and 245.1
ISSUER: Pace ENV – Minneapolis – MIN4

1.0 SCOPE AND APPLICATION

This standard operating procedure (SOP) describes the laboratory procedure for the determination of mercury in mobility procedure extracts, aqueous wastes, ground waters, soils, sediments, bottom deposits, and sludge-type materials using cold vapor atomic absorption (CVAA).

1.1 Target Analyte List and Limits of Quantitation (LOQ)

The default reporting limit (RL) or Limit of Quantitation (LOQ) for mercury in liquid is 0.2 µg/L. The default reporting limit for mercury in soil is 0.02 mg/kg. Reporting limits may vary based on the nature of the individual sample matrix. For certain applications, a lower level method optimized for sensitivity in which the reporting limit is 0.010 µg/L is available. This is for aqueous samples only.

LOQ are established in accordance with Pace policy and SOPs for method validation and for the determination of detection limits (DL) and quantitation limits (LOQ). DL and LOQ are routinely verified and updated when needed.

The reporting limit (RL) is the value to which analytes are reported as detected or not detected in the final report. When the RL is less than the lower limit of quantitation (LLOQ), all detects and non-detects at the RL are qualitative. The LLOQ is the lowest point of the calibration curve used for each target analyte.

DL, LOQ, and RL are always adjusted to account for actual amounts used and for dilution.

1.2 Applicable Matrices

This SOP is applicable to ground, surface, drinking, and storm runoff water samples; industrial, domestic waste waters and solids.

2.0 SUMMARY OF METHOD

2.1 The method, a CVAA technique, is based on the absorption of radiation at the characteristic wavelength of 253.7 nm by mercury vapor. The mercury is reduced to the elemental state 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.

2.2 Chemical Reactions - Organic mercury compounds are decomposed by digestion with potassium permanganate in acid solution. The mercuric ions are then reduced to the elemental state with stannous chloride and mercury vapor is produced.

3.0 INTERFERENCES

3.1 Potassium permanganate is added during digestion of samples to break down organo-mercury compounds which would otherwise not respond to the cold vapor technique. A heating step is required for methyl mercuric chloride when present in or spiked to a natural system. Possible sulfide interferences are also eliminated by the addition of potassium permanganate. EPA studies indicate concentrations as high as 20 mg/L of sodium sulfide do not interfere with the recovery of added inorganic mercury from distilled water.

3.2 Copper has also been reported to interfere; however, EPA studies indicate copper concentrations as high as 10 mg/L had no effect on recovery of mercury from reagent water.

3.3 Sea waters, brines and industrial effluents high in chlorides require additional permanganate. During the oxidation step, chlorides are converted to free chlorine which will also absorb radiation of 253 nm. Care must be taken to assure that free chlorine is absent before the mercury is reduced and swept into the cell. The design of the dedicated mercury analyzer assures that this does not occur.

4.0 DEFINITIONS

Refer to the Laboratory Quality Manual for a glossary of common lab terms and definitions.

5.0 HEALTH AND SAFETY

The toxicity or carcinogenicity of each chemical material used in the laboratory has not been fully established. Each chemical should be regarded as a potential health hazard and exposure to these compounds should be as low as reasonably achievable.

The laboratory maintains documentation of hazard assessments and OSHA regulations regarding the safe handling of the chemicals specified in each method. Safety data sheets for all hazardous chemicals are available to all personnel. Employees must abide by the health, safety and environmental (HSE) policies and procedures specified in this SOP and in the Pace Chemical Hygiene / Safety Manual.

Personal protective equipment (PPE) such as safety glasses, gloves, and a laboratory coat must be worn in designated areas and while handling samples and chemical materials to protect against physical contact with samples that contain potentially hazardous chemicals and exposure to chemical materials used in the procedure.

Concentrated corrosives present additional hazards and are damaging to skin and mucus membranes. Use these acids in a fume hood whenever possible with additional PPE designed for handing these materials. If eye or skin contact occurs, flush with large volumes of water. When working with acids, always add acid to water to prevent violent reactions. Any processes that emit large volumes of solvents (evaporation/concentration processes) must be in a hood or apparatus that prevents employee exposure.

Contact your supervisor or local HSE coordinator with questions or concerns regarding safety protocol or safe handling procedures for this procedure.

6.0 SAMPLE COLLECTION, PRESERVATION, HOLDING TIME, AND STORAGE

Samples should be collected in accordance with a sampling plan and procedures appropriate to achieve the regulatory, scientific, and data quality objectives for the project.

The laboratory does not perform sample collection or field measurements for this test method. To assure sample collection and field checks and treatment are performed in accordance with applicable regulations Pace project managers will inform the client of these requirements at the time of request for analytical services when the request for testing is received prior to sample collection. If samples were already collected, the laboratory will record any nonconformance to these requirements in the

laboratory's sample receipt record when sufficient information about sample collection is provided with the samples.

General Requirements

Matrix	Routine Container	Minimum Sample Amount[1]	Preservation	Holding Time
Aqueous	250 mL Plastic	30 mL	Acidified with nitric acid to pH<2, stored ambient	Must be analyzed within 28 days of collection.
Solid	8 oz glass jar	0.3 gram	<6°C, but above freezing	

[1]*Minimum amount needed for each discrete analysis.*

Thermal preservation is checked and recorded on receipt in the laboratory in accordance with laboratory ENV-SOP-MIN4-0008 *Sample Management*, or equivalent replacement. Chemical preservation is checked and recorded at time of receipt or prior to sample preparation.

After receipt, samples are stored either stored at ambient or 6°C until sample preparation. Prepared samples digestates are stored at ambient temperatures until sample analysis.

After analysis, unless otherwise specified in the analytical services contract, samples are retained for 21 days from date of final report and then disposed of in accordance with Federal, State, and Local regulations.

7.0 EQUIPMENT AND SUPPLIES

7.1 Equipment

Equipment	Description
Mercury analyzer, computer controlled	Cold Vapor Atomic Adsorption (CVAA), Cetac M-7600 or PE FIMS-400. Each instrument has an associated auto-sampler, Cetac ASX 520 or equivalent
Hot Block™ digester	54 place block or equivalent, Environmental Express SC154 or equivalent
Analytical Balance	Sartoriius or equivalent, capable of weighing to 0.01g
Mechanical pipettors	Eppendorf, Fisher brand or equivalent replacement, various sizes
Glassware	Class A volumetric flasks and graduated cylinders of various sizes

7.2 Supplies

Supply	Description
Argon gas	Praxair or equivalent, High purity grade, 99.99%
Peristaltic pump tubing	Fisher Scientific or equivalent
Digestion cups	Moldpro or equivalent, 50 mL disposable digestion cups
Resin Pellets	Environmental Express SC400 or equivalent
Filters	GE Whatman or equivalent
Auto-sampler tubes	Moldpro or equivalent, 15 mL metals free auto-sampler tubes
Digestion cups	Moldpro or equivalent, 50 mL disposable digestion cups

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TEST METHOD STANDARD OPERATING PROCEDURE
TITLE: Mercury Analysis by CVAA
TEST METHOD 7470A, 7471A, 7471B, and 245.1
ISSUER: Pace ENV – Minneapolis – MIN4

8.0 REAGENTS AND STANDARDS

8.1 Reagents

Reagent	Description
Reagent water	ASTM Type II
Nitric Acid (HNO_3)	Fisher Scientific, A-509-P212 or equivalent
Hydrochloric acid (HCl)	Fisher Scientific, A-508-P212 or equivalent
Sulfuric acid	Fisher Scientific P/N A510-P212 or equivalent
Potassium permanganate solution	Dissolve 100 g potassium permanganate in a minimum volume of reagent water and dilute to 2000 mL with reagent water. Filter reagent as needed for lower level procedures. Store the reagent at room temperature in either a plastic or glass container. This solution expires 3 months from preparation date. Fisher Scientific brand reagents or equivalent.
Sodium chloride - Hydroxylamine hydrochloride solution	Dissolve 240 g sodium chloride and 240 g hydroxylamine hydrochloride in reagent water and dilute to 2000 mL with reagent water. Store the standard at room temperature in either a plastic or glass container. Solution expires 1 month from preparation date. Fisher Scientific brand reagents or equivalent.
Potassium persulfate solution (5%)	Dissolve 100 g of potassium persulfate in reagent grade water and dilute to 2000 mL. This solution expires 3 months from the preparation date. Fisher Scientific brand reagents or equivalent.
Rinse solution	Add 48 mL concentrated hydrochloric acid to 800 mL water, add 24 mL concentrated nitric acid and dilute to 1 L with reagent water. Store in 5L Nalgene container at room temperature. The solution expires 1 week from preparation date.
Stannous Chloride	Add 140 mL concentrated hydrochloric acid and 200 grams SNCl2-2H20 to 2000 mL reagent water. Different amounts may be made based on need. Store in bottle marked “Stannous Chloride” at the instrument. Fisher Scientific brand reagents or equivalent.
Aqua Regia	Mix 3 parts concentrated hydrochloric acid with 1 part concentrated nitric acid. Use fresh daily, expires within 24 hours.

8.2 Standards

Standard	Description
Mercury Calibration Stock Solution	1000 mg/mL, NIST traceable standard. Store at room temperature. Expires as specified by manufacturer. Inorganic Ventures or equivalent.
Intermediate Working Calibration Solution[1]	50 ug/L intermediate final concentration. Mercury Calibration Intermediate Standard to be prepared every 6 months or as needed. The calibration standards are prepared using the same type of acid and reagents, at the same concentration range as the samples to be analyzed. See appendix B for composition.
ICV/CCV Mercury Stock Solution	1 ug/mL, NIST traceable standard. Must be from a separate source than the mercury calibration stock source. Spex-Certiprep or equivalent.
Low Level Mercury Calibration Stock Solution	10 mg/L, NIST traceable standard. Store at room temperature. Expires as specified by manufacturer. Inorganic Ventures or equivalent.

Low Level ICV/CCV Mercury Stock Solution	10 mg/L, NIST traceable standard. Must be from a separate source than the mercury calibration stock source. Inorganic Ventures or equivalent.
Low Level Mercury Calibration Intermediate Standard[1]	1 ug/L final concentration. Mercury Calibration Intermediate Standard to be prepared every 6 months or as needed. The calibration standards are prepared using the same type of acid and reagents, at the same concentration range as the samples to be analyzed. See appendix B for composition.

8.2.1 Mercury Calibration Intermediate Standard to be prepared every 6 months or as needed. The calibration standards are prepared using the same type of acid and reagents, at the same concentration range as the samples to be analyzed.

8.2.2 SW-846 series methods for mercury require that calibration standards are processed like samples including heating while EPA 245.1 specifically prohibits the calibration standards from being heated. Daily calibration records are documented in the electronic Prep Log.

9.0 PROCEDURE

9.1 Water

9.1.1 Sample Preparation

9.1.1.1 Prepare a method blank (MB) by transferring 30 mL of reagent grade water to a new 50 mL digestion cup. Label with the LIMS batch number and sample number.

9.1.1.2 Prepare a laboratory control sample (LCS) by transferring a 0.15 mL aliquot of the stock mercury standard to a 50 mL cup. For low level mercury samples, transfer 0.15 mL aliquot of the low level mercury intermediate standard. Bring the total volume to 30 mL with reagent water. Label with the LIMS batch number and sample number.

9.1.1.3 Shake sample to achieve homogeneity. Maximum sample volume is 30 mL. Use this or a smaller volume diluted to 30 mL. Place the sample into the 50 mL cup labeled with the corresponding LIMS sample number. Record sample volume in the Hg CVAA Sample Preparation Log.

9.1.1.4 Prepare an MS/MSD by transferring 0.15 mL aliquot of the stock mercury standard to 50 mL cups. For low level mercury samples, transfer 0.15 mL aliquot of the low level mercury intermediate standard. Bring the total volume of each to 30 mL with sample.

9.1.1.5 To all samples (including QC) add 1.5 mL concentrated sulfuric acid and 0.75 mL concentrated nitric acid, mixing well after each addition.

9.1.1.6 To all samples (including QC) add 5 mL potassium permanganate, and observe physical changes for 15 minutes. If the purple color disappears, the sample is re-batched and re-prepped at a lower volume.

9.1.1.7 To all samples (including QC) add 2.5 mL of potassium persulfate solution and swirl to mix.

9.1.1.8 Loosely cap each cup and place into the digestion block, maintained at a temperature of 95°C ± 3°C and heat for two hours. Observe the initial temperature and time in the block.

9.1.1.9 After the two hour digestion, remove the samples from the block and cool. Observe the time the samples were removed from the block, as well as the final temperature of the block.

9.1.1.10 To all samples (including QC) add 1.8 mL of hydroxylamine hydrochloride to reduce the excess permanganate. The permanganate is reduced when the purple color dissipates. If the purple color does not dissipate, add additional hydroxylamine hydrochloride until the color dissipates. Note this on the preparation log and adjust in LIMS. For example: if an additional mL is needed, then add 1 mL to the final volume.

9.1.2 Documentation – Digestion Records

Record the observations and necessary information in the electronic preplog using template version F-MN-I-342-Rev.02. Information includes batch and sample ID, initial and final times, temperatures, volumes, prep date, prep analyst, supporting equipment, and lot numbers of solutions used. Also include any additional comments if needed. The initial and final times and temperatures will be representative of the elapsed time for the batch.

9.2 Solid/Semi-Solid

9.2.1 Sample Preparation

9.2.1.1 Prepare a method blank (MB) by weighing 0.3 g of resin pellets in a 50 mL cup. Label with the LIMS batch number and sample number.

9.2.1.2 Prepare a LCS by weighing 0.3 g of resin pellets in a 50 mL cup and spiking with a 0.15 mL aliquot of the ICV/CCV working mercury standard. Label with the LIMS batch number and sample number.

9.2.1.3 Weigh a representative 0.3-0.36 g portion of sample in a 50 mL labeled cup.

9.2.1.4 Weigh two additional samples for matrix spike/matrix spike duplicate (MS/MSD) and spike carefully to get these samples as close to the weight of the unspiked sample used for QC, as possible. Spike both the MS and MSD with 0.15 mL of the mercury ICV/CCV working standard.

9.2.1.5 To all samples (including QC) add 3 mL DI water.

9.2.1.6 To all samples (including QC) add 3 mL aqua regia (see 10.1 above).

9.2.1.7 Place in hot block, maintained at 95°C ± 3°C and heat for 2 minutes. Record this time and temperature as the initial start time.

9.2.1.8 Remove from hot block and allow to cool.

9.2.1.9 Bring all samples (including QC) up to a volume of 30 mL with DI water.

9.2.1.10 To all samples (including QC) add 9 mL potassium permanganate and observe physical changes for 15 minutes. If the purple color disappears, re-prepare the sample, MB, and LCS with less DI and the corresponding amount of potassium permanganate added so that final volume does not exceed 30 mL. Additional permanganate is noted as a comment on the prep form.

9.2.1.11 Loosely cap each cup and return samples to hot block digester, maintained at a temperature of 95°C ± 3°C and heat for 30 minutes.

9.2.1.12 Remove the samples from the block and record the final time and the temperature. Allow the samples to cool.

9.2.1.13 To all samples (including QC) add 3.6 mL of hydroxylamine hydrochloride to reduce the excess permanganate. The permanganate is reduced when the purple color dissipates. If the purple color does not dissipate, add additional hydroxylamine hydrochloride until the color dissipates. Note this on the preparation log and adjust in LIMS. For example: if an additional mL is needed, then add 1 mL to the final volume.

9.2.2 Documentation – Digestion Records

Record the necessary information in the electronic preplog using template version F-MN-I-343-Rev.03. Information includes batch and sample ID, initial and final times, temperatures, volumes, prep date, prep analyst, supporting equipment, and lot numbers of solutions used. Also include any additional comments if needed. The initial and final times and temperatures will be representative of the elapsed time for the batch.

9.3 Equipment Preparation & Analysis

9.3.1 Turn on the computer and load the software. Turn on, or 'wake up' the instrument and allow the lamp to warm up for about 90 minutes from a cold shut down (lamp off, main power off and gas off) and 5 minutes from standby (lamp off, main power on and gas off). Check the following:

9.3.2 Prepare any necessary reagents and record the appropriate information (volumes, manufacturer, lot numbers, etc.) in the standard solution log.

9.3.3 Check instrument waste and empty as needed.

9.3.4 Perform any routine maintenance as needed and record in maintenance log.

9.3.5 Check the $KMnO_4$ trap at the back of the instrument to make sure it is filled with crystalline $KMnO_4$ and not wet or spent (the brown MnO_2 color approaches the open end of the trap).

9.3.6 Fill the rinse solution container with rinse solution, if needed, and move the probe down into the rinse well.

9.3.7 Check peristaltic pump tubing installation, make sure tension is adjusted if needed, and turn pump on.

9.3.8 Place the $SnCl_2$ line in DI water.

9.3.9 Initialize the wetting of the GLS by selecting 'wet the gas liquid separator post' option in the software. This increases the gas flow to 300-350 mL/min and ramps the pump speed to 100%. Pinch the waste line tubing shut with your fingers. Watch the bubbles and ensure that 1-2 bubbles completely propels to the top of the chamber, wetting the entire post and the top. As soon as this happens, open the waste line tubing so the GLS can drain.

9.3.10 Inspect the GLS to make sure it is draining completely and liquid is not pooling.

9.3.11 Attach the sample gas line to the nafion dryer cartridge.

9.3.12 Fill the stannous chloride bottle with stannous chloride.

9.3.13 Place the $SnCl_2$ line into the $SnCl_2$ solution bottle.

9.3.14 Create a worksheet for analysis by selecting 'new from' in the file menu. Enter the name, ie 20Aug15 (DDMMMYY), a, b, c etc. (if more than one run is performed that day) soil or water to indicate sample matrix, and instrument ID number. The program will then go to the Method Editor page.

9.3.14.1 In the conditions page in the Method Editor, check the instrument settings including the time profile (baseline correction and read time delays). To do this, read a standard and move the baseline correction window and read time window accordingly if needed.

9.3.14.2 Check the Standards page to ensure the correct calibration parameters and standards are entered.

9.3.14.3 Check the QC tests page to make sure the correct test solutions and parameters are entered if the software is to calculate recoveries during analysis.

9.3.15 Create a sequence in the sequence editor tab and enter sample IDs or import them from LimsLink.

9.3.16 Start analysis, monitor all initial QC checks. If initial QC fails, make adjustments if needed and re-calibrate. If checks pass criteria, continue with sample analysis.

9.3.17 After analysis, print out a report and transfer valid data into LIMS system via LimsLink.

9.3.18 After completing sample analysis for the day, shut down the instrument.

9.3.18.1 Place the $SnCl_2$ line in 10% HNO_3 and run for ~10 minutes. After this move the probe up out of the rinse well and place the $SnCl_2$ line in DI water and run for 2-5 minutes. Remove from DI and allow the line to run dry. Turn off pump, disconnect the clamps, and loosen pump tubing.

9.3.18.2 Disconnect the sample gas line from the nafion dryer cartridge.

9.3.18.3 Turn off the gas and the lamp.

9.3.18.4 If the instrument will be used in the next day or two, leave it in the stand-by mode. If not, do a cold shut down and turn off the software, instrument, auto sampler and auto diluter.

9.4 Routine Instrument Operating Conditions

Parameter	Setting
Sample Probe Depth (mm)	145
ASX Rinse Pump Speed (%)	50
Sample Uptake Time (s)	45
Rinse Time (s)	95
Gas Flow (mL/min)	100
Pump speed (%)	50
Read Delay time (s)	55.50
Replicate read time (s)	1.50
Replicates	4

9.5 Initial Calibration

9.5.1 Calibration Design

9.5.1.1 The calibration curve must consist of a minimum of a calibration blank and five non-zero standards for each mode of analysis. The calibration range for standard level analysis is 0.2 ug/L to 10 ug/L. The calibration range for lower level analysis is 0.010 µg/L to 0.20 µg/L. Use the average of four integrations for both calibration and sample analyses. Using the instrumentation software, prepare a standard curve for each element by plotting absorbance versus concentration. The calibration is a linear regression using equation; $y = mx + b$ The analyst may employ a regression equation that does not pass through the origin, however forcing through zero is not allowed. Instruments must be calibrated at a minimum of once every 24 hours or prior to use. The instrument standardization date and time must be included in the raw data.

9.5.1.2 Additional calibration specifications may be referenced in ENV-POL-CORQ-0005 *Acceptable Calibration Practices for Instrument Testing* (or equivalent replacement).

9.5.2 Calibration Sequence

Calibration Blank (CAL0)
CAL1
CAL2
CAL3
CAL4
CAL5
ICV
ICB
CRDL
CCV
CCB
Client samples
CRDL
CCV
CCB

9.5.3 ICAL Evaluation

9.5.3.1 Curve Fit

With a multi-point calibration, the regression calculation will generate a correlation coefficient (r) that is the measure of the "goodness of fit" of the regression line to the data. In order to be used for quantitative purposes, the correlation coefficient must be > 0.995.

9.5.3.2 Relative Standard Error (RSE)

%RSE is evaluated after all calibration points have been measured. In order for a standard curve to be acceptable, the %RSE acceptance criteria is 80%-120% must be observed.

Note: %RSE is analogous to %RSD. 40CFR Part 136 allow %RSE to be used in place of correlation coefficient (R) or coefficient of determination (r^2) for the acceptability determination of the curve.

9.5.3.3 Initial Calibration Verification

In addition to meeting the linearity requirement, any new calibration curve must be assessed for accuracy in the values generated. To assess the accuracy, a single standard

from a secondary source must be analyzed and the results obtained must be compared to the known value of the standard. This step is referred to as Initial Calibration Verification. The ICV is analyzed immediately following an initial calibration curve.

9.5.4 Continuing Calibration Verification

A CCV followed immediately by a CCB must be analyzed after every 10 samples and at the end of the analytical batch to verify the system is still calibrated.

10.0 DATA ANALYSIS AND CALCULATIONS

See the laboratory SOP ENV-SOP-MIN4-0171 *Laboratory Calculations*, or equivalent replacement, for equations for common calculations.

10.1 The percent recovery in the LCS is calculated using Equation 1:

Equation 1

$$\%\,\mathrm{Recovery} = \frac{SR}{SA}\,\mathrm{x}100$$

Where, SR = LCS result (ug/L or mg/kg)
SA = spike added, ug/L or mg/kg

10.2 The percent recovery of mercury in the matrix spike and matrix spike duplicate is calculated using Equation 2:

Equation 2

$$\%\,\mathrm{Recovery} = \frac{(SSR - SR)}{SA}\,\mathrm{x}100$$

Where, SSR = Spiked sample result, mg/L or mg/kg
SR = Sample result, mg/L or mg/kg
SA = Spike added, mg/L or mg/kg

10.3 Calculate the Relative Percent Difference (RPD) between the matrix spike and matrix spike duplicate using Equation 3:

Equation 3

$$\%RPD = \frac{|S - D|}{(S + D)/2}\,x100$$

Where, S = Sample result, mg/L or mg/kg
D = Duplicate sample result, mg/L or mg/kg

10.4 The corrected dry weight concentration can be calculated using the following:

$$corrected\ dry\ wt\ conc = \frac{\left(c \times \frac{v_f}{wt_i}\right)}{\%\ dry\ wt}$$

Where, c = concentration on instrument, µg/L
v_f = final volume, L
wt_i = initial weight, g

$$\%Dry\ weight = \frac{Sample\ Dry\ Weight}{Sample\ Wet\ Weight} \text{x}100$$

11.0 QUALITY CONTROL AND METHOD PERFORMANCE

11.1 Quality Control

The following QC samples are prepared and analyzed with each batch of samples. Refer to Appendix A for acceptance criteria and required corrective action.

QC Item	Frequency
Method Blank (MB)	1 per batch of 20 or fewer samples.
Laboratory Control Sample (LCS)	1 per batch of 20 or fewer samples.
Laboratory Control Sample Duplicate (LCSD)	As needed
Matrix Spike (MS)	1 per batch of 20 or fewer samples for 7470/7471. 1 per batch of 10 or fewer samples for 245.1
Matrix Spike Duplicate (MSD)	1 per batch of 20 or fewer samples.
Sample Duplicate	Performed at client request.
Serial Dilution	Performed at client request.
Post Digestion Spike	Performed at client request.
Filter Blank (FB)	1 per batch of 20 or fewer samples when applicable.

11.2 Instrument QC

The following Instrument QC checks are performed. Refer to Appendix A for acceptance criteria and required corrective action.

QC Item	Frequency
Initial Calibration	Daily
Initial Calibration Verification	Immediately after each initial calibration
Initial Calibration Blank	Immediately after each initial calibration
Continuing Calibration Verification	Prior to the analysis of any samples and after every 10 injections thereafter. Samples must be bracketed with a closing CCV standard.
Continuing Calibration Blank	Following every CCV injection
CRDL / LLCCV verification	At the beginning of each run. May be run more frequently per state or client requirement.

11.3 Method Performance

11.3.1 Method Validation

11.3.1.1 Detection Limits

Detection limits (DL) and limits of quantitation (LOQ) are established at initial method setup and verified on an on-going basis thereafter. Refer to Pace ENV corporate SOP ENV-SOP-CORQ-0011 *Method Validation and Instrument Verification* and to the laboratory's SOP ENV-SOP-MIN4-0163 *Determination of LOD and LOQ* (or equivalent replacement) for these procedures.

11.4 Analyst Qualifications and Training

Employees that perform any step of this procedure must have a completed Read and Acknowledgment Statement for this version of the SOP in their training record. In addition, prior to unsupervised (independent) work on any client sample, analysts that prepare or analyze samples must have successful initial demonstration of capability (IDOC) and must successfully demonstrate on-going proficiency on an annual basis. Successful means the initial and on-going DOC met criteria, documentation of the DOC is complete, and the DOC record is in the employee's training file. Refer to laboratory SOP ENV-SOP-MIN4-0165 *Orientation and Training Procedures* (or equivalent replacement) for more information.

12.0 Data Review And Corrective Action

12.1 Data Review

Pace's data review process includes a series of checks performed at different stages of the analytical process by different people to ensure that SOPs were followed, the analytical record is complete and properly documented, proper corrective actions were taken for QC failure and other nonconformance(s), and that test results are reported with proper qualification.

The review steps and checks that occur as employee's complete tasks and review their own work is called primary review.

All data and results are also reviewed by an experienced peer or supervisor. Secondary review is performed to verify SOPs were followed, that calibration, instrument performance, and QC criteria were met and/or proper corrective actions were taken, qualitative ID and quantitative measurement is accurate, all manual integrations are justified and documented in accordance with the Pace ENV's SOP for manual integration, calculations are correct, the analytical record is complete and traceable, and that results are properly qualified.

A third-level review, called a completeness check, is performed by reporting or project management staff to verify the data report is not missing information and project specifications were met.

Refer to laboratory SOP ENV-SOP-MIN4-0092 *Data Review Process* (or equivalent replacement) for specific instructions and requirements for each step of the data review process.

12.2 Corrective Action

Corrective action is expected any time QC or sample results are not within acceptance criteria. If corrective action is not taken or was not successful, the decision/outcome must be documented in the analytical record. The primary analyst has primary responsibility for taking corrective action when QA/QC criteria are not met. Secondary data reviewers must verify that appropriate action was taken and/or that results reported with QC failure are properly qualified.

Corrective action is also required when carryover is suspected and when results are over range.

Samples analyzed after a high concentration sample must be checked for carryover and reanalyzed if carryover is suspected. Carryover is usually indicated by low concentration detects of the analyte in successive samples analyzed after the high concentration sample.

Sample results at concentrations above the upper limit of quantitation must be diluted and reanalyzed. The result in the diluted samples should be within the upper half of the calibration range. Results less than the mid-range of the calibration indicate the sample was over diluted and analysis should be repeated with a lower level of dilution. If dilution is not performed, any result reported above the upper range is considered a qualitative measurement and must be qualified as an estimated value.

Refer to Appendix A for a complete summary of QC, acceptance criteria, and recommended corrective actions for QC associated with this test method.

13.0 POLLUTION PREVENTION AND WASTE MANAGEMENT

Pace proactively seeks ways to minimize waste generated during our work processes. Some examples of pollution prevention include but are not limited to: reduced solvent extraction, solvent capture, use of reusable containers for solvent management, and real-time purchasing.

The EPA requires that laboratory waste management practice to be conducted consistent with all applicable federal and state laws and regulations. Excess reagents, samples and method process wastes must be characterized and disposed of in an acceptable manner in accordance with Pace's Chemical Hygiene Plan / Safety Manual.

14.0 MODIFICATIONS

A modification is a change to a reference test method made by the laboratory. For example, changes in stoichiometry, technology, quantitation ions, reagent or solvent volumes, reducing digestion or extraction times, instrument runtimes, etc. are all examples of modifications. Refer to Pace ENV corporate SOP ENV-SOP-CORQ-0011 *Method Validation and Instrument Verification* for the conditions under which the procedures in test method SOPs may be modified and for the procedure and document requirements.

14.1 Use of Block Digestor- Heating is conducted with hot block digestion as the heating equivalent mentioned in SW 846 7471B (section 6.10) and SW 846 7470. This is also compliant with method 245.1 under the Clean Water Act method flexibility in 40CFR section 136.6 (b) (4) (iii).

14.2 The lab utilizes a 30 mL final volume, all solid weights and reagent ratios are conducted based on the 0.3 g versus the 0.5 g initial weight accordingly.

14.3 Mercury calibration standards are prepared and digested weekly for SW-846 analysis of soils and waters. The stability and performance of standards prepared weekly has been evaluated and documented.

15.0 RESPONSIBILITIES

Pace ENV employees that perform any part this procedure in their work activities must have a signed Read and Acknowledgement Statement in their training file for this version of the SOP. The employee is responsible for following the procedures in this SOP and handling temporary departures from this SOP in accordance with Pace's policy for temporary departure.

Pace supervisors/managers are responsible for training employees on the procedures in this SOP and monitoring the implementation of this SOP in their work area.

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TEST METHOD STANDARD OPERATING PROCEDURE
TITLE: Mercury Analysis by CVAA
TEST METHOD 7470A, 7471A, 7471B, and 245.1
ISSUER: Pace ENV – Minneapolis – MIN4

16.0 ATTACHMENTS

Appendix A – QC Summary

Appendix B – Working Standard Summary

17.0 REFERENCES

Pace Quality Assurance Manual- most current version.

TNI Standard, *Management and Technical Requirements for Laboratories Performing Environmental Analyses*, EL-V1-2009.

TNI Standard, *Management and Technical Requirements for Laboratories Performing Environmental Analyses*, EL-VI-2016-Rev.2.1.

Test Methods for Evaluating Water and Solid Waste, Physical/Chemical Methods, SW-846, Method 7470A, 1994.

Test Methods for Evaluating Water and Solid Waste, Physical/Chemical Methods, SW-846, Method 7471A, 1994.

Test Methods for Evaluating Water and Solid Waste, Physical/Chemical Methods, SW-846, Method 7000a, Revision 1, July 1992.

Test Methods for Evaluating Water and Solid Waste, Physical/Chemical Methods, SW-846, Method 7471B, Revision 2, Feb 2011.

Methods for Chemical Analysis of Water and Wastes, Method 245.1. Rev.3.0, 1994.

40 CFR Appendix B to Part 136, *Definition and Procedure for the Determination of the Method Detection Limit - Rev 2*, August 28, 2017.

Minnesota Pollution Control Agency, Laboratory Quality Control and Data Policies, July 2011.

18.0 REVISION HISTORY

This Version:

Section	Description of Change
7.1	Updated the description of the Mercury analyzer, computer controlled from "or equivalent to "PE FIMS-400"
7.2	Added the filters row
8.1	update the description of the Potassium permanganate solution to include "Filter reagent as needed for lower level procedures
9.1.1.6	Added; " and observe physical changes for 15 minutes."
9.1.1.8	Update digestion temperature acceptance range from 95°C ± 2°C to 95°C ± **3**°C
9.2.1.7	Update digestion temperature acceptance range from 95°C ± 2°C to 95°C ± **3**°C
9.2.1.11	Update digestion temperature acceptance range from 95°C ± 2°C to 95°C ± **3**°C
Append A	Remove all references to West Virginia

This document supersedes the following document(s):

TEST METHOD STANDARD OPERATING PROCEDURE
TITLE: Mercury Analysis by CVAA
TEST METHOD 7470A, 7471A, 7471B, and 245.1
ISSUER: Pace ENV – Minneapolis – MIN4

Document Number	Title	Version
ENV-SOP-MIN4-0054	Mercury in Liquid and Solid/Semi-Solid Waste by 7470A, 7471, 7471B, and 245.1	05

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TEST METHOD STANDARD OPERATING PROCEDURE
TITLE: Mercury Analysis by CVAA
TEST METHOD 7470A, 7471A, 7471B, and 245.1
ISSUER: Pace ENV – Minneapolis – MIN4

Appendix A: QC Summary

QC Item	Frequency	Acceptance Criteria	Corrective Action	Qualification
ICAL	Daily	r ≥ 0.995 RSE < 20%	Identify and correct source of problem, repeat.	None. Do not proceed with analysis.
ICV	After each ICAL	± 10% for SW-846 7000 series methods and ± 5% for 245.1	Identify and correct the source of problem, re-analyze.	None. Do not proceed with analysis..
ICB	Immediately after the initial calibration verification	Result must be less than the absolute value of the Reporting Limit (LOQ). NC requires blanks to be clean to ½ RL. WIDNR require samples to be reported to the MDL.	Identify source of problem, re-analyze. Analysis may proceed if it can be demonstrated that the ICB exceedance has no impact on analytical measurements. For example, the ICB has detections and the analyte is not detected in sample(s).	Qualify analytes with ICB out of criteria.
CRDL / LLCCV[4]	At the beginning of each run. Depending on data quality objectives it may be required that a CRDL bracket samples.	± 30% (or specified by the client)	Identify source of problem, re-analyze. Analysis may proceed if it can be demonstrated that the CRDL exceedance has no impact on analytical measurements. For example, the CRDL %R is high and the analyte is not detected in sample(s). For example, the CRDL %R is high and the analyte detections exceed the continuing calibrations verification level (midpoint of the curve). If the CRDL is biased low, no data can be reported for the target elements failing criteria.	Qualify outages and explain in case narrative.
CCV[5]	Daily, before sample analysis, after every 10, and at end of analytical window.	All analytes must be within ± 10% of the true value. (%R):	Identify source of problem, re-analyze. Analysis may proceed if it can be demonstrated that the CCV exceedance has no impact on analytical measurements. For example, the CCV %R is high, and the analyte is not detected in sample(s).	Qualify analytes with CCV out of criteria.
CCB	Daily, before sample analysis, after every 10, and at end of analytical window	Result must be less than the absolute value of the Reporting Limit (LOQ). NC requires blanks to be clean to ½ RL. WIDNR require samples to be reported to the MDL.	Identify source of problem, re-analyze. Analysis may proceed if it can be demonstrated that the CCB exceedance has no impact on analytical measurements. For example, the CCB has detections and the analyte is not detected in sample(s).	Qualify analytes with CCB out of criteria.
Method Blank	One per 20 samples	Method 7470/7471: The method blank is considered to be	Identify source of problem, re-analyze. If reanalysis of the MB fails, all samples affected by the	Qualify outages and explain in case narrative.

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		acceptable if it does not contain the target analytes that exceed the LLOQ or project-specific DQOs. Method 245.1: The method blank is considered to be acceptable if it does not contain the target analytes that exceed 1/2 LLOQ or project-specific DQOs.	failing MB elements need to be re-digested and re-analyzed. If the method blank exceeds the criteria, but the associated samples are either below the reporting level or other DQOs, or detections in the sample are >10x MB detections then the sample data may be reported. J-flag qualification will be applied for blank detections between the LOQ and LOD when DQOs require evaluation to the MDL.	
LCS	One per 20 samples	80-120% for 7470/7470A and 7471/7471B. 85-115% for 245.1.	Identify source of problem, re-analyze. If reanalysis of the LCS fails, all samples affected by the failing LCS elements need to be re-digested and re-analyzed. If LCS recovery is > QC limits and these compounds are non-detect in the associated samples	Qualify analytes with LCS out of criteria.
LCSD[1]	An LCSD must be substituted in the event of insufficient sample volume for a matrix spike duplicate sample.	80-120% for 7470/7470A and 7471/7471B. 85-115% for 245.1 % RPD ≤ 20%	Identify source of problem, re-analyze. If reanalysis of the LCS fails, all samples affected by the failing LCS elements need to be re-digested and re-analyzed. If LCS recovery is > QC limits and these compounds are non-detect in the associated samples	Qualify analytes with LCS out of criteria.
MS/MSD[2,3]	One per 20 samples for 7470/7470A and 7471/7471B. One per 10 samples for 200.8	80-120% for 7470/7470A[3] and 7471/74/1B. 245.1: 70-130% %RPD: 20%	If the percent recovery for the MS and MSD fall outside the control limits, the results are flagged that they are outside acceptance criteria along with the parent sample. If the RPD exceeds the acceptance criteria, the MSD sample and associated parent sample need to be flagged. If MS or MSD fails and spike amount is less than 4 times the native concentration in the sample, remove M1 flag and replace with P6 flag. If the RPD is outside the limit, report the data and footnote the samples with precision outliers. The footnote only applies to samples within the same batch containing the sample used for the MS and MSD analyses.	Qualify analytes with MS out of criteria.
Sample Duplicate	Per client request	%Diff ≤ 20%	Qualify outages	Qualify outages.
Serial Dilution	Per client request	Refer to project specific technical specifications.	Qualify outages	Qualify outages.
Post Digestion Spike	Per client request	Refer to project specific technical specifications.	Qualify outages	Qualify outages.

TEST METHOD STANDARD OPERATING PROCEDURE
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Laboratory Filter Blank (FB)	Analyzed only with batches of lab filtered dissolved metals, one per batch of 20 or less.	Result must be less than the absolute value of the Reporting Limit (LOQ). NC requires blanks to be clean to ½ RL.	Identify source of problem, re-analyze. If reanalysis of the MB fails, all samples affected by the failing MB elements need to be re-digested and re-analyzed. If sample(s) non-detect, report the data. If sample result >10x FB detections, report the data.	Qualify outages and explain in case narrative.

[1]WIDNR requires the use of a lab created matrix solution from unused samples.

[2]In the event that only samples identified as Equipment Blanks and/or Field Blanks are available, and LCS/LCSD will be prepared in place of MS/MSD.

[3]In the absence of method specified recovery limits, results will be evaluated based on specifications outlined by the MPCA guidelines for Inorganic Analysis.

[4]A reporting limit verification is performed by analyzing a CRDL at ± 30% while the method has no low end criteria.

[5] ICV/CCV criteria is ± 10% while the 7000 series indicates ± 20%, the tighter criteria is applied to allow for instrumentation to be utilized for any mercury method throughout an analytical shift.

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TEST METHOD STANDARD OPERATING PROCEDURE
TITLE: Mercury Analysis by CVAA
TEST METHOD 7470A, 7471A, 7471B, and 245.1
ISSUER: Pace ENV – Minneapolis – MIN4

Appendix B: Working Standard Summary

Standard	Standard(s) Used	Standard(s) Amount (mL)	Solvent	Solvent Volume (mL)	Final Total Volume (mL)	Final Concentration (µg/L)
Mercury Calibration Intermediate.	Mercury Stock (10 ug/mL)	5	Reagent water	985	1000	50
	Concentrated nitric acid	10				
Standard 0	Intermediate Standard (50 µg/L)	0	Reagent water	30	30	0
Standard 1		0.12		29.88		0.2
Standard 2		0.6		29.4		1.0
Standard 3		1.8		28.2		3.0
Standard 4		3.0		27		5.0
Standard 5		6.0		24		10
CRDL		0.12		29.88		0.2
ICV/CCV	Mercury Stock 1000 mg/mL	0.15	Reagent water	29.85	30	5.0
ICB/CCB	N/A	N/A	Reagent water	30	30	0
Low Level Mercury Calibration Intermediate Standard; Prepare every 6 months.	Calibration Mercury Stock (10 mg/L)	0.100	Reagent water	984.9	1000	1.0
	Concentrated nitric acid	5.0				
	Concentrated hydrochloric acid	10				
Standard 0	Intermediate Standard (1.0 µg/L)	0	Reagent Water	30	30	0
Standard 1		0.30		29.7		0.010
Standard 2		0.75		29.25		0.025
Standard 3		1.5		28.5		0.050
Standard 4		3.0		27		0.100
Standard 5		6.0		24		0.200
CRDL		0.30		29.7		0.01
Low Level Mercury ICV/CCV **Intermediate** Standard. Prepare every 6 months	ICV/CCV Mercury Stock (10 mg/L)	0.4	Reagent water	184.6	200	20
	Concentrated nitric acid	5.0				
	Concentrated hydrochloric acid	10				
Low Level Mercury ICV/CCV	Low Level Mercury ICV/CCV Intermediate (75 µg/L)	0.15	Reagent water	29.85	30	0.10
Lower Level Mercury ICB/CCB	N/A	N/A	Reagent water	30	30	0

Document Information

Document Number: ENV-SOP-MIN4-0056 **Revision:** 04

Document Title: Metals Preparation of Solid Samples for Analysis by ICP and ICP-MS by 3050B

Department(s): Metals

Date Information

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Notes

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All Dates and Times are listed in: Central Time Zone

Signature Manifest

Document Number: ENV-SOP-MIN4-0056 **Revision:** 04

Title: Metals Preparation of Solid Samples for Analysis by ICP and ICP-MS by 3050B

All dates and times are in Central Time Zone.

ENV-SOP-MIN4-0056

QM Approval

Name/Signature	Title	Date	Meaning/Reason
Janielle Ward (007319)	Manager - Quality	30 Sep 2021, 12:40:17 PM	Approved

Management Approval

Name/Signature	Title	Date	Meaning/Reason
Adam Haugerud (005828)	General Manager 2	01 Oct 2021, 05:17:47 PM	Approved
Andrew Mickelson (009792)	Manager	06 Oct 2021, 02:22:12 PM	Approved

TEST METHOD STANDARD OPERATING PROCEDURE
TITLE: Metals Preparation of Solid Samples for Analysis by ICP and ICPMS
TEST METHOD EPA Method 3050B
ISSUER: Pace ENV – Minneapolis – MIN4

1.0 SCOPE AND APPLICATION

This standard operating procedure (SOP) describes the laboratory procedure for the preparation of solid samples using hot block digestion as described in EPA Method 3050B.

1.1 Target Analyte List and Limits of Quantitation (LOQ)

LOQ are established in accordance with Pace policy and SOPs for method validation and for the determination of detection limits (DL) and quantitation limits (LOQ). DL and LOQ are routinely verified and updated when needed. The current LOQ for each target analyte that can be determined by this SOP as of the effective date of this SOP is provided in the associated analytical SOP; SOP ENV-SOP-MIN4-0052 *Metals Analysis by ICP - Method 6010 and 200.7* or ENV-SOP-MIN4-0043 *Metals Analysis by ICP/MS - Method 6020 and 200.8* (or equivalent replacements).

The reporting limit (RL) is the value to which analytes are reported as detected or not detected in the final report. When the RL is less than the lower limit of quantitation (LLOQ), all detects and non-detects at the RL are qualitative. The LLOQ is the lowest point of the calibration curve used for each target analyte.

DL, LOQ, and RL are always adjusted to account for actual amounts used and for dilution.

1.2 Applicable Matrices

This SOP is applicable to sediments, sludges and soil samples.

2.0 SUMMARY OF METHOD

A one-gram aliquot sample is digested in concentrated nitric acid, hydrochloric acid and hydrogen peroxide. After digestion, samples are brought to a final volume of 50mL. Digestates are then analyzed using Inductively Coupled Plasma (ICP) technologies for the determination of metals in solution.

3.0 INTERFERENCES

Sludge samples can contain diverse matrix types, each of which may present its own analytical challenge. Spiked samples and any relevant standard reference material should be processed in accordance with the quality control requirements given in SW-846 Sec. 8.0 to aid in determining whether Method 3050B is applicable to a given waste.

4.0 DEFINITIONS

Refer to the Laboratory Quality Manual for a glossary of common lab terms and definitions.

5.0 HEALTH AND SAFETY

The toxicity or carcinogenicity of each chemical material used in the laboratory has not been fully established. Each chemical should be regarded as a potential health hazard and exposure to these compounds should be as low as reasonably achievable.

The laboratory maintains documentation of hazard assessments and OSHA regulations regarding the safe handling of the chemicals specified in each method. Safety data sheets for all hazardous chemicals are available to all personnel. Employees must abide by the health, safety and environmental (HSE) policies and procedures specified in this SOP and in the Pace Chemical Hygiene / Safety Manual.

Personal protective equipment (PPE) such as safety glasses, gloves, and a laboratory coat must be worn in designated areas and while handling samples and chemical materials to protect against physical contact with samples that contain potentially hazardous chemicals and exposure to chemical materials used in the procedure.

Concentrated corrosives present additional hazards and are damaging to skin and mucus membranes. Use these acids in a fume hood whenever possible with additional PPE designed for handing these materials. If eye or skin contact occurs, flush with large volumes of water. When working with acids, always add acid to water to prevent violent reactions. Any processes that emit large volumes of solvents (evaporation/concentration processes) must be in a hood or apparatus that prevents employee exposure.

Contact your supervisor or local HSE coordinator with questions or concerns regarding safety protocol or safe handling procedures for this procedure.

6.0 SAMPLE COLLECTION, PRESERVATION, HOLDING TIME, AND STORAGE

Samples should be collected in accordance with a sampling plan and procedures appropriate to achieve the regulatory, scientific, and data quality objectives for the project.

The laboratory does not perform sample collection or field measurements for this test method. To assure sample collection and field checks and treatment are performed in accordance with applicable regulations Pace project managers will inform the client of these requirements at the time of request for analytical services when the request for testing is received prior to sample collection. If samples were already collected, the laboratory will record any nonconformance to these requirements in the laboratory's sample receipt record when sufficient information about sample collection is provided with the samples.

General Requirements

Matrix	Routine Container	Minimum Sample Amount[1]	Preservation	Holding Time
Solid	8 oz glass jar	1 gram	<6°C, but above freezing	Must be analyzed within 180 days of collection. If mercury is requested, analysis must occur within 28 days of sample collection.

[1]*Minimum amount needed for each discrete analysis.*

Thermal preservation is checked and recorded on receipt in the laboratory in accordance with laboratory ENV-SOP-MIN4-0008 *Sample Management*, or equivalent replacement.

After analysis, unless otherwise specified in the analytical services contract, samples are retained for 21 days from date of final report and then disposed of in accordance with Federal, State, and Local regulations.

7.0 EQUIPMENT AND SUPPLIES

7.1 Equipment

Equipment	Description	Vendor/Item #/Description
Mechanical pipettes	Various sizes	Fisher Scientific or equivalent
Hot Block ™	54 Place Hot Block	Environmental Express
Analytical Balance	Ability to weigh to the nearest 0.01g	Fisher Scientific or equivalent

7.2 Supplies

Supply	Description	Vendor/Item #/Description
Digestion Cups	50 mL verified to class A specification	Environmental Express or equivalent
Vapor Recovery Device	Reflux cap or Watch glass	Environmental Express or equivalent
Resin beads	For solid matrix QC	Environmental Express or equivalent

8.0 Reagents And Standards

8.1 Reagents

Reagent/Standard	Concentration/Description	Requirements/Vendor/Item #
De-ionized (DI) water	ASTM Type II	Verify that background levels of volatile compounds are acceptable by analysis
Hydrogen Peroxide	30% ACS Grade	Fisher brand
Hydrogen Peroxide	30%, Optima Grade for tin only	Fisher brand
Concentrated nitric acid (HNO_3)	Trace Metal grade	Fisher brand
Concentrated hydrochloric acid (HCl)	Trace Metal grade	Fisher brand

8.2 Standards

Standard	Concentration/Description	Requirements/Vendor/Item #
Metals Spike - Stock solution standards for LCS and MS/MSD	The solution identifications are METALS-STK1 and METALS-STK2. See Appendix A for composition	Purchased from Inorganic Ventures (or equivalent). Store at room temperature. Expires as specified by manufacturer.
Mercury Spike – Stock solution standards for LCS and MS/MSD	10 µg/mL Hg-STK Stock	Purchased from Spex Certiprep. Store at room temperature. Expires as specified by manufacturer.

9.0 Procedure

9.1 Equipment Preparation

9.1.1 Support Equipment

Verify the calibration of variable and fixed volume pipettes as specified in SOP ENV-SOP-MIN4-0161 *Support Equipment* (or equivalent replacement). Calibration records are kept in the QA Office.

Verify the calibration for the thermometer as specified in SOP ENV-SOP-MIN4-0161 *Support Equipment* (or equivalent replacement). Calibration records are kept in the QA Office.

9.1.2 Equipment

The hot block digestors are set to maintain a digestion temperature of 95 +/- 5°C. Use a NIST-traceable thermometer inserted into a digestion cup filled with 50mL of DI to measure the temperature of the hot block. The temperature should be checked in different wells of the hot blocks such that all wells are evaluated over a period of time. Record the temperature of each hot block daily in the temperature logbook.

Balances shall be checked prior to use on each working day with a NIST traceable reference in the expected range of use. Balances must be verified with weights of a class appropriate for the accuracy of the balance being calibrated. Verify the calibration for the balance as specified in SOP ENV-SOP-MIN4-0161 *Support Equipment* (or equivalent replacement). Record the measurements of each weight in the daily balance verification logbook.

9.2 Sample Preparation

9.2.1 Obtain and label digestion tubes in the order for which samples will be weighed out.

9.2.2 Mix the sample thoroughly to achieve homogeneity. For each digestion procedure, weigh a 1-1.1g portion of sample (to the nearest 0.01g) and transfer to a 50 mL digestion cup. Alternative sample volume may be used based on sample matrix. Weigh out 3 aliquots for the batch QC sample (background, matrix spike (MS), and matrix spike duplicate (MSD) being sure to weigh them as close to the same weight as possible.

9.2.2.1 Create a method blank and a laboratory control sample (LCS) by weighing out 1 gram of resin beads for each.

9.2.2.2 Spike the LCS, MS/MSD each of METALS-STK1 and METALS-STK2. If mercury is requested spike 0.25 mL of Hg-SPK stock.

9.2.3 Add DI to the 10mL marking for each sample.

9.2.4 Add 7.5mL of concentrated HNO3, mix the slurry, and cover with a reflux cap. Heat the sample to 95 +/- 5°C and reflux for 70 minutes without boiling. Record initial Hot Block temperature in the digestion log. Observe the sample during heating for brown fumes indicating oxidation of the sample. If this occurs, add up to an additional 5 mL HNO3 and re-heat. Repeat this process until no fumes are given off during heating. Record on the digestion log to what samples and how much additional acid was added.

NOTE: When mercury is a requested analyte, watch glasses will be used rather than reflux caps.

9.2.5 Cool the sample 10 minutes. Add 2.5mL of 30% hydrogen peroxide. Cover with reflux cap and return to the Hot Block for warming which will start the peroxide reaction. Care must be taken to ensure that losses do not occur due to vigorous effervescence. Heat until effervescence subsides for a total of 10 minutes. Cool the samples in the plastic cups.

NOTE: Use Optima grade hydrogen peroxide if the analysis of tin (Sn) is required. Tin is used as a stabilizer in the ACS grade of hydrogen peroxide.

9.2.5.1 If effervescence does not subside, continue to add 30% hydrogen peroxide in 1mL aliquots with warming until the effervescence is minimal or until the general sample appearance is unchanged. Note in the comments section of prep sheet the additional aliquots.

NOTE: Do NOT add more than a total of 10mL hydrogen peroxide.

9.2.6 Add 5mL of concentrated HCl, return the sample to the Hot Block and reflux for an additional 15 minutes without boiling.

9.2.7 Remove samples from Hot Block and record final temperature in digestion log. Allow samples to cool. Bring samples up to a final volume of 50 ml with DI water. Cap and invert several times for proper mixing.

9.2.8 Samples may be allowed to sit overnight while solid materials settle out or samples may be centrifuged for 15 minutes at a rate of 1000 rpm. If samples are centrifuged, all QC samples including the method blank and laboratory control sample (LCS) must also be centrifuged.

9.3 Documentation

9.3.1 Digestion Records

Record the necessary information in the electronic preplog using template version F-MN-I-330-Rev.01. Information includes batch and sample ID, initial and final volumes, prep date, prep analyst, supporting equipment, and lot numbers of solutions used. Also include any additional comments if needed. Save file in prep log with the naming convention;

"Queue HBN Method" Ie. MPRP 555222 6020A

10.0 Data Analysis And Calculations

10.1 Calculations

Refer to associated analytical SOP for equations and common calculations.

11.0 Quality Control and Method Performance

11.1 Quality Control

The following QC samples are prepared and analyzed with each batch of samples. Refer to associated analytical SOP for acceptance criteria and required corrective action.

QC Item	Frequency
Method Blank (MB)	1 per batch of 20 or fewer samples.
Laboratory Control Sample (LCS)	1 per batch of 20 or fewer samples.
Laboratory Control Sample Duplicate (LCSD)	As needed
Matrix Spike (MS)	Prepared with each batch of samples. Client specific requirements may result in a greater number of MS or MS/MSD sets in a batch
Matrix Spike Duplicate (MSD)	1 per batch of 20 or fewer samples.
Sample Duplicate	Performed at client request.

11.2 Method Performance

11.2.1 Method Validation

11.2.1.1 Detection Limits

Detection limits (DL) and limits of quantitation (LOQ) are established at initial method setup and verified on an on-going basis thereafter. Refer to Pace ENV corporate SOP ENV-SOP-CORQ-0011 *Method Validation and Instrument Verification* and to the laboratory's SOP ENV-SOP-MIN4-0163 *Determination of LOD and LOQ* (or equivalent replacement) for these procedures.

11.3 Analyst Qualifications and Training

Employees that perform any step of this procedure must have a completed Read and Acknowledgment Statement for this version of the SOP in their training record. In addition, prior to unsupervised (independent) work on any client sample, analysts that prepare or analyze samples must have successful initial demonstration of capability (IDOC) and must successfully demonstrate on-going proficiency on an annual basis. Successful means the initial and on-going DOC met criteria, documentation of the DOC is complete, and the DOC record is in the employee's training file. Refer to laboratory SOP ENV-SOP-MIN4-0165 *Orientation and Training Procedures* (or equivalent replacement) for more information.

12.0 Data Review And Corrective Action

12.1 Data Review

Pace's data review process includes a series of checks performed at different stages of the analytical process by different people to ensure that SOPs were followed, the analytical record is complete and properly documented, proper corrective actions were taken for QC failure and other nonconformance(s), and that test results are reported with proper qualification.

The review steps and checks that occur as employee's complete tasks and review their own work is called primary review.

All data and results are also reviewed by an experienced peer or supervisor. Secondary review is performed to verify SOPs were followed, that calibration, instrument performance, and QC criteria were met and/or proper corrective actions were taken, qualitative ID and quantitative measurement is accurate, all manual integrations are justified and documented in accordance with the Pace ENV's SOP for manual integration, calculations are correct, the analytical record is complete and traceable, and that results are properly qualified.

A third-level review, called a completeness check, is performed by reporting or project management staff to verify the data report is not missing information and project specifications were met.

Refer to laboratory SOP ENV-SOP-MIN4-0092 *Data Review Process* (or equivalent replacement) for specific instructions and requirements for each step of the data review process.

12.2 Corrective Action

Corrective action is expected any time QC or sample results are not within acceptance criteria. If corrective action is not taken or was not successful, the decision/outcome must be documented in the analytical record. The primary analyst has primary responsibility for taking corrective action when QA/QC criteria are not met. Secondary data reviewers must verify that appropriate action was taken and/or that results reported with QC failure are properly qualified.

Corrective action is also required when carryover is suspected and when results are over range.

Samples analyzed after a high concentration sample must be checked for carryover and reanalyzed if carryover is suspected. Carryover is usually indicated by low concentration detects of the analyte in successive samples analyzed after the high concentration sample.

Sample results at concentrations above the upper limit of quantitation must be diluted and reanalyzed. The result in the diluted samples should be within the upper half of the calibration range. Results less than the mid-range of the calibration indicate the sample was over diluted and analysis should be repeated with a lower level of dilution. If dilution is not performed, any result reported above the upper range is considered a qualitative measurement and must be qualified as an estimated value.

Refer to the associated analytical SOP for a complete summary of QC, acceptance criteria, and recommended corrective actions for QC associated with this test method.

13.0 POLLUTION PREVENTION AND WASTE MANAGEMENT

Pace proactively seeks ways to minimize waste generated during our work processes. Some examples of pollution prevention include but are not limited to: reduced solvent extraction, solvent capture, use of reusable containers for solvent management, and real-time purchasing.

The EPA requires that laboratory waste management practice to be conducted consistent with all applicable federal and state laws and regulations. Excess reagents, samples and method process wastes must be characterized and disposed of in an acceptable manner in accordance with Pace's Chemical Hygiene Plan / Safety Manual.

14.0 MODIFICATIONS

A modification is a change to a reference test method made by the laboratory. For example, changes in stoichiometry, technology, quantitation ions, reagent or solvent volumes, reducing digestion or extraction times, instrument runtimes, etc. are all examples of modifications. Refer to Pace ENV corporate SOP ENV-SOP-CORQ-0011 *Method Validation and Instrument Verification* for the conditions under which the procedures in test method SOPs may be modified and for the procedure and document requirements.

14.1 The preparation method has been modified in terms of the amounts of reagents used and the individual heating times. The chemistry is maintained. Reason for this modification is better performance for silver and antimony. PT samples are analyzed regularly to validate that the modifications are effective. Per the method, the nitric acid and peroxide amounts are varied based on the sample reaction and this is the case with the Pace method. Overall, the Pace digestion ends up with a higher total acid concentration.

14.2 The final volume for the Pace method is 50 mL, opposed to 100 mL for the reference method.

14.3 Samples are processed using the Hot Block digestion system employing metals free disposable plastic ware rather than glass beakers.

15.0 RESPONSIBILITIES

Pace ENV employees that perform any part this procedure in their work activities must have a signed Read and Acknowledgement Statement in their training file for this version of the SOP. The employee is responsible for following the procedures in this SOP and handling temporary departures from this SOP in accordance with Pace's policy for temporary departure.

Pace supervisors/managers are responsible for training employees on the procedures in this SOP and monitoring the implementation of this SOP in their work area.

16.0 ATTACHMENTS

Appendix A – Stock Standard Summary

17.0 REFERENCES

Pace Quality Assurance Manual- most current version.

TNI Standard, *Management and Technical Requirements for Laboratories Performing Environmental Analyses*, EL-V1-2009.

TNI Standard, *Management and Technical Requirements for Laboratories Performing Environmental Analyses*, EL-VI-2016-Rev.2.1.

Test Methods for Evaluating Solid Waste Physical/Chemical Methods, SW-846, Third Edition. Method 3050B.

40 CFR Appendix B to Part 136, *Definition and Procedure for the Determination of the Method Detection Limit - Rev 2*, August 28, 2017.

18.0 REVISION HISTORY

This Version:

Section	Description of Change
8.2	Updated concentration description for the metals spike
9.1.2	Include balance calibration verification
9.2.2.2	Update spike sources and volumes
9.3.1	Provide greater detail for documentation procedure ie batch nomenclature.
Appendix A	Added/updated spike sources
9.1.2	Include balance calibration verification
9.3.1	Provide greater detail for documentation procedure ie batch nomenclature.
9.2.2.2	Update spike sources and volumes

This document supersedes the following document(s):

Document Number	Title	Version
ENV-SOP-MIN4-0056	Metals Preparation of Solid Samples for Analysis by ICP and ICPMS by EPA Method 3050B	03

TEST METHOD STANDARD OPERATING PROCEDURE
TITLE: Metals Preparation of Solid Samples for Analysis by ICP and ICPMS
TEST METHOD EPA Method 3050B
ISSUER: Pace ENV – Minneapolis – MIN4

Appendix A: Metals Standard Reference

Stock standards used for solid sample preparation

METALS-STK1		METALS-STK2		Hg-SPK	
ZPACEMN-116		ZPACEMN-106		MERC-STK1 Stock	
Element	(mg/L)	Element	(µg/L)	Element	(µg/L)
Ca	2000	Si	500	Hg	10000
Fe	2000	Sb	100		
Mg	2000	Mo	100		
K	2000	Sn	100		
Na	2000	Ti	100		
Al	2000	S	2000		
Ba	100	As	100		
Be	100	Pd	20		
Bi	100	Pt	20		
B	100	Se	100		
Cd	100				
Cs	100				
Cr	100				
Co	100				
Cu	100				
Li	100				
P	100				
Mn	100				
Pb	100				
Ni	100				
Ag	50				
Sr	100				
Tl	100				
V	100				
Zn	100				
U	100				
Th	100				

Appendix C
Forms

Appendix C.1 Chain of Custody Form

Appendix C.2 XRF Field Data Sheet

Appendix C.3 XRF Data Validation Checklist

Appendix C.4 Stage 2A Metals Data Validation Checklist

Appendix C.5 Level A-B Screening Checklist

Appendix C.6 Corrective Action Report Template

Laboratory Management Program (LaMP) Chain of Custody Record

Soil, Sediment and Groundwater Samples

BP Site Node Path: _____ **Req Due Date (mm/dd/yy):** _____ Rush TAT Yes _____ No ____x

BP/RM Facility No: _____ **Lab Work Order Number:** _____

Lab Name:	BP/ARC Facility Address:	
Lab Address:	City, State, ZIP Code:	Consultant/Contractor Project No:
Lab PM:	Lead Regulatory Agency:	Address:
Lab Phone:	California Global ID No.:	Consultant/Contractor PM:
Lab Shipping Accnt:	Enfos Proposal No:	Phone: Email:
Lab Bottle Order No:	Accounting Mode: Provision _____ OOC-BU _____ OOC-RM _____	Send/Submit EDD to:
Other Info:	Stage Activity OMM	Invoice To: BP-RM _____ BP-Other _____

BP/RM PM:	Sample Details	Requested Analyses	Report Type & QC Level
			Limited (Standard) Package ____
PM Phone:		Filt	Limited Plus Package ____
PM Email:		Pres	Full Package ____

Lab No.	Sample Description	Date	Time	Field Matrix	Start Depth	End Depth	Depth Unit	Grab (G) or Composite (C)	Total Number of Containers	Analysis	Comments

Sampler's Name:	Relinquished By / Affiliation	Date	Time	Accepted By / Affiliation	Date	Time
Sampler's Company:						
Ship Method: Ship Date:						
Shipment Tracking No:						

Special Instructions:

THIS LINE - LAB USE ONLY: Custody Seals In Place: Yes / No | Temp Blank: Yes / No | Cooler Temp on Receipt: _____°F/C | Trip Blank: Yes / No | MS/MSD Sample Submitted: Yes / No

BPSOU: Unreclaimed Sites Field XRF and Soil pH Results

Site Number: Operator:

Land Use: XRF Unit #:

pH probe #:

*Reference 2021 UR Confirmation Sample Decision Tree for more information on declaring the need for a confirmation sample.

Soil Action/Screening Levels (mg/kg)							
Residential	250			1,200		147	
Non-Residential				2,300			
Recreational	1,000						
Commercial	500						
Storm Water	200	20	1000	1000	1000	10	

XRF Reading #	Sample Name	Depth (inches)	Soil pH (s.u.)	Date Collected	Time Collected	Date Analyzed	XRF Results (mg/kg)						Lab Sample
							As	Cd	Cu	Pb	Zn	Hg	
	BPSOU-UR												
	BPSOU-UR												
	BPSOU-UR												
	BPSOU-UR												
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Data Validation Checklist XRF Sample Analysis

Site: **Case No:** **Laboratory:**
Project: **Sample Matrix:** **Analyses:**
Sample Date(s): **Analysis Date(s):**
Data Validator: **Validation Date(s):**

1. Holding Times

Analyte	Laboratory	Matrix	Method	Holding Times	Collection Date(s)	Analysis Date(s)	Holding Time Met (Y/N)	Affected Data Flagged (Y/N)

*Reference for Holding Times –

Were any data flagged because of holding time? Y [ ] N [X]
What sample preparation steps were performed (i.e. drying, sieving etc.)?
Were the samples prepped according to the SAP/QAPP? Y [X] N [ ]

Describe Any Actions Taken:

Comments:

2. Energy Calibration (System Check)

Was the energy calibration performed at the frequency of once per day? Y [ ] N [ ]
Was the energy calibration Resolution below 195? Y [ ] N [ ]
Did the energy calibration run for at least 50 seconds? Y [ ] N [ ]

Describe Any Actions Taken:

Comments:

3. SiO_2 Standards

Was the SiO_2 Standard analyzed at the beginning of analysis? Y [ ] N [ ]
Was the SiO_2 Standard analyzed at the frequency of 1 per 20 natural samples? Y [ ] N [ ]
Were the SiO_2 Standard results within the control limits? Y [ ] N [ ]
Were any data flagged because of the SiO_2 Standard results? Y [ ] N [ ]

Describe Any Actions Taken:

Comments:

4. Calibration Check Samples

Were the appropriate Calibration Check Samples (CCS) analyzed at the beginning of analysis? Y [ ] N [ ]
Were the appropriate CCS analyzed at the frequency of 1 per 20 natural samples? Y [ ] N [ ]
Were CCS results within the control limits? Y [ ] N [ ]
Were any data flagged because of CCS problems? Y [ ] N [ ]

Describe Any Actions Taken:

Comments:

5. Duplicate Sample Results

Were Duplicate Samples analyzed at the frequency of 1 per 20 natural samples?	Y ☐	N ☐
Were Duplicate Sample results within the control window of ≤ 35% RPD?	Y ☐	N ☐
Were any data flagged because of duplicate sample results?	Y ☐	N ☐

Describe Any Actions Taken:

Comments:

6. Replicate Sample Results

Were Replicate Samples analyzed at the frequency of 1 per 20 natural samples?	Y ☐	N ☐
Were replicate sample results within the control window of ≤35% RPD?	Y ☐	N ☐
Were any data flagged because of replicate sample results?	Y ☐	N ☐

Describe Any Actions Taken:

Comments:

7. Overall Assessment

Are there analytical limitations of the data that users should be aware of?	Y ☐	N ☐

If so, explain:

Comments:

8. Authorization of Data Validation

Data Validator

Name: **Reviewed by:**

Signature: _____ _____

Date: _____ _____

Stage 2A Data Validation Checklist for Metals Sample Analysis

Site: **Case No:** **Laboratory:**
Project: **Sample Matrix:** **Analyses:**
Sample Date(s): **Analysis Date(s):**
Data Validator: **Validation Date(s):**

1. Holding Times

Analyte	Laboratory	Matrix	Method	Holding Times	Collection Date(s):	Analysis Date(s)	Holding Time Met (Y/N)	Affected Data Flagged (Y/N)

Were any data flagged because of holding time? Y ☐ N ☐
Were any data flagged because of preservation problems? Y ☐ N ☐

Describe Any Actions Taken:

Comments:

2. Blanks

Were Method Blanks (MBs) analyzed at the frequency of 1 per analytical batch? Y ☐ N ☐
Were MBs within the control window? Y ☐ N ☐
Were any data flagged because of blank problems? Y ☐ N ☐

Describe Any Actions Taken:

Comments:

3. Laboratory Control Samples

Were Laboratory Control Samples (LCS) analyzed at the frequency of 1 per batch? Y ☐ N ☐
Were LCS results within the control window? Y ☐ N ☐
Were any data flagged because of LCS problems? Y ☐ N ☐

Describe Any Actions Taken:

Comments:

4. Duplicate Sample Results

Were Laboratory Duplicate Samples (LDS) analyzed at the frequency of 1 per batch? Y ☐ N ☐
Were LDS results within the control window? Y ☐ N ☐
Were any data flagged because of LDS problems? Y ☐ N ☐

Describe Any Actions Taken:

Comments:

5. Matrix Spike Sample Results

Were Laboratory Matrix Spike Samples (LMS) analyzed at the frequency of 1 per batch? Y ☐ N ☐
Were LMS results within the control window? Y ☐ N ☐
Were any data flagged because of LMS problems? Y ☐ N ☐

Describe Any Actions Taken:

Comments:

6. Field Blanks

Were field blanks submitted as specified in the Sampling Analysis Plan (SAP)?	Y		N		N/A	
Were field blanks within the control window?	Y		N		N/A	
Were any data qualified because of field blank problems?	Y		N		N/A	

Describe Any Actions Taken:

Comments:

7. Field Duplicates

Were field duplicates submitted as specified in the Sampling Analysis Plan (SAP)?	Y		N		N/A	
Were results for field duplicates within the control window?	Y		N		N/A	
Were any data qualified because of field duplicate problems?	Y		N		N/A	

Describe Any Actions Taken:

Comments:

8. Overall Assessment

Are there analytical limitations of the data that users should be aware of? Y ☐ N ☐

If so, explain:

Comments:

9. Authorization of Data Validation

Data Validator

Name: **Reviewed by:**

Signature: _____ _____

Date: _____ _____

Level A/B Screening Checklist

1. General Information

Site:
Project:
Client:
Sample Matrix:

2. Screening Result

Data are:
1. Unusable _____
2. Level A _____
3. Level B _____

I. **Level A**

Criteria – The following must be fully documented.	**Yes/No**	**Comments**
1. Sampling date		
2. Sampling team or leader		
3. Physical description of sampling location		
4. Sample depth (soils)		
5. Sample collection technique		
6. Field preparation technique		
7. Sample preservation technique		
8. Sample shipping records		

II**. Level B**

Criteria – The following must be fully documented.	**Yes/No**	**Comments**
1. Field instrumentation methods and standardization complete		
2. Sample container preparation		
3. Collection of field replicates (1/20 minimum)		
4. Proper and decontaminated sampling equipment		
6. Field custody documentation		
7. Shipping custody documentation		
8. Traceable sample designation number		
9. Field notebook(s), custody records in secure repository		
10. Completed field forms		

Corrective Action Report/ Corrective Action Plan

Project ID	Project Name	Document ID

Preparer's Signature/Submit Date	Submitted to:

Description of the requirement or specification	
Reason for the Corrective Action	
Location, affected sample, affected equipment, etc. requiring corrective action	
Suggested Corrective Action	(Continue on Back)
Corrective Action Plan	(Continue on Back) ☐ Approval signature/date:_____
	Approval of corrective actions required by EPA? ☐ Yes ☐ No ☐ EPA approval name/date:_____ ☐ Corrective actions completed name/date:_____
Preventative Action Plan	(Continue on Back) ☐ Preventative actions completed name/date:_____

Corrective Action Report/
Corrective Action Plan

Suggested Corrective Action (Continued)	
Corrective Action Plan (Continued)	
Preventative Action Plan (Continued)	

Appendix D
Revision Log

Appendix D.1
Summary of Revisions

Rev. No.	Year	Description
1	2021	Distribution lists: Updated to current distribution list. Updated text to reference BPSOU CD and Field Sampling Plans (FSPs) rather than sampling and analysis plans (this affected Section 2). Section 2.1: Updated Project Organization and Responsibilities • Updated Atlantic Richfield QAM to David Gratson • Updated Atlantic Richfield Liability Manager Title (Mike Mc Anulty Atlantic Richfield) • Updated Operations Manager (Eric Hassler) • Added Brandon Warner as BSB Field Team Supervisor Section 2.2 and Section 2.3: Updated text to reference the BPSOU CD and specify metals-impacted sediment. Section 2.4. Updated Step 2: Identify the Goals of the Study to include: Are contaminants, if present on site, the result of historical mining operations or related activities? Minor word changes in Step 4 and Step 7 for clarification. Section 2.6.7: Added metals-impacted to clarify type of sediments. Added Section 3.1 Site Evaluation Objectives, which changed all the section 3 headings after it. Section 3.3.2 Sedimentation Analysis (previously Section 3.2.2): Added metals-impacted to clarify type of sediments. Section 6 References: added the BPSOU CD information. Appendix A: Figures/Charts • Updated A.1 – Updated BPSOU Area Map to revised BPSOU boundary in the Consent Decree • Updated A.2 – Organization Chart • Updated A.3 – Decision Logic • Updated A.4 – Precision, Accuracy, and Completeness Calculations Appendix B: SOP Updates • SOP-SA-04 – revised 11/12/2020 • SOP-DE-02 – revised 09/08/2020 Appendix C: Updated forms Appendix D: Added changes to previous revision.
2	2022	Table 1 revised Hg action levels per UR RFC-01.

Rev. No.	Year	Description
		Section 2.1 – Organization name updates. Section 2.4 - DQO steps and Accuracy criteria updated. Table 3 – Minimum Detection Limits updated. Section 2.6 - Data retrieval information for validation added. Section 3 - Table 4 Updated SOPs. Updated sampling procedures for general sampling. Updated sample naming structure. Table 5 updated for mercury. Added Section 4 - Data Management. Section 6 – Data Validation and Usability Revised. Section 7 – References Updated.