Winter 2-28-2022

Residential Metals Abatement Program Quality Assurance Project Plan (Non-Residential Parcels - Indoor Dust)

Elsie King

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Environmental Resource Management (ERM)

UNITED STATES ENVIRONMENTAL PROTECTION AGENCY
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Ref: 8MO

March 22, 2022

Mr. Mike Mc Anulty
Liability Manager
Atlantic Richfield Company
317 Anaconda Road
Butte, Montana 59701

Re: Approval letter for the Butte Priority Soils Operable Unit (BPSOU) Draft Final Residential Metals Abatement Program (RMAP), Quality Assurance Project Plan (QAPP), Non-Residential Parcels – Indoor Dust (dated February 28, 2022)

Dear Mike:

The U. S. Environmental Protection Agency (EPA), in consultation with the Montana Department of Environmental Quality (DEQ), is approving the *Draft Final Residential Metals Abatement Program (RMAP), Quality Assurance Project Plan (QAPP), Non-Residential Parcels – Indoor Dust (dated February 28, 2022),* with the following comments

- If the content or the technical approach provided in the plan has changed or requires modification, please submit the revised plan to EPA and DEQ for review.
- Please submit and distribute the Final QAPP with the attached signature/approval page and the EPA approved crosswalk.

If you have any questions or concerns, please call me at (406) 457-5019.

Sincerely,

Nikia Greene
Remedial Project Manager

Attachments:
EPA crosswalk
EPA and DEQ Signature Page

cc: (email only)
Butte File
Jenny Chambers; DEQ
Matt Dorrington, DEQ
Daryl Reed; DEQ
Will George; DEQ
Jon Morgan; DEQ counsel
Carolina Balliew; DEQ
Harley Harris; NRDP
Katherine Hausrath; NRDP
Jim Ford; NRDP
Ray Vinkey; NRDP
John Gallagher; BSBC
Sean Peterson; BSBC
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Eric Hassler; BSBC
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Chris Wardell; EPA
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Jean Belille; EPA
Ian Magruder; CTEC (Tech Advisor)
Janice Hogan; CTEC
Kristi Carroll; Montana Tech Library

Residential Metals Abatement Program Quality Assurance Project Plan (Non-Residential Parcels - Indoor Dust)

Atlantic Richfield Company and Butte-Silver Bow County

28 February 2022

Project No.: 0612471

The business of sustainability

ERM

Signature Page

28 February 2022

Residential Metals Abatement Program Quality Assurance Project Plan (Non-Residential Parcels - Indoor Dust)

Atlantic Richfield Company and Butte-Silver Bow County

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CONTENTS

List of Tables (in Text)

List of Tables (Attached)

List of Figures

Acronyms and Abbreviations

Name	Description
Agencies	U.S. Environmental Protection Agency and Montana Department of Environmental Quality
AR	Atlantic Richfield Company
BPSOU	Butte Priority Soils Operable Unit
BSB	Butte-Silver Bow
CERCLA	Comprehensive Environmental Response, Compensation, and Liability Act
CFRSSI	Clark Fork River Superfund Site Investigation
COC	constituents of concern
DEQ	Montana Department of Environmental Quality
DM/DV	Data Management/Data Validation
DSR	Data Summary Report
DQA	Data Quality Assessment
DQO	Data Quality Objective
EDD	electronic data deliverable
GPS	Global Positioning System
HAZWOPER	hazardous waste operations and emergency response
HEPA	high-efficiency particulate air
HVS3	high-volume small surface sampler
ICP-MS	inductively coupled plasma mass spectrometry
LCS	laboratory control sample
MDL	method detection limit
mg/kg	milligrams per kilogram
MS	matrix spike
MSD	matrix spike duplicate
LMS	laboratory matrix spike

PARCCS	precision, accuracy, representativeness, comparability, completeness, and sensitivity
Program	Butte-Silver Bow County Multi-Pathway Residential Metals Abatement Program
QA/QC	quality assurance/quality control
QAPP	Quality Assurance Project Plan
RL	reporting limit
RMAP	Residential Metals Abatement Program
ROD	Record of Decision
RPD	relative percent difference
SDG	Sample Delivery Group
SOP	standard operating procedure
USEPA	U.S. Environmental Protection Agency
XRF	x-ray fluorescence

APPROVAL PAGE

Butte Priority Soils Operable Unit Residential Metals Abatement Program Quality Assurance Project Plan (Non-Residential Parcels – Indoor Dust)

Approved: NIKIA GREENE Digitally signed by NIKIA GREENE Date: 2022.03.22 06:18:47 -06'00' Date: _____

Nikia Greene, Remedial Project Manager
U.S. Environmental Protection Agency, Region 8
Quality Assurance Approval Official

Approved: _____ Date: 3/21/2022

Daryl Reed, Project Officer
Montana Department of Environmental Quality

Approved: _____ Date: 3/15/2022

Eric Hassler, Director
Department of Reclamation and Environmental Services
Butte-Silver Bow County

Approved: Michael C McAnulty Date: 3/15/2022

Mike Mc Anulty, Liability Manager
Atlantic Richfield Company

The Quality Assurance Project Plan is effective on date of approval.

DISTRIBUTION LIST

Butte Priority Soils Operable Unit Residential Metals Abatement Program Quality Assurance Project Plan (Non-Residential Parcels – Indoor Dust)

QAPP Recipients	Title	Organization	Telephone Number	E-mail Address
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A complete list of personnel to receive this document is provided on the associated cover letter distribution list. Atlantic Richfield Company will distribute the original Agency approved document.

1. INTRODUCTION

The Butte-Silver Bow County (BSB) Multi-Pathway Residential Metals Abatement Program (RMAP) (BSB and Atlantic Richfield 2020) (hereafter referred to as the Program or the RMAP) is designed to mitigate exposure of residents of the Butte Priority Soils Operable Unit (BPSOU), the larger Butte community as a whole, as well as rural residential development within the Silver Bow Creek/Butte Area Superfund Site to sources of arsenic, lead, and mercury contamination. The current Program boundary (depicted as the 2020 RMAP Area Boundary) is shown on Figure 1.

The contamination may originate from both mining-related (waste rock, tailings, aerial emissions) and non-mining-related sources. The Program uses remediation and abatement of contaminated properties, and community awareness and education to ensure its effectiveness.

The Program requires systematic sampling of residential yard soil and interior dust within the BPSOU. Presently, no interior dust data for schools is available. For areas outside of BPSOU, but within the 2020 RMAP Area Boundary (Figure 1), the Program also requires systematic sampling of playground and play areas (e.g., schools and parks). Interior assessments and sampling of interior dust in non-residential schools, preschools, and non-residential daycares (see Figure 2) will be addressed in this Quality Assurance Project Plan (QAPP). A separate QAPP addresses external soil sampling of non-residential parcels (schools, parks, non-residential daycares) that fall under the RMAP umbrella. Additionally, a separate QAPP addresses the assessment of residential RMAP parcels/properties.

The Program contains additional institutional control measures regarding education, outreach, and tracking programs related to remedial activities at residential properties, as further described in the BPSOU *Institutional Controls Implementation and Assurance Plan (ICIAP)* (Atlantic Richfield 2019a).

1.1 Purpose

The *BPSOU Quality Management Plan (QMP)* (Atlantic Richfield 2016) provides guidance to ensure quality environmental data collected for the BPSOU meet requirements mandated by the U.S. Environmental Protection Agency (USEPA). The purpose of this QAPP is to provide guidance for future RMAP indoor sampling and analyses of non-residential properties (e.g., schools, preschools, and non-residential daycares) and to describe the quality assurance/quality control (QA/QC) policies and procedures to be used during these efforts. This QAPP functions as the RMAP sampling and analysis plan for all future non-residential sampling activities. A separate QAPP has been developed to address residential BPSOU RMAP parcels (including residential daycares and commercial properties containing living space).

This QAPP includes standard recognized elements referenced in the *EPA Requirements for Quality Assurance Project Plans, EPA QA/R-5* (USEPA 2001); the *Guidance on Systematic Planning Using the Data Quality Objectives Process, EPA QA/G4* (USEPA 2006a); and the *EPA Region 8 QA Document Review Crosswalk* checklist (USEPA 2017) provided in Appendix A. This QAPP includes the following four key elements:

- Program management and organization (Section 2)
- Measurement and data acquisition (Section 3)
- Assessment and oversight (Section 4)
- Data review and usability (Section 5)

The sections below provide the project elements and include details for planning, sampling, and analyses within the Program areas. Sections in this QAPP expand on or reference information in other site-wide documents and present project-specific requirements.

2. PROGRAM MANAGEMENT AND ORGANIZATION

This section addresses Program and project administrative functions as well as project background, objectives, and documentation requirements for sampling and analyses activities on each project site within the Program area. Figure 3 summarizes the project personnel involved in the planning, approval, and implementation of this QAPP. Project personnel roles are described below. Responsibilities of personnel in each of these roles are described below.

2.1 Agency Oversight

The USEPA and Montana Department of Environmental Quality (DEQ) (the Agencies) are responsible for project oversight, review, and approval of all Program-generated sampling data and subsequent site-specific remediation plans. The USEPA or a USEPA contractor will provide oversight during site reconnaissance and sampling activities. The USEPA Remedial Project Manager is Nikia Greene and the DEQ Project Officer is Daryl Reed.

The Agencies also review sampling results above action levels listed in Table 1, and project completion reports.

2.2 Atlantic Richfield Company

Atlantic Richfield Company (Atlantic Richfield) provides Program funding through an Allocation Agreement between BSB and Atlantic Richfield. The Atlantic Richfield Liability Manager, Mike Mc Anulty, must authorize all reclamation activities under the Program. An Atlantic Richfield project representative, or designated alternate, may complete a site walkthrough and assist with site-specific work plan approval of all reclamation projects prior to implementation.

At this time, it is anticipated that Atlantic Richfield will elect to self-perform portions of the RMAP sampling and analysis work in consultation with BSB representatives.

2.3 Butte-Silver Bow County Department of Reclamation and Environmental Services

BSB is responsible for supporting the indoor dust investigation effort at schools and daycares, maintaining Program data, and supporting any future abatement activities. Key individuals comprising the BSB County Department of Reclamation and Environmental Services are shown on Figure 3.

2.4 Analytical Laboratory

Pace Analytical Laboratories, LLC, contracted to work on this Program's project, must ensure that the laboratory's QA personnel are familiar with this QAPP and are performing the analytical and QC work as specified per laboratory methods and this QAPP. Laboratory QA personnel are responsible for reviewing final analytical reports produced by the laboratory, coordinating the laboratory analyses schedule, and supervising in-house chain-of-custody procedures.

2.5 Data Validation Consultant

The data validation consultant Environmental Standards, Incorporated provides independent third-party QA oversight and will be primarily responsible for assessing/monitoring the data collection and analysis activities performed by project personnel relative to this QAPP. The consultant is responsible for:

- Evaluating accuracy and condition of sample receipt documentation;
- Coordinating receipt of data packages and electronic data deliverables (EDD) from the laboratories;

- Routinely communicating with the laboratories regarding status and resubmission of data deliverables;
- Coordinating the activities of staff chemists who are validating laboratory-produced data in a manner consistent with the QAPP validation protocols;
- Performing senior review of reports;
- Downloading unqualified EDDs and uploading qualified EDD from/to the Atlantic Richfield (AR) EQuIS database; and
- Notifying the Quality Assurance Officer of issues relating to the quality or validity of laboratory data, and/or delivery schedules.

In addition, the data validation consultant will complete a Level A/B review during the verification process for field documentation related to samples collected for laboratory analyses for determination of screening or enforcement quality data for each school. Finally, the data validation consultant will complete field and laboratory audits in accordance with the QAPP.

2.6 Indoor Dust Investigation Consultant

ERM, the environmental consultant contracted to perform the indoor dust investigations, is responsible for developing planning documents (QAPP, field sampling plans, health and safety plans, etc.), performing the indoor dust investigations, and preparing summary reports to document the results of the indoor dust investigations. The environmental consultant will work with all the entities listed above during the successful completion of the investigation. Elsie King is the ERM Quality Manager responsible for maintaining the official, approved QAPP.

2.7 Problem Definition and Background

The USEPA has included schools (public and private schools, daycares, and preschools) in the RMAP in the First Amendment to the Administrative Order (USEPA Docket No. Comprehensive Environmental Response, Compensation, and Liability Act [CERCLA]-08-2011-0011). Currently, there is no indoor dust data for schools and indoor school dust sampling will be performed to determine if indoor dust levels of lead, arsenic, and mercury are above the current residential cleanup levels. Contamination of schools described herein may originate from both mining-related (waste rock, tailings, aerial emissions) and non-mining-related sources (e.g., lead paint or broken mercury thermometers). This component of the RMAP Program evaluates arsenic, lead, and mercury present in interior dust.

Sampling and assessment are needed to determine remediation or abatement requirements if:

- Accessible interior dust exceeds solid media action levels in areas currently accessible to students or daycare children. Accessible dust is surface dust located in areas that are commonly occupied by students or daycare children, such as classrooms, hallways, bathrooms, and other areas (e.g., cafeterias) within the school or daycare.
- Inaccessible space dust exceeds solid media action levels in areas mainly accessible to facility staff. Inaccessible dust is surface dust found in locations such as boiler or mechanical rooms, tops of ceiling tiles, janitorial closets, on ventilation system ductwork or vents, and storage rooms in areas that are not commonly accessed or occupied by students or daycare children.
- For buildings constructed in or before 1980, dust in attics and/or crawlspaces exceeds solid media action levels where there is an exposure pathway to an interior occupied space. Information on attics and/or crawlspaces with elevated dust levels should made available to facility personnel performing maintenance activities to mitigate the potential for future exposures.

This QAPP was developed in response to the Agencies 2006 *Record of Decision, Butte Priority Soils Operable Unit, Silver Bow Creek/Butte Area NPL Site* (BPSOU ROD) (USEPA 2006b) and *Explanation of Significant Differences to the 2006 Butte Priority Soils Operable Unit Record of Decision* (USEPA 2011a). This QAPP was also developed in response to the Agencies 2020 Unilateral Administrative Order Amendment (UAO Amendment) for "Partial Remedial Design/Remedial Action Implementation and Certain Operation and Maintenance at the Butte Priority Soils Operable Unit/Butte Site (EPA Docket No. CERCLA-08-2011-0011) (USEPA 2020a). The UAO Amendment expanded the RMAP boundary (see Figure 1) and also expanded the Program to include schools, parks, and daycare facilities.

2.8 Project Description and Schedule

The Program is designed to mitigate exposure to sources of arsenic, lead, and mercury contamination to residents of the BPSOU and the 2011 Residential Metals Expanded Area (Expanded Area) shown in Figure 1. Contamination in the Expanded Area may originate from both mining-related (waste rock, tailings, aerial emissions) and non-mining-related sources.

In 2019, the Program was expanded to perform both residential attic and yard sampling within the 2020 RMAP Area Boundary provided on Figure 1. Specific exclusion areas are also identified on Figure 1. Sampling residential yards and attics outside of the BPSOU but within the expanded boundary will be performed on a test-by-request basis. In 2020, the Program boundary was expanded further, and the scope modified to include schools as additional property types to the RMAP statement of work.

Components of the Program include environmental sampling and remediation, long-term tracking and data management, and education and outreach. Medical monitoring is conducted as a sister program to the Program. Long-term tracking and data management ensures properties will be sampled, evaluated, and remediated, if necessary. The tracking portion provides a record of changes in ownership and notes permits issued by BSB government for remodeling homes in which attic dust sampling found contamination above action levels, but a pathway did not exist when the assessment was completed. The long-term tracking and data management will be continued for the life of the Program. The BPSOU *Final Data Management Plan* (Atlantic Richfield 2017) describes data management. The BPSOU Data Management Plan is being updated and the 2020 version of the document is currently under review. The final, approved version of the Data Management Plan will ultimately be the governing document for this QAPP. Only validated data will be uploaded to the Program database.

The Program stipulates sampling residential yard and school playground soil, interior dust, for all constituents of concern (COC) and interior air monitoring for mercury vapor within the BPSOU. The Program includes systematic sampling of additional specific areas within the 2020 RMAP Area such as parks and play areas, schools, and commercial areas with accessible (living and interior school) space based on site-specific conditions and evidence of exposure pathways. Program eligibility is described in the *Revised Final Multi-Pathway Residential Metals Abatement Program (RMAP) Plan* (BSB and Atlantic Richfield Company, 2020).

2.8.1 Project/Task Description

This QAPP will guide data collection activities at the schools in 2021 and 2022. Data generated from the samples will be used to address questions regarding arsenic, lead, and mercury in interior dust that may be identified within the schools and the potential for students and school personnel to contact interior dust with arsenic, lead, and mercury at concentrations that exceed residential cleanup levels (250 milligrams per kilogram [mg/kg] arsenic, 1,200 mg/kg lead, and 147 mg/kg mercury). No interior dust data for schools are currently available. This sampling will address that data gap.

This work is designed to be in general conformance with the residential dust indoor sampling previously conducted by AR. AR conducted this sampling to address concerns by the community over potential arsenic, lead, and mercury concentrations in interior dust.

2.8.2 Project Schedule

A high-level indoor school dust investigation and remediation schedule is provided on Figure 4. Submittal of school/daycare-specific field sampling plans (FSPs) should occur within 30 days following agency approval of this QAPP. Dust investigation field work should begin within 30 days following agency approvals of FSPs, which will occur during school breaks, on weekends, or after hours while school is in session. Samples will be analyzed and Level 4 data packages provided in 10 to 12 business days, and data validation will occur within 7 business days following Level 4 data package receipt. Investigation Summary and Data Summary Reports will be submitted approximately 3 months after all data validation activities are completed.

2.9 Quality Objectives and Criteria

This section discusses the internal QC and review procedures used to ensure that all data collected for this project are of known quality. The Data Quality Objectives (DQO) were developed in accordance with the USEPA's *Guidance on Systematic Planning Using the Data Quality Objectives Process* (USEPA 2006a). The DQOs are statements that define the type, quality, quantity, purpose, and use of data to be collected. The USEPA developed a seven-step process to establish DQOs to help ensure that data collected during a field sampling event are adequate to support reliable site-specific decision-making (USEPA 2001; USEPA 2006a). The sections below outline the QAPP DQOs.

2.9.1 Data Quality Objectives

The DQO process specifies project decisions, the data quality required to support those decisions, specific data types needed, data collection requirements, and analytical techniques necessary to generate the specified data quality. The process also ensures justification of the resources required to generate the data. The DQO process consists of seven steps of which the output from each step influences the choices that will be made later in the process:

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

During the first six steps of the process, the planning team develops decision/performance criteria that will be used to develop the data collection design. The final step of the process involves developing the data collection design based on the information from the other steps. The following provides a brief discussion of these steps and their application to this sampling effort.

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.*

> **Describing the problem**. Properties in Butte and within the 2020 RMAP Expanded Area (see Figure 1) have the potential to be contaminated by historical mining activities and related

contaminants. The proximity of properties to mining wastes and operations may have resulted in contamination of non-residential properties such as schools, preschools, and non-residential daycare facilities.

The presence of contaminants and exposure pathways, related and non-related to historical mining activities, may result in a health-based risk to users of non-residential properties.

Establishing the planning team. Project personnel, roles, and responsibilities are detailed in Sections 2.1 through 2.6 of this document.

Describing the conceptual model of the potential hazard. Historical surface and underground mining activities resulted in the presence of contaminants in soil and interior dust around Butte due to waste dumping and deposition of aerial emissions from smelters/mills. Other, non-mining sources (e.g., lead-based paint, broken mercury thermometers) have also resulted in contamination in some areas. Contaminants in soil may be transferred to indoor dust when people enter the building (e.g., carried in on shoes or clothing) or through open doors and windows via windblown airborne particulates. People may contact contaminated dust at non-residential properties through pathways such as inhalation, which can also result in incidental ingestion when dust particles are inhaled and then swallowed, and through incidental ingestion due to hand-to-mouth contact with dust-laden surfaces. When people contact contaminated dust, they may be exposed to contaminants, which could pose a health risk if concentrations are above health-protective concentrations. The residential lead, arsenic, and mercury soil action levels established for the Program account for and are applicable to indoor dust contribution to total exposures. The Program has also established a residential action level for mercury vapors in indoor air. In order to investigate this problem, data quantifying contaminant concentrations in indoor dust, and when applicable, mercury vapor, will need to be collected, compared to the appropriate project action levels, and used for remedial decision-making.

Identifying available resources, constraints, and deadlines. Atlantic Richfield (Section 2.2), BSB (Section 2.3), and their support contractors will provide necessary project resources (financial and staffing) to properly implement the Program. Project schedule details are provided in Section 2.8 and 2.8.1.

Step 2: Identify the Goals of the Study. *This step identifies what questions the study will attempt to resolve and what actions may result.*

Key elements/questions. The Program requires that all area schools and non-residential daycare facilities within the 2020 RMAP Expanded Area be sampled and assessed based on the sample decision framework specified on Figure 5. The goal is to use best efforts to obtain access to all applicable non-residential schools, daycares, and preschools within the 2020 RMAP Expanded Area (see Figure 1) to complete an interior dust investigation. Exterior soil sampling at schools, preschools, and non-resident daycares was addressed in a separate QAPP (ARCO/BSB 2021). Interior dust investigation/sampling are addressed in this QAPP.

Specifying the primary question. The primary question to be addressed is the following:

Are indoor dust concentrations of arsenic, lead, and/or mercury at these non-residential properties present at levels that may pose a risk to human health (e.g., above the action levels)? If action levels are exceeded, can the source of the exceedance be ascertained (e.g., historic smelter emissions, lead-based paint, track-in from outside, historic mining operations, or some other source)?

Specifically, these study questions can be detailed and broken down further as follows:

i. Are indoor dust concentrations of arsenic, lead, and/or mercury in currently accessible areas of non-residential properties greater than the BPSOU soil/dust action levels?

ii. Are indoor dust concentrations of arsenic, lead, and/or mercury in inaccessible areas of non-residential properties greater than the BPSOU soil/dust action levels?

iii. Do attics and/or crawlspaces have dust concentrations of arsenic, lead, and/or mercury greater than the BPSOU soil/dust action levels?

iv. Is lead, arsenic, and/or mercury being tracked into schools from outside sources?

v. If mercury dust concentrations exceed the action level, are mercury vapor concentrations in indoor air greater than the BPSOU mercury vapor action level?

Determining alternative actions. For all schools and daycares, indoor dust shall be collected from entrance floor mats and floor surfaces in accessible areas. For buildings constructed prior to 1980, indoor dust shall be collected from inaccessible surfaces and attics/crawlspaces. As appropriate, opportunistic sampling of visible surface dust will be performed in accessible areas when present. Possible alternative actions, as depicted in Figure 5, are as follows:

- Take no action. If indoor dust concentrations of lead, arsenic, and mercury are below their respective BPSOU residential soil/dust action level, no further action is needed.
- Perform indoor mercury vapor sampling: if mercury dust results exceed the BPSOU residential soil/dust action level, indoor mercury vapor sampling would be necessary. In this event, a separate site-specific sampling plan will be prepared to investigate the source of the mercury and to measure mercury vapor concentrations in indoor air. The objectives, sampling design, and analytical methods for mercury vapor sampling will be documented in a separate plan that would be submitted to the Agencies for review and approval.
- Perform lead paint analysis. If lead dust concentrations exceed the BPSOU residential soil/dust action level, interior and/or exterior paint analysis may be necessary to identify the lead source. In this event, a separate site-specific sampling plan will be prepared to investigate the source of lead and, if appropriate, discuss the need to perform additional sampling (e.g., interior or exterior paint). This separate plan would be submitted to the Agencies for review and approval.
- Complete remedial action. If indoor dust concentrations of lead, arsenic, and/or mercury are greater than or equal to their respective BPSOU residential soil/dust action level, remedial actions would be necessary. Remedial actions would consist of indoor dust removal or containment. Removal action may include location- and media-specific cleaning, use of a remediation grade/high-efficiency particulate air (HEPA) filter vacuum, carpet replacement, insulation replacement, or other appropriate means. Containment measures may include the use of sealants, coverings, or other physical migration pathway termination options.

Specifying the decision statement. The decision statement is as follows:

- Determine whether mercury vapor sampling is required.
- Determine whether lead paint analysis is required.
- Determine whether remedial action (indoor dust removal or containment) is required.

Step 3: Identify the 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.*

Identifying the type of information that is needed to resolve the decision statement. Arsenic, lead, and mercury concentrations should be determined through sampling indoor dust from non-residential RMAP properties (schools, preschools, and non-residential daycares). The goal of indoor dust collection and analysis is to obtain a reliable estimate of the average concentration of a COC in dust over a specified decision unit area where exposure may occur, for comparison to the appropriate action level for that area. The relationship between the average COC concentration and the action level provides the input needed to resolve the decision statements outlined in Step 2 in order to determine whether abatement is required for non-residential RMAP dust.

Information about the use of, or the presence of exterior soil COC action level exceedances at, the different schools/daycares should inform the sampling design for each property. Property use information should be used to make decisions about the appropriate sample count/density (such as a representative number of floors to be sampled).

Sample coordinates and location information such as the property type (e.g., school, preschool, non-residential daycare), sample type (e.g., floor mat, floor surface, accessible surface), and location sampled (e.g., entrance, classroom, gym, inaccessible area, etc.) should also be documented so that sample results are linked to specific locations to inform remediation decisions. This information will also inform the use of specific data. For example, entrance floor mat and inaccessible area samples are useful for determining the source of contaminants present in dust, while floor or accessible surface samples provide data to assess potential exposures. If chips from building interior lead-based paint are identified in a sampled area, this should also be documented as it is likely to influence lead concentrations in dust.

Identifying the number of variables to be collected. Arsenic, lead, and mercury concentrations (in mg/kg) should be determined for each dust sample collected from entrance floor mats, accessible floor surfaces and inaccessible surface locations, and attics/crawlspaces. Other variables to be collected include:

- Sample coordinates
- Property type
- Locations sampled (i.e., which rooms)
- Sample type (e.g., floor mat, floor surface, etc.)
- Surface area sampled
- Presence of lead paing chips
- Potential exposure routes between occupied spaces and attics/crawlspaces
- Time of year and antecedent weather conditions

Identifying the appropriate action levels. Action levels developed for BPSOU soils are also applicable for dust. For Butte, there are no school-specific soil action levels. Therefore, the basis of the existing soil action levels (as presented in the BPSOU ROD) was reviewed to determine which type of action level is likely to be the most applicable and adequately protective level to employ in making cleanup decisions for the schools. The non-residential soil action level for lead (2,300 mg/kg) has historically been applied to address waste rock dumps and source areas, which are different from the types of materials expected at schools. The recreational soil action level for arsenic (1,000 mg/kg) was developed based on a dirt-bike riding scenario, which is an activity that is quite different from anticipated use of school property. There is no non-residential soil action level for mercury.

Based on a review of the basis of the soil action levels, the residential soil action levels should be employed in evaluating the dust sampling results for the schools. The application of the residential action levels is conservative for a school scenario; however, use of more conservative action levels is appropriate, especially considering the school setting and community sensitivity to childhood exposures. The use of the residential action levels in making cleanup decisions for interior dust is consistent with what has been done historically for Butte parks and exterior school/daycare surface soils.

The BPSOU residential action levels (arsenic: 250 mg/kg, lead: 1,200 mg/kg, mercury: 147 mg/kg) will be utilized for all work completed under this QAPP (see Table 1).

Identifying appropriate sampling and analysis methods. Multiple sampling strategies (discrete, defined surface area, composite, etc.) should be considered for potential use on this project. Given the varying size and configuration of the indoor spaces contemplated for this project, exclusive discrete sampling may not be the most appropriate option given its common deficiencies including poor spatial coverage, inadequate sample density, or data that cannot be used to statistically represent the entire area of interest with a reasonable level of confidence. Composite sampling and defined surface areas sampling methods should also be contemplated to collect data representative of the various decision units that will be defined in school/daycare-specific FSPs. In some instances discrete grab sample collection may be required and appropriate. A minimum of 2 grams of dust is typically needed to perform laboratory analysis; thus, the sampling method must allow for sufficient mass for analysis.

X-ray fluorescence (XRF) has been used historically to analyze arsenic and lead concentrations in Butte soils and may be helpful during interior dust investigations. This method provides a quick output that can be used for immediate decision-making. However, it is less sensitive than laboratory analytical methods, and cannot be used for mercury analysis. Because samples must be packaged and shipped on ice (<6 °C) to a laboratory for mercury analysis, it is more practical to have all three metals analyzed by the laboratory via EPA methods SW6020B and SW7471B. The sensitivity of these methods will meet the data quality objectives for both soil and interior dust. Data from an analytical laboratory can also be validated. Expedited laboratory analysis (5 to 7 business day turn around on data and Level 2 data packages and 10 to 12 business day turn around on data and Level 4 data packages) and data validation (7 business day turn around after Level 4 data packages are received) options should be investigated in order to achieve the project assessment and remediation goals.

Step 4: Define the Boundaries of the Study. *The purpose of this step is to define the spatial and temporal boundaries of the problem.*

Specifying the target population. The 2020 RMAP/Program area addressed under this QAPP will include indoor dust within schools and non-residential daycares identified on Figure 1.

Describing what constitutes a sampling unit. Sampling units should be defined based on interior school use information. Sampling unit extents are defined as the maximum area to be sampled to support decision-making (see Step 3). The USEPA's Superfund Lead-Contaminated Residential Sites Handbook (USEPA 2003), previous RMAP QAPP, and procedures for sampling schools in nearby Anaconda were reviewed to inform sampling unit extents appropriate for the interior dust investigation. The recommendations below were developed consistent with USEPA recommendations, other RMAP sampling efforts, and sampling of schools where similar types of contamination are present. These recommended sampling unit extents should inform development of the sampling plans for each appropriate school or daycare building.

Sampling units should be defined based on the area where dust may be contacted (for accessible spaces) or from which a pathway might occur currently, or in the future (for inaccessible spaces). Because dust and vapor can move around within an indoor space, the samples collected from these media should be representative of the entire space where students and faculty spend time. For example, the routinely accessible interior space where students and faculty may contact dust includes entryways, hallways, classrooms, etc.; all of these spaces together should be considered part of one or more sampling units since they are connected and transfer between areas can occur. Other spaces routinely accessed by faculty include administrative and engineering offices, store rooms, boiler rooms etc., which should also be considered as part of one or more sampling units. If part of the school or daycare has a different use, such as a gymnasium or lunch room separated from the classrooms and hallways, or if the accessible space is separated by dedicated entrances, multiple floors, or separate buildings, the areas within or adjacent to the structure should be considered separate sampling units because different exposures may be applicable for each.

The availability of dust, the presence of dust accumulation on structures (counter tops, window sills/tracks, suspended lighting, I-beams, HVAC vents, etc.), and the relative ease or difficulty to clean an area or available infrastructure within the subject area must be factored into decisions regarding sampling unit size or extent. Additionally, the type of remedial measure, or the extent of remediation that may be required if constituent concentrations exceed action levels must be considered.

Interior inaccessible spaces from which a pathway to accessible spaces may originate include attics and crawl spaces. As with accessible spaces, since dust can move around within an attic or crawl space, the sampling unit should include the whole space. Pathways for transport of dust from inaccessible space to accessible space within the schools and daycares should be determined during pre-sampling site visits and re-confirmed, as necessary, during subsequent dust sampling visits.

Schools and non-residential daycares differ in size, shape, and complexity. As a result, setting standards for establishing sampling units at all schools and daycares is impractical. Sampling units will be defined in school/daycare-specific FSPs. The onsite USEPA representative will be consulted to determine the number of representative rooms, hallways, etc. to establish sampling units for dust sample collection at each school/daycare.

Time frame for collecting data and making the decision. Interior school/daycare sampling should be completed when school is in session, in a manner that does not interfere with student learning. Outreach meetings should be conducted with each school to better understand individual schedule restraints (beginning and end of the school day and any after-school activities, construction projects, etc.) The collection of floor mat dust samples will occur during a season when track-in will be maximized (e.g., moist spring conditions).

Specifying the scale for decision-making. For the non-residential RMAP schools/daycares, the sampling unit extents for each building subarea should be specified as the maximum area for decision-making to identify any location where arsenic, lead, or mercury concentrations are above health-protective action levels and need to be remediated. By setting the decision unit equal to the sampling unit, decisions to remediate can be made for subareas of a building, rather than on a building-wide basis, and any subarea with analyte concentrations above action levels can be addressed even if building-wide remediation is not warranted. A decision to remediate a larger area could be informed by multiple sampling or decision units. There is potential for multiple sampling units and decision units within a building and within a subarea of the building. A sampling unit could be: a single large room or space (i.e. – a gymnasium); a group of collocated

rooms with similar characteristics; a single floor; an attic or a crawl space; or a small school or building. A sampling unit could also include specific parts of a room, group of collocated rooms, or other space such as counter tops, window sills/tracks, suspended lighting, I-beams, HVAC vents, etc. The number and types of locations and surfaces that constitute a single sampling unit will depend on the details (e.g., layout and use) of the property being sampled. Due to the varying size, configuration, and complexity of the various RMAP Area schools and daycares, the scale and number of school/daycare-specific sampling units and decision units will be defined in their respective FSPs.

Step 5: Develop the Analytic Approach. *The purpose of this step is to define the parameters of interest and integrate any previous DQO inputs into a single statement that describes a logical basis for choosing among alternative actions.*

Identifying the population parameters most relevant for making inferences and conclusions on the target population. Arsenic, lead, and mercury concentrations should be measured for each sampling unit as determined by analysis of each corresponding dust sample collected. As described in Step 3, the relationship between the average COC concentration and the action level provides the input needed to resolve the decision statements outlined in Step 2 in order to determine whether abatement is required for non-residential RMAP dust. Therefore, the average concentration is the population parameter of interest. Because estimation of average concentrations from a population of sample results can lead to decision errors (as described further in Step 6), collection of individual samples that capture the average concentration across a potential exposure area (such as composite samples) is preferable to reduce decision error. Each sample result, representing the average concentration for each sampling unit, can then be compared to the action level. The average concentration measured in each sampling unit is the population parameter that should be used to make inferences and conclusions for each decision unit (i.e., the decision unit should be set equal to the sampling unit to support health-protective decision-making).

Specifying the theoretical decision rule. The theoretical decision rule is as follows. If the analyte concentration measured in the sampling unit (i.e., the average concentration within each decision unit for either arsenic, lead, or mercury) exceeds the appropriate residential action level detailed in Table 1, then remedial action to remove or contain the dust must be performed. This includes accessible spaces and inaccessible spaces where a pathway exists allowing dust transport to accessible spaces.

Step 6: Specify Performance or Acceptance Criteria. *The purpose of this step is to identify baseline conditions, limits, and ranges for decisions and consequences of decision errors.*

The decision question identified in Step 2 is: Are dust concentrations of arsenic, lead, and/or mercury at non-residential properties present at levels that may pose a risk to human health (e.g., above the action levels)? In this case, the baseline condition for each decision unit is that the analyte concentration in dust is below the action level, and the alternative condition is that there is an exceedance. Because this is a decision question, the potential exists for decision error to occur due to variability and uncertainty in the data. Potential decision errors include Type I (or false positive) and Type II (or false negative) errors. In the context of the RMAP non-residential sampling decision question, a false positive would mean determining that the arsenic, lead, or mercury concentration in dust is above the action level when in fact it is not. Consequences of this type of error include unnecessary remedial action and increased costs. A false negative would mean concluding that the arsenic, lead, or mercury concentration in dust is below the action level when it is actually above the action level. Consequences of this type of error include

leaving dust in place that contains a metal at concentrations above the action level, resulting in a potential risk to human health.

Because the goal of the RMAP is to protect human health, the tolerance for making a Type II (false negative) error is lower than the tolerance for making a Type I (false positive) error.

Therefore, a sampling design and analysis method that minimizes the potential for false negative decision errors should be selected. Due to the potential for work to occur over more than one semester and the need to make decisions on a building-by-building, or room-by-room basis as determined by the property-specific FSP, the experiment-wise error rate will likely be difficult to assess and efforts should be made to reduce the Type II error rate at the decision unit, rather than at the project-wide level.

When discrete sampling methods are used and the resulting population of sample data representing each decision unit are compared to a standard using hypothesis testing, the chance of making a Type I error can be reduced by setting a lower significance level (i.e., a lower Type I error rate). The chance of making a Type II error is reduced by setting a higher statistical power. The significance level and power can be raised or lowered to control the probability of each type of error depending on the tolerance for each. With this type of approach, there is a set tolerance for reaching a conclusion (the action level is or is not exceeded) that is correct for most, but not all, values in a population. Typically, the probability of a Type I error is lower than that of a Type II error; for example, a significance level of 0.05 and a power of 80 percent (0.2 probability of Type II error) are often selected. It can be difficult to obtain the sample size needed to achieve a much higher statistical power due to limitations such as the area available for sampling and associated analytical costs.

For the non-residential RMAP, the tolerance for Type II decision errors is lower than that for Type I errors. Because of the difficulties in lowering the Type II error rate that are associated with approaches such as hypothesis testing, an alternative approach may be preferable. Instead of addressing the decision question through hypothesis testing or estimating an upper confidence limit on the mean concentration using a population of discrete samples collected across a non-residential building (i.e., setting the entire building as the decision unit), the size of the decision unit can be reduced to maximize the potential to find an exceedance where present (i.e., to lower the Type II error rate). If each sample result is compared individually to the action level, this eliminates the chance for a percentage of the sample results to be incorrectly identified as being below the action level, as can occur when the entire population is being compared across a larger decision unit.

In addition to lowering the potential for Type II errors, study error should be minimized through proper training of the field sampling team, sample documentation and handling, the use of appropriate analytical methods that achieve method detection limits (MDLs) below the action levels, analysis of field and analytical QC samples, analysis of precision, accuracy, and other measurement performance criteria (described in detail in Section 2.9.2), and data validation.

Decisions should be made using data that meet the performance and acceptance criteria; if these criteria are not met, corrective action steps should be taken.

Step 7: Develop the Plan for Obtaining Data. *The purpose of this step is to develop an optimized plan to complete the task.*

Selecting the sampling design. The data collection scheme is designed to ensure that the information will be of sufficient quality and quantity to determine the component(s) of individual schools, preschools, and non-residential daycares requiring remedial action (and the extent to which remedial action is required). The information and outputs generated in Steps 1 through 6 of

the DQO process informed selection of the optimized approach for dust sampling and analyses at non-residential RMAP properties described in this final step of the process. The data collection design (sampling program) is described in detail in Section 3.

Specifying the QA/QC procedures. Sufficient data quality will be achieved through the field and laboratory quality control measures (Sections 3.9 and 3.10) including the use of appropriate sample collection, handling, and chain-of-custody procedures and laboratory analytical methods, quality control sample analysis (field and laboratory), assessment of the performance criteria described in Section 2.9.2, following the corrective action procedures detailed in Sections 4.1 and 4.2, and analytical data validation (Section 5).

2.9.2 Measurement Performance Criteria for Data

Measurement performance criteria are established by defining acceptance criteria and quantitative or qualitative goals (e.g., control limits) for precision, accuracy, representativeness, comparability, completeness, and sensitivity (PARCCS) of measurement data. The definitions of PARCCS are provided below. Acceptance limits are detailed in Section 3.9.2 for each measurement performance criteria. Equations for calculation of precision, accuracy, and completeness are provided in Table 2. Additional QC acceptance criteria are provided in Table 3.

2.9.2.1 Precision

Precision is the amount of scatter or variance that occurs in repeated measurements of a particular analyte. Precision is assessed using the relative percent difference (RPD) between a primary sample result and its paired field or laboratory duplicate sample result (for field and laboratory precision, respectively). For example, perfect precision would be a 0 percent RPD between the primary sample result and its paired field or laboratory duplicate sample result (both samples have the same analytical result). For these sampling events, precision will be assessed based on laboratory prepared and field duplicate sample analysis.

2.9.2.2 Accuracy/Bias

Accuracy is the ability of the analytical procedure to determine the actual or known quantity of a particular substance in a sample. Accuracy is assessed based on the percent recovery and percent difference of various laboratory QC samples. Perfect percent recovery is 100 percent and perfect percent difference is 0 percent (the analysis result is exactly the known concentration of the QC sample). The laboratory control sample (LCS) and laboratory matrix spike (LMS) are used to measure accuracy, based on the percent recovery of the LMS and LCS. Additional laboratory QC samples (serial dilution samples, interference check samples, calibration standards, calibration blanks and method blanks) may be used to assess accuracy as appropriate to the analytical method.

Bias is the systematic or persistent distortion of a measurement process that causes error in one direction (e.g., consistently higher or lower than the true concentration). As with accuracy, analytical bias can also be assessed based on percent recovery of laboratory QC samples. Sampling bias is addressed by use of proper sampling design and methods.

2.9.2.3 Representativeness

Representativeness is the degree to which sample data represent a characteristic of a population, parameter, or environmental condition. Representativeness is a qualitative parameter that is most concerned with proper design of the sampling and analytical schemes. Representativeness is achieved by determining the number and locations of samples and the appropriate sampling techniques needed to depict, as accurately and precisely as necessary, the conditions being measured. Representativeness

deals with protocols for sample storage, preservation, and transportation; analyzing samples with appropriate methods, techniques, and instrumentation; and using the methods to document these protocols. Representativeness will be achieved through judicious selection of sampling locations and methods. This QAPP requires that samples are representative of the medium being sampled and that there are enough samples to meet the project DQOs and satisfy the project remedial action design elements.

2.9.2.4 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 and applicable laboratory standard operating procedures (SOP), as well as the Program SOPs, which are comparable to the sampling methods used during previous investigations at the site (Appendix B contains various field and laboratory SOPs). 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. Dust data will be reported in units of mg/kg.

2.9.2.5 Completeness

Completeness is a measure of the amount of valid data obtained from the measurement system. Proposed sample collection points may fail to produce usable data for many reasons (e.g., non-traceable sample identification, sample container breakage, elevated storage temperature, exceeded sample holding time, or data loss). When samples are analyzed, but the data are rejected, the numerator of this calculation becomes the number of valid results minus the number of possible results rejected. Valid data are data not rejected or deemed unusable during the data validation process. Completeness describes the amount of valid data that meets the DQOs for representativeness, accuracy, and precision versus the amount of data obtained or considered necessary to achieve a specific level of confidence in decision-making. For relatively clean, homogeneous matrices, data would be expected to be 100 percent complete. As matrix complexity and sample heterogeneity increases, however, completeness may decrease. Based on the complexity of sample matrices anticipated to be collected from the project sites; the analytical data completeness goal following validation is stated to be greater than or equal to 90 percent and will be generated on a Sample Delivery Group (SDG) basis.

Project completeness with regard to the collection of samples and identified data gaps will be addressed by the data generators and users. A goal of 90 percent is anticipated for each project location (e.g., each school location).

In order to more accurately depict the percent analytical completeness, individual analyte completeness will be calculated and reported. In the event re-analyses are performed by the laboratory, only a single analytical set (possibly a mixture of original and re-analyses data based on usability) will be included in the analytical completeness calculation so as not to count duplicate data. Valid results used to meet completeness objectives are those results that provide a defensible estimate of the true concentration of an analyte in a sample. These valid results include data that are not qualified and data that are qualified but that can still be used to meet project objectives. Invalid data are those results for which there is an indication that the prescribed sampling or analytical protocol was not followed, or results did not meet QC specifications.

2.9.2.6 Sensitivity

Sensitivity is related to the ability to compare analytical results with project-specific action levels. Analytical quantitation limits for the sample analytes should be below the level of interest to allow an effective comparison. The MDLs for arsenic, lead and mercury are included in Table 1.

Achieving proper sensitivity (i.e., reporting limits [RL]) will depend on instrument sensitivity and potential matrix effects. Data sensitivity is the ability of the analytical method to differentiate the target analyte from instrument "noise." It is important to monitor the instrument performance to verify consistent instrument performance at the low end of the calibration range. Instrument sensitivity will be monitored through analysis of method blanks and calibration check samples. Project data will be reported to the MDL with variations due to sample amount digested, potential dilutions and percent moisture correction for mercury analysis. The MDLs are below the action limits defined in the DQO steps above.

Additional details regarding bias, sensitivity, and QC acceptance criteria are included in Section 3.9.2.

2.10 Special Training

All ERM field personnel will review the requirements of this QAPP and receive training on Program-related tasks during a project meeting held prior to the beginning of fieldwork. A review of sampling procedures and requirements will be completed prior to field activities so that sample collection and handling methods are performed according to QAPP requirements. Field personnel will be trained in proper use of field equipment, sample collection tools, etc., and procedures according to field data collection SOPs (Appendix B) and methods described in the Program. Field personnel performing sampling activities or members who can potentially contact contaminated materials should receive hazardous waste operations and emergency response (HAZWOPER) training.

One hard copy of the approved version of this QAPP will be maintained for reference in the field vehicle and/or field office. All field team personnel will have access to Portable Document Format (.pdf) files of the complete QAPP.

2.11 Documents and Records

This section describes procedures for documentation management and record keeping for this QAPP from initial record generation through final data formatting and storage. All sampling data conducted for all media under the Program and records of property access requests are housed within the Program (RMAP) database. The Program database is housed in an Access Structured Query Language (SQL) server database and maintained by BSB. Document backups are contained in the BPSOU document SharePoint and USEPA document repository. Refer to the BPSOU *Final Data Management Plan* (Atlantic Richfield 2017 or most current revision) for additional details regarding data management, backup, and storage. Atlantic Richfield and BSB will coordinate Agency testing of the Program database with the Program architects and primary users in a manner to minimize provision of written comments and the potential misinterpretation of those comments. All data collected during interior dust investigation of the Butte RMAP schools, preschools, and non-residential daycares, as described in this QAPP will be uploaded to the Program database.

2.11.1 Property Access Agreements

An executed sampling access agreement (see Appendix C) must be obtained before sampling takes place. Program access agreements are also described in detail within the *Institutional Controls Implementation and Assurance Plan (ICIAP)* (Atlantic Richfield 2019a). The agreements represent a temporary agreement between Atlantic Richfield and school/daycare officials stating that Atlantic Richfield and its contractors are permitted to conduct certain sampling activities at the specified school/daycare.

Completed agreements will be photocopied, scanned, and the electronic version stored. The status of property access will be tracked in the Program database tracking system. A copy of the access agreements (Appendix C) will also be included in the project record files.

2.11.2 Field Sampling Plans

FSPs will be prepared for individual schools or daycares, or for groups of schools and daycares. Grouping will occur with similarly sized or closely located schools and daycares, where applicable and appropriate. The FSPs will be prepared for review and approval by the Agencies prior to conducing field sampling activities.

2.11.3 Field Documentation

Field documentation provides a description of site conditions during sampling activities and provides a permanent record of all field activities. Field documentation will primarily be achieved through field notes, data collection forms or electronic means (i.e., field tablets). Field documentation includes a sample location map that shows school buildings, rooms, structures, and features relevant to the interior dust sampling effort.

Documentation for each site will include the information listed below, at a minimum:

- A description of the field task
- Time and date fieldwork started
- Location and description of the work area including sketches, if possible, map references, and references to photographs collected
- Names and titles of field personnel
- Name, address, and phone number of any field contacts or site visitors (e.g., Agency representatives, auditors, etc.)
- Details of the fieldwork performed with special attention noted to any deviation from the QAPP or applicable field SOPs. Such deviations will be brought to the attention of and discussed with Agency field oversight personnel. If the deviations are deemed to be minor by the Agency representative, a resolution and path forward will be determined in the field. If the Agency representative determines that the deviation is major in scope, it will be his/her responsibility to elevate the question internally and to receive Agency direction.
- All field measurements made (e.g., areas sampled, HVS3 pressure readings, micro-vacuum flow rates, sample masses)
- Personnel and equipment decontamination procedures

For any field sampling work, the field documentation will include all applicable items from the Level A/B assessment checklist (see Section 5.1.2.1 and Appendix D). At a minimum this includes documentation of the following:

- Sample team and/or leader
- Sample location, and traceable sample designation number
- Sample type collected
- Date and time of sample collection
- Sampling method, particularly any deviations from the field SOPs (Appendix B)

- Documentation or reference of preparation procedures for reagents or supplies that will become an integral part of the sample (if any used in the field); specify if sample bottles/preservatives are not provided by the laboratory and certified as cleaned
- Collection of field duplicates and information on the associated parent sample
- Decontamination of sampling equipment
- Sample custody documentation
- Sample preservation (if used)

Sufficient information should be recorded to allow the sampling event to be reconstructed without having to rely on the sampler's memory.

A report containing all the above-listed information will be provided to the school/daycare official and the information recorded in the Program database and tracking system and uploaded to cloud-based databases managed by BSB (BPSOU *Final Data Management Plan* [Atlantic Richfield 2017 or most recent revision]). Sample results will be validated, and Agency approved prior to submission to property owners unless otherwise approved by the Agencies.

2.11.4 Field Photographs

Field personnel will use a digital camera to take photographs at the site. Photographs may be taken of sampling locations, field activities, and documenting site conditions, as necessary.

Photographs should include a scale in the picture when practical. Documentation of all photographs taken during sampling activities will be recorded in a bound field logbook or appropriate field collection device and will specifically include the following for each photograph taken:

- The date, time, and site identification
- A brief description of the subject and the fieldwork portrayed in the picture
- Sequential number of photograph

Electronic files will be placed in project files with copies of supporting documentation from the bound field logbooks/data collection device.

2.11.5 Chain-of-Custody Records

Each sample collected will be assigned a unique sample number, and the sample container will be labeled with sample designation number, date and time of collection, and requested analyses. Then the information will be recorded in the field documentation. Chain-of-custody records document the traceability of samples from the time of collection until final disposition. After samples have been collected, they will be maintained under strict chain-of-custody protocols in accordance with the SOPs (Appendix B). A chain-of-custody record will be initiated by the individual physically in charge of the sample collection. The chain-of-custody form may be completed concurrently with the field sampling or before shipping or hand delivery of samples to the laboratory. The sampler is personally responsible for the care and custody of the samples until they are shipped, or hand delivered to the laboratory. When transferring the sample possession, the individual relinquishing and receiving the sample will sign and record the date and time of day on the chain-of-custody record.

A copy of each as-transmitted chain-of-custody form will be scanned and stored on a hard drive. Chain-of-custody records will also be copied to the project record files (refer to Section 3.15). The chain-of-custody records will be included in the laboratory data packages.

2.11.6 Analytical Laboratory Records

Results received from the laboratories will be documented both in report form and in an electronic format. Laboratory documentation includes laboratory confirmation reports such as information on how samples have been batched, the analyses requested, data packages containing the laboratory report and the EDD, and any change requests or corrective action requests. Section 5.1.2.2 lists the laboratory reporting requirements in detail. The deliverable (data package or report) issued by the laboratory must include data necessary to complete Stage 2B and Stage 4 validation of laboratory results. Original reports and electronic files received from laboratories will be maintained with the Program quality records. Refer to the BPSOU *Final Data Management Plan* (Atlantic Richfield 2017 or most recent revision) for additional requirements.

2.11.7 Project Data Reports

Upon receipt of laboratory results and completion of the data review/validation process, all analytical data will be uploaded into a Program database and submitted to the Agencies for review and approval. For the school sampling portion of this project, these data would be anticipated to be submitted on a per school/daycare basis to decrease the turnaround time required for reporting as much as possible. Upon receiving Agency approval, the sample results (for all analytes) will be reported to school/daycare officials along with a letter explaining what the results indicate (see result letter templates in Appendix E). The action levels for arsenic, lead, and mercury will be reported along with sample results.

Following landowner notification, sample results will be used to develop an individual site work plan for each school/daycare remedial action where sample results exceeded BPSOU action levels (Table 1). In addition to the “real time” submittals described above, all sampling data will be forwarded to the Agencies for review and approval in the form of a Data Summary Report (DSR). This DSR will include figures displaying location of buildings/rooms sampled, analytical results, and copies of all field data. As described above, all sampling data will reside in the project records.

Sampling for remedial design/remedial action under the RMAP will be documented through an interior dust sampling DSR submitted for review and approval by the Agencies. Sample data, with their laboratory and data usability qualifiers, will be maintained electronically by BSB/Atlantic Richfield and reported in an interior dust sampling report. The interior dust sampling report will be a DSR prepared based on the guidelines in *Clark Fork River Superfund Site Investigations (CFRSSI) Pilot Data Report Addendum* (AERL 2000) following interior dust data collection. The final report will describe the interior dust sampling activities, provide a summary of the data obtained, discuss the results of data validation, and provide a detailed listing of any deviations from the QAPP. The DSR will also include a data usability assessment for laboratory data. A data summary table with all the samples and analyte concentrations listed, along with the laboratory- and data validation-assigned qualifiers will also be included. The Level A/B checklists, laboratory data validation checklists, and data validation summary will provide an overall assessment of the quality and usability of the data. Furthermore, the DSR will also contain copies of all analytical reports, EDDs, and data validation reports. The DSR will be submitted to the Agencies for review approximately 3 months after all data validation activities are completed for the interior dust sampling.

2.11.8 Quality Records

Quality records are defined as completed, legible 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 consultant, and will include the following at a minimum:

- This QAPP and any approved revisions or addenda
- Approved versions of the Health and Safety Plan and any addenda

- Copies of field SOPs for field data collection, with any updates, revisions, or addenda to those SOPs
- Incoming and outgoing project correspondence (letters, telephone conversation records, and faxes)
- Copies of completed access agreements (Appendix C) for the individual schools/daycares sampled
- Individual school/daycare maps, including any field drawings and field photographs
- Field documentation forms
- Copies of all field documentation/records
- Copies of all sample chain-of-custody forms
- Copies of all laboratory agreements and amendments
- Laboratory data packages (electronic version)
- Documentation of field and/or laboratory audit findings and any corrective actions
- Draft and final delivered versions of all reports and supporting procedures such as statistical analyses, numerical models, etc.

3. MEASUREMENT AND DATA ACQUISITION

This section addresses all aspects of project design and implementation for generating and acquiring data. Adhering to the procedures provided in Appendix B in this QAPP and described in this section result in conformance to requirements specified in the appropriate methods or procedures for sampling, sample handling, laboratory analyses, field and laboratory QC, instrument/equipment testing, inspection, maintenance, instrument/equipment calibration, data management, and data security.

3.1 Property Access

Non-residential RMAP sampling will occur at public and private schools, daycares, and preschools. Prior to conducting any sampling or cleanup activities, access must be provided from authorized school/daycare officials in the form of an executed sampling access agreement (see Appendix C).

Any dispute concerning access should be brought to the attention of the Agencies. It is essential to begin access procurement as early as possible in the remedial process to avoid potentially lengthy delays. If access for response work cannot be reasonably obtained, the USEPA may choose to use its authorities under CERCLA to secure access, as provided in the current Unilateral Administrative Order (USEPA 2011b) and any updated Unilateral Administrative Orders.

3.2 RMAP Indoor Dust Sampling Design

The primary goal of the sampling is to provide data to measure concentrations of COCs in dust in representative accessible areas within the schools and daycares in the Program area. All school/daycare RMAP dust sampling work will be conducted in accordance with Figure 5, and as described below to determine the presence of the COCs listed in Table 1. Field personnel will follow the procedures in the SOPs (Appendix B) and will record all information in the field logbook/data collection device. The procedures for RMAP dust sampling are summarized below.

3.2.1 Sample Locations

Sample locations will be defined in individual school/daycare field sampling plans or grouped school/daycare field sampling plans developed separately from this QAPP.

3.2.2 Entrance Floor Mat Dust Sampling

Schools and daycares typically use floor mats just inside the buildings at points of entry to reduce tracking of dirt through the interiors. The field sampling team will consult with USEPA to obtain replacement mats for collection of dirt at building entrances. At all schools/daycares, replacement mats will be put in place the week prior to the interior sampling to collect samples under typical conditions to determine if COCs are being tracked into the schools. This will provide useful information should concentrations of COCs be found above the residential cleanup levels in the accessible interior floor and surface dust samples. Results from floor mat sampling are intended to provide information on the potential source of those contaminants (interior versus exterior), not to measure exposure.

3.2.3 Floor Surface Sampling

A representative number of floors will be vacuumed using the HSV3 under typical conditions to obtain dust samples for analysis of COCs in readily accessible interiors within all schools/daycares. These data will be compared to residential cleanup levels to determine if COCs are present in concentrations exceeding cleanup levels. Efforts will be made to collect sufficient sample mass with the HSV3, including sampling in additional room areas. If dust is not present in sufficient concentrations to sample or if the

concentrations are below the residential cleanup levels, potential exposure to COCs in interior dust will be considered negligible and no additional investigation will be required of the school interiors.

3.2.4 Surface Dust Sampling

3.2.4.1 Accessible Surface Sampling

Floor surface sample results will be used to assess surface dust in accessible areas of schools and daycares. However, there may be circumstances where an opportunistic micro-vacuum surface dust sample may be collected to provide useful information on surface dusts within accessible areas (e.g., top of cabinets, bookshelves) if visible dust is observed. These surface sampling results will be used to determine if arsenic, lead, and/or mercury is present in concentrations exceeding cleanup levels.

3.2.4.2 Inaccessible Surface Sampling

For buildings constructed prior to 1980 (that have not undergone remodeling or had an interior remediation since this time), micro-vacuum surface dust samples will be collected from areas typically inaccessible to students (e.g., boiler or mechanical rooms, tops of ceiling tiles, janitorial closets, ventilation system ductwork or vents, storage rooms, I-beams, etc.). These sample results are intended to provide information on exposure potential to facility staff performing maintenance or other functions in these areas. In addition, these samples may also provide information on the potential source of contaminants if elevated concentrations are present in floor dust samples.

3.2.4.3 Attic and Crawlspace Sampling

For buildings constructed prior to 1980 (that have not undergone remodeling or had an interior remediation since this time), micro- vacuum surface dust samples will be collected from attic and crawlspaces if there is an exposure pathway to an occupied space. These dust samples will provide information on the potential source of contaminants if elevated concentrations are present in floor dust samples.

3.2.5 Grab Samples

Grab dust samples may be collected at certain locations where sufficient quantities of dust are present, or where composite vacuum sampling cannot be completed due to sample media limitations (i.e., insulation in attics). In these instances, dust samples may be collected using new, disposable paintbrushes and properly decontaminated dust pans.

3.3 RMAP Indoor Soil Sampling

All RMAP soil sampling work inside school properties will be conducted as described below to determine the presence of the COCs listed in Table 1. Field personnel will follow the procedures in the SOPs (Appendix B) and will record all information in the field logbook/data collection device. The procedures for RMAP soil sampling are summarized in section 3.3.1 and 3.5.5.

For non-residential earthen basement sampling components, subsamples will be collected from a minimum of 3 subsample locations or at a rate of approximately 5 subsamples per 5,000 square feet (ft^2) in surface area per sampling component, whichever is greater. Subsamples from these locations will be composited in the field, and a single composite sample from the 0- to 2-inch depth interval will be analyzed for arsenic, lead, and mercury. Each subsample should have similar mass so that each location is equally represented in the total sample mass. The maximum area represented by a single composite sample will be 10,000 ft^2 (meaning a maximum of 10 subsamples will be collected from any non-residential sampling component).

3.4 Mercury Vapor and Paint Sampling

When RMAP mercury vapor and/or paint sampling is required, the procedures to be used will be included in an agency approved site-specific field sampling plan.

3.5 Field Procedures

The field sampling includes floor mat, floor surface, surface sample dust, and earthen basement soil collection. Each of these activities is described below. Digital photographs with a minimum resolution of at least 640x480 pixels will be taken at each sample site and appropriate information will be recorded in the field logbook following the protocols set forth in the SOPs in Appendix B. The location of the sample will be sketched in the field book.

3.5.1 Floor Mat Sampling

Floor mats will be placed just inside the main entryways of the schools/daycares 1 week prior to performing interior dust sampling. The mats will be secured with duct tape to make sure they are not cleaned or removed. Placement of the mats will be coordinated with the school/daycare. The mats will be checked daily and will be left in place for a period of 5 days, or until the surface appears to be overloaded with tracked dirt, whichever comes sooner. At the end of the 5-day period or when the mat becomes overloaded, it will be sampled in place. The mat will be vacuumed by the high-volume small surface sampler (HVS3) by subjecting it to three to four passes over the entire carpeted area of the mat, until all the dust has been removed. The HVS3 high-volume vacuum will be used to collect dust from the mat as specified in ASTM International (ASTM) D5438-17, *Standard Practice for Collection of Floor Dust for Chemical Analysis* (Appendix B). A floor mat blank sample will be collected at the beginning of each sampling event as described in Section 3.10.5.

There is a possibility that due to weather conditions (frozen ground, spring snowstorm, etc.) that insufficient soil will be tracked in to generate dust. In that event, a second floor mat sampling event will be scheduled later when school is session. The decision to conduct a second round of floor mat sampling event will be made by AR and USEPA after obtaining the first round of sampling results.

3.5.2 Floor Surface Sampling

Dust sampling will be performed on flooring in a representative number of typically accessible interior spaces. The locations will be selected following a field reconnaissance of the school/daycare buildings, and as specified in an Agency-approved Field Sampling Plan. The HVS3 vacuum will be used to collect dust from the flooring as specified in ASTM D 5438-17, *Standard Practices for Collection of Floor Dust for Chemical Analysis* (Appendix B). Before samples are collected, the date of the last cleaning will be determined and recorded on the sampling form. The sampling team will vacuum the selected floor location until enough dust (ideally 6 to 8 grams) has been collected. The sampling team will then estimate the floor area sampled so that an estimate of dust density can be provided in the data summary report. Acceptable methods to estimate floor area include counting floor tiles or using a measuring tape.

Based on the type of surface, the HVS3 will be set up to the appropriate pressure drop and flow rate. The sample collection bottle will be pre-weighed and recorded and attached to the vacuum. Sampling will attempt to collect 6 to 8 grams of dust to allow an adequate amount for duplicates, matrix spikes (MS), and re-analysis. This may be difficult due to local COVID-19 pandemic cleaning requirements. A minimum of 2 grams of dust is typically needed to perform laboratory analysis for both USEPA Methods 6020B and 7471B. If a smaller amount is collected, the RLs may be elevated, and it may not be possible to analyze both methods. The analysis of arsenic and lead will be prioritized over the mercury analysis. The HSV3 will be cleaned with reagent grade methanol between each sample per the ASTM D 5438-17 specification.

3.5.3 Surface Sampling

Dust samples will be collected from a representative number of typically inaccessible areas within the schools/daycares. The locations to be sampled will be determined by a field reconnaissance of the buildings and documented in Agency-approved field sampling plans. Samples will typically be micro-vacuumed from multiple sub-locations (a minimum of two) within the area sampled to form a composite sample, typically in the same room or space (e.g., mechanical room). Samples in inaccessible locations with heavy dust may also be collected using a disposable paintbrush and properly decontaminated dustpan

The samples will be collected using a micro-vacuum as specified in ASTM D 7144-21, *Standard Practice for Collection of Surface Dust by Micro-vacuum Sampling for Subsequent Determination of Metals and Metalloids* (Appendix B). The micro-vacuum collects dust using a collection nozzle attached to a filter holder (sampling cassette) connected to an air sampling pump. Samples will be collected on 37-millimeter (mm) two-piece air sampling cassettes with matched-weight mixed cellulose ester (MCE) filters. Prior to sampling, ten unused filters (from the same filter lot) will be weighed to establish an average filter weight. Sample weights will be calculated by subtracting the average filter weight from the measured filter plus sample weight for each sample. A separate filter cassette will be collected for each method: USEPA Methods 6020B and 7471B. A minimum sample mass of 0.05 grams will be needed for each method. Filter lot blank samples will be analyzed for arsenic, lead, and mercury prior to use of the cassettes in the field. A sampling pump flow rate of 2.5 liters per minute (L/min) will be used initially for surface dust sampling. If this does not allow collection of adequate sample mass, the flow rate will be increased to 6.0 ± 0.5 L/min.

3.5.4 Grab Samples

Grab samples may be collected using a disposable paintbrush and properly decontaminated dustpan. Other opportunistic samples may be collected with the HVS3 or micro-vacuum, based on observations by the field sampling team and any accompanying oversight.

3.5.5 Soil Samples

Interior soil sample collection is not expected to be a component of the interior dust investigation at the majority of RMAP Area schools and daycares. Soil sampling will be included in school/daycare-specific FSPs, following Agency coordination and site inspection. Such sampling will only be included and performed for interior soils where student and faculty exposure concerns exist.

Soil samples will be collected from the 0 to 2-inch depth interval, when necessary. Sampling crew personnel will follow the steps listed below:

1. Ensure that an executed sample request form (refer to Section 2.11.1 and Section 3.1) exists prior to beginning any sampling event.
2. Visually inspect the property to determine the number of polygons needed for composite sampling.
3. Take photographs to create a record to document the pre-sampling condition of all portions of the property scheduled to be sampled. At the end of the project, a copy of the record is provided to the owner. Copies will also be made available for review by the Agencies.
4. Create a scaled sample location map of each basement that shows boundaries of exposed soil. The sample location map will be developed using conventional and representative methods (i.e., computer or tablet devices). Use measuring devices (standard measuring tape, or laser measuring devices) to accurately measure basement features within an accuracy of approximately plus or minus 2.0 feet. Divide each basement into polygons for sampling and identify these areas on the map. All

subsample locations will be plotted on the sample location map by sampling crews in the field. The map should include the following at a minimum:

- Surface area applicable to each individual basement component
- Number of subsamples required from each basement component (based upon component surface area).
- Surface area applicable to the exposed basement soil boundary of each property
- Location of miscellaneous structures (walls, doors).
- Any noticeably dissimilar soil material types or surface conditions (i.e., bare ground areas, areas where paint chips were observed, locations of obvious imported fill materials, etc.).

5. For each composite sample, label the bag with the correct sample identification number (see Section 3.8).
6. Collect composite samples as dictated by the Sample Location Map (placing each composite sample in the corresponding bag).
7. Follow chain of custody procedures outlined in the Sample Management work instructions (Attachment C).
8. Ensure all sampling identification information is entered into the Program's database tracking system.
9. Duplicate field samples will be collected as described in Section 3.10.1

3.6 Field Equipment

The following field equipment is required:

- QAPP, field notebook, pens, camera, and batteries
- Maps of proposed sampling locations and Global Positioning System (GPS)
- HSV3 vacuum floor sampler (1)
- Surface dust micro-vacuum (1)
- Tweezers to remove hair balls and dust balls from samples, dry brush, and wet wipes
- Floor mats
- Heavy-duty contractor trash bags and duct tape
- Digital scale for weighing sample bottles before and after vacuuming
- Sample bottles for HVS3 vacuum
- Filters for micro-vacuum
- Paper towels, deionized water, sprayer, lab-grade methanol
- Health and safety gear (work gloves, flashlight, safety glasses, first aid kit, and ear protection, as the HVS3 is noisy)

3.7 Sample Handling and Chain of Custody

After collection and labeling, the samples will be maintained under strict chain-of-custody protocols, in accordance with the sample packaging SOP (Appendix B). The field sampling personnel will complete a chain-of-custody form for each individual school/daycare shipment/delivery (i.e., batch of coolers) of

samples to be delivered to the laboratory for analysis. The coolers containing dust samples will be shipped from the field on ice to the Pace Analytical Laboratory located in Minneapolis, Minnesota (1700 Elm Street SE, Minneapolis, MN 55414) for analysis. Jennifer Anderson is the Pace Analytical point of contact.

The sampler is responsible for initiating and filling out the chain-of-custody form. The chain of custody for a shipment/delivery will list only those samples in that shipment/delivery. Any documentation, including chain of custody, should be placed inside a re-sealable plastic bag, within the shipment/delivery container. Coolers that are to be shipped will be custody sealed, securely taped shut, and have a shipping label securely adhered to the cooler.

The sampling personnel 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 of the samples is transferred to a designated laboratory, a courier, or to another project employee for the purpose of shipping the samples to the designated laboratory. Custody is transferred when both parties to the transfer complete the portion of the chain of custody under "Relinquished by" and "Received by." Signatures, printed names, company names, dates, and times are required. Upon transfer of custody, the sampling personnel who relinquished the samples will retain the third sheet (pink copy), photocopy, or electronic copy of the chain of custody. When the samples are shipped by a common carrier, a Bill of Lading supplied by the carrier will also be used to document the sample custody, and its identification number will be entered on the chain of custody.

Copies, receipts, and carbons of bills of lading will be retained as part of the permanent documentation in the project file. It is not necessary for courier personnel to sign the chain of custody.

Upon receipt by the laboratory, the samples will be inspected for sample integrity. The chain of custody will be immediately signed, dated, and reviewed by laboratory personnel to verify completeness. Any discrepancies between the chain of custody and sample labels and any problems or questions noted upon sample receipt will be communicated immediately to the field team leader. The laboratory will provide the field team leader and/or the consultant QA manager with a copy of the chain of custody and associated sample-receipt information within 2 working days of receipt of samples. The sample-receipt information routinely provided will include sample receipt date, sample IDs transcribed from the chain-of-custody sample matrix type, and list of analyses to be performed for each sample. Broken custody seals, damaged sample containers, sample labeling discrepancies between container labels and the chain-of-custody form and analytical request discrepancies will be noted on the chain-of-custody form. This information is reviewed by the data validation consultant to verify sample labeling and resolve integrity issues. The field team leader and QA manager will be notified of any such problems and the discrepancies or non-conformances resolved and addressed before the samples are analyzed.

The laboratory will be responsible for following their internal custody procedures from the time of sample receipt until sample disposal. Samples and extracts will be stored in a secure area controlled by the laboratory's designated sample custodian. Samples will be removed from the shipping container and stored in their original containers unless damaged. Damaged samples will be disposed of in an appropriate manner after notifying the field team leader and consultant QA manager, and authorization to dispose is received and documented. In addition, samples will be stored after completion of analyses in accordance with contractual requirements.

3.8 Sample Identification

The RMAP sample identification procedures are detailed in this section. An alphanumeric coding system will be used to uniquely identify each sample collected during RMAP sampling events. Sample identifiers will begin with the matrix, followed by the RMAP Database School ID. The School ID is a unique identifier that is associated with a specific property (address and/or geocode specific). Following the School ID will

be the parcel component, location number, QC code (when applicable), and sample date. The sample ID format is [school ID]- [matrix]- [component type]-##[QC code]-YYYYMMDD.

RMAP Database School ID: (example of S-0001)

Site Property Codes:

S – School

D – Daycare

P – Preschool

School ID:

0001 – Associated with a specific address or geocode

Matrix:

D – Dust

S - Soil

Component: Component IDs will be derived on a site-specific basis during development of the FSP Sample Location Map and refined by the sampling team (as necessary). Examples of Component IDs are listed below.

A – Attic

AV – Air Vent

CS – Crawlspace

CT – Ceiling Tile

F – Floor

FM – Floor Mats

G – Grab

O - Other

S – Surface

QC Codes:

D – Field Duplicate

An example sample identification would be S-0001-D-AV-02-20211205. This indicates that the sample was collected at the school with the School ID S-0001 (corresponding to a physical address and/or geocode), was a dust sample collected in an air vent at location number two on December 5, 2021

The sample identification for a field duplicate collected at this location would be S-0001-D-AV-02D-20211205.

3.9 Analyses Methods

The subsections below describe analytical methods the laboratory must use to analyze RMAP samples.

3.9.1 Dust Sample Analysis Methods

All RMAP dust samples will be analyzed to determine metal concentrations via standard laboratory analytical methodologies for arsenic, lead, and mercury. Sample preparations and analyses will be in accordance with the referenced USEPA analytical method specifications as well as standard laboratory practices. The dust samples will be digested according to modified USEPA Method 3050B, and arsenic and lead concentrations will be determined per USEPA Method 6020B (inductively coupled plasma mass spectrometry [ICP-MS]). Mercury concentrations will be determined per USEPA Method 7471B (Manual Cold-Vapor Technique).

3.9.2 Laboratory Quality Control Samples

As outlined above in Section 3.9.1, RMAP dust samples will be analyzed to determine metals concentrations (arsenic, lead, and mercury) via standard laboratory analytical methodologies. Laboratory QC procedures are outlined below.

The analyses calibration procedures and frequencies of QC samples are specified in the laboratory's SOPs (see Appendix B). Instrument QC samples include calibration verification standards, calibration blanks, and contract required detection limit standards. ICP-MS QC samples also include tuning standards, interference check standards, and internal standards.

Laboratory QC samples will be analyzed in addition to the calibration samples with each QC batch. Laboratory QC samples are introduced into the measurement process to evaluate laboratory performance and sample measurement bias. Control samples may be prepared from environmental samples or generated from standard materials in the laboratory.

Laboratory method blanks, LCSs, analytical duplicates, and serial dilutions at a frequency of 1 each per 20 field samples. If less than 20 field samples are submitted, then 1 set of these QA/QC samples will still be run with a set of less than 20 samples. MS samples will be analyzed when additional amounts of dust are collected. For filter samples, an additional filter for MSs or duplicates must be provided for each method analyzed. When additional samples are not provided for dust, a LCS duplicate may also be included. A second MS sample is not necessary for all laboratory QC batches that already have one MS/matrix spike duplicate (MSD).

3.9.2.1 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. Method blank results outside of specified control limits will be re-run/re-digested and re-analyzed with all associated samples and/or flagged by the laboratory per the QC requirements of the analytical method.

Initial and continuing calibration blanks are also analyzed every 10 samples and samples are re-analyzed within compliant blank analyses. All elements of interest must be evaluated to +/- the RL for USEPA Method 6020B.

3.9.2.2 Laboratory Control Samples

An LCS, or a blank spike, is an aqueous or solid control sample of known composition that is analyzed using the same sample preparation, reagents, and analytical methods employed for the Program 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 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 LCS is re-analyzed once. If re-analysis of the LCS fails, all samples affected by the failing LCS elements need to be re-digested and re-analyzed.

3.9.2.3 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. Analytical duplicates are a pair of subsamples from a field sample that are 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 MSDs monitor the precision of the analytical process. The frequency of analyses, precision goals, and corrective action information pertaining to analytical duplicates are provided in the laboratory SOPs (Appendix B). 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.

3.9.2.4 Serial Dilutions

Serial dilutions are performed in conjunction with USEPA Method 6020B to determine whether significant physical or chemical interferences exist due to sample matrix. A serial dilution is performed by analyzing a 5-fold dilution of a field sample (field blanks may not be used) and calculating the percent difference between the original determination and the serial dilution result. Serial dilutions are only applicable for analyte concentrations that are greater than 50 times the MDL. The frequency of analyses, precision goals, and corrective action information pertaining to serial dilutions are provided in the laboratory SOPs in Appendix B.

3.9.2.5 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/MSDs 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 un-spiked samples. A post-digestion spike is performed on any elements that fail to meet criteria. If the percent recovery for the MS and MSD falls outside the control limits, the results are flagged by the laboratory that they are outside acceptance criteria along with the parent sample.

For dust samples collected with the micro-vacuum method, additional filter cassettes will be required for MS analyses. If adequate dust is not present, the analysis of MSs on filter cassette samples will not be included.

3.9.2.6 Additional Quality Control Samples

The laboratory will also analyze ICP-MS interference check, internal standards, and ICP-MS instrument tunes as part of the analytical sequence for USEPA Method 6020B. These instrument QC samples will be evaluated against the method requirements during data validation.

Table 3 contains acceptance criteria for the QC samples detailed above.

3.10 Field Quality Control 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.

Sampling protocols will be consistent with the field SOPs included in Appendix B and will include 1 field duplicate collected for every 20 primary samples or once per sampling event (e.g., once per sampling day), whichever is more frequent (in accordance with Level A/B field screening/data review criteria, Appendix D). Sampling equipment for soils and indoor dust filter cassettes are anticipated to be "one time use"; therefore, no external contamination blank/cross-contamination blank samples will be submitted. The HVS3 vacuum equipment is decontaminated between samples; equipment blank samples will be collected to ensure decontamination procedures are effective. Any deviation from the SOPs or this QAPP will be identified in the logbook/data collection device and discussed in the interior dust sampling DSR.

3.10.1 Field Duplicate (Dust Samples)

Field duplicate samples associated with dust sampling will be collected as side-by-side duplicates in separate cartridges rather than a split sample. Each duplicate sample will have its own sample number. Both the original and duplicate sample will be analyzed for identical chemical parameters. The results of the field duplicate will be compared to determine laboratory precision. Field duplicate samples will be collected at a frequency of 1 per 20 samples.

The RPD field precision goal for dust field duplicates will be 35 percent for sample pairs with both sample results being greater than five times the RL. For dust field duplicate/primary sample pairs with one or both sample results being less than five times the RL, an absolute difference of less than or equal to two times the RL (difference less than or equal to two times the RL) will be used as the precision goal. Laboratory precision goals are laboratory specific.

3.10.2 Filter Blanks

Filter blanks are collected to determine if micro-vacuum dust samples for metals analysis are collected with metals-free filters. A filter blank is a randomly selected filter cassette from a manufactured lot. For this sampling effort, one filter blank will be selected at random from each lot number of cassettes to be used for the collection of micro-vacuum dust samples. The filter blank remains unopened prior to being submitted to the laboratory. The entire batch of cassettes may be rejected if any metals are detected in a lot blank.

3.10.3 Field Blanks

Field blanks are collected to evaluate potential contamination introduced during sample collection, shipping and handling, or analysis. For this sampling effort, field blanks for surface dust and air will be collected at a rate of one each per school. Field blanks are collected by removing the end cap of the sample cassette to expose the filter in the same area where sample collection occurs for about 30 seconds before re-capping the sample cassette. The field blanks are then analyzed for metals.

3.10.4 Equipment Blanks

Equipment blanks are collected to evaluate potential cross-contamination between samples collected with the HVS3 vacuum. For this sampling effort, equipment blanks will be collected at a rate of one per sampling day Equipment blanks will be collected after the first sample has been collected and the HSV3

has been decontaminated. Approximately five grams of acid-washed glass beads will be poured through the sample collection chamber into the sample catchment container.

3.10.5 Floor Mat Blanks

Floor mat blanks are collected to evaluate potential contamination introduced from the floor mats used for dust collection. For this sampling effort, floor mat blanks will be collected at a rate of one per sampling event. Approximately five grams of acid-washed glass beads will be poured onto a floor mat and then collected with the HSV3 vacuum.

3.11 Sample Disposal

Dust samples shipped to the laboratory for analyses will be held until the laboratory analyses have been completed, the Agencies have reviewed and approved all subsequent project laboratory data and work plans, and the sample hold times have expired. At this point, the laboratory may dispose of samples. Any excess sample mass that was not included in the aliquot submitted to the laboratory will be subject to the same disposal criteria. The laboratory will notify ARCO/BSB when they will be disposing of samples.

3.12 Instrument/Equipment Testing, Inspection, and Maintenance

To document continual quality performance of any instruments or equipment, the testing, inspection, and maintenance activities listed in the sections below will be performed and recorded.

3.12.1 Field Equipment

Field equipment will be examined daily to certify that it is in proper operating order prior to its use. Equipment, instruments, tools, and other items requiring preventative maintenance will be serviced in accordance with the manufacturer's specified recommendations. Field equipment will be cleaned and safely stored between each use. Any routine maintenance recommended by the equipment manufacturer will also be performed and documented in field logbooks.

Equipment will be inspected, and the calibration checked, if applicable, before it is transported to a field setting for use.

3.12.2 Laboratory Equipment

Instruments used by the laboratories 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 preventative maintenance program.

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

3.13 Inspection/Acceptance of Supplies and Consumables

All supplies and consumables received for the project (e.g., sampling equipment, supplies, 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. Inspections of field supplies will be performed by the ERM field team leader.

The personnel at each laboratory will be responsible for performing inspections of laboratory supplies in accordance with their QA plan.

3.14 Non-Direct Measurement Data Acquisition Requirements

Non-direct measurement data include information from site reconnaissance, literature searches, previous sampling events, and interviews. The acceptance criteria for such data include a review by someone other than the author. Any measurement data included in information obtained from these sources will determine further action at the Site only to the extent that those data can be verified.

Types of data being used for the indoor dust assessments include but are not limited to:

- As-built floor plans of schools and daycares
- Interviews. School or daycare employees will be interviewed prior to the sampling event to determine building usage and determine appropriate sample locations
- Surveys. Visual surveys of the properties will be made by the field team during the sampling event and documented following ERM protocols for site photography and field notes

3.15 Data Management Procedures

This section describes the management of data for the project including field and laboratory data. The Program quality records will be maintained by the data management division manager, as described in the BPSOU *Final Data Management Plan* (Atlantic Richfield 2017).

These records, either electronic or hard copy in form, may include the following:

- Project work plans with any approved modifications, updates, and addenda
- Individual school/daycare maps (hard copy or scanned field drawings and electronic files)
- Individual school/daycare result letters (both no action and remedial action required)
- Project QAPP, including this QAPP, with any approved modifications, updates, addenda, and corrective or preventative actions
- Access agreements from school officials
- Field documentation
- Chain-of-custody records
- Laboratory documentation (results received from the laboratory will be documented both in report form and in an electronic format)
- Data validation documentation
- Annual completion report

Hard copy field and laboratory records will be maintained in the project's central data file, where original field and laboratory documents are filed chronologically for future reference. These records are also scanned to produce electronic copies. The electronic versions of these records are maintained on a central server system with backup scheduled daily.

Before field and laboratory data are incorporated into the Program databases, the data and supporting documentation will be subject to appropriate review to document the accuracy and completeness of original data records. Field data that have been reviewed in a hard-copy format will be entered into electronic data files for upload to the Program database. All manual data entry into an electronic format will be reviewed by a separate party before the information is incorporated. Laboratory EDDs and related data packages will be reviewed as part of the internal data review process. The data management division manager, or designated alternate, will be responsible for ensuring data integrity prior to Program

database uploads. Following these review steps, field and laboratory electronic data files will be imported to the Program database.

Standardized data import formats and procedures will be used to upload both field and laboratory data into the Program database. An existing EDD format will be used for data upload. Standardized parameter names, numerical formats, and units of measure may be applied to the original information to facilitate comparability across all datasets and within the Program database. Data management activities for the RMAP are further defined in the BPSOU *Data Management Plan* (Atlantic Richfield 2017).

3.15.1 Requests for Data

Requests for data can be made to the data management division manager or to the Agencies who can access data directly through the secure Program database. Refer to the *Institutional Controls Management System Plan* (BSB and Atlantic Richfield 2019b) for additional details and specific examples of the Program's database and tracking system. The *Institutional Controls Management System Plan* (BSB and Atlantic Richfield Company 2019b) is in Appendix F of the *Institutional Controls Implementation and Assurance Plan (ICIAP)* (BSB and Atlantic Richfield 2019a).

4. ASSESSMENT AND OVERSIGHT

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 USEPA or a USEPA contractor will provide oversight during site reconnaissance and sampling activities. The audits of field and laboratory activities include two independent parts: internal and external audits. Internal audits may be conducted by Atlantic Richfield's contractor Environmental Standards, Inc. as necessary (i.e., if concerns are raised during work execution, or observed the Agency oversight, internal audits will be scheduled). External audits may be performed by the Agencies as necessary. Audits are not currently scheduled for this project.

Performance and system audits of field and laboratory data collection and reporting procedures are described in this section.

4.1 Corrective Actions

Corrective action is the process of identifying, recommending, approving, and implementing measures to counter unacceptable procedures or out-of-QC performance, which can affect data quality. Corrective action can occur during field activities, laboratory analyses, and data assessment. A corrective action template is provided in Appendix F.

Non-conforming equipment, items, activities, conditions, and unusual incidents that could affect data quality and attainment of the project's quality objectives will be identified, controlled, and reported in a timely manner. For the purpose of this QAPP, a non-conformance is defined as a malfunction, failure, deficiency, or deviation that renders the quality of an item unacceptable or indeterminate in meeting the project's quality objectives.

Corrective action in the laboratory may occur prior to, during, and after initial analyses. Several conditions such as broken sample containers, preservation or holding-time issues, and potentially high-concentration samples may be identified during sample log-in or just prior to analyses.

Corrective actions to address these conditions will be taken in consultation with the Atlantic Richfield Liability Manager, the consultant project manager, and/or the consultant QA manager. If corrective action requests are not in complete accordance with approved project planning documents, the USEPA will be consulted, and concurrence will be obtained before the change is implemented, or new samples may be obtained.

If during analyses of the samples the associated laboratory QC results fall outside of the project's performance criteria, the laboratory should initiate corrective actions immediately. Following consultation with laboratory analysts and section leaders, it may be necessary for the contract laboratory's QA officer to approve implementing a corrective action. These conditions may include dilution of samples, additional sample extract cleanup, or automatic re-injection/re-analysis when certain QC criteria are not met, etc. If the laboratory cannot correct the situation that caused the non-conformance and an out-of-control situation continues to occur or is expected to occur, then the laboratory will immediately contact the Atlantic Richfield Liability Manager, the consultant project manager, and/or the consultant QA manager and request instructions regarding how to proceed with sample analyses.

Completion of any corrective action should be evidenced by data once again falling within the project's performance criteria. If this is not the case, and an error in laboratory procedures or sample collection and handling procedures cannot be found, the results will be reviewed by the consultant QA manager to assess whether re-analysis or re-sampling is required.

All corrective actions taken by the laboratory will be documented in writing by the laboratory project manager and reported to the consultant QA manager. If corrective action requests are not in complete

accordance with approved project planning documents, the USEPA will be consulted, and concurrence will be obtained before the change is implemented. All corrective action records will be included in the Program quality records.

4.2 Corrective Actions during Data Assessment

The need for corrective action may be identified by any member of the project team during data assessment. Potential types of corrective action may include re-sampling by the field team, re-analyses of samples by the laboratory, or re-submitting data packages with corrected clerical errors. The appropriate and feasible corrective actions are dependent upon the ability to mobilize the field team and whether the data to be collected is necessary to meet the required QA objectives (e.g., the holding time for samples is not exceeded). If corrective action requests are not in complete accordance with approved project planning documents, the USEPA will be consulted, and concurrence will be obtained before the change is implemented. Corrective actions of this type will be documented by the consultant QA manager on a Corrective Action Report (Appendix F) and will be included in any subsequent reports.

4.3 Reports to Management

Upon receipt of laboratory results and completion of the data review/validation process, all analytical data will be uploaded into the Program database and submitted to the Agencies for review and approval. For the school sampling portion of this project, these submittals would be anticipated to be submitted on a per school basis to decrease the turnaround time required for landowner reporting as much as possible. Upon receiving Agency approval, the sample results (for all analytes) will be reported to school and daycare officials along with a letter explaining what the results indicate (see result letter templates in Appendix E). The action levels for arsenic, lead, and mercury will be reported along with sample results.

After site investigations and remedial actions are complete, the consultant QA manager will prepare an interior dust sampling DSR summarizing the sampling activities. The laboratory and data validation turnaround times for providing sample results will be expedited in order to achieve project assessment and remediation goals while also allowing timely completion of the DSR. This is estimated to be a 5 to 7 business day turnaround time on lab data and Level 2 data packages and 10 to 12 business day turn around on lab data and Level 4 data packages. Data validation is estimated to be a 7-business day turnaround time after data packages are received from the lab. The report will describe specific field sampling activities performed during implementation of the QAPP. Each report will include field documentation, documentation of field QC procedures, results of all field and laboratory data, data validation results, and data usability assessments.

A separate report will be prepared by the consultant QA manager, as needed, to communicate the results of performance evaluations or program audits to identify specific significant QA issues and provided to the USEPA for review. Any corrective action reporting described in Section 4.2 above will be summarized and included as appropriate.

5. DATA REVIEW AND USABILITY

The following sections address the final project checks conducted after the data collection phase of the project is completed to confirm that the data obtained meet the project objectives and to estimate the effect of any deviations on data usability for the express purposes of achieving the stated DQOs (Section 2.9.1). Data review/validation process under this QAPP is streamlined to support the post-BPSOU ROD (USEPA 2006b) decision-making process. The analytical data collected under this QAPP and produced by analytical laboratories will undergo a combination of Stage 4 and 2B data validation which are described in Section 5.2. The field documentation will be subject to Level A/B criteria review, and analytical data will be validated per the *Clark Fork River Superfund Site Investigation (CFRSSI) Data Management/Data Validation Plan* (CFRSSI DM/DV Plan) (ARCO 1992a), the *EPA Contract Laboratory Program National Functional Guidelines for Inorganic Superfund Data* Review (USEPA 2020b), and the project DQOs. Data review and validation will be conducted by a qualified technical consultant who is independent from the sampling consultant (i.e., an individual other than the individual who performed sampling).

5.1 Data Review, Verification, and Validation

This section describes the review, verification, and validation process for field data and laboratory data. The section also details laboratory data reporting requirements, which describe how results are conveyed to data users.

5.1.1 Data Review Requirements

Data review is performed by the data producer to determine if the data have been recorded, transmitted, and processed correctly.

5.1.1.1 Field Data Review

Raw field data will be entered in field logbooks/data collection device and reviewed for accuracy and completeness by the field team leader before those records are considered final. The overall quality of the field data from any given sampling round will be further evaluated during the process of data reduction and reporting. The field data will be reviewed quarterly by the consultant QA manager, or designated alternate.

Field data reduction procedures will be minimal in scope compared to those implemented in the laboratory setting. Field data review will include verification that any QC checks and calibrations, if necessary, are recorded properly in the field logbooks/data collection device and that any necessary and appropriate corrective actions were implemented and recorded. Such data will be recorded in the field logbook/data collection device immediately after measurements are taken. If errors are made, results will be legibly crossed out, initialed, and dated by the field member, and corrected in a space adjacent to the original (erroneous) entry. Later, the field team leader will review the field logbooks/data collection device to determine whether any transcription errors have been made by the field crew. If transcription errors have been made, the field team leader and field crew will address the errors to provide resolution.

As appropriate, field measurement data will be entered into electronic files for import to the Program database. Data entries will be made from the reviewed logbooks/data collection device, and all data entries will be reviewed for accuracy and completeness by a separate party before the electronic file is provided to the Program database manager. Electronic files of field measurement data will be maintained as part of the project's quality records.

5.1.1.2 Laboratory Data Review

Internal laboratory data reduction procedures will be according to each laboratory's quality management plan. At a minimum, paper 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, reagent concentrations, instrument settings, and the raw data. These records will be signed and dated by the analyst. Secondary review of these records by the laboratory supervisor (or designee) will take place prior to final data reporting. The laboratory is responsible for assigning appropriate flags/qualifiers in accordance with the analytical method and internal laboratory SOPs.

5.1.2 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.

5.1.2.1 Field Data Verification

The Level A/B review (see checklist in Appendix D), as described in the CFRSSI DM/DV Plan (ARCO 1992a) and the DM/DV Addendum (AERL 2000), will be used in the verification process for field documentation related to samples collected for laboratory analyses.

Level A criteria includes:

- Sampling date
- Sample team and/or leader
- Physical description of sample location
- Sample collection technique
- Field preparation technique
- Sample preservation technique
- Sample shipping records

Level B criteria includes:

- Field instrumentation methods and standardization complete
- Sample container 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

5.1.2.2 Laboratory Data Verification

The laboratory will prepare Level 2 and Level 4 data packages for transmittal of results and associated QC information to the Atlantic Richfield Liability Manager or consultant designee within a standard turnaround time unless otherwise required.

These data packages will be prepared in general accordance with the *EPA Contract Laboratory Program Statement of Work for Superfund Analytical Methods (Multi-Media, Multi-Concentration) SFAM01.1* (USEPA 2020c). Deviations from these specifications may be acceptable based on the SW-846 methods provided the report presents all the requested types of information in an organized, consistent, and readily reviewable format.

Each data package, as described above, will be accompanied by an EDD prepared by the laboratory. A non-validated EDD is uploaded to the BP RM EQuIS database by the laboratory to capture the laboratory supplied EDD. Once the laboratory supplied EDD is loaded, the data validator is notified and downloads the non-validated EDD from the database for the verification and validation process. Once data verification and validation is complete, the qualifiers will be added to the downloaded EDD, the enforcement “E” and screening “S” qualifiers are added and the revised EDD is uploaded to the database by the validator for final reporting.” Additional laboratory QC data can be included in the EDD. The EDDs will be cross-checked against corresponding data reports to confirm consistency in results reported in these two separate formats. This cross-check will take place as part of the data verification process. All data will be submitted in both Level 2 and Level 4 format.

5.1.2.3 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.

5.1.3 Data Validation Requirements

The purpose of analytical data validation is to provide an assessment of data quality. Data validation will be performed by qualified, independent data validation personnel, who are not associated with data collection or sampling responsibilities, and that have applicable training. Data validation categorizes data as acceptable for use, unacceptable for use, or qualified for select use. The validation effort routinely identifies data use limitations and corrects reporting and quantitation errors. The data packages provided for validation will be evaluated for compliance with respect to the requested analytical methods and/or the QAPP and completeness of requested deliverables. Concurrent with the data validation efforts, analytical data usability will also be assessed. Analytical data usability is the determination of whether a data set is sufficiently complete and of sufficient quality for further evaluation by the data user as detailed in Section 5.3 of the QAPP to support a decision or action.

The data will be validated during the data validation process with guidance from the CFRSSI QAPP (ARCO 1992b), the CFRSSI DM/DV Plan (ARCO 1992a), the CFRSSI DM/DV Plan Addendum (AERL 2000), the *EPA Contract Laboratory Program National Functional Guidelines for Inorganic Superfund Data Review* (USEPA 2020b), laboratory-specific QC criteria, and/or method-specific criteria where applicable. The use of the functional guidelines versions listed above is important to maintain consistency between data validation and qualification of data currently being performed and future work to be performed under the RMAP. It should be noted that the USEPA National Functional Guidelines, which were developed for the validation of data generated in accordance with the Contract Laboratory Program, are not directly applicable to the type of analyses/protocols associated with the analyses for this project. USEPA National Functional Guidelines qualifies data based on strict contractual Contract Laboratory Program method requirements and acceptance criteria, which may not be consistent with the requirements and acceptance criteria presented in SW-846 methods. Data validators will apply the

USEPA guidelines as appropriate, assess the data relative to method QC protocols and DQOs in this QAPP, and use professional judgment according to the documents listed above. Finally, reason codes for qualification will be included in the data validation report and entered to the qualified EDD.

5.2 Verification and Validation Methods

The Level A/B assessment checklists included in Appendix D are based on the CFRSSI DM/DV Plan Addendum (AERL 2000) guidance and will be used for field data verification as detailed in Section 5.1.2.1.

Data qualifiers will follow those used in the *EPA Contract Laboratory Program National Functional Guidelines for Inorganic Superfund Data Review* (USEPA 2020b). Data validation for each laboratory data package will be documented on the data validation checklists based on the CFRSSI DM/DV Plan Addendum (AERL 2000) guidance (Appendix G).

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 data validation report, which will be reviewed by the consultant project manager and QA manager. The data validation reports for each SDG will be included as an appendix to the DSR.

Qualifiers that may be applied to the data during the data validation process are listed in Table 5-1.

Table 5-1: Validation Qualifiers

Qualifier	Definition
U	The analyte was analyzed for, but was not detected above the level of the adjusted detection limit or quantitation limit, as appropriate.
J	The analyte was positively identified; the associated numerical value is an estimate of the concentration of the analyte in the sample. This will also include results reported between the MDL and RL.
J+	The result is an estimated quantity, but the result may be biased high.
J-	The result is an estimated quantity, but the result may be biased low.
UJ	The analyte was not detected above the sample MDL. However, the MDL is approximate and may or may not represent the actual limit of quantitation necessary to accurately and precisely measure the analyte in the sample.
R	The sample results are rejected due to serious deficiencies in the ability to analyze the sample and meet quality control criteria. The presence or absence of the analyte cannot be verified.
No Flag	Result accepted without qualification.

5.2.1 Differences between Stage 2B and Stage 4 Validation

The content and scope of the Stage 2B and Stage 4 data validation will be performed with guidance from *Guidance for Labeling Externally Validated Laboratory Analytical Data for Superfund Use, OSWER No. 9200.1-85, EPA 540-R-08-005, 13* (USEPA 2009). The major difference between Stage 2B and Stage 4 data validation is the detail level of the data evaluation. Stage 4 data validation is an in-depth process that consists of a comparison between raw data and summary forms to check for inconsistencies between reported data and raw data. Stage 2B data validation does not involve evaluating raw data or checking reported data and raw data and assumes that all results and recoveries are correctly reported.

Stage 2B and Stage 4 data validations and reports are generated by an initial reviewer on a per-SDG or sampling location basis from the complete Level 4 data package to ensure completeness and data usability of data packages. Level 2 data packages are a condensed version of final data prior to completion and receipt of Level 4 data packages. Level 2 data packages contain the same information as the Level 4 data packages with the exception that instrumental QC (i.e., instrument tunes and raw data) to support the sample and the QA/QC results are not provided.

Each validation report is reviewed by a senior chemist for accuracy to ensure that the initial reviewer has rigorously evaluated the recoveries/results and applied the applicable qualifiers to the data.

5.2.2 Stage 2B and Stage 4 Validation Procedure

A comprehensive QA review will be performed to independently verify compliance with the required analytical protocols and to determine the qualitative and quantitative reliability of the data. Stage 4 data validation includes a detailed review and interpretation of the data generated by the laboratory. Stage 4 data validation includes the review of the summary forms for all QC procedures and all sample and quality control raw data (including instrument calibration) to support the results reported. The purpose of a Stage 2B validation is to qualify data based on identified data quality limitations.

For each of the inorganic analytes, the Stage 4 verification and validation checks include an evaluation of the following, as applicable for each analytical method. A Stage 2B validation focuses solely on data usability and does not include a review of raw data.

- Completeness of laboratory data package
- Requested analytical methods performed
- Compliance with the QAPP, analytical method, and analyte list
- Proper sample collection, custody, preservation, and handling procedures
- Holding times
- Reported detection limits
- Dilution factors
- ICP-MS tuning
- Instrument calibration
- Initial and continuing calibration verification standards
- Initial and continuing calibration blanks
- ICP-MS interference check samples
- Method blanks
- LCSs
- RL check standard recoveries
- Field duplicate results
- MS/MSDs (pre-digestion and post-digestion)
- ICP-MS internal standard recoveries
- ICP-MS serial dilutions
- Results verification and reported detection limits

- Sample Preparation and Analytical Run Log

5.2.3 Data Validation Ratios

Initially, 10% of the project data will undergo Stage 4 validation. The data validator will perform Stage 4 data validation on the first SDG of each designated school sampling event to verify that the laboratory is analyzing the project samples in accordance with the applicable analytical methods and QAPP procedures, and is providing all required data deliverables. This process will ensure Stage 4 validation is performed for each school and periodically throughout the entire sampling event. However, in some instances, where multiple small project SDGs containing the same analytical list are being prepared, validation of the first data package of each project school may represent the entire data set for the project, thereby raising the percentage of Stage 4 validation performed. This approach should allow the data validator to identify and have the laboratory correct any non-compliances early on in the data collection process. In the event significant problems or issues are identified during the 10% Stage 4 data validation effort, it may be necessary to increase the percent of data subjected to Stage 4 validation to ensure that all errors and non-compliances have been appropriately corrected. The remaining 90% of the data will be validated at a Stage 2B level. In addition, the Consultant PM can also offer guidance or request greater percentage of Stage 4 data validation as the required level of validation based on project DQOs.

5.3 Reconciliation and User Requirements

A 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, 2000) 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: Select the statistical test/method. *There are no statistical tests that are planned in the interpretation of the non-residential soils results; laboratory results will be compared directly to action limits defined in the DQOs (Section 2.9.1).*

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 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 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 data limitations.

The PARCCS data quality indicators (Section 2.9.2) will be used when conducting the DQA. If the PARCCS assessment satisfies the project DQOs, then usability of the data will follow the enforcement/screening/unusable data categories as described in the CFRSSI DM/DV Plan (ARCO 1992a):

- Enforcement Quality (Unrestricted Use). Enforcement quality data may be used for all purposes under the Superfund program including the following: site characterization, health and safety, environmental evaluation/cost analysis, remedial investigation/feasibility study, alternatives evaluation, conformational purpose, risk assessment, and engineering design.

- Screening Quality (Restricted Use). 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.
- Unusable Data. These data are not usable for Superfund-related activities.

Data that meet the Level A and Level B field data verification 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 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 usability designations defined in Table 5-2.

Table 5-2: Data Usability Designation Definitions

Designation	Definition	Data Validation Criteria	Field Verification Criteria
E	Enforcement quality	No qualifiers, U qualifier, or J qualifier (see note below)	Meets both Level A and B criteria
S	Screening quality	J or UJ qualifier	Meets only Level A criteria
R	Unusable	R qualifier	Does not meet Level A or B criteria

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

The selection process for the appropriate enforcement designation is presented in Table 5-3.

Table 5-3: Enforcement/Screening Designation Selection

Validation Qualifier	Field Screening Criteria		
	Meets Level A and B	Meets Level A	Does not meet Level A or B
No qualifier, U, or laboratory results reported between the MDL and RL with a J qualifier	E	S	R
J, J+, J-, or UJ	S	S	R
R	R	R	R

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. The enforcement/screening designations are also added to the qualified AR EQuIS EDDs by the data validation consultant for upload to the AR EQuIS database.

5.3.1 Evaluation of Results

The analytical results that have been validated in accordance with Sections 5.1 and 5.2 of this QAPP will be compared to the BPSOU residential action levels (Arsenic – 250 mg/kg, Lead – 1,200 mg/kg, Mercury - 147 mg/kg) for all work completed under this QAPP (see Table 1). Analytical results will be compared to the action levels and the three statements below will be used for identifying data groupings for decision-making purposes. These statements assume the primary and duplicate results are valid and not qualified for other QA/QC deficiencies. If either the primary and/or duplicate sample are qualified for other reasons, professional judgement will be used with agency engagement and approval in the decision making process.

1. Undetected results (MDL< action level) or positive sample results are less than the action level(s).
2. Primary and field duplicate sample results are greater than the action level(s).
3. Primary and field duplicate sample results where one result is above the action level(s) and the other result is below the action level(s). The sample results will be evaluated using the following criteria.

    a. If the RPD between the primary and field duplicate results is <35% and the results are unqualified for field duplicate precision, then the highest of the primary and duplicate results will be used for decision making.

    b. If the RPD between the primary and field duplicate results is >35% and the results are qualified for field duplicate precision, the data is considered screening quality “S” in accordance with the QAPP. For interior soils, repreparation and reanalysis of the sample pairs will occur when the RPD is greater than 35%. For interior dust where sample volumes are limited or where samples were collected using filter cartridges, repreparation and reanalysis of the sample pairs is not possible; recollection of samples and analysis may be necessary. If resampling is not possible then the highest of the primary and duplicate results will be used for decision making.

        If these conditions are met for soil samples, then both the parent and the field duplicate sample will be reprepared from the air-dried, sieved soil and reanalyzed by the laboratory.

    Upon re-analysis no further action will be taken if:

    c. The parent sample and field duplicate sample results are below the action level(s), and the RPD is less than 35%, Statement 1 above will applied to the results. If the above conditions were not met, the highest of the primary and duplicate results will be used for decision making.

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TABLES

Table 1

RMAP ACTION LEVELS AND SAMPLE PROTOCOL

RMAP Non-Residential Parcels

Butte, Montana

Matrix	Exposure Scenario	Contaminant of Concern:	Residential Action Levels	Analytical Method	Method Detection Limit (MDL)[1]	Sample Frequency	Sample Density
Indoor dust and soil	Dust inhalation or ingestion	Lead	1200 mg/kg	EPA 6020B	0.087 mg/kg	See field sampling plans	See field sampling plans
		Arsenic	250 mg/kg	EPA 6020B	0.156 mg/kg		
		Mercury	147 mg/kg	EPA 7471B	0.008 mg/kg		
Indoor air	Vapor inhalation	Mercury	0.43 µg/m^3	Mercury Tracker 3000	0.1 µg/m^3		

Notes:

1 Detection limits will be re-evaluated and may change on a quarterly basis.

mg/kg = milligrams per kilogram

µg/m^3 = microgram per cubic meter

Table 2
PRECISION, ACCURACY AND COMPLETENESS CALCULATION EQUATIONS
RMAP Non-Residential Parcels
Butte, Montana

Characteristic	Formula	Symbols
Precision (as relative percent difference, RPD)	$RPD = \frac{(x_i - x_j)}{\frac{(x_i + x_j)}{2}} \times 100$	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 collected

Table 3
QUALITY CONTROL SAMPLE ACCEPTANCE CRITERIA
RMAP Non-Residential Parcels
Butte, Montana

Analyte	Residential Action Limit (mg/kg)	Analytical Method	Method Detection Limit (MDLs)[1] (mg/kg)	Reporting Limit (RL) (mg/kg)[1]	Laboratory Control Sample (LCS) Recovery Limits	Matrix Spike/ Matrix Spike Duplicate (MS/MSD) Recovery Limits[2]	MS/MSD Relative Percent Difference (RPD)[2]	Laboratory Duplicate Precision (RPD)	Field Duplicate Precision[3]
Lead	1,200	EPA 6020B	0.087	0.50	70-130%	75-125%	20	20	35
Arsenic	250	EPA 6020B	0.156	0.20	70-130%	75-125%	20	20	35
Mercury	147	EPA 7471B	0.008	0.02	70-130%	75-125%	20	20	35

Notes:

[1] The MDLs and RLs are considered the laboratory base values. Soil samples for arsenic and lead will be dried prior to sample digestion and will not be dry weight corrected. Sample results for mercury will be reported on a dry weight basis, since soil samples will be digested on an "as received" basis. MDLs and RLs may also be affected based on the actual weight of sample digested and potential dilutions required for high concentration samples.

[2] The percent recovery for each analyte in the MS and MSD and the RPD should be within the limits on the table with the exception when native sample results exceed the concentration of the added spike by 4 or more. Sample results will not be qualified in the event of this condition.

[3] The RPD field precision goal for soil field duplicates will be 35% for sample pairs with both sample results being greater than 5 times the reporting limit (RL). For soil field duplicate/primary sample pairs with 1 or both sample results being less than 5 times the RL, an absolute difference of less than or equal to 2 times the RL (difference ≤ 2xRL) will be used as the precision goal.
mg/kg = milligrams per kilogram

FIGURES

DRAWN BY: Jimmy Holcomb

SCALE: 1:130,800 when printed at 11x17

REVISED: 09/24/2021

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DEER LODGE COUNTY

SILVER BOW COUNTY

JEFFERSON COUNTY

Fairmont

Ramsay

Solvay

Rocker

Continental Mine

Beal Mountain

4N 11W | 4N 10W | 4N 9W | 4N 8W | 4N 7W | 4N 6W

3N 11W | 3N 10W | 3N 9W | 3N 8W | 3N 7W | 3N 6W

2N 11W | 2N 10W | 2N 9W | 2N 8W | 2N 7W | 2N 6W

1N 10W | 1N 9W | 1N 8W

0 10,000 20,000 Feet

0 95 190 380 Miles

MONTANA

Missouri

Legend

- BPSOU Boundary
- Areas Excluded From RMAP (Continental Mine, Solvay, Beal Mountain)
- 2020 RMAP Area Boundary
- 2011 Residential Metals Expanded Area
- County Boundary
- US Forest Service

N

0 2 4 Miles

Source: Esri - World Topoographic Map; NAD 1983 StatePlane Montana FIPS 2500 Feet Intl

Figure 1

BPSOU 2020 RMAP Area Boundary

Atlantic Richfield Company

Butte, Montana

Environmental Resources Management

www.erm.com

ERM

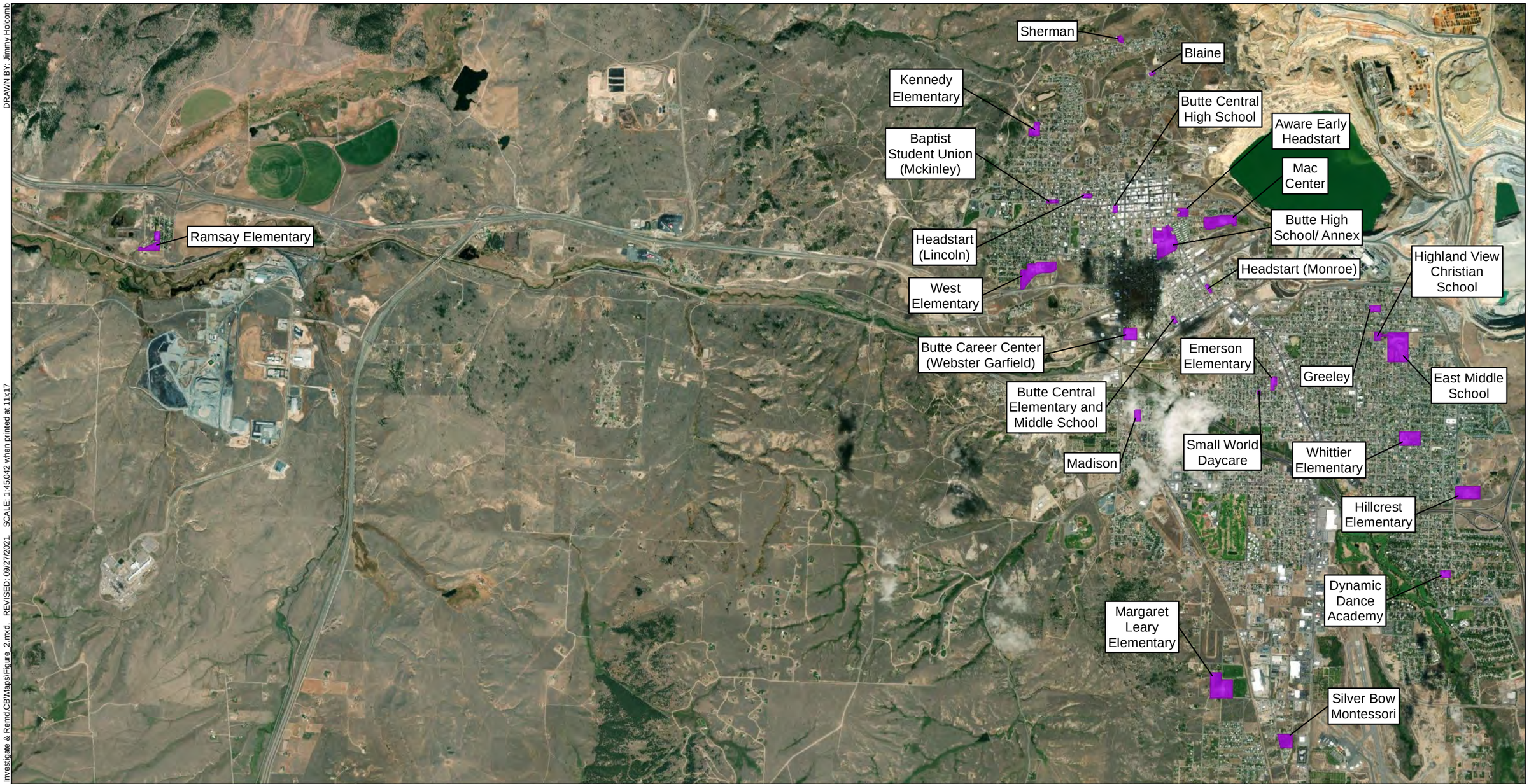


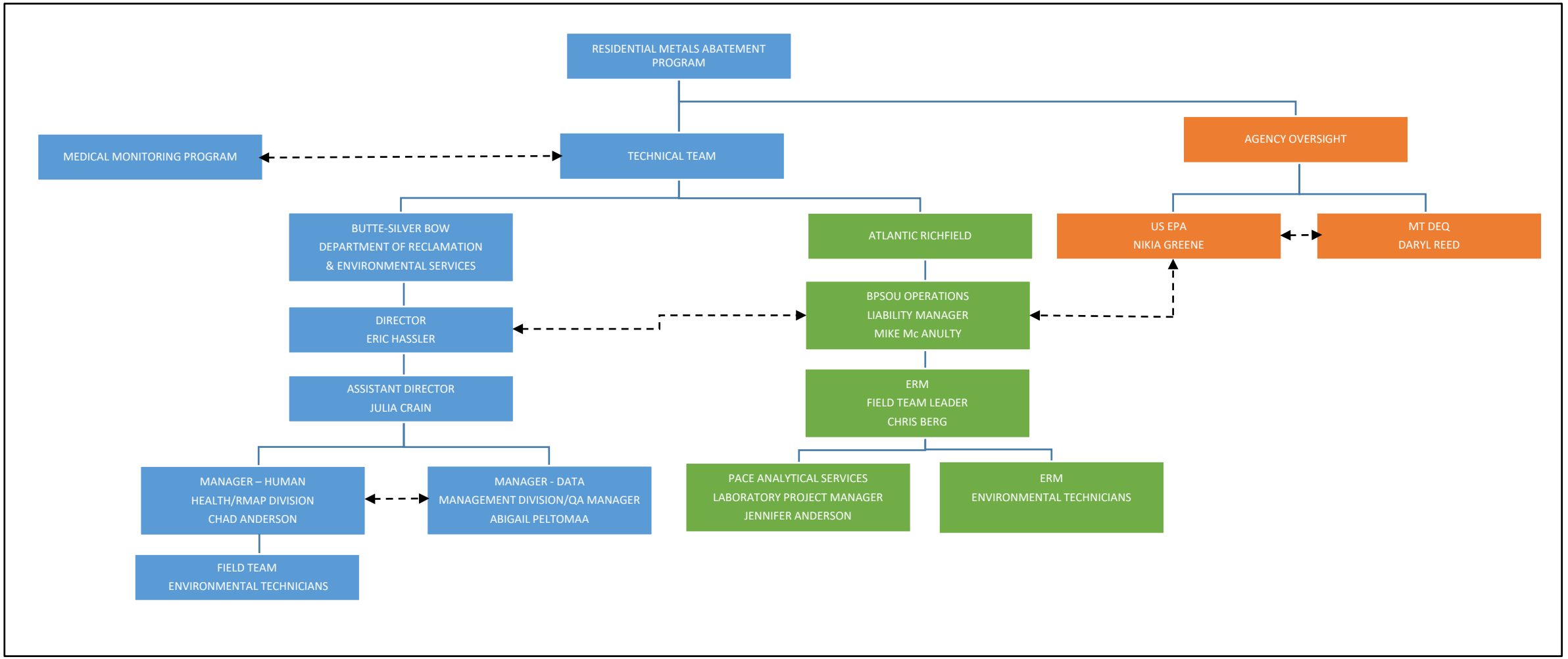
Figure 2
Butte-Silver Bow Schools and Former Schools
Atlantic Richfield Company
Butte, Montana

Environmental Resources Management
www.erm.com

Source: Esri - World Topoographic Map; NAD 1983 StatePlane Montana FIPS 2500 Feet Intl

M:\Projects\0612471 BP Remediation Management Investigate & Remd.CBWMaps\Figure 2.mxd, REVISED: 09/27/2021, SCALE: 1:45,042 when printed at 11x17

DRAWN BY: Jimmy Holcomb



AUTHORITY

COMMUNICATION

BLUE – BUTTE REMEDIAL ABATEMENT PROGRAM
GREEN – ATLANTIC RICHFIELD
ORANGE – AGENCY OVERSIGHT

	DISPLAYED AS:
COORD SYS/ZONE:	NA
DATUM:	NA
UNITS:	NA
SOURCE:	ERM

SCALE IN FEET
0 NTS NTS

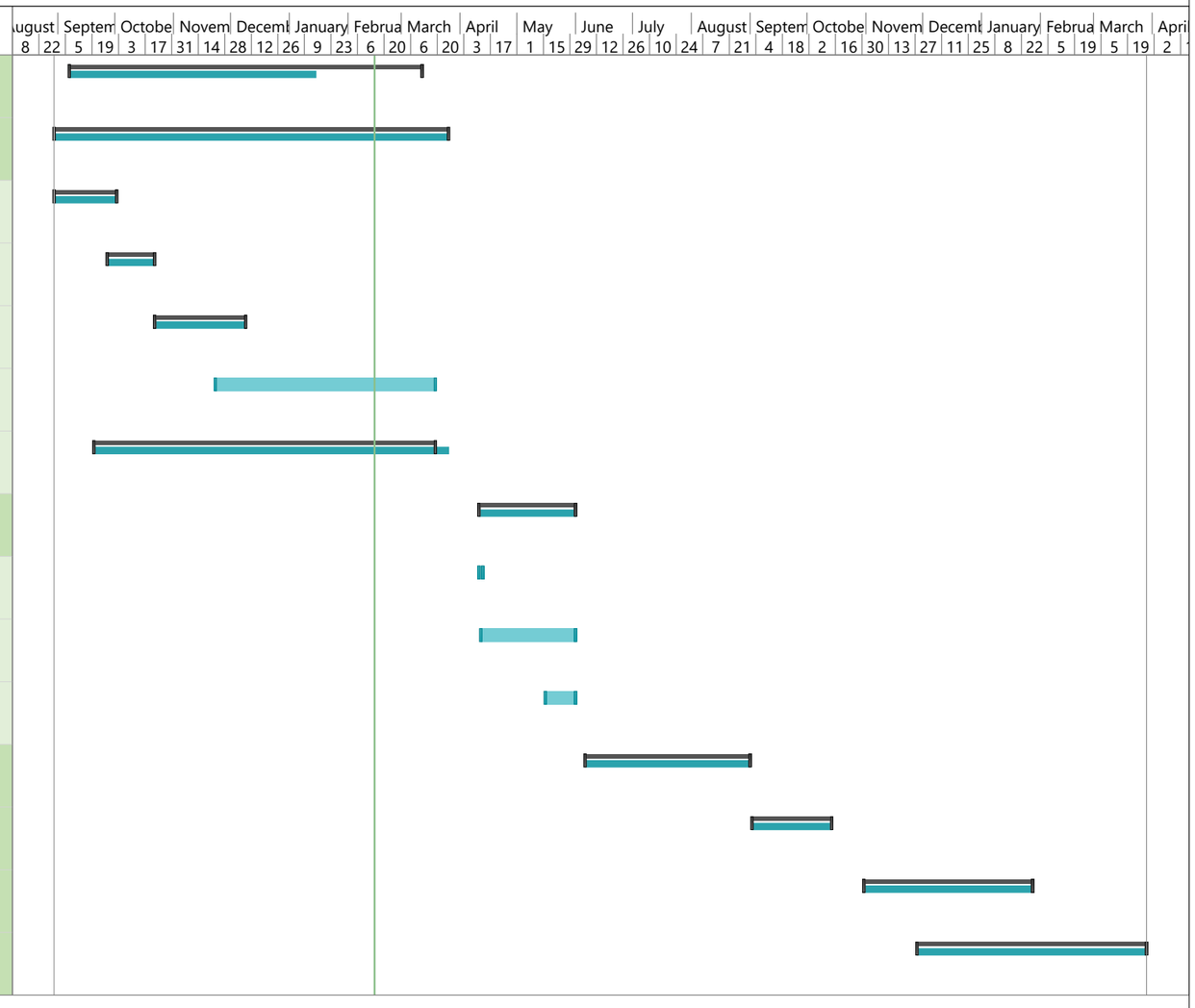
FIGURE 3

RMAP ORGANIZATIONAL & COMMUNICATIONS STRUCTURE

Figure 4 - Butte Priority Soils Operable Unit - Residential Metals Abatement Program
School Indoor Dust Investigation Schedule
Updated Tue 2/15/22

Tue 2/15/22

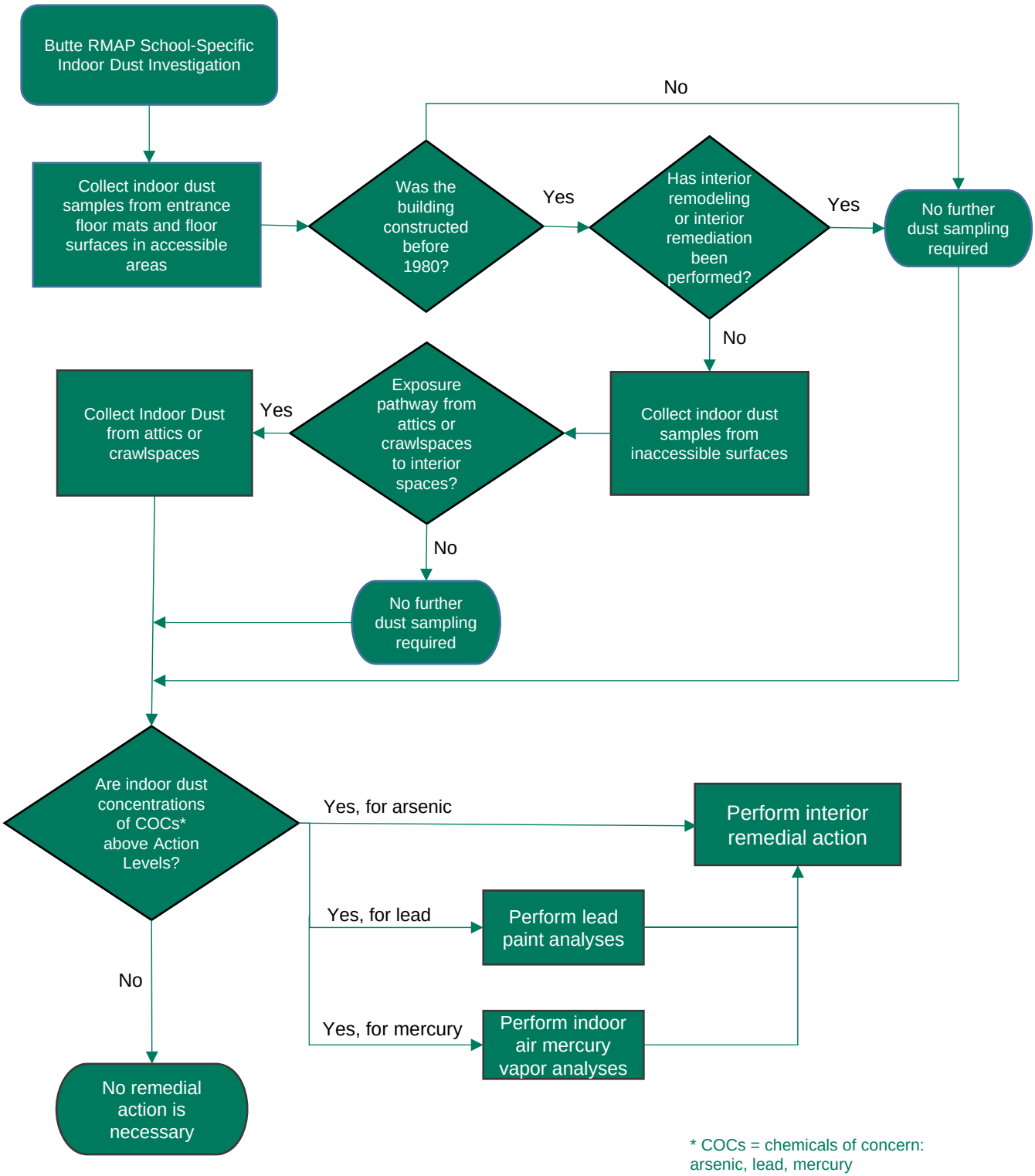
ID	Task Mode	Task Name	Start	Finish
1		**Quality Assurance Project Plan Development & Submittal**	**Tue 9/7/21**	**Fri 3/11/22**
8		**Field Sampling Plans Development & Submittal**	**Mon 8/30/21**	**Fri 3/25/22**
9		**School Pre-Coordination**	**Mon 8/30/21**	**Fri 10/1/21**
12		**Info Graphic Flyers Development & Submitttal**	**Mon 9/27/21**	**Thu 10/21/21**
17		**Perform Preliminary School Site Walks**	**Fri 10/22/21**	**Wed 12/8/21**
23		**Butte High School Tunnel - Field Sampling Plan Development**	**Tue 11/23/21**	**Fri 3/18/22**
24		**Field Sampling Plan Development**	**Mon 9/20/21**	**Fri 3/18/22**
30		**Perform School Dust Sampling**	**Mon 4/11/22**	**Tue 5/31/22**
31		**Butte High School Tunnel - Soil Sampling Event**	**Mon 4/11/22**	**Tue 4/12/22**
32		**Phase 1 - Inaccesible Space Sampling Events**	**Tue 4/12/22**	**Tue 5/31/22**
33		**Phase 2 - Accessible Space Sampling Events**	**Mon 5/16/22**	**Tue 5/31/22**
34		**Dust Investigation and Data Summary Reports Development**	**Mon 6/6/22**	**Wed 8/31/22**
43		**Remedial Action Work Plans Development**	**Fri 9/2/22**	**Thu 10/13/22**
56		**Remedial Action Implementation**	**Mon 10/31/22**	**Fri 1/27/23**
60		**Remedial Action Completion Reports Development**	**Mon 11/28/22**	**Tue 3/28/23**



Project: Butte School Dust Inve
Date: Tue 2/15/22

Task		Project Summary		Manual Task		Start-only		Deadline	
Split		Inactive Task		Duration-only		Finish-only		Progress	
Milestone		Inactive Milestone		Manual Summary Rollup		External Tasks		Manual Progress	
Summary		Inactive Summary		Manual Summary		External Milestone			

Figure 5. BPSOU Non-Residential School/Daycare Indoor Dust Sampling Decision Framework



* COCs = chemicals of concern: arsenic, lead, mercury

APPENDIX A QAPP CROSSWALK

EPA REGION 8 QA DOCUMENT REVIEW CROSSWALK

QAPP/FSP/SAP for: *(check appropriate box)* ☐ **GRANTEE** ☐ **CONTRACTOR** ☐ **EPA** ☐ **Other**	**Entity** *(grantee, contract, EPA AO, EPA Program, Other)* Atlantic Richfield	**Regulatory Authority and/or Funding Mechanism**	___ **2 CFR 1500 for Grantee/Cooperative Agreements** ___ **48 CFR 46 for Contracts** ___ **Interagency Agreement (FFA, USGS,)** ___ **EPA/Court Order** ___ **EPA Program Funding** ___ **EPA Program Regulation** ___ **EPA CIO 2105**
Document Title *[Note: Title will be repeated in Header]*	Draft Residential Metals Abatement Program Quality Assurance Project Plan (Non-Residential Parcels – Indoor Dust) (12/17/2021)		
QAPP/FSP/SAP Preparer			
Period of Performance *(of QAPP/FSP/SAP)*	**2021-2022**	**Date Submitted for Review**	2/28/22
EPA Project Officer **EPA Project Manager**	Nikia Greene	**PO Phone #** **PM Phone #**	
QA Program Reviewer or Approving Official	Nikia Greene	**Date of Review**	3/15/22

Documents Submitted for QAPP Review (QA Reviewer must complete):

1. QA Document(s) submitted for review:

QA Document	Document Date	Document Stand-alone	Document with QAPP
QAPP	**10/08/2021**	**Yes** / No	
FSP		Yes / No	Yes / No
SAP		Yes / No	Yes / No
SOP(s)			**Yes** / No

2. WP/SOW/TO/PP/RP Date _____
WP/SOW/TO/RP Performance Period _____

3. QA document consistent with the:
WP/SOW/PP for grants? Yes / No
SOW/TO for contracts? Yes / No

4. QARF signed by R8 QAM Yes / No / NA
Funding Mechanism IA / contract / grant / NA
Amount _____

Notes for Document Submittals:

1. A QAPP written by a Grantee, EPA, or Federal Partner must include for review: Work Plan(WP) / Statement of Work (SOW) / Program Plan (PP) / Research Proposal (RP) and funding mechanism

2. A QAPP written by Contractor must include for review:
- **a)** Copy of Task Order Work Assignment/SOW
- **b)** Reference to a hard or electronic copy of the contractor's approved QMP
- **c)** Copy of Contract SOW if no QMP has been approved
- **d)** Copy of EPA/Court Order, if applicable
- **e)** The QA Review must determine (with the EPA CO or PO) if a QARF was completed for the environmental data activity described in the QAPP.

3. a. Field Sampling Plan (FSP) and/or Sampling & Analyses Plan (SAP) must include the Project QAPP ***or*** must be a stand-alone QA document that contain all QAPP required elements (Project Management, Data Generation/Acquisition, Assessment and Oversight, and Data Validation and Usability).

b. SOPs must be submitted with a QA document that contains all QAPP required elements.

Summary of Comments *(highlight significant concerns/issues)*:

1. Comment #1 – Please address the comments contained within the comment letter. Atlantic Richfield Response (12/16/2021): Comments addressed in comment letter. EPA comments resolved (3/15/22)

2. Comment #2 Atlantic Richfield Response (12/17/2021): The document title and period of performance have been revised. EPA comments resolved (3/15/22)
3. Comment #3
4. Atlantic Richfield **must address the comments in the Summary of Comments, as well as those identified in the Comment section(s) that includes a "Response (date)" and Resolved (date)".** Atlantic Richfield Response (12/17/2021): Comments addressed in comments sections below. EPA comments resolved (3/15/22)

Element	Acceptable *Yes/No/NA*	Page/ Section	Comments
A. Project Management			
A1. Title and Approval Sheet			
a. Contains project title	Yes	Title page and Signature page	EPA no comment (11/22/21)
b. Date and revision number line (for when needed)	Yes	Title page and Signature page	EPA no comment (11/22/21)
c. Indicates organization's name	Yes	Title page	EPA no comment (11/22/21)
d. Date and signature line for organization's project manager	No	Signature Page	EPA comment (11/22/21) – Please provide signatures with the revised plan Atlantic Richfield Response (12/16/2021): Signatures inserted. EPA comment resolved (3/15/22)
e. Date and signature line for organization's QA manager	No	Signature Page	EPA comment (11/22/21) – Please provide signatures with the revised plan Atlantic Richfield Response (12/16/2021): Signatures inserted EPA comment resolved (3/15/22)
f. Other date and signatures lines, as needed	Yes	Signature Page	EPA no comment (11/22/21)
A2. Table of Contents			
a. Lists QA Project Plan information sections	Yes	Pages iii to vi	EPA no comment (11/22/21)
b. Document control information indicated	Yes	Page v	EPA no comment (11/22/21)
A3. Distribution List			
Includes all individuals who are to receive a copy of the QA Project Plan and identifies their organization	No	Page ii	EPA comment (11/22/21) – Please provide this information with the revised plan Atlantic Richfield Response (12/16/2021): Distribution List inserted EPA comment resolved (3/15/22)
A4. Project/Task Organization			

a. Identifies key individuals involved in all major aspects of the project, including contractors	Yes	Sections 2.0 to 2.6	EPA no comment (11/22/21)
b. Discusses their responsibilities	Yes	Sections 2.0 to 2.6	EPA no comment (11/22/21)
c. Project QA Manager position indicates independence from unit generating data	Yes	Sections 2.0 to 2.6	EPA no comment (11/22/21)
d. Identifies individual responsible for maintaining the official, approved QA Project Plan	Yes	Section 2.6	EPA comment (11/22/21) – the name of the individual should be identified in this section Atlantic Richfield Response (12/16/2021): The name of the ERM QA Manager has been inserted. EPA comment resolved (3/15/22)
e. Organizational chart shows lines of authority and reporting responsibilities	Yes	Figure 3	Atlantic Richfield Response (12/16/2021): Figure 3 has been updated based on EPA comment letter.
A5. Problem Definition/Background			
a. States decision(s) to be made, actions to be taken, or outcomes expected from the information to be obtained	Yes	Sections 1.0 and 2.9	EPA comment (11/22/21) – please update based on the EPA comment letter Atlantic Richfield Response (12/16/2021): Sections have been updated based on EPA comment letter. EPA comment resolved (3/15/22)
b. Clearly explains the reason (site background or historical context) for initiating this project	Yes	Sections 2.7 and 2.8	EPA comment (11/22/21) – please update based on the EPA comment letter Atlantic Richfield Response (12/16/2021): Sections have been updated based on EPA comment letter. EPA comment resolved (3/15/22)
c. Identifies regulatory information, applicable criteria, action limits, etc. necessary to the project	Yes	Section 2.9	EPA comment (11/22/21) – please update based on the EPA comment letter Atlantic Richfield Response (12/16/2021): Section has been updated based on EPA comment letter. EPA comment resolved (3/15/22)
A6. Project/Task Description			
a. Summarizes work to be performed, for example, measurements to be made, data files to be obtained, etc., that support the project's goals	Yes	Sections 1.0 and 2.7	EPA comment (11/22/21) – please update based on the EPA comment letter Atlantic Richfield Response (12/16/2021): Sections have been updated based on EPA comment letter. EPA comment resolved (3/15/22)

b. Provides work schedule indicating critical project points, e.g., start and completion dates for activities such as sampling, analysis, data or file reviews, and assessments	Yes	Section 2.8	EPA comment (11/22/21) – please update based on the EPA comment letter Atlantic Richfield Response (12/16/2021): Section has been updated based on EPA comment letter. EPA comment resolved (3/15/22)
c. Details geographical locations to be studied, including maps where possible	Yes	Sections 1.0 and 2.8	EPA comment (11/22/21) – please update based on the EPA comment letter Atlantic Richfield Response (12/16/2021): Section has been updated based on EPA comment letter. EPA comment resolved (3/15/22)
d. Discusses resource and time constraints, if applicable	Yes	Section 2.8.1	EPA no comment (11/22/21)
A7. Quality Objectives and Criteria			
a. Identifies - performance/measurement criteria for all information to be collected and acceptance criteria for information obtained from previous studies, - including project action limits and laboratory detection limits and - range of anticipated concentrations of each parameter of interest	Yes	Section 2.9.1; Table 1	EPA comment (11/22/21) – please update based on the EPA comment letter Atlantic Richfield Response (12/16/2021): Sections have been updated based on EPA comment letter. EPA comment resolved (3/15/22)
b. Discusses precision	Yes	Sections 2.9.2, 3.9.2 and 3.10.1 (formerly 3.7.2, and 3.8.2)	EPA comment (11/22/21) – please update based on the EPA comment letter Atlantic Richfield Response (12/16/2021): Sections have been updated based on EPA comment letter. EPA comment resolved (3/15/22)
c. Addresses bias	Yes	Sections 2.9.2 and 3.9.2 (formerly 3.7.2)	EPA comment (11/22/21) – please update based on the EPA comment letter Atlantic Richfield Response (12/16/2021): Sections have been updated based on EPA comment letter. EPA comment resolved (3/15/22)
d. Discusses representativeness	Yes	Sections 2.9.2 and 3.9.2 (formerly 2.7.2 and 3.7.2)	EPA comment (11/22/21) – please update based on the EPA comment letter Atlantic Richfield Response (12/16/2021): Sections have been updated based on EPA comment letter. EPA comment resolved (3/15/22)

e. Identifies the need for completeness	Yes	Sections 2.9.2 and 3.9.2 (formerly 3.7.2)	EPA comment (11/22/21) – please update based on the EPA comment letter Atlantic Richfield Response (12/16/2021): Sections have been updated based on EPA comment letter. EPA comment resolved (3/15/22)
f. Describes the need for comparability	Yes	Sections 2.9.2 and 3.9.2 (formerly 3.7.2)	EPA comment (11/22/21) – please update based on the EPA comment letter Atlantic Richfield Response (12/16/2021): Sections have been updated based on EPA comment letter.
g. Discusses desired method sensitivity	Yes	Sections 2.9.2 and 3.9.2 (formerly 3.7.2)	EPA comment (11/22/21) – please update based on the EPA comment letter Atlantic Richfield Response (12/16/2021): Sections have been updated based on EPA comment letter. EPA comment resolved (3/15/22)
A8. Special Training/Certifications			
a. Identifies any project personnel specialized training or certifications	Yes	Section 2.10	EPA no comment (11/22/21)
b. Discusses how this training will be provided	Yes	Section 2.10	EPA no comment (11/22/21)
c. Indicates personnel responsible for assuring training/certifications are satisfied	Yes	Section 2.10	EPA no comment (11/22/21)
d. identifies where this information is documented	Yes	Section 2.10	EPA no comment (11/22/21)
A9. Documentation and Records			
a. Identifies report format and summarizes all data report package information	Yes	Section 2.11	EPA no comment (11/22/21)
b. Lists all other project documents, records, and electronic files that will be produced	Yes	Section 2.11	EPA no comment (11/22/21)
c. Identifies where project information should be kept and for how long	Yes	Section 2.11	EPA no comment (11/22/21)
d. Discusses back up plans for records stored electronically	Yes	Section 2.11	EPA no comment (11/22/21)
e. States how individuals identified in A3 will receive the most current copy of the approved QA Project Plan, identifying the individual responsible for this	Yes	Section 2.11	EPA no comment (11/22/21)
B. Data Generation/Acquisition			

B1. Sampling Process Design (Experimental Design)			
a. Describes and justifies design strategy, indicating size of the area, volume, or time period to be represented by a sample	Yes	Section 3.0	EPA comment (11/22/21) – please update based on the EPA comment letter Atlantic Richfield Response (12/16/2021): Sections have been updated based on EPA comment letter. EPA comment resolved (3/15/22)
b. Details the type and total number of sample types/matrix or test runs/trials expected and needed	Yes	Section 3.2	EPA comment (11/22/21) – please update based on the EPA comment letter Atlantic Richfield Response (12/16/2021): Sections have been updated based on EPA comment letter. EPA comment resolved (3/15/22)
c. Indicates where samples should be taken, how sites will be identified/located	Yes	Section 3.2.1; FSP	EPA comment (11/22/21) – please update based on the EPA comment letter Atlantic Richfield Response (12/16/2021): Sections have been updated based on EPA comment letter. EPA comment resolved (3/15/22)
d. Discusses what to do if sampling sites become inaccessible	Yes	Section 3.2.2 to 3.2.5 (formerly 3.2.6)	EPA comment (11/22/21) – please update based on the EPA comment letter Atlantic Richfield Response (12/16/2021): Sections have been updated based on EPA comment letter. EPA comment resolved (3/15/22)
e. Identifies project activity schedules such as each sampling event, times samples should be sent to the laboratory, etc.	Yes	Sections 3.2 to 3.5	EPA comment (11/22/21) – please update based on the EPA comment letter Atlantic Richfield Response (12/16/2021): Sections have been updated based on EPA comment letter. EPA comment resolved (3/15/22)
f. Specifies what information is critical and what is for informational purposes only	Yes	Sections 3.2 to 3.5	EPA comment (11/22/21) – please update based on the EPA comment letter Atlantic Richfield Response (12/16/2021): Sections have been updated based on EPA comment letter. EPA comment resolved (3/15/22)
g. Identifies sources of variability and how this variability should be reconciled with project information	Yes	Sections 3.7 and 3.8	EPA comment (11/22/21) – please update based on the EPA comment letter Atlantic Richfield Response (12/16/2021): Sections have been updated based on EPA comment letter. EPA comment resolved (3/15/22)
B2. Sampling Methods			

a. Identifies all sampling SOPs by number, date, and regulatory citation, indicating sampling options or modifications to be taken	Yes	Sections 3.2, 3.3, and 3.5	EPA comment (11/22/21) – please update based on the EPA comment letter Atlantic Richfield Response (12/16/2021): Sections have been updated based on EPA comment letter. EPA comment resolved (3/15/22)
b. Indicates how each sample/matrix type should be collected	Yes	Sections 3.2 to 3.5	EPA comment (11/22/21) – please update based on the EPA comment letter Atlantic Richfield Response (12/16/2021): Sections have been updated based on EPA comment letter. EPA comment resolved (3/15/22)
c. If in situ monitoring, indicates how instruments should be deployed and operated to avoid contamination and ensure maintenance of proper data	NA	NA	NA
d. If continuous monitoring, indicates averaging time and how instruments should store and maintain raw data, or data averages	NA	NA	NA
e. Indicates how samples are to be homogenized, composited, split, or filtered, if needed	Yes (formerly NA)	Section 3.3.1 (formerly NA)	NA Atlantic Richfield Response (12/16/2021): Section has been inserted based on request to collect earthen basement soil samples. EPA comment resolved (3/15/22)
f. Indicates what sample containers and sample volumes should be used	Yes	Sections 3.2 to 3.5	EPA comment (11/22/21) – please update based on the EPA comment letter Atlantic Richfield Response (12/16/2021): Sections have been updated based on EPA comment letter. EPA comment resolved (3/15/22)
g. Identifies whether samples should be preserved and indicates methods that should be followed	Yes	Sections 3.5	EPA comment (11/22/21) – Section 3.5 is field equipment. Please update to the appropriate section. Atlantic Richfield Response (12/16/2021): Section numbers have been updated. Section 3.5 (formerly 3.4) is Field Procedures. EPA comment resolved (3/15/22)
h. Indicates whether sampling equipment and samplers should be cleaned and/or decontaminated, identifying how this should be done and by-products disposed of	Yes	Section 3.5.2 (incorrectly listed as 3.2.4), FS-WI -010	EPA no comment (11/22/21) Atlantic Richfield Response (12/16/2021): Section numbers have been updated. Section 3.5.2 (formerly 3.4.2) is now Floor Surface Sampling. EPA comment resolved (3/15/22)

i. Identifies any equipment and support facilities needed	Yes	Sections 3.6 (incorrectly listed as 3.4 and 3.10)	EPA comment (11/22/21) – please update to the appropriate section. Atlantic Richfield Response (12/16/2021): Section numbers have been updated. Section 3.6 (formerly 3.5) is now Field Equipment. EPA comment resolved (3/15/22)
j. Addresses actions to be taken when problems occur, identifying individual(s) responsible for corrective action and how this should be documented	Yes	Section 4.1	EPA no comment (11/22/21)
B3. Sample Handling and Custody			
a. States maximum holding times allowed from sample collection to extraction and/or analysis for each sample type and, for in-situ or continuous monitoring, the maximum time before retrieval of information	Yes	Section 3.7 (incorrectly listed as 3.5)	EPA comment (11/22/21) – please update to the appropriate section. Atlantic Richfield Response (12/16/2021): Section numbers have been updated. Section 3.7 (formerly 3.6) is now Sample Handling and Chain of Custody. EPA comment resolved (3/15/22)
b. Identifies how samples or information should be physically handled, transported, and then received and held in the laboratory or office (including temperature upon receipt)	Yes	Section 3.7 (incorrectly listed as 3.5)	EPA comment (11/22/21) – please update to the appropriate section. Atlantic Richfield Response (12/16/2021): Section numbers have been updated. Section 3.7 (formerly 3.6) is now Sample Handling and Chain of Custody. EPA comment resolved (3/15/22)
c. Indicates how sample or information handling and custody information should be documented, such as in field notebooks and forms, identifying individual responsible	Yes	Sections 2.11.2, 2.11.4 and 3.7 (incorrectly listed as 3.5)	EPA comment (11/22/21) – please update to the appropriate section. Atlantic Richfield Response (12/16/2021): Section numbers have been updated. Section 3.7 (formerly 3.6) is now Sample Handling and Chain of Custody. Added reference to Section 2.11.2 Field Documentation. EPA comment resolved (3/15/22)
d. Discusses system for identifying samples, for example, numbering system, sample tags and labels, and attaches forms to the plan	Yes	Section 3.8 (incorrectly listed as 3.6)	EPA comment (11/22/21) – please update to the appropriate section. Atlantic Richfield Response (12/16/2021): Section numbers have been updated. Section 3.8 (formerly 3.7) is now Sample Identification EPA comment resolved (3/15/22)
e. Identifies chain-of-custody procedures and includes form to track custody	Yes	Sections 2.11.4 and 3.7	EPA comment (11/22/21) – please update to the appropriate section. Atlantic Richfield Response (12/16/2021): Section numbers have been updated. Section 3.7 (formerly 3.6) is now Sample Handling and Chain of Custody. EPA comment resolved (3/15/22)
B4. Analytical Methods			

a. Identifies all analytical SOPs (field, laboratory and/or office) that should be followed by number, date, and regulatory citation, indicating options or modifications to be taken, such as sub-sampling and extraction procedures	Yes	Section 3.9 (incorrectly listed as 3.7), Appendix C	EPA comment (11/22/21) – please update based on the EPA comment letter Atlantic Richfield Response (12/16/2021): Section numbers have been updated. Section 3.9 (formerly 3.8) is now Analyses Methods EPA comment resolved (3/15/22)
b. Identifies equipment or instrumentation needed	Yes	Section 3.9.1 (incorrectly listed as 3.7)	EPA comment (11/22/21) – please update to the appropriate section. Atlantic Richfield Response (12/16/2021): Section numbers have been updated. Section 3.9.1 (formerly 3.8.1) is now Dust Sample Analysis Methods EPA comment resolved (3/15/22)
c. Specifies any specific method performance criteria	Yes	Sections 2.9.2 and 3.9.2 (incorrectly listed as 3.7.2)	EPA comment (11/22/21) – please update to the appropriate section. Atlantic Richfield Response (12/16/2021): Section numbers have been updated. Section 3.9.1 (formerly 3.8.1) is now Dust Sample Analysis Methods EPA comment resolved (3/15/22)
d. Identifies procedures to follow when failures occur, identifying individual responsible for corrective action and appropriate documentation	Yes	Sections 3.9.2.1, 3.9.2.2, 3.9.2.3, 4.1, Appendix B	EPA comment (11/22/21) – please update to the appropriate section. Atlantic Richfield Response (12/16/2021): Included specific laboratory control sample subsections from Sections 3.9.2 that discuss corrective action. Section 4.1 paragraph 5 discusses corrective action during analysis. Appendix B, Laboratory SOPs also include method specific corrective action procedures. EPA comment resolved (3/15/22)
e. Identifies sample disposal procedures	Yes	Section 3.11 (incorrectly listed as 3.9)	EPA comment (11/22/21) – please update to the appropriate section. Atlantic Richfield Response (12/16/2021): Section numbers have been updated. Section 3.11 (formerly 3.10) is now Sample Disposal EPA comment resolved (3/15/22)
f. Specifies laboratory turnaround times needed	Yes	Sections 2.9.1 and 4.3	EPA comment (11/22/21) – please update to the appropriate section. Section 2.9.1 **Step 3 Identifying appropriate sampling and analytical methods,** paragraph 2 discusses laboratory turnaround times. Section 4.3 Reports to Management, paragraph 2 discusses turnaround times. EPA comment resolved (3/15/22)
g. Provides method validation information and SOPs for nonstandard methods	Yes	Section 5.0	EPA no comment (11/22/21)
B5. Quality Control			

a. For each type of sampling, analysis, or measurement technique, identifies QC activities which should be used, for example, blanks, spikes, duplicates, etc., and at what frequency	Yes	Sections 3.9.2 and 3.10 (formerly 3.7 and 3.8)	EPA no comment (11/22/21) Atlantic Richfield Response (12/16/2021): Section numbers have been updated. Section 3.9.2 (formerly 3.8.2) is Laboratory Quality Control Samples. Section 3.10 (formerly 3.9) is Field Quality Control Samples. EPA no comment (3/15/22)
b. Details what should be done when control limits are exceeded, and how effectiveness of control actions will be determined and documented	Yes	Section 5.0	EPA no comment (11/22/21)
c. Identifies procedures and formulas for calculating applicable QC statistics, for example, for precision, bias, outliers and missing data	Yes	Sections 2.9.2, 3.9.2, 3.10 (formerly 3.7, 3.8), and Table 2	EPA no comment (11/22/21) Atlantic Richfield Response (12/16/2021): Section numbers have been updated. Section 3.9.2 (formerly 3.8.2) is Laboratory Quality Control Samples. Section 3.10 (formerly 3.9) is Field Quality Control Samples. EPA no comment (3/15/22)
B6. Instrument/Equipment Testing, Inspection, and Maintenance			
a. Identifies field and laboratory equipment needing periodic maintenance, and the schedule for this	Yes	Section 3.12 (incorrectly listed as 3.10)	EPA comment (11/22/21) – please update to the appropriate section. Atlantic Richfield Response (12/16/2021): Section numbers have been updated. Section 3.12 (formerly 3.11) is Instrument/Equipment Testing, Inspection, and Maintenance EPA comment resolved (3/15/22)
b. Identifies testing criteria	Yes	Section 3.12 (incorrectly listed as 3.10)	EPA comment (11/22/21) – please update to the appropriate section. Atlantic Richfield Response (12/16/2021): Section numbers have been updated. Section 3.12 (formerly 3.11) is Instrument/Equipment Testing, Inspection, and Maintenance EPA comment resolved (3/15/22)
c. Notes availability and location of spare parts	Yes	Section 3.12 (incorrectly listed as 3.10)	EPA comment (11/22/21) – please update to the appropriate section. Atlantic Richfield Response (12/16/2021): Section numbers have been updated. Section 3.12 (formerly 3.11) is Instrument/Equipment Testing, Inspection, and Maintenance EPA comment resolved (3/15/22)
d. Indicates procedures in place for inspecting equipment before usage	Yes	Section 3.12 (incorrectly listed as 3.10)	EPA comment (11/22/21) – please update to the appropriate section. Atlantic Richfield Response (12/16/2021): Section numbers have been updated. Section 3.12 (formerly 3.11) is Instrument/Equipment Testing, Inspection, and Maintenance EPA comment resolved (3/15/22)

e. Identifies individual(s) responsible for testing, inspection and maintenance	Yes	Section 3.12 (incorrectly listed as 3.10)	EPA comment (11/22/21) – please update to the appropriate section. Atlantic Richfield Response (12/16/2021): Section numbers have been updated. Section 3.12 (formerly 3.11) is Instrument/Equipment Testing, Inspection, and Maintenance EPA comment resolved (3/15/22)
f. Indicates how deficiencies found should be resolved, re-inspections performed, and effectiveness of corrective action determined and documented	Yes	Section 3.12 (incorrectly listed as 3.10)	EPA comment (11/22/21) – please update to the appropriate section. Atlantic Richfield Response (12/16/2021): Section numbers have been updated. Section 3.12 (formerly 3.11) is Instrument/Equipment Testing, Inspection, and Maintenance EPA comment resolved (3/15/22)
B7. Instrument/Equipment Calibration and Frequency			
a. Identifies equipment, tools, and instruments that should be calibrated and the frequency for this calibration	Yes	Sections 3.12.1, 3.12.2, and Appendix B (Incorrectly listed 2.9.2, and 3.8)	EPA comment (11/22/21) – please update to the appropriate section. Atlantic Richfield Response (12/16/2021): Section numbers have been updated. Section 3.12.1 (formerly 3.11.1) is Field Equipment and Section 3.12.2 is Laboratory Equipment. Calibration is also included in the Appendix B SOPs for laboratory methods. EPA comment resolved (3/15/22)
b. Describes how calibrations should be performed and documented, indicating test criteria and standards or certified equipment	Yes	Sections 3.12.1, 3.12.2, and Appendix B (Incorrectly listed as 2.9.2 and 3.10; Appendix C)	EPA comment (11/22/21) – please update to the appropriate section. Atlantic Richfield Response (12/16/2021): Section numbers have been updated. Section 3.12.1 (formerly 3.11.1) is Field Equipment and Section 3.12.2 is Laboratory Equipment. Calibration is also included in the Appendix B SOPs for laboratory methods. EPA comment resolved (3/15/22)
c. Identifies how deficiencies should be resolved and documented	Yes	Section 4.1 (incorrectly listed as 5.1)	EPA comment (11/22/21) – please update to the appropriate section. Atlantic Richfield Response (12/16/2021): Section numbers have been updated. Section 3.12.1 (formerly 3.11.1) is Field Equipment and Section 3.12.2 is Laboratory Equipment. EPA comment resolved (3/15/22)
B8. Inspection/Acceptance for Supplies and Consumables			

a. Identifies critical supplies and consumables for field and laboratory, noting supply source, acceptance criteria, and procedures for tracking, storing and retrieving these materials	Yes	Sections 3.6, 3.13 (incorrectly listed as 3.11), and Appendix B	EPA comment (11/22/21) – please update to the appropriate section. Atlantic Richfield Response (12/16/2021): Section numbers have been updated. Section 3.13 (formerly 3.12) is Inspection/Acceptance of Supplies and Consumables. Section 3.6 is Field Equipment. Appendix B SOPs for laboratory methods include laboratory supplies. EPA comment resolved (3/15/22)
b. Identifies the individual(s) responsible for this	Yes	Section 3.13 (incorrectly listed as 3.11)	EPA comment (11/22/21) – please provide this information and update the section accordingly. Atlantic Richfield Response (12/16/2021): Section numbers have been updated. Section 3.13 (formerly 3.12) is Inspection/Acceptance of Supplies and Consumables. Field supplies will be inspected by the Field Team Leader (may vary). Laboratory supplies are inspected by laboratory personnel (may vary). EPA comment resolved (3/15/22)
B9. Use of Existing Data (Non-direct Measurements)			
a. Identifies data sources, for example, computer databases or literature files, or models that should be accessed and used	Yes	Section 3.14 (Incorrectly listed as 5.0)	EPA comment (11/22/21) – please provide this information and update the section accordingly. Atlantic Richfield Response (12/16/2021): Inserted Section 3.14 Non-Direct Measurement Data Acquisition Requirements EPA comment resolved (3/15/22)
b. Describes the intended use of this information and the rationale for their selection, i.e., its relevance to project	Yes	Section 3.14 (Incorrectly listed as 5.0)	EPA comment (11/22/21) – please provide this information and update the section accordingly. Atlantic Richfield Response (12/16/2021): Inserted Section 3.14 Non-Direct Measurement Data Acquisition Requirements EPA comment resolved (3/15/22)
c. Indicates the acceptance criteria for these data sources and/or models	Yes	Section 3.14 (Incorrectly listed as 5.0)	EPA comment (11/22/21) – please provide this information and update the section accordingly. Atlantic Richfield Response (12/16/2021): Inserted Section 3.14 Non-Direct Measurement Data Acquisition Requirements EPA comment resolved (3/15/22)
d. Identifies key resources/support facilities needed	Yes	Section 3.14 (Incorrectly listed as 5.0)	EPA comment (11/22/21) – please provide this information and update the section accordingly. Atlantic Richfield Response (12/16/2021): Inserted Section 3.14 Non-Direct Measurement Data Acquisition Requirements EPA comment resolved (3/15/22)

e. Describes how limits to validity and operating conditions should be determined, for example, internal checks of the program and Beta testing	Yes	Section 3.14 (Incorrectly listed as 5.0)	EPA comment (11/22/21) – please provide this information and update the section accordingly. Atlantic Richfield Response (12/16/2021): Inserted Section 3.14 Non-Direct Measurement Data Acquisition Requirements EPA comment resolved (3/15/22)
B10. Data Management			
a. Describes data management scheme from field to final use and storage	Yes	Section 3.15 (incorrectly listed as 3.12)	EPA comment (11/22/21) – please provide this information and update the section accordingly. Atlantic Richfield Response (12/16/2021): Section numbers have been updated. Section 3.15 (formerly 3.13) is Date Management Procedures) EPA comment resolved (3/15/22)
b. Discusses standard record-keeping and tracking practices, and the document control system or cites other written documentation such as SOPs	Yes	Section 3.15 (incorrectly listed as 3.12)	EPA comment (11/22/21) – please provide this information and update the section accordingly. Atlantic Richfield Response (12/16/2021): Section numbers have been updated. Section 3.15 (formerly 3.13) is Date Management Procedures) EPA comment resolved (3/15/22)
c. Identifies data handling equipment/procedures that should be used to process, compile, analyze, and transmit data reliably and accurately	Yes	Section 3.15 (incorrectly listed as 3.12)	EPA comment (11/22/21) – please provide this information and update the section accordingly. Atlantic Richfield Response (12/16/2021): Section numbers have been updated. Section 3.15 (formerly 3.13) is Date Management Procedures) EPA comment resolved (3/15/22)
d. Identifies individual(s) responsible for this	Yes	Section 3.15 (incorrectly listed as 3.12)	EPA comment (11/22/21) – please provide this information and update the section accordingly. Atlantic Richfield Response (12/16/2021): Section numbers have been updated. Section 3.15 (formerly 3.13) is Date Management Procedures) EPA comment resolved (3/15/22)
e. Describes the process for data archival and retrieval	Yes	Section 3.15 (incorrectly listed as 3.12)	EPA comment (11/22/21) – please provide this information and update the section accordingly. Atlantic Richfield Response (12/16/2021): Section numbers have been updated. Section 3.15 (formerly 3.13) is Date Management Procedures) EPA comment resolved (3/15/22)
f. Describes procedures to demonstrate acceptability of hardware and software configurations	Yes	Section 3.15 (incorrectly listed as 3.12)	EPA comment (11/22/21) – please provide this information and update the section accordingly. Atlantic Richfield Response (12/16/2021): Section numbers have been updated. Section 3.15 (formerly 3.13) is Date Management Procedures)

g. Attaches checklists and forms that should be used	Yes	Section 3.15 (incorrectly listed as 3.12)	EPA comment (11/22/21) – please provide this information and update the section accordingly. Atlantic Richfield Response (12/16/2021): Section numbers have been updated. Section 3.15 (formerly 3.13) is Date Management Procedures) EPA comment resolved (3/15/22)
C. Assessment and Oversight			
C1. Assessments and Response Actions			
a. Lists the number, frequency, and type of assessment activities that should be conducted, with the approximate dates	Yes	Section 4. (formerly 4.0)	EPA comment (11/22/21) – please provide this information and update the section accordingly. Atlantic Richfield Response (12/16/2021): Section 4. has been updated based on EPA comment letter. EPA comment resolved (3/15/22)
b. Identifies individual(s) responsible for conducting assessments, indicating their authority to issue stop work orders, and any other possible participants in the assessment process	Yes	Sections 4.1 and 4.2 (formerly 4.0)	EPA comment (11/22/21) – please provide this information and update the section accordingly. Atlantic Richfield Response (12/16/2021): Section references have been updated to include information. EPA comment resolved (3/15/22)
c. Describes how and to whom assessment information should be reported	Yes	Sections 4.1, 4.2, and 4.3	EPA comment (11/22/21) – please provide this information and update the section accordingly. Atlantic Richfield Response (12/16/2021): Section references have been updated to include information. EPA comment resolved (3/15/22)
d. Identifies how corrective actions should be addressed and by whom, and how they should be verified and documented	Yes	Sections 4.2, 4.3 (formerly 4.1 and 4.2), and Appendix F	EPA comment (11/22/21) – please provide this information and update the section accordingly. Atlantic Richfield Response (12/16/2021): Section references have been updated to include information. EPA comment resolved (3/15/22)
C2. Reports to Management			
a. Identifies what project QA status reports are needed and how frequently	Yes	Section 4.3	EPA comment (11/22/21) – please provide this information and update the section accordingly. Atlantic Richfield Response (12/16/2021): Section 4.3, Paragraph 3 "A separate report will be prepared by the consultant QA manager, as needed, to communicate the results of performance evaluations or program audits to identify specific significant QA issues and provided to the USEPA for review. Any corrective action reporting described in Section 4.2 above will be summarized and included as appropriate." EPA comment resolved (3/15/22)

b. Identifies who should write these reports and who should receive this information	Yes	Section 4.3	EPA comment (11/22/21) – please provide this information and update the section accordingly. Atlantic Richfield Response (12/16/2021): Section 4.3, Paragraph 3 "A separate report will be prepared by the consultant QA manager, as needed, to communicate the results of performance evaluations or program audits to identify specific significant QA issues and provided to the USEPA for review. Any corrective action reporting described in Section 4.2 above will be summarized and included as appropriate." EPA comment resolved (3/15/22)
D. Data Validation and Usability			
D1. Data Review, Verification, and Validation			
Describes criteria that should be used for accepting, rejecting, or qualifying project data	Yes	Sections 5.2 and 5.3 (formerly 5.0)	EPA comment (11/22/21) – please provide this information and update the section accordingly. Atlantic Richfield Response (12/16/2021): Section references have been updated to include information. Sections 5.2.3 and 5.3 were revised to include missing information. EPA comment resolved (3/15/22)
D2. Verification and Validation Methods			
a. Describes process for data verification and validation, providing SOPs and indicating what data validation software should be used, if any	Yes	Sections 5.1.2, 5.1.3, and 5.2 (formerly 5.0)	EPA comment (11/22/21) – please provide this information and update the section accordingly. Atlantic Richfield Response (12/16/2021): Section references have been updated to include information. EPA comment resolved (3/15/22)
b. Identifies who is responsible for verifying and validating different components of the project data/information, for example, chain-of-custody forms, receipt logs, calibration information, etc.	Yes	Sections 5.1.2 and 5.1.3 (formerly 5.0)	EPA comment (11/22/21) – please provide this information and update the section accordingly. Atlantic Richfield Response (12/16/2021): Section references have been updated to include information. EPA comment resolved (3/15/22)
c. Identifies issue resolution process, and method and individual responsible for conveying these results to data users	Yes	Sections 5.1.1, 5.1.2, and 5.2.2 (formerly 5.0)	EPA comment (11/22/21) – please provide this information and update the section accordingly. Atlantic Richfield Response (12/16/2021): Section references have been updated to include information. EPA comment resolved (3/15/22)

d. Attaches checklists, forms, and calculations	Yes	Section 5.2 (formerly 5.0), Appendix D, and Appendix G	EPA comment (11/22/21) – please provide this information and update the section accordingly. Atlantic Richfield Response (12/16/2021): Section references have been updated to include information. EPA comment resolved (3/15/22)
D3. Reconciliation with User Requirements			
a. Describes procedures to evaluate the uncertainty of the validated data	Yes	Section 5.3 (formerly 5.0)	EPA comment (11/22/21) – please provide this information and update the section accordingly. Atlantic Richfield Response (12/16/2021): Sections 5.3 has been inserted. EPA comment resolved (3/15/22)
b. Describes how limitations on data use should be reported to the data users	Yes	Section 5.3 (formerly 5.0)	EPA comment (11/22/21) – please provide this information and update the section accordingly. Atlantic Richfield Response (12/16/2021): Section 5.3 has been inserted. EPA comment resolved (3/15/22)

EPA REGION 8 QA DOCUMENT REVIEW CROSSWALK

QAPP/FSP/SAP for: *(check appropriate box)* ☐ **GRANTEE** ☐ **CONTRACTOR** ☐ **EPA** ☐ **Other**	**Entity** *(grantee, contract, EPA AO, EPA Program, Other)* Atlantic Richfield	**Regulatory Authority** **and/or** **Funding Mechanism**	___ **2 CFR 1500 for Grantee/Cooperative Agreements** ___ **48 CFR 46 for Contracts** ___ **Interagency Agreement (FFA, USGS,)** ___ **EPA/Court Order** ___ **EPA Program Funding** ___ **EPA Program Regulation** ___ **EPA CIO 2105**
Document Title ***[Note: Title will be repeated in Header]***	Draft Residential Metals Abatement Program Quality Assurance Project Plan (Non-Residential Parcels – Indoor Dust) (12/17/2021)		
QAPP/FSP/SAP Preparer			
Period of Performance *(of QAPP/FSP/SAP)*	**2021-2022**	**Date Submitted for Review**	10/08/2021
EPA Project Officer **EPA Project Manager**	Nikia Greene	**PO Phone #** **PM Phone #**	
QA Program Reviewer or Approving Official	Nikia Greene	**Date of Review**	11/22/21

Documents Submitted for QAPP Review (QA Reviewer must complete):

1. QA Document(s) submitted for review:

QA Document	Document Date	Document Stand-alone	Document with QAPP
QAPP	**10/08/2021**	**Yes** / No	
FSP		Yes / No	Yes / No
SAP		Yes / No	Yes / No
SOP(s)			**Yes** / No

2. WP/SOW/TO/PP/RP Date _____
WP/SOW/TO/RP Performance Period _____

3. QA document consistent with the:
WP/SOW/PP for grants? Yes / No
SOW/TO for contracts? Yes / No

4. QARF signed by R8 QAM Yes / No / NA
Funding Mechanism IA / contract / grant / NA
Amount _____

Notes for Document Submittals:

1. A QAPP written by a Grantee, EPA, or Federal Partner must include for review: Work Plan(WP) / Statement of Work (SOW) / Program Plan (PP) / Research Proposal (RP) and funding mechanism

2. A QAPP written by Contractor must include for review:
- **a)** Copy of Task Order Work Assignment/SOW
- **b)** Reference to a hard or electronic copy of the contractor's approved QMP
- **c)** Copy of Contract SOW if no QMP has been approved
- **d)** Copy of EPA/Court Order, if applicable
- **e)** The QA Review must determine (with the EPA CO or PO) if a QARF was completed for the environmental data activity described in the QAPP.

3. a. Field Sampling Plan (FSP) and/or Sampling & Analyses Plan (SAP) must include the Project QAPP ***or*** must be a stand-alone QA document that contain all QAPP required elements (Project Management, Data Generation/Acquisition, Assessment and Oversight, and Data Validation and Usability).

b. SOPs must be submitted with a QA document that contains all QAPP required elements.

Summary of Comments *(highlight significant concerns/issues)***:**

1. Comment #1 – Please address the comments contained within the comment letter. Atlantic Richfield Response (12/16/2021): Comments addressed in comment letter.

2. Comment #2 Atlantic Richfield Response (12/17/2021): The document title and period of performance have been revised.
3. Comment #3
4. Atlantic Richfield **must address the comments in the Summary of Comments, as well as those identified in the Comment section(s) that includes a "Response (date)" and Resolved (date)".** Atlantic Richfield Response (12/17/2021): Comments addressed in comments sections below.

Element	Acceptable *Yes/No/NA*	Page/ Section	Comments
A. Project Management			
A1. Title and Approval Sheet			
a. Contains project title	Yes	Title page and Signature page	EPA no comment (11/22/21)
b. Date and revision number line (for when needed)	Yes	Title page and Signature page	EPA no comment (11/22/21)
c. Indicates organization's name	Yes	Title page	EPA no comment (11/22/21)
d. Date and signature line for organization's project manager	No	Signature Page	EPA comment (11/22/21) – Please provide signatures with the revised plan Atlantic Richfield Response (12/16/2021): Signatures inserted.
e. Date and signature line for organization's QA manager	No	Signature Page	EPA comment (11/22/21) – Please provide signatures with the revised plan Atlantic Richfield Response (12/16/2021): Signatures inserted
f. Other date and signatures lines, as needed	Yes	Signature Page	EPA no comment (11/22/21)
A2. Table of Contents			
a. Lists QA Project Plan information sections	Yes	Pages iii to vi	EPA no comment (11/22/21)
b. Document control information indicated	Yes	Page v	EPA no comment (11/22/21)
A3. Distribution List			
Includes all individuals who are to receive a copy of the QA Project Plan and identifies their organization	No	Page ii	EPA comment (11/22/21) – Please provide this information with the revised plan Atlantic Richfield Response (12/16/2021): Distribution List inserted
A4. Project/Task Organization			
a. Identifies key individuals involved in all major aspects of the project, including contractors	Yes	Sections 2.0 to 2.6	EPA no comment (11/22/21)
b. Discusses their responsibilities	Yes	Sections 2.0 to 2.6	EPA no comment (11/22/21)

c. Project QA Manager position indicates independence from unit generating data	Yes	Sections 2.0 to 2.6	EPA no comment (11/22/21)
d. Identifies individual responsible for maintaining the official, approved QA Project Plan	Yes	Section 2.6	EPA comment (11/22/21) – the name of the individual should be identified in this section Atlantic Richfield Response (12/16/2021): The name of the ERM QA Manager has been inserted.
e. Organizational chart shows lines of authority and reporting responsibilities	Yes	Figure 3	Atlantic Richfield Response (12/16/2021): Figure 3 has been updated based on EPA comment letter.
A5. Problem Definition/Background			
a. States decision(s) to be made, actions to be taken, or outcomes expected from the information to be obtained	Yes	Sections 1.0 and 2.9	EPA comment (11/22/21) – please update based on the EPA comment letter Atlantic Richfield Response (12/16/2021): Sections have been updated based on EPA comment letter.
b. Clearly explains the reason (site background or historical context) for initiating this project	Yes	Sections 2.7 and 2.8	EPA comment (11/22/21) – please update based on the EPA comment letter Atlantic Richfield Response (12/16/2021): Sections have been updated based on EPA comment letter.
c. Identifies regulatory information, applicable criteria, action limits, etc. necessary to the project	Yes	Section 2.9	EPA comment (11/22/21) – please update based on the EPA comment letter Atlantic Richfield Response (12/16/2021): Section has been updated based on EPA comment letter.
A6. Project/Task Description			
a. Summarizes work to be performed, for example, measurements to be made, data files to be obtained, etc., that support the project's goals	Yes	Sections 1.0 and 2.7	EPA comment (11/22/21) – please update based on the EPA comment letter Atlantic Richfield Response (12/16/2021): Sections have been updated based on EPA comment letter.
b. Provides work schedule indicating critical project points, e.g., start and completion dates for activities such as sampling, analysis, data or file reviews, and assessments	Yes	Section 2.8	EPA comment (11/22/21) – please update based on the EPA comment letter Atlantic Richfield Response (12/16/2021): Section has been updated based on EPA comment letter.
c. Details geographical locations to be studied, including maps where possible	Yes	Sections 1.0 and 2.8	EPA comment (11/22/21) – please update based on the EPA comment letter Atlantic Richfield Response (12/16/2021): Section has been updated based on EPA comment letter.
d. Discusses resource and time constraints, if applicable	Yes	Section 2.8.1	EPA no comment (11/22/21)
A7. Quality Objectives and Criteria			

a. Identifies - performance/measurement criteria for all information to be collected and acceptance criteria for information obtained from previous studies, - including project action limits and laboratory detection limits and - range of anticipated concentrations of each parameter of interest	Yes	Section 2.9.1; Table 1	EPA comment (11/22/21) – please update based on the EPA comment letter Atlantic Richfield Response (12/16/2021): Sections have been updated based on EPA comment letter.
b. Discusses precision	Yes	Sections 2.9.2, 3.9.2 and 3.10.1 (formerly 3.7.2, and 3.8.2)	EPA comment (11/22/21) – please update based on the EPA comment letter Atlantic Richfield Response (12/16/2021): Sections have been updated based on EPA comment letter.
c. Addresses bias	Yes	Sections 2.9.2 and 3.9.2 (formerly 3.7.2)	EPA comment (11/22/21) – please update based on the EPA comment letter Atlantic Richfield Response (12/16/2021): Sections have been updated based on EPA comment letter.
d. Discusses representativeness	Yes	Sections 2.9.2 and 3.9.2 (formerly 2.7.2 and 3.7.2)	EPA comment (11/22/21) – please update based on the EPA comment letter Atlantic Richfield Response (12/16/2021): Sections have been updated based on EPA comment letter.
e. Identifies the need for completeness	Yes	Sections 2.9.2 and 3.9.2 (formerly 3.7.2)	EPA comment (11/22/21) – please update based on the EPA comment letter Atlantic Richfield Response (12/16/2021): Sections have been updated based on EPA comment letter.
f. Describes the need for comparability	Yes	Sections 2.9.2 and 3.9.2 (formerly 3.7.2)	EPA comment (11/22/21) – please update based on the EPA comment letter Atlantic Richfield Response (12/16/2021): Sections have been updated based on EPA comment letter.
g. Discusses desired method sensitivity	Yes	Sections 2.9.2 and 3.9.2 (formerly 3.7.2)	EPA comment (11/22/21) – please update based on the EPA comment letter Atlantic Richfield Response (12/16/2021): Sections have been updated based on EPA comment letter.

A8. Special Training/Certifications			
a. Identifies any project personnel specialized training or certifications	Yes	Section 2.10	EPA no comment (11/22/21)
b. Discusses how this training will be provided	Yes	Section 2.10	EPA no comment (11/22/21)
c. Indicates personnel responsible for assuring training/certifications are satisfied	Yes	Section 2.10	EPA no comment (11/22/21)
d. identifies where this information is documented	Yes	Section 2.10	EPA no comment (11/22/21)
A9. Documentation and Records			
a. Identifies report format and summarizes all data report package information	Yes	Section 2.11	EPA no comment (11/22/21)
b. Lists all other project documents, records, and electronic files that will be produced	Yes	Section 2.11	EPA no comment (11/22/21)
c. Identifies where project information should be kept and for how long	Yes	Section 2.11	EPA no comment (11/22/21)
d. Discusses back up plans for records stored electronically	Yes	Section 2.11	EPA no comment (11/22/21)
e. States how individuals identified in A3 will receive the most current copy of the approved QA Project Plan, identifying the individual responsible for this	Yes	Section 2.11	EPA no comment (11/22/21)
B. Data Generation/Acquisition			
B1. Sampling Process Design (Experimental Design)			
a. Describes and justifies design strategy, indicating size of the area, volume, or time period to be represented by a sample	Yes	Section 3.0	EPA comment (11/22/21) – please update based on the EPA comment letter Atlantic Richfield Response (12/16/2021): Sections have been updated based on EPA comment letter.
b. Details the type and total number of sample types/matrix or test runs/trials expected and needed	Yes	Section 3.2	EPA comment (11/22/21) – please update based on the EPA comment letter Atlantic Richfield Response (12/16/2021): Sections have been updated based on EPA comment letter.
c. Indicates where samples should be taken, how sites will be identified/located	Yes	Section 3.2.1; FSP	EPA comment (11/22/21) – please update based on the EPA comment letter Atlantic Richfield Response (12/16/2021): Sections have been updated based on EPA comment letter.

d. Discusses what to do if sampling sites become inaccessible	Yes	Section 3.2.2 to 3.2.5 (formerly 3.2.6)	EPA comment (11/22/21) – please update based on the EPA comment letter Atlantic Richfield Response (12/16/2021): Sections have been updated based on EPA comment letter.
e. Identifies project activity schedules such as each sampling event, times samples should be sent to the laboratory, etc.	Yes	Sections 3.2 to 3.5	EPA comment (11/22/21) – please update based on the EPA comment letter Atlantic Richfield Response (12/16/2021): Sections have been updated based on EPA comment letter.
f. Specifies what information is critical and what is for informational purposes only	Yes	Sections 3.2 to 3.5	EPA comment (11/22/21) – please update based on the EPA comment letter Atlantic Richfield Response (12/16/2021): Sections have been updated based on EPA comment letter.
g. Identifies sources of variability and how this variability should be reconciled with project information	Yes	Sections 3.7 and 3.8	EPA comment (11/22/21) – please update based on the EPA comment letter Atlantic Richfield Response (12/16/2021): Sections have been updated based on EPA comment letter.
B2. Sampling Methods			
a. Identifies all sampling SOPs by number, date, and regulatory citation, indicating sampling options or modifications to be taken	Yes	Sections 3.2, 3.3, and 3.5	EPA comment (11/22/21) – please update based on the EPA comment letter Atlantic Richfield Response (12/16/2021): Sections have been updated based on EPA comment letter.
b. Indicates how each sample/matrix type should be collected	Yes	Sections 3.2 to 3.5	EPA comment (11/22/21) – please update based on the EPA comment letter Atlantic Richfield Response (12/16/2021): Sections have been updated based on EPA comment letter.
c. If in situ monitoring, indicates how instruments should be deployed and operated to avoid contamination and ensure maintenance of proper data	NA	NA	NA
d. If continuous monitoring, indicates averaging time and how instruments should store and maintain raw data, or data averages	NA	NA	NA
e. Indicates how samples are to be homogenized, composited, split, or filtered, if needed	Yes (formerly NA)	Section 3.3.1 (formerly NA)	NA Atlantic Richfield Response (12/16/2021): Section has been inserted based on request to collect earthen basement soil samples.

f. Indicates what sample containers and sample volumes should be used	Yes	Sections 3.2 to 3.5	EPA comment (11/22/21) – please update based on the EPA comment letter Atlantic Richfield Response (12/16/2021): Sections have been updated based on EPA comment letter.
g. Identifies whether samples should be preserved and indicates methods that should be followed	Yes	Sections 3.5	EPA comment (11/22/21) – Section 3.5 is field equipment. Please update to the appropriate section. Atlantic Richfield Response (12/16/2021): Section numbers have been updated. Section 3.5 (formerly 3.4) is Field Procedures.
h. Indicates whether sampling equipment and samplers should be cleaned and/or decontaminated, identifying how this should be done and by-products disposed of	Yes	Section 3.5.2 (incorrectly listed as 3.2.4), FS-WI -010	EPA no comment (11/22/21) Atlantic Richfield Response (12/16/2021): Section numbers have been updated. Section 3.5.2 (formerly 3.4.2) is now Floor Surface Sampling.
i. Identifies any equipment and support facilities needed	Yes	Sections 3.6 (incorrectly listed as 3.4 and 3.10)	EPA comment (11/22/21) – please update to the appropriate section. Atlantic Richfield Response (12/16/2021): Section numbers have been updated. Section 3.6 (formerly 3.5) is now Field Equipment.
j. Addresses actions to be taken when problems occur, identifying individual(s) responsible for corrective action and how this should be documented	Yes	Section 4.1	EPA no comment (11/22/21)
B3. Sample Handling and Custody			
a. States maximum holding times allowed from sample collection to extraction and/or analysis for each sample type and, for in-situ or continuous monitoring, the maximum time before retrieval of information	Yes	Section 3.7 (incorrectly listed as 3.5)	EPA comment (11/22/21) – please update to the appropriate section. Atlantic Richfield Response (12/16/2021): Section numbers have been updated. Section 3.7 (formerly 3.6) is now Sample Handling and Chain of Custody.
b. Identifies how samples or information should be physically handled, transported, and then received and held in the laboratory or office (including temperature upon receipt)	Yes	Section 3.7 (incorrectly listed as 3.5)	EPA comment (11/22/21) – please update to the appropriate section. Atlantic Richfield Response (12/16/2021): Section numbers have been updated. Section 3.7 (formerly 3.6) is now Sample Handling and Chain of Custody.
c. Indicates how sample or information handling and custody information should be documented, such as in field notebooks and forms, identifying individual responsible	Yes	Sections 2.11.2, 2.11.4 and 3.7 (incorrectly listed as 3.5)	EPA comment (11/22/21) – please update to the appropriate section. Atlantic Richfield Response (12/16/2021): Section numbers have been updated. Section 3.7 (formerly 3.6) is now Sample Handling and Chain of Custody. Added reference to Section 2.11.2 Field Documentation.

d. Discusses system for identifying samples, for example, numbering system, sample tags and labels, and attaches forms to the plan	Yes	Section 3.8 (incorrectly listed as 3.6)	EPA comment (11/22/21) – please update to the appropriate section. Atlantic Richfield Response (12/16/2021): Section numbers have been updated. Section 3.8 (formerly 3.7) is now Sample Identification
e. Identifies chain-of-custody procedures and includes form to track custody	Yes	Sections 2.11.4 and 3.7	EPA comment (11/22/21) – please update to the appropriate section. Atlantic Richfield Response (12/16/2021): Section numbers have been updated. Section 3.7 (formerly 3.6) is now Sample Handling and Chain of Custody.
B4. Analytical Methods			
a. Identifies all analytical SOPs (field, laboratory and/or office) that should be followed by number, date, and regulatory citation, indicating options or modifications to be taken, such as sub-sampling and extraction procedures	Yes	Section 3.9 (incorrectly listed as 3.7), Appendix C	EPA comment (11/22/21) – please update based on the EPA comment letter Atlantic Richfield Response (12/16/2021): Section numbers have been updated. Section 3.9 (formerly 3.8) is now Analyses Methods
b. Identifies equipment or instrumentation needed	Yes	Section 3.9.1 (incorrectly listed as 3.7)	EPA comment (11/22/21) – please update to the appropriate section. Atlantic Richfield Response (12/16/2021): Section numbers have been updated. Section 3.9.1 (formerly 3.8.1) is now Dust Sample Analysis Methods
c. Specifies any specific method performance criteria	Yes	Sections 2.9.2 and 3.9.2 (incorrectly listed as 3.7.2)	EPA comment (11/22/21) – please update to the appropriate section. Atlantic Richfield Response (12/16/2021): Section numbers have been updated. Section 3.9.1 (formerly 3.8.1) is now Dust Sample Analysis Methods
d. Identifies procedures to follow when failures occur, identifying individual responsible for corrective action and appropriate documentation	Yes	Sections 3.9.2.1, 3.9.2.2, 3.9.2.3, 4.1, Appendix B	EPA comment (11/22/21) – please update to the appropriate section. Atlantic Richfield Response (12/16/2021): Included specific laboratory control sample subsections from Sections 3.9.2 that discuss corrective action. Section 4.1 paragraph 5 discusses corrective action during analysis. Appendix B, Laboratory SOPs also include method specific corrective action procedures.
e. Identifies sample disposal procedures	Yes	Section 3.11 (incorrectly listed as 3.9)	EPA comment (11/22/21) – please update to the appropriate section. Atlantic Richfield Response (12/16/2021): Section numbers have been updated. Section 3.11 (formerly 3.10) is now Sample Disposal

f. Specifies laboratory turnaround times needed	Yes	Sections 2.9.1 and 4.3	EPA comment (11/22/21) – please update to the appropriate section. Section 2.9.1 **Step 3 Identifying appropriate sampling and analytical methods,** paragraph 2 discusses laboratory turnaround times. Section 4.3 Reports to Management, paragraph 2 discusses turnaround times.
g. Provides method validation information and SOPs for nonstandard methods	Yes	Section 5.0	EPA no comment (11/22/21)
B5. Quality Control			
a. For each type of sampling, analysis, or measurement technique, identifies QC activities which should be used, for example, blanks, spikes, duplicates, etc., and at what frequency	Yes	Sections 3.9.2 and 3.10 (formerly 3.7 and 3.8)	EPA no comment (11/22/21) Atlantic Richfield Response (12/16/2021): Section numbers have been updated. Section 3.9.2 (formerly 3.8.2) is Laboratory Quality Control Samples. Section 3.10 (formerly 3.9) is Field Quality Control Samples.
b. Details what should be done when control limits are exceeded, and how effectiveness of control actions will be determined and documented	Yes	Section 5.0	EPA no comment (11/22/21)
c. Identifies procedures and formulas for calculating applicable QC statistics, for example, for precision, bias, outliers and missing data	Yes	Sections 2.9.2, 3.9.2, 3.10 (formerly 3.7, 3.8), and Table 2	EPA no comment (11/22/21) Atlantic Richfield Response (12/16/2021): Section numbers have been updated. Section 3.9.2 (formerly 3.8.2) is Laboratory Quality Control Samples. Section 3.10 (formerly 3.9) is Field Quality Control Samples.
B6. Instrument/Equipment Testing, Inspection, and Maintenance			
a. Identifies field and laboratory equipment needing periodic maintenance, and the schedule for this	Yes	Section 3.12 (incorrectly listed as 3.10)	EPA comment (11/22/21) – please update to the appropriate section. Atlantic Richfield Response (12/16/2021): Section numbers have been updated. Section 3.12 (formerly 3.11) is Instrument/Equipment Testing, Inspection, and Maintenance
b. Identifies testing criteria	Yes	Section 3.12 (incorrectly listed as 3.10)	EPA comment (11/22/21) – please update to the appropriate section. Atlantic Richfield Response (12/16/2021): Section numbers have been updated. Section 3.12 (formerly 3.11) is Instrument/Equipment Testing, Inspection, and Maintenance
c. Notes availability and location of spare parts	Yes	Section 3.12 (incorrectly listed as 3.10)	EPA comment (11/22/21) – please update to the appropriate section. Atlantic Richfield Response (12/16/2021): Section numbers have been updated. Section 3.12 (formerly 3.11) is Instrument/Equipment Testing, Inspection, and Maintenance

d. Indicates procedures in place for inspecting equipment before usage	Yes	Section 3.12 (incorrectly listed as 3.10)	EPA comment (11/22/21) – please update to the appropriate section. Atlantic Richfield Response (12/16/2021): Section numbers have been updated. Section 3.12 (formerly 3.11) is Instrument/Equipment Testing, Inspection, and Maintenance
e. Identifies individual(s) responsible for testing, inspection and maintenance	Yes	Section 3.12 (incorrectly listed as 3.10)	EPA comment (11/22/21) – please update to the appropriate section. Atlantic Richfield Response (12/16/2021): Section numbers have been updated. Section 3.12 (formerly 3.11) is Instrument/Equipment Testing, Inspection, and Maintenance
f. Indicates how deficiencies found should be resolved, re-inspections performed, and effectiveness of corrective action determined and documented	Yes	Section 3.12 (incorrectly listed as 3.10)	EPA comment (11/22/21) – please update to the appropriate section. Atlantic Richfield Response (12/16/2021): Section numbers have been updated. Section 3.12 (formerly 3.11) is Instrument/Equipment Testing, Inspection, and Maintenance
B7. Instrument/Equipment Calibration and Frequency			
a. Identifies equipment, tools, and instruments that should be calibrated and the frequency for this calibration	Yes	Sections 3.12.1, 3.12.2, and Appendix B (Incorrectly listed 2.9.2, and 3.8)	EPA comment (11/22/21) – please update to the appropriate section. Atlantic Richfield Response (12/16/2021): Section numbers have been updated. Section 3.12.1 (formerly 3.11.1) is Field Equipment and Section 3.12.2 is Laboratory Equipment. Calibration is also included in the Appendix B SOPs for laboratory methods.
b. Describes how calibrations should be performed and documented, indicating test criteria and standards or certified equipment	Yes	Sections 3.12.1, 3.12.2, and Appendix B (Incorrectly listed as 2.9.2 and 3.10; Appendix C)	EPA comment (11/22/21) – please update to the appropriate section. Atlantic Richfield Response (12/16/2021): Section numbers have been updated. Section 3.12.1 (formerly 3.11.1) is Field Equipment and Section 3.12.2 is Laboratory Equipment. Calibration is also included in the Appendix B SOPs for laboratory methods.
c. Identifies how deficiencies should be resolved and documented	Yes	Section 4.1 (incorrectly listed as 5.1)	EPA comment (11/22/21) – please update to the appropriate section. Atlantic Richfield Response (12/16/2021): Section numbers have been updated. Section 3.12.1 (formerly 3.11.1) is Field Equipment and Section 3.12.2 is Laboratory Equipment.
B8. Inspection/Acceptance for Supplies and Consumables			

a. Identifies critical supplies and consumables for field and laboratory, noting supply source, acceptance criteria, and procedures for tracking, storing and retrieving these materials	Yes	Sections 3.6, 3.13 (incorrectly listed as 3.11), and Appendix B	EPA comment (11/22/21) – please update to the appropriate section. Atlantic Richfield Response (12/16/2021): Section numbers have been updated. Section 3.13 (formerly 3.12) is Inspection/Acceptance of Supplies and Consumables. Section 3.6 is Field Equipment. Appendix B SOPs for laboratory methods include laboratory supplies.
b. Identifies the individual(s) responsible for this	Yes	Section 3.13 (incorrectly listed as 3.11)	EPA comment (11/22/21) – please provide this information and update the section accordingly. Atlantic Richfield Response (12/16/2021): Section numbers have been updated. Section 3.13 (formerly 3.12) is Inspection/Acceptance of Supplies and Consumables. Field supplies will be inspected by the Field Team Leader (may vary). Laboratory supplies are inspected by laboratory personnel (may vary).
B9. Use of Existing Data (Non-direct Measurements)			
a. Identifies data sources, for example, computer databases or literature files, or models that should be accessed and used	Yes	Section 3.14 (Incorrectly listed as 5.0)	EPA comment (11/22/21) – please provide this information and update the section accordingly. Atlantic Richfield Response (12/16/2021): Inserted Section 3.14 Non-Direct Measurement Data Acquisition Requirements
b. Describes the intended use of this information and the rationale for their selection, i.e., its relevance to project	Yes	Section 3.14 (Incorrectly listed as 5.0)	EPA comment (11/22/21) – please provide this information and update the section accordingly. Atlantic Richfield Response (12/16/2021): Inserted Section 3.14 Non-Direct Measurement Data Acquisition Requirements
c. Indicates the acceptance criteria for these data sources and/or models	Yes	Section 3.14 (Incorrectly listed as 5.0)	EPA comment (11/22/21) – please provide this information and update the section accordingly. Atlantic Richfield Response (12/16/2021): Inserted Section 3.14 Non-Direct Measurement Data Acquisition Requirements
d. Identifies key resources/support facilities needed	Yes	Section 3.14 (Incorrectly listed as 5.0)	EPA comment (11/22/21) – please provide this information and update the section accordingly. Atlantic Richfield Response (12/16/2021): Inserted Section 3.14 Non-Direct Measurement Data Acquisition Requirements
e. Describes how limits to validity and operating conditions should be determined, for example, internal checks of the program and Beta testing	Yes	Section 3.14 (Incorrectly listed as 5.0)	EPA comment (11/22/21) – please provide this information and update the section accordingly. Atlantic Richfield Response (12/16/2021): Inserted Section 3.14 Non-Direct Measurement Data Acquisition Requirements

B10. Data Management			
a. Describes data management scheme from field to final use and storage	Yes	Section 3.15 (incorrectly listed as 3.12)	EPA comment (11/22/21) – please provide this information and update the section accordingly. Atlantic Richfield Response (12/16/2021): Section numbers have been updated. Section 3.15 (formerly 3.13) is Date Management Procedures)
b. Discusses standard record-keeping and tracking practices, and the document control system or cites other written documentation such as SOPs	Yes	Section 3.15 (incorrectly listed as 3.12)	EPA comment (11/22/21) – please provide this information and update the section accordingly. Atlantic Richfield Response (12/16/2021): Section numbers have been updated. Section 3.15 (formerly 3.13) is Date Management Procedures)
c. Identifies data handling equipment/procedures that should be used to process, compile, analyze, and transmit data reliably and accurately	Yes	Section 3.15 (incorrectly listed as 3.12)	EPA comment (11/22/21) – please provide this information and update the section accordingly. Atlantic Richfield Response (12/16/2021): Section numbers have been updated. Section 3.15 (formerly 3.13) is Date Management Procedures)
d. Identifies individual(s) responsible for this	Yes	Section 3.15 (incorrectly listed as 3.12)	EPA comment (11/22/21) – please provide this information and update the section accordingly. Atlantic Richfield Response (12/16/2021): Section numbers have been updated. Section 3.15 (formerly 3.13) is Date Management Procedures)
e. Describes the process for data archival and retrieval	Yes	Section 3.15 (incorrectly listed as 3.12)	EPA comment (11/22/21) – please provide this information and update the section accordingly. Atlantic Richfield Response (12/16/2021): Section numbers have been updated. Section 3.15 (formerly 3.13) is Date Management Procedures)
f. Describes procedures to demonstrate acceptability of hardware and software configurations	Yes	Section 3.15 (incorrectly listed as 3.12)	EPA comment (11/22/21) – please provide this information and update the section accordingly. Atlantic Richfield Response (12/16/2021): Section numbers have been updated. Section 3.15 (formerly 3.13) is Date Management Procedures)
g. Attaches checklists and forms that should be used	Yes	Section 3.15 (incorrectly listed as 3.12)	EPA comment (11/22/21) – please provide this information and update the section accordingly. Atlantic Richfield Response (12/16/2021): Section numbers have been updated. Section 3.15 (formerly 3.13) is Date Management Procedures)
C. Assessment and Oversight			
C1. Assessments and Response Actions			

a. Lists the number, frequency, and type of assessment activities that should be conducted, with the approximate dates	Yes	Section 4. (formerly 4.0)	EPA comment (11/22/21) – please provide this information and update the section accordingly. Atlantic Richfield Response (12/16/2021): Section 4. has been updated based on EPA comment letter.
b. Identifies individual(s) responsible for conducting assessments, indicating their authority to issue stop work orders, and any other possible participants in the assessment process	Yes	Sections 4.1 and 4.2 (formerly 4.0)	EPA comment (11/22/21) – please provide this information and update the section accordingly. Atlantic Richfield Response (12/16/2021): Section references have been updated to include information.
c. Describes how and to whom assessment information should be reported	Yes	Sections 4.1, 4.2, and 4.3	EPA comment (11/22/21) – please provide this information and update the section accordingly. Atlantic Richfield Response (12/16/2021): Section references have been updated to include information.
d. Identifies how corrective actions should be addressed and by whom, and how they should be verified and documented	Yes	Sections 4.2, 4.3 (formerly 4.1 and 4.2), and Appendix F	EPA comment (11/22/21) – please provide this information and update the section accordingly. Atlantic Richfield Response (12/16/2021): Section references have been updated to include information.
C2. Reports to Management			
a. Identifies what project QA status reports are needed and how frequently	Yes	Section 4.3	EPA comment (11/22/21) – please provide this information and update the section accordingly. Atlantic Richfield Response (12/16/2021): Section 4.3, Paragraph 3 "A separate report will be prepared by the consultant QA manager, as needed, to communicate the results of performance evaluations or program audits to identify specific significant QA issues and provided to the USEPA for review. Any corrective action reporting described in Section 4.2 above will be summarized and included as appropriate."
b. Identifies who should write these reports and who should receive this information	Yes	Section 4.3	EPA comment (11/22/21) – please provide this information and update the section accordingly. Atlantic Richfield Response (12/16/2021): Section 4.3, Paragraph 3 "A separate report will be prepared by the consultant QA manager, as needed, to communicate the results of performance evaluations or program audits to identify specific significant QA issues and provided to the USEPA for review. Any corrective action reporting described in Section 4.2 above will be summarized and included as appropriate."
D. Data Validation and Usability			
D1. Data Review, Verification, and Validation			

Describes criteria that should be used for accepting, rejecting, or qualifying project data	Yes	Sections 5.2 and 5.3 (formerly 5.0)	EPA comment (11/22/21) – please provide this information and update the section accordingly. Atlantic Richfield Response (12/16/2021): Section references have been updated to include information. Sections 5.2.3 and 5.3 were revised to include missing information.
D2. Verification and Validation Methods			
a. Describes process for data verification and validation, providing SOPs and indicating what data validation software should be used, if any	Yes	Sections 5.1.2, 5.1.3, and 5.2 (formerly 5.0)	EPA comment (11/22/21) – please provide this information and update the section accordingly. Atlantic Richfield Response (12/16/2021): Section references have been updated to include information.
b. Identifies who is responsible for verifying and validating different components of the project data/information, for example, chain-of-custody forms, receipt logs, calibration information, etc.	Yes	Sections 5.1.2 and 5.1.3 (formerly 5.0)	EPA comment (11/22/21) – please provide this information and update the section accordingly. Atlantic Richfield Response (12/16/2021): Section references have been updated to include information.
c. Identifies issue resolution process, and method and individual responsible for conveying these results to data users	Yes	Sections 5.1.1, 5.1.2, and 5.2.2 (formerly 5.0)	EPA comment (11/22/21) – please provide this information and update the section accordingly. Atlantic Richfield Response (12/16/2021): Section references have been updated to include information.
d. Attaches checklists, forms, and calculations	Yes	Section 5.2 (formerly 5.0), Appendix D, and Appendix G	EPA comment (11/22/21) – please provide this information and update the section accordingly. Atlantic Richfield Response (12/16/2021): Section references have been updated to include information.
D3. Reconciliation with User Requirements			
a. Describes procedures to evaluate the uncertainty of the validated data	Yes	Section 5.3 (formerly 5.0)	EPA comment (11/22/21) – please provide this information and update the section accordingly. Atlantic Richfield Response (12/16/2021): Sections 5.3 has been inserted.
b. Describes how limitations on data use should be reported to the data users	Yes	Section 5.3 (formerly 5.0)	EPA comment (11/22/21) – please provide this information and update the section accordingly. Atlantic Richfield Response (12/16/2021): Section 5.3 has been inserted.

APPENDIX B STANDARD OPERATING PROCEDURES

Attachment B
ERM Field SOPs
Index

SOP Number	SOP Title	# Pages
FS-WI-003	Surface Soil	9
FS-WI-008	Waste Management	6
FS-WI-010	Equipment Decontamination	5
FS-WI-014	Sample Management	15
FS-WI-017	Field Reporting	4
FS-WI-020	Field Logbook	7
ASTM D5438-05	Standard Practice for Collection of Floor Dust for Chemical Analysis	8
ASTM D7144-21	Standard Practice for Collection of Surface Dust by Micro-vacuum Sampling for Subsequent Determination of Metals and Metalloids	5

1. PURPOSE/SCOPE OF PROCEDURE

1.1 Purpose

The purpose of this field sampling work instruction (FS-WI) is to describe a standard process for collecting surface soil samples to ensure personnel supporting the field activities are prepared to follow consistent protocols, which enable the objectives defined in project-specific work plans and the Quality Assurance Project Plan (QAPP) to be met.

1.2 Scope

This procedure was developed to guide and support work conducted within the Butte Priority Soils Operable Unit in Butte, Montana in support of Residual Metals Abatement Program Plan (RMAP) Administrative Order on Consent (United States Environmental Protection Agency [USEPA] Docket No. CERCLA-08-2011-0011) for Corrective Action.

2. DEFINITIONS

°C	Degrees Celsius
CoC	Chain-of-Custody
DQO	Data Quality Objective
FS-WI	Field Sampling-Work Instruction
FTL	Field Team Leader
MS/MSD	Matrix Spike / Matrix Spike Duplicate
PM	Project Manager
QAPP	Quality Assurance Project Plan
USEPA	United States Environmental Protection Agency

3. PROCEDURE/PROCESS

Sampling strategies will be defined within project-specific work plans and may include systematic, biased, or random sampling techniques. Because of the nature of the media, soil samples can vary considerably across a site and often more than one sampling technique can be used to collect the desired samples. The sampling strategy can be based on historic information regarding the site, knowledge about the behavior of the contaminant(s), and/or knowledge about the effects of the physical system on the fate of the contaminant. Sampling requirements defined in the project-specific work plan supersedes directions provided in this FS-WI.

The type of sample required to meet project goals should be considered prior to selecting a sampling method. Application techniques for sample methods include discrete (grab) samples, composite, and multi-incremental samples. A grab sample is a discrete aliquot representing a specific location at any point in time. The sample is collected immediately and at one particular point in the sample matrix. A composite is a sample composed of two or more discrete samples collected at various, non-specific, sampling locations and/or depths. Multi-incremental samples are collected from a clearly defined decision unit and are comprised of typically at least 30-100 discrete samples that are composited into one sample.

Samples are collected using a variety of methods depending on the sampling strategy, location, and type of sampling required and defined in the work plan and data quality objectives (DQOs). When collecting soils for metals analysis the project-specific work plan shall designate the sample collection equipment appropriate for DQOs as applicable to the metals of interest.

Sampling frequency will depend on project objectives and site conditions. For example, if the objective of the event is to determine if a site is contaminated, a limited number of samples from properly chosen locations will yield useful information. If, however, the site is known to be contaminated, and delineation of the contamination is the objective, greater number of samples may be needed.

Surface soil samples can be collected using various techniques that are defined by applicability to the DQO for the project. Selection of soil sampling equipment is usually based on the matrix, location, and depth of the samples and manual techniques are usually selected for surface or shallow subsurface soil sampling.

Acceptable processes for collecting soil samples in thawed conditions are described in detail below:

3.1 Grab Sampling

The simplest, most direct method for collecting surface soil samples from thawed soils is to use a spade and stainless steel scoop; a hand auger or hand coring device may also be used. A clean household spade can be used to remove the top cover of soil to the required depth, but the smaller stainless steel scoop should be used to acquire the sample. Likewise, the manual auger or coring device can be driven to the desired depth for sample collection. Alternatively, the sampler may choose to use their gloved hand to collect the soil (field samplers must replace their gloves prior to collecting the next samples with new/clean non-powdered nitrile gloves). The sampling equipment should be decontaminated prior to and after each use. Spades plated with chrome or other metals are inappropriate when analyzing for metals.

The sampling procedure is defined as follows:

- Sketch and or photograph the sample area or decision unit and note any recognizable features for future reference.
- Remove any debris or oversize material from the ground surface and surface soil to the depth above where sample will be collected.
- Insert clean sampling device into material and collect a sample.
- Use a stainless steel trowel, scoop, or spoon; to transfer soil to appropriate sample container. Never use plastic or wooden spoons to collect samples.
- Carefully plan your sampling locations and minimal aliquots of sample to be collected. Using a stainless steel spoon, scoop a small volume of soil, and place it directly into the container with methanol. Continue to collect small aliquots of soil until you have a sample representative of the decision area or sample depth. Make sure you have collected adequate volume so the laboratory can achieve the required detection limits established for your project.
- After volatile analysis samples are collected, homogenize the remaining sample to prepare for collection of the remaining analytical parameters. To homogenize the remaining sample, transfer the

soil to a re-sealable gallon freezer bag or stainless steel mixing bowl. Volatile parameters are never homogenized and shall always be transferred directly to the sample container (4-ounce jar).

- After thoroughly mixing the representative soil remove large rocks and organic material such as roots, twigs, etc. Transfer to appropriate sample containers filling each to the rim of the jar and compressing the soil to maximize total volume.
- Secure cap but do not over tighten. Over tightening may cause the cap to break during transit to the offsite analytical laboratory.
- Label the sample container; wrap sample in bubble wrap, place container in a re-sealable freezer bag and place sample on gel ice in a pre-chilled cooler immediately. By placing the sample in a re-sealable bag, sample is not lost if container breaks in transit to the analytical laboratory.
- Provide field notes, completed field data collection forms (Soil Sample Sheets), and the samples to the designated sample management person to complete all CoCs and ship samples to the contract laboratory.
- Decontaminate sampling equipment in accordance with the decontamination procedure (FS-WI-008 and FS-WI-010) after use and between sample locations unless disposable sampling equipment is used.

3.2 Composite Sampling

Composite samples will consist of discrete aliquots of equal amounts of soil from each subsample location. Sample collection devices include disposable plastic scoops. The following procedure is designed to be used to collect soil samples from the 0-12 inch horizon. 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.

The sampling procedure is defined as follows:

- Sketch and or photograph the sample area or decision unit and note any recognizable features for future reference.
- Dig a 6 to 12-inch square pit to a depth of approximately 12 inches. The size and depth of the sample pit required would depend on the amount of material needed for sample analysis and the interval to be sampled. If a sod mat is present, it shall be separated from the mineral soil surface with the chosen sampling tool. The removed sod mat shall be shaken and scraped over the sample collection bowl to dislodge any mineral soil particles. All dislodged particles shall be placed in the sample
- Measure the interval to be sampled (0-12 inches) with a stainless steel tape measure, a ruler or other calibrated marking device and mark the appropriate interval.
- Scrape the walls of the sample pit within the marked interval with a disposable plastic scoop to expose a clean surface.

- 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, a new cleaned foil pan or gallon Ziploc bag.
- The soil aliquots will be thoroughly mixed. During the homogenization process, large particles (greater than 0.5 inch in diameter) will be discarded. After mixing, the sample will be placed in a one quart plastic bag and labeled. 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.
- After thoroughly mixing the representative soil remove large rocks and organic material such as roots, twigs, etc. Transfer to appropriate sample containers filling each to the rim of the jar and compressing the soil to maximize total volume.
- Secure cap but do not over tighten. Over tightening may cause the cap to break during transit to the offsite analytical laboratory.
- Label the sample container; wrap sample in bubble wrap, place container in a re-sealable freezer bag and place sample on gel ice in a pre-chilled cooler immediately. By placing the sample in a re-sealable bag, sample is not lost if container breaks in transit to the analytical laboratory.
- Provide field notes, completed field data collection forms (Soil Sample Sheets), and the samples to the designated sample management person to complete all CoCs and ship samples to the contract laboratory.
- Decontaminate sampling equipment in accordance with the decontamination procedure (FS-WI-008 and FS-WI-010) after use and between sample locations unless disposable sampling equipment is used.

3.3 Field Documentation

Field documentation shall be reviewed by the field team leader (FTL) daily to ensure recorded information is accurate and complete. Field documentation will be recorded in field logbooks and supplemented on field data collection forms as described in below subsections. All photograph and video documentation shall be downloaded onto the field computer daily. At the conclusion of the field effort all photographs, video, scanned copies of field forms, manifests and logbooks shall be transferred to a thumb drive. The thumb drive shall be sent to the consultant project manager (PM) at the completion of each field effort.

3.3.1 Field Logbook

See work instruction FS-WI-020 Field Logbook for instructions and reasoning for keeping field logbooks as part of field documentation.

3.3.2 Field Forms

The field form (Soil Sampling Form provided in Attachment 1) shall be completed immediately upon sample collection. All fields on the forms must be completed. Use NA to indicate a field is not applicable where appropriate. The forms shall be provided to the FTL, who will provide them to the PM at the completion of the project, along with all logbooks used during field activities. This will ensure that all information is available to office personnel preparing post field event summary reports.

3.3.2.1 Soil Sampling Form

The Soil Sampling Form (Attachment 1) shall be completed while the field sampler is at the sample location and shall include at a minimum the following information:

- Sample location
- Sample identification
- Site name
- Equipment used to collect the sample
- Date and time
- Samplers name
- Field parameters per form
- Analytical parameters
- Associated quality assurance / quality control samples, such as MS/MSD, duplicate samples, etc.
- Diagram of sample locations and reference to photographs (photo log), as applicable

3.3.3 Materials

- Clean plastic sheeting
- Metal clipboard box case (container for field forms)
- Required health and safety equipment (e.g., dig permit, photo ionization detector, personal protective equipment, etc.)
- Soil sample collection equipment (e.g., core sampler, scoop/trowel, tube sampler, split spoon sampler, stainless steel spoons)
- Decontamination materials (FS-WI-010) and appropriate storage container for transport or disposal (FS-WI-008)
- Sample collection, storage and management materials (e.g., jars, preservation, gel ice, coolers, re-sealable bags, bubble wrap, CoCs, custody seals, etc.)
- Digital or disposable camera
- Logbook with lot numbers and glassware inventory
- Field logbook

4. KEY RESPONSIBILITIES

The key project responsibilities should be clearly defined in the project-specific work plan and QAPP.

5. KEY DOCUMENTS/TOOLS/REFERENCES

ERM (ERM Alaska, Inc.). 2021. FS-WI-008. Waste Management.

ERM. 2021. FS-WI-010. Equipment Decontamination.

ERM. 2021. FS-WI-014. Sample Management.

ERM. 2021. FS-WI-017. Field Reporting.

ERM. 2021. FS-WI-020 Field Logbook.

USEPA 2008. Test Methods for SW846 Third Edition to include Updates I through IVB. January.

Attachment:

Soil Sampling Form

6. CONTROLLED DOCUMENT ADMINISTRATION INFORMATION

Authority:	Program Manager	**Issue Date:**	December 17, 2021
Custodian:	Document Custodian	**Revision Date:**	

7. REVISION LOG

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December 17, 2021	Thomas Beckman	Nicole Beier	Initial

ATTACHMENT 1

Soil Sampling Form

Soil Sampling Worksheet

Location _____
Project # : _____ Date: _____
Project Name: _____ Start Time: _____
End Time: _____
Field Team: _____
Sample ID: _____ Time: _____ primary dup split ms/msd
Sample ID: _____ Time: _____ primary dup split ms/msd
Weather Conditions:

Notable Observations (circle all that apply)		PID Readings
Description:	Sandy Gravel, Organic Material, Tundra Mat, Other:	1
Odor:	None, Low, Medium, High, Very Strong, H2S, Fuel like, Chemical ?, Unknown	2
Organic Matter:	Yes, No	3
Collection Method:	Grab, Composite, Multi-Incremental	4
Other:		5
		6
		7

Location Diagram/Notes

General Information

Sample Method	Sample Depth(ft)	Sample Collection Equipment	Extraction Method

Analyses	# of Bottles Collected	Bottle Type (preservative)	Comments:

Signed: _____ Date: _____

Signed/reviewer: Date:

1. PURPOSE/SCOPE OF PROCEDURE

1.1 Purpose

The purpose of this field sampling work instruction (FS-WI) is to describe a standard process for waste management to ensure personnel supporting the field activities follow consistent protocols, which enable the objectives defined in project-specific work plans, waste management plans, and the Quality Assurance Project Plan (QAPP) to be met. The project-specific waste management plan is an internal document and is not reviewed/approved by regulators. However, it may be reviewed by Atlantic Richfield Company (AR) upon request.

1.2 Scope

This procedure was developed to guide and support work conducted within the Butte Priority Soils Operable Unit in Butte, Montana in support of Residual Metals Abatement Program Plan (RMAP) Administrative Order on Consent (United States Environmental Protection Agency [USEPA] Docket No. CERCLA-08-2011-0011) for Corrective Action.

2. DEFINITIONS

AR	Atlantic Richfield Company
CoC	Chain-of-custody
FS-WI	Field Sampling Work Instruction
HASP	Health and Safety Plan
IDW	Investigation-Derived Waste
PM	Project Manager
PPE	Personal Protective Equipment
QAPP	Quality Assurance Project Plan
RCRA	Resource Conservation Recovery Act
SAA	Satellite Accumulation Area
USEPA	United States Environmental Protection Agency
USDOT	United State Department of Transportation

3. PROCEDURE/PROCESS

Non-investigative waste, such as litter and household garbage, shall be collected on a daily basis to maintain each site in a clean and orderly manner. This waste shall be containerized and transported to the designated sanitary landfill or collection bin. Acceptable containers shall be sealed boxes or plastic garbage bags.

The investigative-derived waste (IDW) shall be segregated at the site according to matrix (i.e., solid or liquid), hazardous vs. non-hazardous and hazard classification, and as to how it was derived (e.g., drill cuttings, drilling fluid, decontamination fluids, and purged groundwater). IDW shall be properly containerized and labeled at the site before transfer to the staging or disposal

facility. The containers shall be transported in such a manner to prevent spillage or particulate loss to the atmosphere. The contractor shall use acceptable containers and shall be sealed, (United State Department of Transportation [USDOT] approved steel 55-gallon drums or 5-gallon containers with lids). Each container shall be properly labeled with point of contact information (contractor name and telephone number), site identification, matrix, constituents of concern, and other pertinent information for handling.

Final waste management decisions will be based on location, season, facilities available in the area, facility-specific operating limitations, and owner-company policies as well as local, state and federal laws and regulations.

Used personal protective equipment (PPE) is selected based on type of material handled and hazardous characteristics. These requirements are defined in the project Health and Safety Plan (HASP) and associated Job Hazard Analysis. Used PPE will be disposed based on hazardous characteristics of waste handled and will generally not be sampled. If PPE are considered non-hazardous, material will be disposed with other project trash.

3.1 Planning

It is important to not only plan ahead when managing waste on a project site, but also to include the appropriate people in all stages of the planning to ensure the success of the project. During planning, field team members shall evaluate at a minimum the following:

1. Safe and secure location for staging waste and placement of signage.
2. Appropriate type of container for temporary storage and type required for transferring waste (keep in mind the container may be dependent upon the type of equipment being used and defined by the facility involved in the waste transfer).
3. Type of treatment and location.
4. Schedule for pickup and transfer of waste.
5. Type of staging area. Some questions that must be identified when establishing the type of staging area and the limitations associated with each (90-Day Accumulation Area vs. SAA) type of staging area are identified as follows:
    - How long can waste be stored at the location?
    - What type of waste can be stored at the site? Can hazardous waste be stored in the staging area (RCRA vs. Toxic Substances Control Act)?
    - What are signage and labeling requirements?
    - How much waste can be stored at the location at a time (i.e., only one 55-gallon drum of waste per matrix can be staged onsite at a time if site is designated as a SAA)?
    - Is analytical testing required?
    - What testing is required?

- Are field personnel required to inspect staging area and document the results of the inspection? How often? What are reporting requirements?
- What information is required to manifest and transfer waste?
- Can soil with free water be transferred as a solid waste? Should the free liquid be decanted off the soil and transferred to a separate drum for liquid waste?
- Is a USEPA identification number required for labeling and transfer of waste?
- Who has the Contractor identified as the responsible person to coordinate waste staging, tracking, inspections, and disposal or transfer? Do they have proper training?
- Will staging areas be set up at each point of generation or can it all be managed at the sample management trailer staging area?
- Where and how will non-hazardous materials (identify various waste streams) be disposed?

3.2 Field Documentation

3.2.1 Field Logbook

See work instruction FS-WI-020 Field Logbook for instructions and reasoning for keeping field logbooks as part of field documentation.

3.2.2 Materials

Materials that may be required are listed below and will be site- and project-specific. This list may not be all inclusive and site personnel should refer to the project-specific work plans to identify required equipment and materials needed to complete defined sampling events:

- Metal clipboard box case
- Appropriate PPE (respirator may be required depending on nature of contaminants)
- 55-gallon drums, 5-gallon buckets, lids, super sacks, trash bags or appropriate container for waste type and volume
- Tubs, duck ponds, etc. for decontamination of large pieces of equipment
- Towels, rags, paper towels
- Required health and safety equipment (e.g., photo ionization detector, splash protection apron or face shield, PPE, etc.)
- Sample collection, storage and management materials (e.g., jars, preservation, ice, coolers, re-sealable baggies, bubble wrap, chains-of-custody [CoCs], custody seals, etc.) for waste characterization
- Field logbook

- Liner and tarps
- Strapping to secure drums
- Drum cart is necessary to move drums
- Pallets
- USDOT acceptable drums or buckets
- Spill and sorbent material
- Labels and placards
- Waste sampling equipment (such as drum thieves)
- Waste characterization field kits and materials (Dexsil Clor-N-Oil® [polychlorinated biphenyls], Clor-D-Tect® [halogens], Hazard Categorization Kits [biological and chemical], Drager Tubes, Lead in Paint Kit, etc.)
- User knowledge, historical data or user information (essential for combining similar waste and waste reduction)
- Filters (e.g., granular activated carbon, clay anthracite, filters); these filters and spent carbon may be considered hazardous and must be tested prior to transport or disposal
- Photoionization detector and multi-gas meter to evaluate volatile levels (for PPE upgrade considerations) and explosivity.

3.2.3 Sample Collection for Characterization of Waste

Depending on the type of waste, offsite analytical testing may be required before waste can be moved, transferred, injected, disposed, or treated. The project-specific work plan should address sampling, analysis, and the analytical and quality control requirements for all waste streams.

Quality control samples for waste characterization differ from those required for site characterization or investigation as follows due to high level concentrations of contamination in most matrices:

- Matrix spike/matrix spike duplicate is not required
- Field duplicates are not required
- Trip blanks are not required
- Equipment blanks are not required as disposable equipment will be used to collect samples
- Solid waste samples are not preserved (i.e., volatile organic compounds and gasoline range organics will not be preserved with methanol)

- The contractor project chemist or data manager shall be contacted if field personnel are uncertain on sample collection and preservation techniques.

3.2.4 Waste Accumulation Site Inspections

Waste accumulation sites must be set up in a safe and secure location. Waste accumulation sites may require daily or monthly inspection by trained field personnel and condition of containers and secondary containment documented in the field logbook.

4. KEY RESPONSIBILITIES

The key project responsibilities should be clearly defined in the project-specific work plan and QAPP.

5. KEY DOCUMENTS/TOOLS/REFERENCES

ERM Alaska, Inc. 2021. FS-WI-020 *Field Logbook*.

6. CONTROLLED DOCUMENT ADMINISTRATION INFORMATION

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7. REVISION LOG

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September 21, 2021	Thomas Beckman	Courtney Pijanowski	Initial

1. PURPOSE/SCOPE OF PROCEDURE

1.1 Purpose

The purpose of this field sampling work instruction (FS-WI) is to describe a standard process for equipment decontamination to ensure personnel supporting the field activities follow consistent protocols that enable the objectives defined in project-specific work plans and the Quality Assurance Project Plan (QAPP) to be met.

1.2 Scope

This procedure was developed to guide and support on-going work conducted within the Butte Priority Soils Operable Unit in Butte, Montana in support of Residual Metals Abatement Program Plan (RMAP) Administrative Order on Consent (United States Environmental Protection Agency [USEPA] Docket No. CERCLA-08-2011-0011) for Corrective Action.

2. DEFINITIONS

AR	Atlantic Richfield Company
CoC	Chain-of-Custody
DQO	Data Quality Objective
FS-WI	Field Sampling Work Instruction
FTL	Field Team Leader
PM	Project Manager
PPE	Personal Protective Equipment
QAPP	Quality Assurance Project Plan
USEPA	United States Environmental Protection Agency

3. PROCEDURE/PROCESS

The work plan and waste management plan will provide project-specific details for the site. The appropriateness of the decontamination protocol is vital to the eventual validity of the analytical results and decisions made based upon those results. All non-disposable sampling equipment that will contact sampled media must be properly decontaminated prior to use and between sampling locations. Devices may include dust pans, etc.

Contaminant carryover between samples and/or from leaching of the sampling device is very complex and requires special attention. When equipment is reused, project equipment blanks/rinsate blanks shall be collected at the frequency of at least one per 20 samples per matrix, weekly, or as defined in the project-specific work plan. A rinsate (equipment) blank is collected by passing clean deionized water over decontaminated sampling equipment that is collected in appropriate sample containers, preserved and submitted for analysis. This sample receives a unique sample identification number and is submitted to the laboratory in such a way that the laboratory is not aware it is a quality control sample (i.e., blind). This sample is used to assess cross-contamination from the sampling equipment and effectiveness of the decontamination process in addition to incidental contamination from the sample container and/or preservatives.

3.1.1 Sampling Devices

The following procedure shall be used to decontaminate sampling devices, such as dust pans and the re-usable micro-vacuum filter cassettes. Spray the equipment with a solution of potable water and Alconox™, or an equivalent non-phosphate laboratory-grade detergent and wipe dry with clean paper towels. Then spray or rinse the equipment with potable water, followed by two spray or rinses with deionized water. Wipe the equipment dry using clean paper towels and allow to fully dry on a clean surface or rack located in a clean secure area. Brushes used to collect dust samples in dust pans will be single-use and disposable.

Ideally, disposable sample collection equipment will be used to collect, handle, or measure solid samples; no decontamination is planned for the micro-vacuum samples. The HVS3 sampler will be decontaminated as described in Section 13 of the ASTM D 5438-05 procedure. After use, disposable equipment will be immediately bagged in garbage bags, so it does not cross contaminate unused disposable equipment and for easy disposal in a dumpster.

It is the consultant's responsibility to ensure that deionized water, and potable water stored onsite for decontaminating sampling equipment remain free of contaminants. Field personnel should dispose of unused water after each field effort. All material used to decontaminate equipment must be stored in a secure and clean environment to ensure material does not become contaminated during storage.

All equipment must be allowed to air dry in between sampling, and therefore, extra equipment must be available onsite. All sampling equipment shall be stored in a secure clean environment.

3.1.2 Waste Management

Pre-planning is critical to the successful management of all waste generated by the project activities. Waste generated from decontamination procedures shall be managed on a site-by-site basis. Waste may be classified as non-investigative waste or investigative waste and managed according to and work instruction FS-WI-008.

The investigative derived waste shall be segregated at the site according to matrix (personal protective equipment [PPE], solids, water, etc.), site, and type of waste. Only similar wastes will be consolidated; for example, when transferring waste from 5-gallon containers to a 55-gallon drum, field personnel must ensure all waste streams are similar and from the same site prior to consolidation within the same drum. Hazardous or potentially hazardous waste shall not be mixed with non-hazardous waste. Each container shall be properly labeled with site identification, sampling location, matrix, hazardous or nonhazardous determination, and the name and telephone number for the primary point of contact.

Specific decontamination procedures that differ from those listed herein may be outlined in project-specific work plans. Decontamination procedures shall be developed accurately, shall meet the DQOs, and shall take into account the site-specific conditions, such as temperature and type of material being sampled.

3.2 Field Documentation

Field documentation shall be reviewed daily to ensure recorded information is accurate and complete. Field documentation will also be reviewed by the project manager (PM) to ensure records are complete

and accurate. Field documentation will be recorded in field logbooks and supplemented on field data collection forms as described below.

3.2.1 Field Logbook

See work instruction FS-WI-020 Field Logbook for instructions and reasoning for keeping field logbooks as part of field documentation.

3.2.2 Field Forms

The field forms shall be completed immediately upon completion of field activities. All fields on the form must be completed. Use NA to indicate a field is not applicable where appropriate. The forms shall be provided to the field team leader (FTL), who will provide them to the PM at the completion of the project along with all logbooks used during field activities. This will ensure that all information is available to office personnel preparing post field event summary reports.

3.2.3 Materials

Materials needed by field personnel are listed below. This list may not be all inclusive and site personnel should refer to the project-specific work plans to identify required equipment and materials needed to complete defined sampling events.

- Metal clipboard box case (container for well logs)
- Plastic sheeting
- Appropriate PPE (respirator may be required depending on nature of contaminants)
- Brushes and scrapers to remove surface debris
- Hand-held spray washer
- Alconox™ or other detergent wash appropriate to contaminant
- Tap water and deionized water
- 5-gallon buckets, 55-gallon drums, trash bags or appropriate container for waste type and volume
- Towels, rags, paper towels
- Required health and safety equipment (e.g., PPE, etc.)
- Sample collection, storage and management materials (e.g., bags, jars, preservation, ice, coolers, re-sealable bags, bubble wrap, chains-of-custody [CoCs], custody seals, etc.)
- Field Logbook

4. KEY RESPONSIBILITIES

The key project responsibilities shall be clearly defined in the project-specific work plan and QAPP.

5. KEY DOCUMENTS/TOOLS/REFERENCES

ERM (ERM Alaska, Inc.). 2021. FS-WI-008. Waste Management.

ERM. 2021. FS-WI-020. Field Logbook.

6. CONTROLLED DOCUMENT ADMINISTRATION INFORMATION

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7. REVISION LOG

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September 20, 2021	Thomas Beckman	Courtney Pijanowski	Initial

1. PURPOSE/SCOPE OF PROCEDURE

1.1 Purpose

The purpose of this field sampling work instruction (FS-WI) is to describe a standard process for sample management to ensure personnel supporting the field activities follow consistent protocols, which enable the objectives defined in project-specific work plans and the Quality Assurance Project Plan (QAPP) to be met.

1.2 Scope

This procedure was developed to guide and support work conducted within the Butte Priority Soils Operable Unit in Butte, Montana in support of Residual Metals Abatement Program Plan (RMAP) Administrative Order on Consent (United States Environmental Protection Agency [USEPA] Docket No. CERCLA-08-2011-0011) for Corrective Action.

2. DEFINITIONS

AR	Atlantic Richfield Company
CoC	Chain-of-Custody
ºC	Degrees Celsius
DQO	Data Quality Objective
FS-WI	Field Sampling Work Instructions
FTL	Field Team Lead
GPS	Global Positioning System
HASP	Health and Safety Plan
mL	milliliters
MS/MSD	Matrix Spike / Matrix Spike Duplicate
PM	Project Manager
PPE	Personal Protective Equipment
QA	Quality Assurance
QAC	Quality Assurance Coordinator
QAPP	Quality Assurance Project Plan
QC	Quality Control
RCRA	Resource Conservation and Recovery Act
USEPA	United States Environmental Protection Agency

3. PROCEDURE/PROCESS

The sample management process begins when the analytical laboratory subcontract is established and sample collection supplies are provided by the laboratory. Sample collection supplies include sample containers, coolers, preservatives, and quality control (QC) samples *I.e., trip blanks, temperature blanks, etc.). Sample management does not end until the analytical data have been reviewed, validated, and reported. At this point, the laboratory may be authorized to dispose of the project samples and properly archive the analytical data.

3.1 Planning

Planning is critical to the success of any project or any aspect of an investigation. The project-specific work plan and QAPP must clearly define the project and data quality objectives (DQOs) before the subcontractor laboratory, analytical methods, or sampling procedures are identified.

The analytical data are collected to support real-time, as well as future decisions, and therefore, the collection and management of the samples must follow standard procedures to ensure data are usable to meet project objectives. If samples are not managed properly, data may be considered rejected or unusable.

If changes in site conditions or approach are made after the work plan has been finalized, the variances must be documented in an addendum to the work plan or in the field logbook. Changes must be discussed with the Environmental Consultant Project Manager (PM), Quality Assurance (QA) and Data Management Consultant Chemist, and analytical laboratory to ensure the changes do not impact data usability.

The project-specific work plan shall include the following at a minimum:

- A figure providing the locations where samples will be collected
- Global Positioning System (GPS) coordinates or pre-located sample points based on pre-established locations
- Sample identification nomenclature information
- Description of field screening techniques and field action limits
- Description of field instrumentation to be used on the project and associated user's manuals
- Analytical methods required for each sample and matrix per location
- Definition of sample collection techniques
- The sample collection frequency for field screening, offsite analytical, and QC samples
- Laboratory information for all laboratories involved including name, address, and telephone numbers for the laboratory point of contact and backup in the event the primary is not reachable
- An example of a completed chain-of-custody (CoC) form
- Table containing requirements for analytical methods, sample preservation, holding times, storage temperature, and number of containers, lid type and size per method to include trip blanks
- Waste management plan, section or table defining waste streams, and sample testing requirements
- Health and safety concerns and precautions for personnel collecting or managing the samples and generated waste
- Required personnel protective equipment (PPE) and training requirements

3.2 Sample Containers

Sample containers will be purchased by the laboratory as certified clean from the supplier. The containers shall be provided with a certificate and lot number. The certificate should list the serial numbers associated with each lot. During the process of inventorying the glassware and preservatives, the field personnel must ensure each lot of glassware and preservative is designated for use or associated with planned future environmental sampling events. The certificates will be stored in a folder in the same location as the glassware. The serial/product numbers placed on the sample containers by the manufacturer provide the link to the lot number. The lot numbers will be recorded in a glassware and preservative inventory logbook. It is critical that field sampler does not place the sample label over the manufacturer's label indicating serial/product/lot numbers to simplify the process of identifying glassware and preservative lot numbers by the laboratory should lot-specific problems be identified.

Containers will be stored in a clean secure area to prevent cross-contamination from fuels, solvents, and other contaminants at the site. Amber glass bottles will be used routinely where glass containers have been specified in the sampling protocol to reduce photo-degradation. Containers shipped from the laboratory for volatile organic compound (VOC) analyses will be accompanied by a trip blank(s) for each matrix type. The laboratory will also provide empty temperature blank containers that hold a minimum of 500 milliliters (mL).

The lot numbers for the bottleware submitted by the laboratory directly to the site or consultant must be traceable to project-specific samples. Sample containers provided by the laboratory will be shipped with a packing list that details the number and type of bottles shipped, chemical preservatives, the bottle and preservative lot numbers, and the packer's signature.

Lot numbers of preservatives (acids, bases, and surrogated methanol) added to bottleware must be traceable to the specific lots provided for the project. Each lot of preservative must be labeled with the name of the preservative, the preparation date, the lot number, the concentration, and the expiration date. All preservatives must undergo documented pre-testing to ensure that the preservative is not contaminated. Data obtained from the pre-testing of preservatives must be maintained by the laboratory and available on file for inspection.

In summary, the following actions shall be taken by field personnel:

1. A record of receipt, including the name of the supplier, quantity, lot number if applicable, condition, date received, and the receiver's name, will be recorded in the glassware and preservative inventory logbook.

2. Field personnel must sign and date all bottle order packing slips and maintain copies of this documentation.

3. Field personnel must record which lot numbers of bottles were used for each sample collection event by recording the lot number in the field logbook to ensure traceability.

4. All container lids shall be Teflon®-lined and lids provided with volatile sample containers shall also contain a septum. Field personnel shall inventory and inspect each shipment of glassware to ensure the glassware type and quantity is adequate for the sampling program defined in the project-specific work plan.

3.3 Sample Volumes, Container Types, and Preservation Requirements

Sample volumes, container types, and preservation requirements for the analytical methods are listed in the RCRA Order QAPP and shall also be defined in the project-specific work plan. Because laboratory and method requirements change, the project-specific work plan shall define all project-specific requirements for field screening and offsite analytical sample collection.

Sample holding time tracking begins at the time the sample is collected from the field location and continues until the analysis of the sample and associated QC samples is complete. Holding times for the analytical methods are specified in the QAPP and project-specific work plan.

Field personnel must label all of the bottles with the sample location. Field personnel will place samples in a cooler containing gel ice as samples are collected. Samples will be taken to the field work area where they may be stored in the refrigerator or in sample coolers with gel ice. The refrigerator and coolers containing samples will be maintained at a temperature between 2 degrees Celsius (ºC) and 6 ºC when actively storing samples. A designated field person will record the temperature of the refrigeration/freezer unit 24 hours before it will be used. The refrigeration/freezer unit temperature reading will be recorded in the temperature logbook. Temperature will not be recorded when refrigeration/freezer unit is not in use. If the temperature of the refrigerator is found to be outside the acceptable range, samples will be transferred to coolers and maintained at temperature with gel ice until control of the refrigeration temperature is established for a period of at least 3 hours.

When coolers are used to store samples for an extended period (more than 12 hours) or until refrigeration unit temperature is in control, the field personnel will randomly check the temperature of a representative cooler (i.e., one cooler per five total coolers storing like samples) periodically using a temperature probe (thermometer). This will ensure adequate gel ice is being added to the coolers to maintain samples at the appropriate temperature. Temperature of the samples will be collected by placing the temperature probe into the temperature blank for at least 60 seconds. The lid of the cooler will remain closed for this duration to maintain cooler temperature. The temperature of the cooler will not be recorded in the temperature logbook; however, if the temperature of the samples is not within the specified temperature the spent gel ice will be replaced with new gel ice or extra gel ice may be added to the cooler.

If the temperature of the samples in the cooler or refrigeration unit has exceeded 6 ºC or falls below 2 ºC, the field quality assurance coordinator (QAC) or Environmental Consultant Field Team Lead (FTL) shall contact the Environmental Consultant PM to determine potential impact to data quality and to determine if samples require recollection. When storage temperatures fall below 2 ºC samples do not typically require recollection unless the sample containers break as a result of freezing.

Samples collected in the field shall be transported to the laboratory or field-testing site as expeditiously as possible and maintained at the specified storage temperature. Note some preservation methods will extend a normal holding time. It is critical to plan ahead to ensure there is enough frozen gel ice available to properly chill or keep samples frozen during transit to the laboratory.

Samples shall be placed in a cooler containing gel ice immediately upon sample collection (no wet ice will be used) to maintain samples at the required temperature during collection, storage and transport to the laboratory. When possible, the Environmental Consultant FTL will request that the shipper store samples in a walk-in cooler (not freezer) when not in transit to the laboratory or while waiting for laboratory to pick up the coolers. A temperature blank (minimum 500-mL container) shall be included in every cooler and used to determine the internal temperature of the cooler upon receipt at the laboratory. Container shall be

clearly marked “Temperature Blank” so that laboratory does not mistake the temperature blank for an unlabeled sample.

3.4 Sample Packaging & Custody

Procedures to ensure the custody and integrity of the samples begin at the time the sample is removed from the project location (e.g., dust is collected) and continue through consolidation, packaging, shipping, transport, sample receipt, storage, preparation, analysis, data generation, reporting, and finally sample disposal. Records concerning the custody and condition of the samples will be maintained in field and laboratory records.

The field team shall maintain CoC records for all field and field QC samples. A sample is defined as being under a person's custody if any of the following conditions exist: (1) it is in their possession, (2) it is in their view, after being in their possession, (3) it was in their possession and they locked it up, (4) it is in a designated secure area, or (5) custody seals are used to evaluate whether or not cooler was opened during transit to the laboratory.

The field sampler may transfer custody of collected samples to a designated sample management person for subsequent CoC preparation, packaging, and shipping. The date and time of this transfer will be noted in the field sampler’s logbook. At this time, the field sampler will have relinquished sample custody. The designated sample management person will complete the CoC and sample labeling using information from the field sampler’s logbook. The field sampler or designated sample management person will sign the CoC to show transfers of custody before the CoC is placed in the cooler with samples and shipped to the laboratory. The designated sample management person is responsible for packing the cooler, shipping, and tracking the cooler to the laboratory.

Sample cooler packing will follow the process outlined below:

- Verify that all sample container lids were tightened securely and liquid will not leak out.
- Verify that the lid of the cooler is insulated and if present, drain port has been taped shut.
- Verify the cooler is large enough for samples and appropriate volume of gel ice. Do not use 6-pack coolers as the lids are typically not insulated and they rarely have enough room for the sample, temperature blank and sufficient gel ice.
- Place a sorbent pad into the bottom of the cooler to absorb moisture, condensation, and spilled liquids (methanol or water).
- Optional process (when shipping during hot weather or with extended transit time) - Place a large trash bag into the cooler and place the gel ice, samples, temperature blank and QC samples inside the trash bag and tie trash bag shut. This procedure will add a level of additional insurance that the cooler temperature will be maintained.
- Place a layer of frozen gel ice packs (lying flat) in the bottom of the trash bag or cooler. Gel ice obtained for this purpose should be laid out in a flat position prior to freezing for subsequent use when frozen solid. Partially melted or soft gel ice shall not be used to pack coolers for transport. A minimum of eight frozen gel ice packs are required to maintain sample temperature during 24-hour

transit. When considering addition of more gel ice, field personnel shall understand that small sample containers of water may freeze in transit and sample integrity may be lost.

- Cover the bottom layer of gel ice with bubble wrap to protect the sample containers in transit.
- Place all sample containers in bubble bags, boxes, re-sealable bags with sorbent pad, or wrap with bubble wrap, depending on the type of bottle. Double-bag 1-liter glass bottles in order to prevent damage during transport. All samples shall be placed in an upright and secured location to prevent leaks from occurring if lids are loose or seals ineffective. Sample containers shall never be placed on their side during sample handling, storage or shipping. Sample containers shall be tightly packed in the cooler to reduce movement or tipping during shipping.
- Position gel ice inside the cooler with the sample bottles in a manner that maximizes surface area contact with the samples.
- Place a temperature blank in the cooler, at the same level and next to the samples, preferably in the center of the cooler. Samples and temperature blanks should have been collected, placed in a refrigerator or in a cooler and allowed to stabilize at a temperature of 0 °C to 6 °C prior to packaging for transport.
- Place a layer of bubble wrap over the samples and layer on more flat frozen gel ice, if possible. A top layer of gel ice should not be added over the top of 1-liter glass sample bottles to minimize the possibility of breakage during transport.
- Fill in any empty space in the bottom, sides or top of cooler with paper, bubble wrap, or other packing material to minimize shifting.
- Tape the re-sealable gallon freezer bag containing the CoCs and any other paperwork to the inside lid of the cooler. This allows the laboratory to quickly retrieve the CoC during the login process. If multiple coolers are associated with a CoC, a copy is placed in each cooler.
- Close the lid and seal (using strapping tape) in a manner that shall prevent or detect opening or tampering if it occurs. In no case shall adhesive tape be placed on sample containers.
- Place two signed custody seals on the taped portions of the cooler over the lid opening and place additional layers of clear strapping tape over the signed custody seals. Wrap tape completely around the cooler and overlap ends. This will ensure the tape does not come off in transit and the custody seals will remain attached to the cooler (e.g., tape does not always adhere to the cooler surface during dusty or extremely cold conditions).
- Fill out the appropriate shipping paperwork and attach it to cooler.
    - Declare ALL materials that are classified as dangerous goods or hazardous materials by applying the Dangerous Goods in Excepted Quantities sticker.
    - Each line must be filled out completely.
    - Excepted quantities limits for transport by air are found in the International Air Transportation Association Dangerous Goods Regulations manual, Section 2.7 and ground transport limits can be located in the Code of Federal Regulation for Transportation in 49 Code of Federal Regulations, Parts 100 to 185.

- Attach a shipping address label to the top of the cooler. Attach other stickers such as “Refrigerate”, “Do not Freeze”, “Fragile”, and “up arrows”, indicating which end of the package is upright. The “up arrows” stickers should be placed on opposite sides of the cooler pointing in the same up direction as the sample containers within the cooler.

The laboratory PM shall be notified prior to collection of samples requiring rapid turnaround reporting and immediately after all coolers are shipped to the laboratory. The air bill number and date and time of arrival shall be provided to laboratory for tracking purposes.

All samples shall be uniquely identified, labeled, and documented in the field at the time of collection per the QAPP.

The following minimum information concerning the sample shall be documented on the CoC:

- Project name and number
- CoC identification number
- Contact information
- Unique sample identification (per project-specific work plan specifications)
- Date and time of sample collection, and grab or composite sample designation
- Source of sample (including name, location, and matrix)
- Number of sample containers
- Point of contact and contact information
- Sampler name, signature of field personnel who collected the samples and involved in the sample transfer
- Preservative used
- Analyses required
- Requested analytical turn-around-time
- Analytical laboratory performing the analysis
- Method of sample shipment, courier name and bill of lading or transporter tracking number (if applicable)
- Project Information Form
- Any additional information the laboratory must know to perform the requested analyses, such as holding time, if laboratory filtering is required for dissolved metals and matrix spike / matrix spike duplicates (MS/MSDs)
- Pertinent field data (“strong fuel odor”, field instrument readings for highly contaminated samples, etc.)

- Custody transfer signatures, dates, and times of sample transfer from the field to transporters and to the laboratory or laboratories

Upon arrival at the designated laboratory, the CoC will be completed with:

- Name of the person receiving the container and date of arrival or receipt of samples
- Name of the person opening the shipping container, along with date, time, temperature of temperature blank, shipping container, seal number, and condition of shipping container. If a temperature blank is not included with the cooler, the laboratory will record the air temperature inside the cooler or the temperature of one of the non-volatile samples.
- Any remarks regarding sample condition upon arrival such as temperature, breakage, leakage, incorrectly identified samples, inadequate sample volume, lack of QC shall be recorded on the CoC or cooler receipt form. An example cooler receipt form is provided in Attachment 1.

3.5 Field Documentation

Field documentation shall be reviewed by the Environmental Consultant FTL, field QAC, and/or Environmental Consultant's Project Manager (PM) daily to ensure recorded information is accurate and complete. Field documentation will also be reviewed prior to report development to ensure records are complete and accurate. Field documentation will be recorded in field logbooks and supplemented on field data collection forms.

3.5.1 Field Logbook

See work instructions FS-WI-020 Field Logbook for instructions and reasoning for keeping field logbooks as part of field documentation

3.5.2 Field Forms

The field forms shall be completed immediately upon completion of each field activity. All fields on the form must be completed. If a field is not applicable, then enter "NA". The forms shall be provided to the field QAC or Environmental Consultant FTL, who will provide them to the Environmental Consultant PM at the completion of the project, along with all logbooks used during field activities. This will ensure that all information is available to office personnel preparing post field event summary reports. Field forms are provided with associated work instructions. The laboratory uses a cooler receipt form in addition to the CoC to document sample receiving observations. A copy of this form is provided in Attachment 1. This form may be used by the field personnel to perform a quality check of the sample coolers prior to shipping them to the laboratory.

3.5.3 Materials

A list of anticipated materials needed for sample collection and management are described below. This list may not be all inclusive and site personnel should refer to the project-specific work plans to identify required equipment and materials needed to complete defined sampling events.

3.5.3.1 Field Samplers

- Copy of the project-specific work plan and Health and Safety Plan (HASP)
- Field logbooks (one per team) and data collection forms

- Required health and safety equipment (e.g., work permits, PPE, etc.)
- Sample collection equipment (e.g., HSV3 vacuum floor sampler, surface dust micro-vacuum, tweezers, floor mats, heavy-duty contractor trash bags and duct tape, digital scale, sample bottles, filters for micro-vacuum, paper towels, deionized water, sprayer)
- Decontamination materials (FS-WI-010)
- Sample collection, storage and management materials (e.g., jars, preservation, gel ice, coolers, re-sealable bags, bubble wrap, CoCs, custody seals, etc.)
- Investigation-derived waste containers, labels, spill containment material
- Digital camera

3.5.3.2 Sample Management Personnel

- Copy of the project-specific work plan and Health and Safety Plan (HASP)
- Thumb drive with EDGE sample management Software
- Clean warm work area (should not be area where solvents or fuels are stored or used)
- Field sampler's data collection forms and a copy of field logbook notes
- Required health and safety equipment (e.g., PPE, etc.)
- Decontamination materials (FS-WI-010), waste container, and waste labels
- Sample collection, storage and management materials (e.g., jars, preservation, gel ice, coolers, re-sealable bags, bubble wrap, CoCs, custody seals, etc.)
- Digital camera to document condition of samples (e.g., if duplicate samples look different after they are thawed)
- Logbook with lot numbers and glassware inventory
- Field logbook
- Computer and software to prepare electronic CoC
- Shipping labels and forms
- QA/QC Verification Task List (Attachment 2)

4. KEY RESPONSIBILITIES

The key project responsibilities shall be clearly defined in the project-specific work plan and QAPP.

5. KEY DOCUMENTS/TOOLS/REFERENCES

ERM (ERM Alaska, Inc.). 2021. FS-WI-008. Waste Management.

ERM. 2021. FS-WI-010. Equipment Decontamination.

ERM. 2021. FS-WI-020. Field Logbook.

United States Environmental Protection Agency Test Methods for SW846 Third Edition to include Updates I through IVB.

Attachments:

Example Laboratory Cooler Receipt Form

QA/QC Verification Task List

6. CONTROLLED DOCUMENT ADMINISTRATION INFORMATION

Authority:	Program Manager	**Issue Date:**	September 21, 2021
Custodian:	Document Custodian	**Revision Date:**	

7. REVISION LOG

Revision Date	Authority	Custodian	Revision Details
September 21, 2021	Thomas Beckman	Courtney Pijanowski	Initial

ATTACHMENT 1

Cooler Receipt Form Example

Pace Analytical®	Document Name: Sample Condition Upon Receipt Form - ESI	Revised Date: 02Jun2011 Page 1 of 1
	Document Number: F-L-210 Rev.04	Issuing Authority: Pace Minnesota Quality Office

Sample Condition Upon Receipt - ESI Tech Specs

Client Name: _____ Project # _____

Courier: ☐ Fed Ex ☐ UPS ☐ USPS ☐ Client ☐ Commercial ☐ Pace Other _____

Tracking #: _____

Optional: Proj Due Date: Proj Name:

Custody Seal on Cooler/Box Present: ☐ yes ☐ no Seals intact: ☐ yes ☐ no

Packing Material: ☐ Bubble Wrap ☐ Bubble Bags ☐ None ☐ Other _____ Temp. Blank Yes ____ No ____

Thermometer Used 80344042 or 80512447 **Type of Ice:** Wet Blue None ☐ Samples on ice, cooling process has begun

Cooler Temperature _____ **Biological Tissue is Frozen:** Yes No

Temp should be above freezing ≤ 6°C **Comments:**

Date and initials of person examining contents: _____

Chain of Custody Present:	☐Yes ☐No ☐N/A	1.
Chain of Custody Filled Out:	☐Yes ☐No ☐N/A	2.
Chain of Custody Relinquished:	☐Yes ☐No ☐N/A	3.
Sampler Name & Signature on COC:	☐Yes ☐No ☐N/A	4.
Samples Arrived within Hold Time:	☐Yes ☐No ☐N/A	5.
Short Hold Time Analysis (<72hr):	☐Yes ☐No ☐N/A	6.
Rush Turn Around Time Requested:	☐Yes ☐No ☐N/A	7.
Sufficient Volume: triple volume provided for MS/MSD	☐Yes ☐No ☐N/A	8.
Correct Containers Used: -Pace Containers Used:	☐Yes ☐No ☐N/A ☐Yes ☐No ☐N/A	9.
Containers Intact:	☐Yes ☐No ☐N/A	10.
Filtered volume received for Dissolved tests	☐Yes ☐No ☐N/A	11.
Sample Labels match COC: -Includes date/time/ID/Analysis Matrix:	☐Yes ☐No ☐N/A	12.
All containers needing acid/base preservation have been checked. All containers needing preservation are found to be in compliance with EPA recommendation. Per method, VOA pH is checked after analysis	☐Yes ☐No ☐N/A ☐Yes ☐No ☐N/A	13. ☐ HNO3 ☐ H2SO4 ☐ NaOH ☐ HCl Samp # Initial when completed / Lot # of added preservative
Samples checked for dechlorination:	☐Yes ☐No ☐N/A	14.
Headspace in VOA Vials (>6mm):	☐Yes ☐No ☐N/A	15.
3 Trip Blanks Present: Trip Blank Custody Seals Present Pace Trip Blank Lot # (if purchased):	☐Yes ☐No ☐N/A ☐Yes ☐No ☐N/A	16.

Client Notification/ Resolution: Field Data Required? Y / N

Person Contacted: _____ Date/Time: _____

Comments/ Resolution:

Temp Log: Temp must be maintained at <6 C during login, record temp every 20 mins	
Opened time: Temp:	
Time: put in cooler	
Time: Temp:	

Project Manager Review: _____ **Date:** _____

Note: Whenever there is a discrepancy affecting North Carolina compliance samples, a copy of this form will be sent to the North Carolina DEHNR Certification Office (i.e out of hold, incorrect preservative, out of temp, incorrect containers)

QA/QC Verification Task List

QA/QC Verification Task List

1. Field Sampling Forms and field notes are complete and reviewed (Yes/No):
   a. Check for completeness
   b. Proper units and significant figures
   c. Errors crossed out with single line, initial and date
   d. Initials and dates of field sampler and reviewed
   e. Any field monitoring or sample problems were noted (i.e., dissolved oxygen over range, dry well, poor re-charge, incomplete sample sets)
2. Field Instrument Records are complete and reviewed (Yes/No):
   a. Calibration forms complete
   b. Pre- and post-operations calibration and calibration check performed
   c. Maintenance documented when needed
   d. Field instruments removed from service if not calibrating or operating per manufacturer's specifications
3. Sample collections procedures were followed (Yes/No):
   a. The appropriate FS-WI sample procedures were followed
   b. The correct sample containers were used
   c. The samples were properly labeled
   d. The samples for volatiles were inspected for headspace
   e. Sample lids were tightly sealed
   f. Special sample handling procedures were followed (i.e, PFAS, Acrolein, low-level soil VOCs)
   g. Appropriate decontamination procedures were followed between sample locations
4. Sample handling procedures were followed (Yes/No):
   a. Samples stored on ice in cooler or in ERM field refrigerator
   b. Refrigerator and freezer temperatures within acceptable ranges
   c. Samples were kept in custody of field team member during collection
   d. Samples were stored securely in ERM field trailer until shipment
5. Chain of Custody procedures were followed (Yes/No):
   a. Holding times reviewed and samples shipped to the laboratory with adequate time left for preparation/analysis
   b. Chain of custody completed, signed and dated for each sample delivery group
   c. Sample IDs on field sample forms, sample container labels and chain of custody were reviewed for agreement
   d. Samples listed on the chain of custody were present in the cooler
   e. Chain of custody included with each cooler
   f. Sample coolers were packed with adequate gel ice and packing material
   g. Sample cooler labels were correct; cooler number was added to each label
   h. Shipping documents were reviewed and complete
6. Quality Control Samples collected/included as required (Yes/No):
   a. Laboratory temperature blanks added to each cooler containing samples
   b. Trip blank sets included daily with the sample containers for each volatiles method (I.e., 8260, 8260 SIM, AK101)
   c. Ambient blank sets collected when required for each volatiles method
   d. Equipment blanks collected when required, at beginning of sample event
   e. Storage blanks were placed in refrigerators and freezer weekly when used for sample storage
   f. Field duplicates were collected as required in work plan. Any deviations were documented.
   g. Extra volume for matrix spikes was collected as required in work plan. Any deviations were documented.

1. PURPOSE/SCOPE OF PROCEDURE

1.1 Purpose

The purpose of this field sampling work instruction (FS-WI) is to provide guidance to personnel performing site activities that require various types of reporting. Understanding this FS-WI ensures personnel supporting the field activities are prepared and follow consistent protocol, which enable the objectives defined in the project-specific work plan and Quality Assurance Project Plan (QAPP) to be met.

1.2 Scope

This procedure was developed to guide and support ongoing work conducted within the Butte Priority Soils Operable Unit in Butte, Montana in support of Residual Metals Abatement Program Plan (RMAP) Administrative Order on Consent (United States Environmental Protection Agency [USEPA] Docket No. CERCLA-08-2011-0011) for Corrective Action.

2. DEFINITIONS

AR	Atlantic Richfield Company
FS-WI	Field Sampling Work Instruction
GPS	Global Positioning System
PM	Project Manager
QAPP	Quality Assurance Project Plan
USEPA	United States Environmental Protection Agency

3. PROCEDURE/PROCESS

Accurate recordkeeping is an important piece of all reporting activities and processes. This procedure is written for various activities conducted in the field with reporting requirements. The project-specific work plans will define the reporting requirements for each project.

3.1.1 Daily Reports

Daily reports document the field activities and are requested by AR on a project-by-project basis. Daily reports allow the project team members who are not actively involved in the field to track the status of the projects and provide assistance, where warranted. An example report is provided as an attachment to this work instruction. The following information shall be included in the daily reports at a minimum:

1. Health and safety (summary of tailgate meeting topics, incidents, near misses, and unsafe conditions).

1. Progress achieved on the day the report was written.

2. Cumulative work/sampling progress.

3. Quality assurance / quality control verification.

4. Activities planned for the following day.

5. Problems and unresolved issues (identify person taking the lead to resolve issues).

6. If appropriate, attach figures showing locations where activities were conducted or completed, photographs, or other supplemental documents that can be included in an email.

Identify the person that will develop the daily reports, person in office to review reports (if appropriate), and project team members that will receive a copy of the daily reports. These reports are a great opportunity to show case proactive safety programs or actions and lessons learned, which may provide immediate benefit to others managing similar projects.

3.1.2 Leaks, Spills, Releases

3.1.2.1 Definitions

- Leak: Defined as a release that is not reportable to external agencies; however, should be reported to the field team leader (FTL) and contractor project manager (PM).
- Spill (release): An unplanned loss of primary containment, irrespective of secondary containment or recovery and also as any loss (planned or unplanned) of primary containment that impacts the ground, water, or air.

All leaks, spills, and releases regardless of size must be reported immediately to the consultant PM, and the consultant's health, safety, and environmental (HSE) manager.

3.2 Accidents and Equipment Damage

3.2.1 Accidents

If you are involved in an accident, be calm and assess the situation to determine if there are any injuries or unsafe conditions. If there are any injuries or unsafe conditions (e.g., fire), call for emergency, and begin first aid (if necessary) ensuring you do not place yourself into a more dangerous situation.

Be prepared to provide the following information to accident responders:

- Your name and possibly names of others involved.
- The company you work for.
- Your supervisor's name.
- A current driver's license.
- The circumstances that lead to the accident. Be truthful as there will be an accident investigation.

Assess the area to see if there are any spills. If a spill has occurred, report it immediately following the instructions in Section 3.2. Finally, contact the contractor PM and contractor HSE manager.

3.3 Field Documentation

Field documentation shall be reviewed daily to ensure recorded information is accurate and complete. Field documentation will be recorded in field logbooks and data collection forms or electronic means (i.e., field tablets). Clear and accurate written and photographic documentation is a critical aspect of the tasks

performed under this work instruction. This documentation may be used to support cost estimates or confirm completion and accuracy of field tasks. Site photographs may be taken of sampling locations, field activities, and to document site conditions, as necessary. Photographs should include a scale in the picture when practical.

3.3.1 Field Logbook

See work instructions FS-WI-020 Field Logbook for instructions and reasoning for keeping field logbooks as part of field documentation.

3.3.2 Field Forms

The field forms shall be completed while performing activities or as soon as it is finished. Field forms may be limited to tail gate safety forms or Job Hazard Analysis forms for this activity. A form may be generated to capture specific field information associated with the purpose of the site visit. For example, a special diagram may be generated to show location of sensitive areas, buildings, metal debris, cliff, and seeps that will be transferred into figures in the work plan; a checklist (form) may be needed to document information collected during a site inspection or site audit.

3.3.3 Materials

Materials needed are dependent upon the tasks that will be performed. At a minimum, the following may be required:

- Sample location map that shows school buildings, rooms, structures;
- Project-specific work plans, design drawings;
- GPS coordinates;
- Digital camera;
- Cones or barriers if working in high traffic area;
- Copy of contract (definable features of work, client expectations);
- Metal clipboard box case (store daily reports, personnel training records, work plan);
- Required health and safety processes and equipment (e.g., ground disturbance permit, completed Task Hazard Analysis, personal protective equipment); and
- Field logbook.

4. KEY RESPONSIBILITIES

The key project responsibilities shall be clearly defined in the project-specific work plan and QAPP.

5. KEY DOCUMENTS/TOOLS/REFERENCES

ERM. 2021. FS-WI-020. Field Logbook.

6. CONTROLLED DOCUMENT ADMINISTRATION INFORMATION

Authority:	Program Manager	**Issue Date:**	September 20, 2021
Custodian:	Document Custodian	**Revision Date:**	

7. REVISION LOG

Revision Date	Authority	Custodian	Revision Details
September 20, 2021	Thomas Beckman	Courtney Pijanowski	Initial

1. PURPOSE/SCOPE OF PROCEDURE

1.1 Purpose

The purpose of this field sampling work instruction (FS-WI) is to provide guidance to personnel developing field documentation in the field logbooks to ensure personnel supporting the field activities are prepared and follow consistent protocol, which enable the objectives defined in the project-specific work plan and Quality Assurance Project Plan (QAPP) to be met.

1.2 Scope

This procedure was developed to guide and support work conducted within the Butte Priority Soils Operable Unit in Butte, Montana in support of Residual Metals Abatement Program Plan (RMAP) Administrative Order on Consent (United States Environmental Protection Agency [USEPA] Docket No. CERCLA-08-2011-0011) for Corrective Action.

2. DEFINITIONS

AR	Atlantic Richfield Company
FS-WI	Field Sampling Work Instruction
PM	Project Manager
QAPP	Quality Assurance Project Plan
RMAP	Residential Metals Abatement Program
USEPA	United States Environmental Protection Agency

3. FIELD DOCUMENTATION PROCEDURE

This field documentation procedure has been developed to outline a standardized methodology for use when collecting field notes. Section 3.1 describes the layout of field notes, including the information that shall be included at the start of each workday and the information that shall be included on every subsequent page. Section 3.2 lists basic parameters that shall be used when entering information into the field notebook. Section 3.3 describes the type of detailed information that may be included in the field note body text. Section 3.4 describes the procedure for storing and retaining field documentation.

3.1 Field Notebook Layout

This section details the pertinent information that is to be included on the front cover, the inside cover, and the title block of each page in the field logbook.

On the front page or cover of the logbook:

- Site name
- Project name
- Logbook number
- Owner of logbook

- Date started and ended
- Address of company

On the inside cover of the logbook:

- Point of contacts and telephone numbers (e.g., laboratory, airlines, expeditor, client contacts, emergency numbers, subcontractors)

Each page shall contain the following in the page header:

- Date of entry
- Purpose of site visit or activity
- Location of site of investigation or point of interest
- Names of all field staff including contractors
- Project name
- Project number
- Weather conditions and temperature (first page of the day)

Each page shall contain the following in the page footer:

- Page number and number of pages
- Initials of person writing field notes

See Attachment 1 for an example of the field logbook format.

3.2 Basic Information

Each field sampling team will have a field logbook. When documenting information in the field notebook, the following parameters shall be followed:

- The logbook will be bound with numbered pages (Rite in the Rain notebooks are preferred).
- Field notes shall be entered legibly, using Rite in the Rain pens.
- Use every line in field logbook. If a line is skipped to organize information more clearly, put a dash in the line and initial next to it.
- Place a single line through any mistake; initial and date the mistake.

3.3 Body Text Information

This section outline more detailed information that may be included in the field notes body text. This information may vary depending on the type of work being performed. Project field logbooks shall contain sufficient information to enable the sampling activity to be reconstructed without relying on the collector's

memory. All pertinent information shall be documented as near to real-time as possible. At the conclusion of each day, the person maintaining the logbook shall sign and date the documentation entries.

- Time and date work stated and ended
- Date and times of entries
- Date and time of arrivals and departures at site
- Document meetings and personnel in attendance
- Names and responsibilities of environmental consultant personnel working on site
- Names, affiliations, and purpose of environmental consultant site visitors
- Level of personal protective equipment required
- Special personal monitoring equipment needed
- Points of contact for future reference (cell number, office number, alternate's name and phone number)
- Special coordination requirements for site access
- Field instrumentation or equipment used, and purpose of use (e.g., health and safety screening, sample selection for laboratory analysis)
- Note source, quality, or lot numbers for any supplies or reagents (e.g., calibration standards, preservatives such as methanol)
- Document where certificates or information supplied with the equipment used are retained
- Lot numbers of reagents
- Photographic documentation, including date, time, and other site description information
- Sampling procedures (e.g., filtered, field screened, composite, multi-incremental, preservation techniques)
- Field screening results, field measurement results and type of instrument used
- Calibration information for field measurements, including results and frequency
- Sample location (draw a sketch with corresponding sample identification [ID]; reference photographs or figures)
- Description of samples with sample ID, collection date/time and associated quality control sample ID (e.g., field duplicates, trip blanks, equipment blanks)
- Number of coolers, cooler IDs, chain-of-custody sent to the laboratory, laboratory name, the shipping method used, and shipment tracking number

- Document measurements (e.g., future excavation, size of building), global positioning system coordinates or swing tie measurements, identify distance from potential hazards to area where activities will be performed
- Document appropriate references to maps (work plan) and photographic logs of sampling sites
- Decontamination procedures
- Types of waste, volumes generated, final disposition, and contractor point of contact for waste disposal
- Changes or variances to work plan
- Phone conversations and directions

If any of the information above is already listed on field sampling forms or other field documents, the information does not have to be recorded twice, but it must be referenced in the chronological order of events.

3.4 Document Retention

This section describes the methods that shall be used to properly store and retain field documentation after the field effort. Proper document retention is essential to avoid inadvertent loss or damage to field documents.

After field activities have concluded, the following steps shall be taken to ensure document retention:

- Scan field documents and store in the project folder on the secured consultant server
- Organize field document hard copies and return to the project manager (PM)

3.4.1 Materials

Materials needed are dependent on the tasks that will be performed. At a minimum, the following may be required:

- At least one field logbook per team
- Rite in the Rain waterproof pen(s)

4. KEY RESPONSIBILITIES

The key project responsibilities shall be clearly defined in the project-specific work plan and QAPP.

5. KEY DOCUMENTS/TOOLS/REFERENCES

Attachment:

Example Field Logbook Format

6. CONTROLLED DOCUMENT ADMINISTRATION INFORMATION

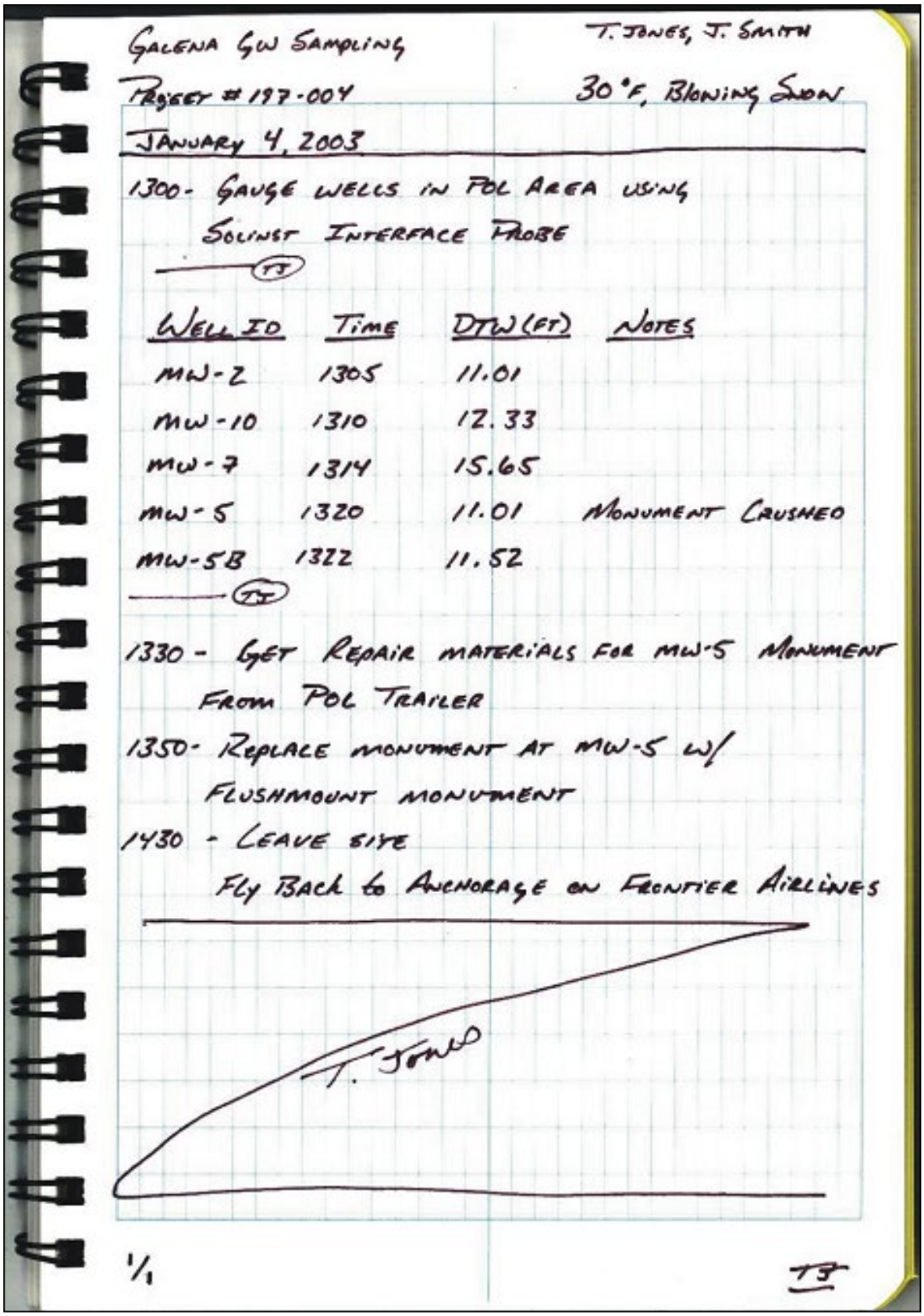
Authority:	Program Manager	**Issue Date:**	September 20, 2021
Custodian:	Document Custodian	**Revision Date:**	

7. REVISION LOG

Revision Date	Authority	Custodian	Revision Details
September 20, 2021	Thomas Beckman	Courtney Pijanowski	Initial

ATTACHMENT 1

Example Field Logbook Format



Galena GW Sampling — T. Jones, J. Smith

Project # 197-004 — 30°F, Blowing Snow

January 4, 2003

1300 - Gauge wells in POL area using Solinst Interface Probe

TT

Well ID	Time	DTW (ft)	Notes
MW-2	1305	11.01	
MW-10	1310	12.33	
MW-7	1314	15.65	
MW-5	1320	11.01	Monument Crushed
MW-5B	1322	11.52	

TT

1330 - Get Repair materials for MW-5 Monument from POL Trailer

1350 - Replace monument at MW-5 w/ Flushmount monument

1430 - Leave site

Fly Back to Anchorage on Frontier Airlines

T. Jones

1/1

TJ

Sheet No.: _____

RMAP FIELD SAMPLE DATA SHEET (FSDS) FOR SURFACE DUST

School: _____ Sampling Date: _____

Other_____ Field Logbook No: _____

Page No: _____

Sampling Team: ERM Other _____ Name(s):_____

Data Item	1	2	3
Sample ID			
Filter Number	FL_____	FL_____	FL_____
Location (e.g., room number, etc.)			
Sample Group (circle)	Surface, Ceiling Tile, Air Vent, Boiler Room, Light Fixture, Attic Other_____	Surface, Ceiling Tile, Air Vent, Boiler Room, Light Fixture, Attic Other_____	Surface, Ceiling Tile, Air Vent, Boiler Room, Light Fixture, Attic Other_____
Location Description (circle)	Basement, Ground Floor, 1st Floor, 2nd Floor, 3rd Floor, Main Floor Other_____	Basement, Ground Floor, 1st Floor, 2nd Floor, 3rd Floor, Main Floor Other_____	Basement, Ground Floor, 1st Floor, 2nd Floor, 3rd Floor, Main Floor Other_____
Matrix Type (circle)	Horizontal Surfaces Other_____	Horizontal Surfaces Other_____	Horizontal Surfaces Other_____
Category (circle)	FS FB-(field blank) LB-(lot blank) D-(duplicate)	FS FB-(field blank) LB-(lot blank) D-(duplicate)	FS FB-(field blank) LB-(lot blank) D-(duplicate)
Sample Parent ID (if a duplicate sample)			
Approximate Sample Area (circle units)	_____ cm^2 m^2 in^2 ft^2	_____ cm^2 m^2 in^2 ft^2	_____ cm^2 m^2 in^2 ft^2
Flow Meter Type (circle)	Rotometer Dry-Cal NA	Rotometer Dry-Cal NA	Rotometer Dry-Cal NA
Pump ID No.			
Flow Meter ID No.			
Start Time			
Start Flow (L/min)			
Stop Time			
Stop Flow (L/min)			
Pump Fault? (circle)	No Yes	No Yes	No Yes
Field Comments **Cassette Lot Number:** (circle) Other_____			

v 032118 Lab: Pace Analytical Micro-Vac Cassette Filter Diameter = 37mm MCE; Pore Size = 0.45µm

For Field Team Completion (Initials)	Completed by: _____ QC by: _____	For Data Entry	Entered by: _____ QC by: _____

Sheet No.: _____

RMAP FIELD SAMPLE DATA SHEET (FSDS) FOR HVS3 FLOOR DUST

School: _____ Sampling Date: _____

Other_____ Field Logbook No: _____

Page No: _____

Sampling Team: ERM Other _____ Name(s):_____

Data Item	1	2	3
Sample ID			
Location (e.g., room number, etc.)			
Sample Group (circle)	Bare Floor: Tile, Laminate, Wood Carpet: Plush, Level Loop, Multilevel, Shag, Floor Mat Other_____	Bare Floor: Tile, Laminate, Wood Carpet: Plush, Level Loop, Multilevel, Shag, Floor Mat Other_____	Bare Floor: Tile, Laminate, Wood Carpet: Plush, Level Loop, Multilevel, Shag, Floor Mat Other_____
Location Description (circle)	Basement, Ground Floor, 1st Floor, 2nd Floor, 3rd Floor, Main Floor Other_____	Basement, Ground Floor, 1st Floor, 2nd Floor, 3rd Floor, Main Floor Other_____	Basement, Ground Floor, 1st Floor, 2nd Floor, 3rd Floor, Main Floor Other_____
Matrix Type (circle)	Floor Dust Tracked in Dirt Other_____	Floor Dust Tracked in Dirt Other_____	Floor Dust Tracked in Dirt Other_____
Category (circle)	FS D-(duplicate) RB-(rinsate) SB-(sand blank)	FS D-(duplicate) RB-(rinsate) SB-(sand blank)	FS D-(duplicate) RB-(rinsate) SB-(sand blank)
Sample Parent ID (if a duplicate sample)			
Approximate Sample Area (circle units)	_____ cm^2 m^2 in^2 ft^2	_____ cm^2 m^2 in^2 ft^2	_____ cm^2 m^2 in^2 ft^2
HVS3 Vacuum ID No.			
Leak Check? (circle)	No Yes	No Yes	No Yes
20 sec cleaning @ end? (circle)	No Yes	No Yes	No Yes
Total Sample Time	_____minutes	_____minutes	_____minutes
Flow Drop	_____inches of water	_____inches of water	_____inches of water
Nozzle Drop	_____inches of water	_____inches of water	_____inches of water
Field Comments Bottle Lot Number: (circle) Other_____			

v 032118 Lab: Pace Analytical Container: HVS3 Catch Bottle = 250 mL LDPE

For Field Team Completion (Initials)	Completed by: _____ QC by: _____	For Data Entry	Entered by: _____ QC by: _____

Sheet No.:_____

RMAP FIELD SAMPLE DATA SHEET (FSDS) FOR PERSONAL AIR

School: _____ Sampling Date: _____

Other_____ Field Logbook No: _____

Page No: _____

Data Item	1	2	3
Sample ID			
Sampling Activities (circle all that apply)	Surface, Ceiling Tile, Air Vent, Boiler Room, Light Fixture, Attic, HVS3 Floor Other_____	Surface, Ceiling Tile, Air Vent, Boiler Room, Light Fixture, Attic, HVS3 Floor Other_____	Surface, Ceiling Tile, Air Vent, Boiler Room, Light Fixture, Attic, HVS3 Floor Other_____
Location Description (circle all that apply)	Basement, Ground Floor, 1st Floor, 2nd Floor, 3rd Floor, Main Floor Other_____	Basement, Ground Floor, 1st Floor, 2nd Floor, 3rd Floor, Main Floor Other_____	Basement, Ground Floor, 1st Floor, 2nd Floor, 3rd Floor, Main Floor Other_____
Sample Venue	Indoor Outdoor Both NA	Indoor Outdoor Both NA	Indoor Outdoor Both NA
Sample Type	FS FB LB Other____	FS FB LB Other____	FS FB LB Other____
Personnel Information: ID_____ Name_____ Task_____			
Sample Air Type	NA PA-EXC PA-TWA	NA PA-EXC PA-TWA	NA PA-EXC PA-TWA
Flow Meter Type	NA Rotameter DryCal	NA Rotameter DryCal	NA Rotameter DryCal
Cassette Lot No _____ **Flow Meter ID** _____		(*For Blanks "Z" through "Pump ID" to "Sample Air Stop Flow" then circle NA for "Pump Fault" & enter 0 for Total Time & Quantity)*	
Pump ID			
Sample Air Start Date			
Sample Air Start Time			
Sample Air Start Flow (L/min)			
Sample Air Stop Date			
Sample Air Stop Time			
Sample Air Stop Flow (L/min)			
Pump Fault	No NA Yes	No NA Yes	No NA Yes
Sample Total Time (min)			
Sample Quantity (L)			
Field Comments **Cassette Lot Number:** (circle) Other_____			

v 032118 Lab: Pace Analytical Air Filter Diameter = 37mm; Pore Size = 0.8µm

For Field Team Completion: Completed by: _____ QC by:_____	For Data Entry: Entered by:_____ QC by:_____

Designation: D5438 – 17

Standard Practice for Collection of Floor Dust for Chemical Analysis[1]

This standard is issued under the fixed designation D5438; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This practice covers a procedure for the collection of a sample of dust from carpets and bare floors that can be analyzed for lead, pesticides, or other chemical compounds and elements.

1.2 This practice is applicable to a variety of carpeted and bare floor surfaces. It has been tested for level loop and plush pile carpets and bare wood floors, specifically.

1.3 This practice is not intended for the collection and evaluation of dust for the presence of asbestos fibers.

1.4 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

1.5 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]

D422 Test Method for Particle-Size Analysis of Soils (Withdrawn 2016)[3]

D1356 Terminology Relating to Sampling and Analysis of Atmospheres

E1 Specification for ASTM Liquid-in-Glass Thermometers

E337 Test Method for Measuring Humidity with a Psychrometer (the Measurement of Wet- and Dry-Bulb Temperatures)

E1137/E1137M Specification for Industrial Platinum Resistance Thermometers

E2251 Specification for Liquid-in-Glass ASTM Thermometers with Low-Hazard Precision Liquids

F608 Test Method for Evaluation of Carpet Embedded Dirt Removal Effectiveness of Household/Commercial Vacuum Cleaners

3. Terminology

3.1 *Definitions*—For definitions of terms used in this practice, refer to Terminology D1356.

3.1.1 *carpet-embedded dust*—soil and other particulate matter, approximately 5-µm equivalent aerodynamic diameter and larger, embedded in carpet pile and normally removable by household vacuum cleaners.

3.1.2 *surface dust*—soil and other particulate matter, approximately 5-µm equivalent aerodynamic diameter and larger, adhering to floor surfaces and normally removable by household vacuum cleaners.

4. Summary of Practice

4.1 The sampling method described in this practice is taken from work published in Roberts et al. (**1-3**),[4] and Stamper et al. (**4**).

4.2 Particulate matter is withdrawn from the carpet or bare floor by means of vacuum-induced suction which draws through a sampling nozzle at a specific velocity and flow rate, and the particles are separated mechanically by a cyclone. The cyclone is designed to efficiently separate and collect particles approximately 5-µm mean aerodynamic diameter and larger. However, much smaller particles are also collected at unknown efficiencies. The sampling system allows for height, air flow, and suction adjustments to reproduce systematically a specific air velocity for the removal of particulate matter from carpeted and bare floor surfaces, so that these sampling conditions can be repeated.

NOTE 1—Side-by-side comparison of the HVS3 and a conventional upright vacuum cleaner revealed that both collected particles down to at least 0.2 µm and that the HVS3 was more efficient at collecting particles smaller than 20 µm than conventional vacuum cleaners (**5**). If desired, a fine-particle filter may be added downstream of the cyclone to collect 99.9 % of particles above 0.2 µm aerodynamic mean diameter.

4.3 The particulate matter in the air stream is collected in a catch bottle attached to the bottom of the collection cyclone.

[1] This practice is under the jurisdiction of ASTM Committee D22 on Air Quality and is the direct responsibility of Subcommittee D22.05 on Indoor Air.

Current edition approved March 1, 2017. Published March 2017. Originally approved in 1993. Last previous edition approved in 2011 as D5438 – 11. DOI: 10.1520/D5438-17.

[2] For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

[3] The last approved version of this historical standard is referenced on www.astm.org.

[4] The boldface numbers in parentheses refer to the list of references at the end of this standard.

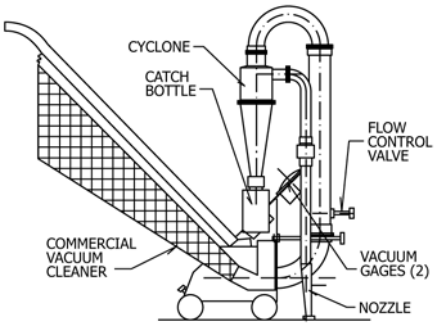


FIG. 1 Floor Dust Sampler Using a Commercial Vacuum Cleaner as the Suction Source

This catch bottle shall be capped for storage of the sample and transported to the laboratory for analysis.

5. Significance and Use

5.1 This practice may be used to collect dust from carpeted or bare floor surfaces for gravimetric or chemical analysis. The collected sample is substantially unmodified by the sampling procedure.

5.2 This practice provides for a reproducible dust removal rate from level loop and plush carpets, as well as bare floors. It has the ability to achieve relatively constant removal efficiency at different loadings of surface dust.

5.3 This practice also provides for the efficient capture of semivolatile organic chemicals associated with the dust. The test system can be fitted with special canisters downstream of the cyclone for the capture of specific semivolatile organic chemicals that may volatilize from the dust particles during collection.

5.4 This practice does not describe procedures for evaluation of the safety of floor surfaces or the potential human exposure to carpet dust. It is the user's responsibility to evaluate the data collected by this practice and make such determinations in the light of other available information.

6. Interferences

6.1 There are no known interferences to the determination of dust loadings covered by this practice.

7. Apparatus

7.1 *Sampling Apparatus,* which may be acquired commercially[5] (as shown in Fig. 1) or constructed as follows:

7.1.1 The dimensions of the sampling apparatus (nozzle size, cyclone diameter, cyclone inlet diameter, etc.) are interdependent. The flow rate must produce a sufficient velocity both at the sampled surface and in the cyclone. The cyclone must have a cut diameter of 5 µm at the same velocity that will provide a horizontal velocity of 40 cm/s at 10 mm from the nozzle in the carpet material, or 5 mm from the nozzle on bare floors. The fundamental principles of this device have been discussed in detail in Roberts et al. (**1-3**).

7.1.2 *Nozzle*—The edges and corners of the sampling nozzle shall be rounded to prevent catching the carpet material. The nozzle must be constructed to allow for sufficient suction to separate loose particles from the carpet or bare floor and carry them to the cyclone. It must have an adjustment mechanism to establish the nozzle lip parallel to the surface and to achieve the proper suction velocity and pressure drop across the nozzle. A nozzle 12.4 cm long and 1 cm wide, with a 13-mm flange and tapered to the nozzle tubing at no more than 30°, will yield the appropriate velocities when operated as specified in Section 11.

7.1.3 *Gaskets*—Gaskets in joints should be of a material appropriate to avoid sample contamination.

7.1.4 *Cyclone*—The cyclone shall be of a specific size such that a given air flow allows for separation of the particles 5-µm mean aerodynamic diameter and larger. The cyclone must be made of aluminum or stainless steel, and the catch bottle must be made of clear glass or fluorinated ethylene propylene (FEP) to avoid contamination and allow the operator to see the sample.

7.1.5 *Flow Control System*—The flow control system shall allow for substantial volume adjustment. The suction source must be capable of drawing 12 L/s through the system with no restrictions other than the nozzle, cyclone, and flow control system connected. An upright commercial vacuum cleaner with a seven amp or greater motor capable of pulling a vacuum of 6.5 kPa may be used for this purpose.

7.1.6 *Flow Measuring and Suction Gauges*—Two vacuum gauges are required— one with a range of 0 to 3.7 kPa is used for setting flow rate and another with a range of 0 to 2.5 kPa is used to set the pressure drop across the vacuum nozzle.

7.1.7 Optional filter holder assembly with appropriate fine particle filter, such as a 25-cm micro-quartz-fibre, binderless, acid-washed filter.[6]

7.2 *Other Equipment:*

7.2.1 *Stopwatch.*

7.2.2 *Masking Tape and Marking Pen,* for outlining sections for sampling.

[5] The sampling device used in the development and performance evaluation of this test method (P/N HVS3) was manufactured by CS-3, Inc., http://www.cs-3.com, which is the sole source of supply of the sampler known to the committee at this time. If you are aware of alternative suppliers, please provide this information to the Committee on Standards, ASTM Headquarters, 100 Barr Harbor Dr., West Conshohocken, PA 19428. Your comments will receive careful consideration at a meeting of the responsible technical committee, which you may attend.

[6] A filter holder for circular 25–cm particle filters and flow control valve assembly which replaces the normal flow control assembly is available from the manufacturer of the floor vacuum device.

7.2.3 *Clean Aluminum Foil and Clean Glass or FEP Jars,* for the collection and storage of samples.

7.2.4 *Thermometer* (see Specification E1, E1137/E1137M, or E2251).

7.2.5 *Relative Humidity Meter* (see Test Method E337, Method A, which allows use of alternative thermometers).

7.2.6 *Shaker Sieve,* as specified in Test Method D422, with 100 mesh-screen above the pan to separate the fine dust below 150 µm.

7.2.7 *Analytical Balance,* sensitive to at least 0.1 mg and having a weighing range from 0.1 mg to 1000 g.

8. Reagents and Materials

8.1 *Purity of Reagents*—Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available (**6**).

8.2 Methanol is required for sampling train cleaning after sample collection.

9. Sampling Strategy

9.1 The overall sampling strategy should be designed to address the goals of the study. Users should consider factors such as foot traffic volume, types of activities, proximity to potential sources, etc. The sampling strategy should be described in the sampling report so it can be taken into consideration when readers are comparing loadings or concentrations, or both, to those obtained from other studies. The ideal sampling location(s) for the beginning of the test procedure are an area that conforms with the protocol for the user's overall sampling strategy. For example, when sampling in a home for child exposure assessment, protocol may require the selection of a carpeted area for sampling where small children play or are likely to play.

10. Pretest Preparation and Calibration

10.1 *Calibration*—The sampling system described in this practice does not have any calibrated flow devices other than the cyclone and the Magnehelic gauges. The cyclone used for the separation of the particles must be designed to give proper separation at varying flow rates throughout the sampling range of the system. The pressure gauges and any other devices (that is, temperature gauge) used for testing purposes should be calibrated against a primary standard.

10.1.1 *Pressure Gauges*—Pressure gauges shall be calibrated against an inclined manometer or other primary standard prior to any field test. One means of checking a Magnehelic gauge is to set a flow rate through the sampling system with a manometer and then switch to the Magnehelic gauge. If the difference in the readings is more than 3 %, the gauge is leaking or is in need of repair or calibration. This should be done at two different flow rates when checking the gauge.

10.1.2 The cyclone flow measurement is calibrated with a laminar flow element, spirometer, or roots meter. See the appendix for cyclone calibration with a laminar flow element.

10.2 *Pretest Preparation:*

10.2.1 Each catch bottle to be used shall be clean and inspected for any contamination. The bottles should be marked with masking tape and a marking pen for identification of the test site, time, and date.

10.2.2 The sampling train shall be inspected to ensure that it has been cleaned and assembled properly.

10.2.3 The sampling train shall be leak-checked prior to sampling. This can be accomplished by placing a mailing envelope or a piece of cardboard beneath the nozzle and switching on the suction source. The flow Magnehelic gauge should read 5 Pa (0.02 in. H_2O) or less to ensure that the system is leak free. If any leakage is detected, the system shall be inspected for the cause and corrected before use.

11. Sampling

11.1 *Sampling a Carpeted Floor:*

11.1.1 *Pre-Test Survey*—Immediately prior to testing, complete a data form recording all requested information and sketch the area to be sampled. (See Fig. 2 for a sample data form.)

11.1.2 Select a sampling area in accordance with the established protocol for your sampling campaign. This should be determined prior to testing.

11.1.3 A typical sampling procedure may use measuring tapes placed on the carpet so that they are parallel to each other and on either side of the portion of carpet to be sampled (Fig. 3). The measuring tapes should be between 0.5 and 1.5-m apart and extended as far as practical. They should be taped to the carpet with masking tape every 30 cm.

11.1.4 Place the sampler in one corner of the sampling area and adjust the flow rate and pressure drop according to the type of carpet (see 11.1.8). The two factors that affect the efficiency of the sampling system are the flow rate and pressure drop at the nozzle. The pressure drop at the nozzle is a function of the flow rate and distance between the surface and the nozzle flange.

11.1.5 Clean the wheels and nozzle lip with a clean laboratory tissue immediately before sampling. Begin sampling by moving the nozzle between the ends of the two measuring tapes. The sampler is then moved back and forth four times on the first strip, moving the sampler at approximately 0.5 m/s. (The widths of the strips are defined by the width of the sampling nozzle.) Effective nozzle width is 13 cm for the CS_3 sampler. Move in a straight line between the numbers on the measuring tape. Angle over to the second strip on the next pass gradually, and repeat four double passes. After sampling approximately 0.5 m^2, determine the amount of collected material in the bottom of the catch bottle. As a rough estimate, the collection of dust to a depth of 6 mm in a 55-mm diameter catch bottle corresponds to approximately 6 to 8 g. If there is less than 6 mm of dust, sample an additional 0.5 m^2 next to the area already sampled. Hair, carpet fibers, and other large objects should be excluded from the sample when estimating the quantity collected.

11.1.6 Continue sampling in the area laid out until an adequate sample is collected. Switch off the vacuum. The catch

SAMPLE DATA SHEET

Operator _____ Date _____ Sample Ident. #:

Sampling site _____

Type of Carpet: Plush _____ Level Loop _____ Multilevel _____ Shag _____
Type of Vacuum: Upright _____ Canister _____ Other _____

Last Vacuumed _____ Temp. _____ Humidity _____ %

Comments: _____

_____

Location of Area Sampled: _____ Area _____ m^2

Sketch of Area Sampled:

Leak Check: Yes _____ No _____; 20 second cleaning @ end: Yes _____ No _____

Total Sample Time: _____ minutes _____ seconds Flow ▲P _____ Nozzle ▲P _____

Bottle final Wt: _____ g Tare Wt: _____ g Net Wt: _____ g

Pan & Sample Wt: _____ g Pan Tare Wt: _____ g Net Wt: _____ g

Total Dust: _____ grams/m^2
Fine Dust: _____ grams/m^2

Cyclone Sample #: _____

Lab Sample #: _____

FIG. 2 Sample Data Sheet for Sampling for Floor Dust

bottle can now be removed, labeled, and capped for storage and analysis. Record the dimensions of the sampled area on the data sheet.

11.1.7 If the rug area to be sampled is very dirty, or has not been cleaned frequently, care must be taken to avoid filling up the cyclone catch bottle on the first sample area. If it is

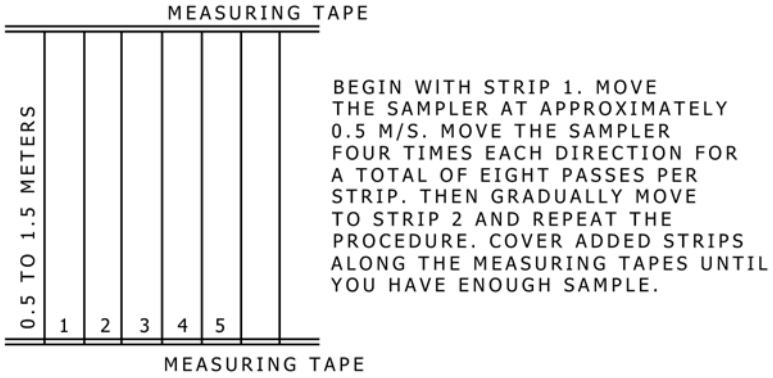


FIG. 3 Example of a Typical Sampling Procedure

suspected that this will be the case, start with a 0.25-m^2 sampling area. Then take a second and a third area as before, until the catch bottle is 75 % full.

11.1.8 Adjust the flow rate and nozzle pressure drop to values that approximate those given in Table 1. Use the same flow rate and pressure drop on multilevel and shag carpets as that used for plush carpets.

11.2 *Sampling a Bare Floor:*

11.2.1 *Pre-Test Survey*—Immediately prior to testing, complete a data form recording all requested information and sketch the area to be sampled. (See Fig. 2 for sample data form.)

11.2.2 Select a sampling area that is as large as possible and in accordance with the established protocol for your sampling campaign. This should be determined prior to testing. Divide the area into parallel areas 0.5 to 1.5 m apart.

11.2.3 A typical sampling procedure may utilize measuring tapes placed on the floor so that they are parallel to each other and on either side of the portion of floor to be sampled (Fig. 3). The measuring tapes should be between 0.5 and 1.5 m apart and extended as far as practical. They should be taped to the floor every 30 cm with masking tape.

11.2.4 Place the sampler in one corner of the sampling area. Set the height of the nozzle above the floor at approximately 1 mm (a U.S. penny under the nozzle lip will hold it at this height) and adjust the flow rate (see 11.2.7). The two factors that affect the efficiency of the sampling system are the flow rate and the pressure drop at the nozzle. The pressure drop at the nozzle is a function of the flow rate and the distance between the surface and nozzle flange.

11.2.5 Clean the wheels and nozzle lip immediately before sampling with a clean laboratory tissue. Begin sampling by moving the nozzle between the ends of the two tapes. The sampler is then moved back and forth two times on the first strip, moving the sampler at approximately 0.5 m/s. (The width of the strips are defined by the width of the sampling nozzle. For the CS_3 sampler, effective nozzle width is 13 cm. Move in a straight line between the numbers on the measuring tape. Gradually angle over to the second strip on the next pass and repeat two double passes. After sampling approximately 10 m^2, check the amount of collected material in the bottom of the catch bottle. As a rough estimate, the collection of dust to a depth of 6 mm in a 55 mm diameter catch bottle corresponds to approximately 6 to 8 g. If there is less than 6 mm of dust, sample additional areas as available. It may not be possible to obtain 6 g of dust from a clean or small bare floor.

TABLE 1 Approximate Values for Flow Rate and Nozzle Pressure Drop

Carpet Type	Flow Rate	Nozzle Pressure Drop
Plush	9.5 L/s	2.2 kPa
Level loop	7.6 L/s	2.5 kPa

11.2.6 Continue sampling in the area laid out until an adequate sample is collected. Switch off the vacuum. The catch bottle can now be removed, capped, and labeled for storage and analysis. Record the dimensions of the sampled area on the data sheet.

11.2.7 Adjust the flow rate to a flow of 9.5 L/s.

12. Sample Packaging and Transport

12.1 After collection of the sample in the catch bottle, the sample may be left in the same bottle or transferred to another container for transport to the laboratory. The procedure for sample handling is different for metals and organic chemicals. Samples for organic analysis should be maintained at 4°C to the extent possible. (Samples should not be frozen before sieving, as this could alter the particle size distribution.) Storage at ambient temperature is appropriate for samples that will be analyzed only for metals, but cooling the sample is also acceptable.

12.2 If the sample will be analyzed for pesticides or other organic chemicals, transfer the dust from the cyclone catch bottle onto the middle of a piece of aluminum foil that has been cleaned by washing with pesticide-free methanol or hexane. Fold the foil into a small package carefully, keeping the dust in the middle. Place the foil pouch in a clean glass jar. Cover the jar opening with another piece of precleaned foil and secure the lid to the jar. Seal the seam of the lid to the jar with polytetrafluoroethylene tape. Place the sample jar in an ice chest to keep it cool during transport to the laboratory. Label the jar for reference.

12.3 If the sample will be analyzed for metals, it can be transferred from the catch bottle to a new polyethylene "zipper" seal sample bag. Seal the zipper, and tape the seal with any marking tape that will adhere well to the polyethylene bag. Label the sample for reference.

TABLE 2 Sampling Efficiency Using Modified Laboratory Test Method F608[A]

Parameters	Carpet Type	
	Plush	Level Loop
Flow rate (L/s)	9.4	7.6
Delta P (kPa)[B]	2.3	2.5
Mean % of mass collected in cyclone	69.5	66.8
Standard deviation	1.2	2.8
Number of tests	3	3

[A] Carpet dust loading was 15.9 g/m^2.
[B] Pressure drop at nozzle.

12.4 Sieve the samples for 5 min in a shaker in accordance with Test Method D422, with a 100-mesh screen above the pan, to determine the weight of fine dust below 150-µm mean diameter.

12.5 Alternative methods for the storage, shipment, and preparation of samples for analysis may be required for some analytes and should be prescribed for specific sampling protocols. The FEP catch-bottle may be used for storage and shipping.

13. Sampler Cleaning

13.1 After the sample bottle is removed, open the flow control valve to maximum flow, tip the sampler back so that the nozzle is approximately 5 cm off the floor, and switch the vacuum on. Place a hand covered by a rubber glove over the bottom of the cyclone and alternate closing and opening the cyclone for 10 s to free any loose material adhering to the walls of the cyclone and tubing. It is not necessary to catch this small amount of dust, as it is usually much less than 1 % of the collected sample.

13.2 Remove the sampler to a well-ventilated cleaning area free of dust. Remove the cyclone and elbow at the top of nozzle tubing from the sampler. Use a 50-cm long by 3-cm diameter brush to clean the nozzle, and clean all related items up to and including the cyclone and catch bottle with reagent grade methanol. This wash can be analyzed at the discretion of the operator. The total amount of dust removed in the air and wet cleaning is usually much less than 1 % of the collected dust. The air and wet cleaning is performed to prevent contamination from passing from one sample to another.

14. Data Analysis

14.1 Weigh the sieved dust sample with an analytical balance accurate to 0.1 mg.

14.2 Calculate the dust weight by subtracting the weight of the pan sample from the final weight in accordance with Test Method D422.

14.3 Calculate the loading for dust per square metre (g/m^2) by dividing the final dust weight by the area sampled (expressed in m^2).

14.4 When the analysis results are received from the laboratory, it is possible to calculate the loading of lead, pesticides, or other analytes per square metre of carpet or bare floor area (µg/m^2) in the same way.

14.5 The concentration of any element or chemical associated with the dust may be determined by analysis.

15. Dust Collection Efficiency[7]

15.1 Tests for dust collection efficiency have been performed using Test Method F608 modified by passing it through a 100-mesh sieve (**1, 2**). The results are given in Table 2.

15.2 Tests performed with a fine particle filter downstream of the cyclone showed that 99 % or more of the collected test dust was retained in the cyclone catch bottle (**1, 2**).

15.3 Tests performed as in 15.2, but with test dust containing lead, showed that 99 % or more of the lead collected was retained in the cyclone catch bottle (**1, 2**).

15.4 Tests performed as in 15.2, but with test dust fortified with pesticides, showed that 97 % or more of the pesticides collected were retained in the cyclone catch bottle. The pesticides tested were chlordane, aldrin, chlorpyrifos, heptachlor, and diazinon.

15.5 Tests were conducted on conditioned carpets, as described in Test Method F608.

16. Keywords

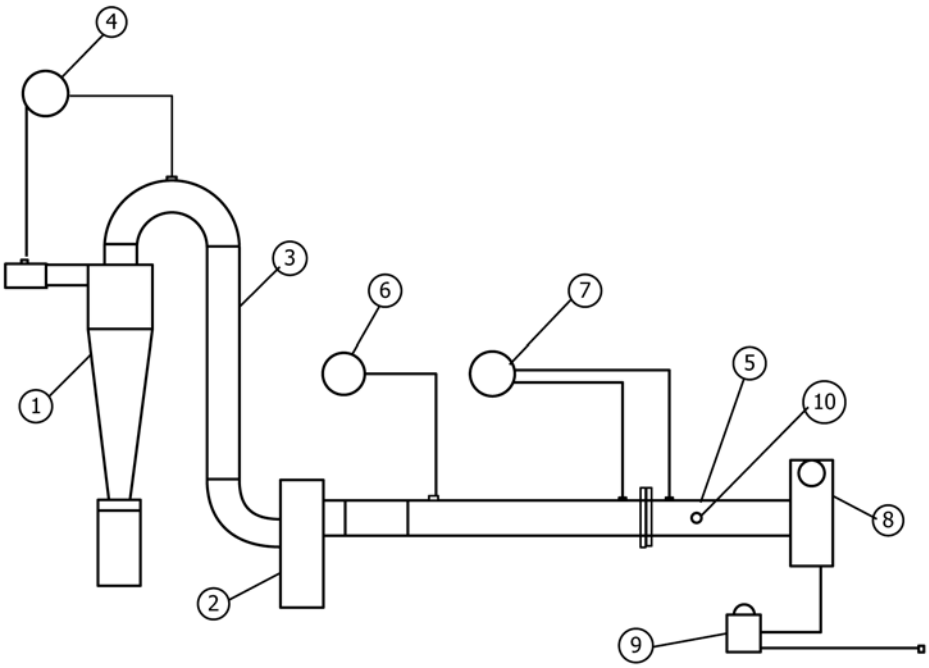
16.1 carpet; cyclone; dust; floors; metals; organic chemicals; particle size; particulate matter; vacuum

[7] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:D22-1010. Contact ASTM Customer Service at service@astm.org.

APPENDIX

(Nonmandatory Information)

X1. CALIBRATION OF CYCLONE USING A LAMINAR-FLOW ELEMENT



1. Cyclone
2. Suction/blower
3. Flow control valve
4. 0 to 2.5 kPa vacuum gauge
5. Laminar flow element
6. ±0.25 kPa vacuum gauge
7. 0 to 1 kPa vacuum gauge
8. Suction/blower
9. Variable autotransformer
10. Temperature gauge

FIG. X1.1 Calibration Using a Laminar Flow Element

X1.1 Assemble the necessary components (see Fig. X1.1).

X1.1.1 *Cyclone.*

X1.1.2 *Suction/Blower.*

X1.1.3 *Flow Control Valve,* 1 to 2.5 kPa.

X1.1.4 *Magnehelic Gauge,* 1 to 2.5 kPa.

X1.1.5 *Laminar Flow Element* (with manufacturer's certified calibration), with pressure gauges and dial thermometer.

X1.1.6 *Suction/Blower,* with power transformer; leak check the system by plugging the inlet to the cyclone and observing the pressure gauge.

X1.1.7 Activate Blowers 2 and 8.

X1.1.8 Open the flow control valve on Flow Control Valve 3 so that 2.0 kPa registers on Pressure Gauge 4. Then adjust Variable Autotransformer 9 so that 0.0 kPa registers on Pressure Gauge 6. Some adjusting of the flow control valve will be necessary.

X1.1.9 Check Pressure Gauge 7 for the gas flow reading and record the flow.

X1.1.10 Adjust the flow through the cyclone to 2.5 kPa, and repeat the procedure. This action should provide a gas flow rate through the cyclone. This should be between 7.1 and 8.5 L/s.

REFERENCES

(1) Roberts, J. W., Budd, W. T., Ruby, M. G., Stamper, V. R., Camann, D. E., Fortmann, R. C., Sheldon, L. S., and Lewis, R. G., "A Small High Volume Surface Sampler HVS3 for Pesticides, and Other Toxic Substances in House Dust," *Paper No. 91-150.2*, 84th Annual Meeting, Air & Waste Management Association, Vancouver, British Columbia, June 16-21, 1991.

(2) Roberts, J. W., and Ruby, M. G., "Development of a High Volume Surface Sampler for Pesticides," *U.S. Environmental Protection Agency Report No. EPA 600/4-88/036*, Research Triangle Park, NC, January 1989.

(3) Roberts, J. W., Han, W., and Ruby, M. G., "Evaluation of Dust Samplers for Bare Floors and Upholstery," Battelle Subcontract No. 46534(g21733808)-00 03EQ, EPA Prime Contract No. 68-00-0007, U.S. EPA, AREAL, Office of Research and Development, 1993.

(4) Stamper, V. R., Roberts, J. W., and Ruby, M. G., "Development of a High Volume Small Surface Sampler for Pesticide and Toxics in House Dust," *Research Triangle Institute Report No. RTI/171-01/02F*, Research Triangle Park, NC, June 1990. Included in supporting data, which are on file at ASTM Headquarters. Request Research Report RR:D22-1010.

(5) Lewis, R. G., Fortune, C. R., Willis, R. D., Camann, D. E., and Antley, J. T., "Distribution of Pesticides and Polycyclic Aromatic Hydrocarbons in House Dust as a Function of Particle Size," *Environmental Health Perspectives*, Vol 107, 1999, pp. 721–726.

(6) *Reagent Chemicals, American Chemical Society Specifications*, American Chemical Society, Washington, DC. For suggestions on the testing of reagents not listed by the American Chemical Society, see *Analar Standards for Laboratory Chemicals*, BDH Ltd., Poole, Dorset, U.K., and the *United States Pharmacopeia and National Formulary*, U.S. Pharmacopeial Convention, Inc. (USPC), Rockville, MD.

Designation: D7144 – 21

Standard Practice for Collection of Surface Dust by Micro-vacuum Sampling for Subsequent Determination of Metals and Metalloids[1]

This standard is issued under the fixed designation D7144; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This practice covers the micro-vacuum collection of surface dust for subsequent determination of metals and metalloids. The primary intended application is for sampling from soft, rough, or porous surfaces.

1.2 Micro-vacuum sampling is carried out using a collection nozzle attached to a filter holder (sampling cassette) that is connected to an air sampling pump.

1.3 This practice allows for the subsequent determination of metals and metalloids on a loading basis (mass of element(s) per unit area sampled), or on a concentration basis (mass of element(s) per unit mass of sample collected), or both.

1.4 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

1.5 *Limitations*—Due to a number of physical factors inherent in the micro-vacuum sampling method, analytical results for vacuum dust samples are not likely to reflect the total dust contained within the sampling area prior to sample collection. Indeed, dust collection will generally be biased towards smaller, less dense dust particles. Nevertheless, the use of this standard practice will generate data that are consistent and comparable between operators performing micro-vacuum collection at a variety of sampling locations and sites.[2]

1.6 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety, health, and environmental practices and determine the applicability of regulatory limitations prior to use.*

1.7 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

2. Referenced Documents

2.1 *ASTM Standards:*[3]

D1356 Terminology Relating to Sampling and Analysis of Atmospheres

D3195 Practice for Rotameter Calibration

D4840 Guide for Sample Chain-of-Custody Procedures

D5438 Practice for Collection of Floor Dust for Chemical Analysis

D5337 Practice for Flow Rate Adjustment of Personal Sampling Pumps

D6966 Practice for Collection of Settled Dust Samples Using Wipe Sampling Methods for Subsequent Determination of Metals

D7035 Test Method for Determination of Metals and Metalloids in Airborne Particulate Matter by Inductively Coupled Plasma Atomic Emission Spectrometry (ICP-AES)

3. Terminology

3.1 *Definitions*—For definitions of terms relating to sampling and analysis of dust not given here, refer to Terminology D1356.

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *air sampling pump, n*—a portable pump that is used to draw air through a filter holder/collection nozzle assembly for micro-vacuum collection of surface dust. An example would include a personal sampling pump.

3.2.2 *batch, n*—a group of field or quality control samples, or both, that are collected together in a similar environment and are processed together using the same reagents and equipment.

3.2.3 *collection nozzle, n*—a piece of flexible plastic tubing cut at a 45° angle at the inlet end, and connected at the outlet end to the inlet orifice of a filter holder (sampling cassette).

[1] This practice is under the jurisdiction of ASTM Committee D22 on Air Quality and is the direct responsibility of Subcommittee D22.04 on Workplace Air Quality.

Current edition approved May 1, 2021. Published May 2021. Originally approved in 2005. Last previous edition approved in 2016 as D7144 – 05a (2016). DOI: 10.1520/D7144-21.

[2] Reynolds, S. J., et al.,"Laboratory Comparison of Vacuum, OSHA, and HUD Sampling Methods for Lead in Household Dust," *American Industrial Hygiene Association Journal*, Vol 58, 1997, pp. 439–446.

[3] For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

3.2.4 *field blank, n*—a sample that is handled in exactly the same way that field samples are collected, except that no air is drawn through it.

3.2.5 *filter holder, n*—an apparatus that supports and contains the filter medium upon which dust is collected. It is also often referred to as a sampling cassette.

3.2.6 *internal capsule, n*—a device inserted into a filter holder (sampling cassette) that allows complete capture of contaminant within its envelope and prevents deposition of collected material on the internal walls of the sampling cassette. Use of an internal capsule is necessary for gravimetric analysis purposes.

3.2.6.1 *Discussion*—Such capsules are commercially available.

3.2.7 *sampling device (assembly), n*—for micro-vacuum sampling, an apparatus consisting of the collection nozzle, filter holder (containing internal capsule, if necessary), and air sampling pump, used to collect surface dust. The collection nozzle is attached to the inlet end of the filter holder. The filter holder houses the filter, through which air is drawn by using the air sampling pump. The filter holder is attached to the pump by flexible tubing.

3.2.8 *surface dust, n*—particulate matter on a given surface which has been transported to its present location by various means, such as settling through the air or tracking from other sources.

4. Summary of Practice

4.1 Samples of surface dust are collected from selected sampling locations into individual filter holders by using a micro-vacuum collection technique that employs a personal sampling pump.[4] The sample is then processed for transport and subsequent laboratory analysis for determination of metals and metalloids content.

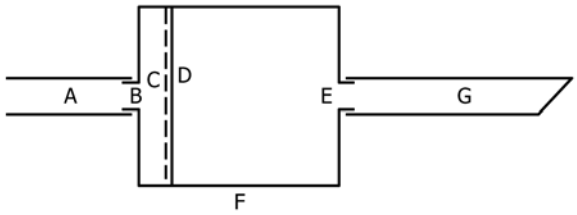
4.2 The collected sample may include particles which adhere to the internal walls of the filter holder. This material should be rinsed or wiped off and added to the sample meant for subsequent chemical analysis. However, this material cannot be included in gravimetric determination unless an internal capsule that can be accurately weighed is used during sample collection.

5. Significance and Use

5.1 Human exposure to toxic metals and metalloids present in surface dust can result from dermal contact with or ingestion of contaminated dust. Also, inhalation exposure can result from disturbing dust particles from contaminated surfaces. Thus, standardized methods for the collection and analysis of metals and metalloids in surface dust samples are needed in order to evaluate the potential for human exposure to toxic elements.

5.2 This practice involves the use of sampling equipment to collect surface dust samples that may contain toxic metals and metalloids, and is intended for use by qualified technical professionals.

[4] Ashley, K., et al., "Evaluation of a Standardized Micro-vacuum Sampling Method for Collection of Surface Dust," *Journal of Occupational and Environmental Hygiene*, Vol 4, 2007, pp. 215–223.



A: Flexible tubing connecting the filter holder to the sampling pump (not shown);
B: Outlet of filter holder;
C: Back-up pad/support;
D: Filter;
E: Inlet of filter holder;
F: Housing of filter holder; and
G: Flexible tubing collection nozzle.

FIG. 1 Schematic of Sampling Assembly for Micro-Vacuum Surface Dust Sampling

5.3 This practice allows for the subsequent determination of collected elemental concentrations on an area (loading) or mass concentration basis, or both.

5.4 Because particle losses can occur due to collection of dust onto the inner surfaces of the nozzle, the length of the collection nozzle is specified in order that such losses are comparable from one sample to another.

5.5 This practice is suitable for the collection of surface dust samples from, for example: *(a)* soft, porous surfaces such as carpet or upholstery; *(b)* hard, rough surfaces such as concrete or roughened wood; *(c)* confined areas that cannot be easily sampled by other means (such as wipe sampling as described in Practice D6966). A companion sampling technique that may be used for collection of surface dust from hard, smooth surfaces is wipe sampling (Practice D6966). A companion vacuum sampling technique that may be used for sampling carpets is described in Practice D5438.

5.6 Procedures presented in this practice are intended to provide a standardized method for dust collection from surfaces that cannot be reliably sampled using wipe collection methods (for example, Practice D6966). Additionally, the procedure described uses equipment that is readily available and in common use for other environmental and occupational hygiene sampling applications.

5.7 The entire contents of the filter holder, that is, the filter plus collected dust, is targeted for subsequent analysis for metals and metalloids content. An internal capsule is used if gravimetric analysis is necessary.

6. Apparatus

6.1 *Dust Sampling Equipment*—The sampling assembly (see Fig. 1) for the micro-vacuum collection of surface dust samples has the following components:

6.1.1 *Filters,* of a diameter suitable for use with the filter holders, and with a collection efficiency of not less than 99.5 % for particles with a diffusion diameter of 0.3 µm, and with a very low metal content (typically less than 0.1 µg of each metal of interest per filter) (see Test Method D7035).

6.1.1.1 Weight-stable filters shall be used if it is desired to determine the mass of collected dust. If the filters are to be

weighed in order to determine the mass of dust collected, it is important that they be resistant to moisture retention, so that blank weight changes that can occur as a result of changes in temperature and humidity are as low and repeatable as possible. Also, filters selected for weight stability should not be excessively brittle, since this can introduce weighing errors due to loss of filter material.

6.1.2 *Filter holders,* for 25-mm or 37-mm diameter filters.

6.1.3 *Internal Capsules, For Gravimetric Analysis*—If it is desired to determine the mass of collected dust, internal capsules shall be weighed to the nearest 0.1 mg. If pre-weighed internal capsules and filters are used, it will be necessary to tare the internal capsules, plus backup pads, prior to use. Procedures for accurate weighing of internal capsules are described in detail elsewhere.

6.1.4 *Back-up Pads, Cellulosic; or Metallic Screen Back-up Support*—If pre-weighed filters are used, it is not necessary to know the mass of each back-up pad. However, if pre-weighed internal capsules and pre-weighed filters are used, it will be necessary to know the influence of the mass of each back-up pad on the overall mass of the entire sampling assembly (to the nearest 0.1 mg).

6.1.5 *Collection nozzle,* consisting of a piece of flexible polyvinyl chloride (PVC) tubing of length 5.5 cm ± 0.5 cm and 0.60 cm ± 0.005 cm inside diameter, cut at a 45° (± 1°) angle at the inlet end.

6.1.6 *Tubing, flexible,* inside diameter 0.60 cm ± 0.005 cm for connecting the sampling device to the air sampling pump (maximum length 1 m).

6.1.7 *Air sampling pump, portable,* capable of sampling at a flow rate of 2.5 L/min ± 0.5 L/min. The pump flow rate shall be adjusted and set with a representative sampling assembly in line so that the volume of air sampled can be measured to an accuracy of ±5 % or better.

6.1.8 *Calibration device,* for air sampling pumps; soap bubble meter or equivalent, as specified in Practice D3195.

6.1.9 *Rotameter, calibrated,* as specified in Practice D3195.

6.1.10 *Sampling templates,* minimum dimensions 10 cm by 10 cm, maximum dimensions 30 cm by 30 cm; reusable metallic or plastic; or disposable plastic or cardboard.

6.1.11 *Gloves, powderless, latex-free,* for handling of filters, back-up pads/supports, samplers, tubing, collection nozzles, and other sample collection components.

6.1.12 *Tape, adhesive,* for immobilization of sampling templates; and for delineation of sampling areas where the use of templates is impractical.

6.1.13 *Tape measure or ruler, metric,* for measurement of sampling areas when the use of templates is impractical, and for measurement of tubing, collection nozzles, and so forth.

6.1.14 *Tweezers,* plastic or plastic-tipped metallic, for handling of filters.

6.1.15 *Sealable plastic bags, or boxes, or other airtight containers, or a combination of the three,* for transporting collected samples.

7. Procedure

7.1 *Assembly of Micro-vacuum Sampling Device*—The following shall be carried out in an uncontaminated area while wearing clean gloves:

7.1.1 Assemble the filter in the filter holder, with the filter supported on a back-up pad or metallic screen. To prevent contamination, the filter should be handled only with tweezers.

7.1.2 If pre-weighed filters and internal capsules are used, record their masses to the nearest 0.1 mg using established acceptance criteria.

NOTE 1—If desired, pre-loaded filter holders and capsules with pre-weighed filters and internal capsules may be purchased, already assembled, from the manufacturer.

7.1.3 Close and seal the sampling device to prevent leakage of air around the filter or into/out of the sampler. Label the sampler with a unique sample identifier.

7.1.4 Attach the outlet end of the collection nozzle to the inlet end of the filter holder, and secure tightly.

7.2 *Flow Adjustment Sampling Train for Micro-vacuum Sampling:*

7.2.1 Ensure that sampling pumps, if battery-powered, are sufficiently charged prior to use.

7.2.2 Using a calibrated and traceable flow measurement device (for example, a calibrated rotameter or soap bubble meter; see Practice D3195), set the flow rate of the air sampling pump, with a sampling assembly in the line, to 2.5 ± 0.1 L/min.

NOTE 2—While soap bubble meters are useful for applications in the laboratory and in the field, calibrated and traceable rotameters are especially convenient for on-site flow rate checks.

7.2.3 The flow of sampling pumps shall be checked prior to and following use in accordance with Practices D3195 and D5337.

7.3 *Preparation for Sampling*—The following shall be carried out while wearing clean gloves:

7.3.1 Attach the sample collection device (that is, the assembly with the collection nozzle attached to filter holder) to the flow adjusted sampling pump by means of a piece of flexible tubing.

7.3.2 Using indelible ink, uniquely label the sampling cassette of each sample collection assembly.

7.3.3 If possible, demarcate the area of the surface to be sampled (for example, 10 cm by 10 cm) using a template, and secure the outside edges of the template with tape. If it is not practical to use a template, carefully measure the area (in cm by cm) to be sampled using a tape measure or ruler, and delineate the sampling area with tape.

NOTE 3—Areas where template-assisted sampling may not be possible include, for example, locations where: *(a)* the surface to be sampled is confined or otherwise not easily accessible; *(b)* the surface to be sampled is smaller (in at least one dimension) than the template; *(c)* the surface to be sampled is curved (not flat); and so forth.

7.3.4 Activate the sampling pump and allow for a suitable warm-up period. To ensure that the specified flow rate is obtained, sufficient pump warm-up shall be determined by using a flow check device (that is, soap bubble meter or rotameter).

NOTE 4—Warm-up times may differ for sampling pumps of different manufacture and age. A 5-min warm-up period is usually sufficient.

7.4 *Sample Collection*—The following shall be carried out while wearing clean gloves:

7.4.1 Hold the collection nozzle immediately adjacent to the surface being sampled, but avoid strongly contacting the surface with the nozzle. The inlet of the nozzle (cut at a 45° (± 1°) angle) should be approximately parallel to the surface being sampled. Endeavor to keep the nozzle lightly in touch with the surface; do not press the nozzle hard against the surface being sampled.

7.4.2 Move the collection nozzle from one side of the sampling area to the other. The rate of movement of the nozzle across the surface shall be no more than 1 s/10 cm. Repeat this sweeping motion in the same direction until the entire sampling area has been "vacuumed" with the collection nozzle. If the nozzle becomes clogged during sampling, dislodge the obstruction using a clean knife or other suitable tool.

7.4.3 Repeat the procedure described in 7.4.2 in a direction 90º from the initial sampling direction. Be sure to cover the entire sampling area.

7.4.4 Continue sample collection (use various sampling directions, if desired) until a total of 1 min sampling time per 100 cm^2 area is reached. Larger sampling areas will require longer sampling times.

7.4.5 Avoid excessive overloading of the filter; this problem can be identified by a >10 % drop in the measured flow rate. If overloading becomes evident, reduce the sampling area; alternatively, use additional sample collection assemblies to sample the defined area. Record this information.

7.4.6 Use a separate, clean collection nozzle and filter holder (with clean filter) for each micro-vacuum sample. Use of a separate collection nozzle for each sample is essential for prevention of cross-contamination.

7.4.7 Prepare field blanks at the same time that sampling is carried out; these shall represent no less than 5 % of the total number of samples, or at least one per batch minimum. Field blanks shall be handled in the same fashion as field samples, but no air is drawn through the filters.

NOTE 5—Some laboratories also require that media blanks are submitted along with field blanks and samples.

7.4.8 Following collection of a surface dust sample, disconnect the sampling assembly from the sampling pump and collection nozzle, and then turn off the sampling pump. When disconnecting and capping the filter holder, hold it upright to ensure that no loose dust is lost from the sampling assembly. After removing the connecting tubes, cap the inlet and outlet ends of the filter holder with plugs.

7.4.9 Place the filter holder in a suitable container for transport, such as a sealable plastic bag or box.

7.5 *Sample Transport*—Samples shall be transported to the laboratory in sample containers. This shall be done in such a manner that the filter holders or internal capsules containing collected dust vacuum samples are neither disturbed nor contaminated. Sample transport shall be carried out and documented so that a chain of custody is established (in accordance with Guide D4840).

7.6 *Records*—Record the following information in a bound notebook (with numbered pages) or on data sampling forms, or both. Record pertinent sampling data for each sample, for example:

7.6.1 Sample location,
7.6.2 Sampling site,
7.6.3 Date and time,
7.6.4 Sampling flow rates,
7.6.5 Calibration certificates,
7.6.6 Pre-sampling filter/sampler mass (if pre-weighed filters and internal capsules are used),
7.6.7 Surface type sampled,
7.6.8 Filter type,
7.6.9 Personal identifier,
7.6.10 Pump type and identifier,
7.6.11 Sampling rate,
7.6.12 Air volume sampled, and
7.6.13 Surface area sampled.

8. Report

8.1 Parameters to be reported include items and information such as:

8.1.1 Flow rate used,
8.1.2 Number of samples and field blanks,
8.1.3 Air sampling pumps used,
8.1.4 Pump settings,
8.1.5 Calibration data and equipment used for calibration,
8.1.6 Sampling areas,
8.1.7 Type/description of collection nozzle used,
8.1.8 Date and time,
8.1.9 Sampling site and locations,
8.1.10 Personal identifier(s) of individual(s) who carried out sampling,
8.1.11 Sample identifiers,
8.1.12 Pertinent information (that is, masses) on pre-weighed filters/internal capsules, if used, and
8.1.13 Type of sampler/filter used.

9. Keywords

9.1 dust; metalloids; metals; surfaces; vacuum sampling

Attachment B
Pace SOPs
Index

SOP Number	SOP Title	# Pages
ENV-SOP-GBAY-0164	Soil Sieve	13
ENV-SOP-MIN4-0043	Metals Analysis by ICP/MS - Method 6020 and 200.8	24
ENV-SOP-MIN4-0054	Mercury in Liquid and Solid/Semi-Solid Waste by 7470A, 7471, 7471B, and 245.1	20
ENV-SOP-MIN4-0056	Metals Preparation of Solid Samples for Analysis by ICP and ICP-MS by 3050B	15
ENV-SOP-MIN4-0059	Preparation of Attic Dust and Filter Cartridge Samples by 3050B for Analysis by ICPMS	8

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ENVIRONMENTAL SCIENCES

Document Information

Document Number: ENV-SOP-GBAY-0164 **Revision:** 00

Document Title: Soil Sieve

Department(s): Wet Chemistry

Date Information

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All Dates and Times are listed in: Central Time Zone

Signature Manifest

Document Number: ENV-SOP-GBAY-0164 **Revision:** 00
Title: Soil Sieve

All dates and times are in Central Time Zone.

ENV-SOP-GBAY-0164-Rev.00 Soil Sieve

QM Approval

Name/Signature	Title	Date	Meaning/Reason
Elizabeth Turner (007857)	Manager - Quality Program	09 Apr 2021, 02:09:58 PM	Approved

Management Approval

Name/Signature	Title	Date	Meaning/Reason
Chad Rusch (007163)	General Manager 2	08 Apr 2021, 09:50:26 AM	Approved

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TEST METHOD STANDARD OPERATING PROCEDURE
TITLE: Soil Sieve
TEST METHOD ENV-SOP-GBAY-0164
ISSUER: Pace ENV – Green Bay Quality – GBAY

1.0 Scope And Application

This standard operating procedure (SOP) describes the laboratory procedure for drying and sieving soil samples to obtain a portion of soil for analysis.

1.1 Target Analyte List and Limits of Quantitation (LOQ) - Not applicable to this SOP.

1.2 Applicable Matrices: Soils and sediments.

1.3 Personnel: The policies and procedures contained in this SOP are applicable to all personnel involved in the analytical method or non-analytical process.

2.0 Summary of Method

A sample is homogenized and air dried. After air-drying, the sample is then sieved through a selected sieve size. The portion that passes the sieve is then ready for analysis.

3.0 Interferences

Not applicable to this SOP.

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.

Requirements for container type, preservation, and field quality control (QC) for the common list of test methods offered by Pace are included in the laboratory's quality manual.

General Requirements

Matrix	Routine Container	Minimum Sample Amount[1]	Preservation	Holding Time
Hg Samples	Ziplock Bag	2 cups	Thermal: ≤ 6°C Chemical: None	28 Days
All Other Metals	Ziplock Bag	2 cups	Thermal: ≤ 6°C Chemical: None	6 Months
Organic Parameters	16 oz glass jar	2 cups	Thermal: ≤ 6°C Chemical: None	VOA 14 Days SVOA 7 Days

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

Thermal preservation is checked and recorded on receipt in the laboratory in accordance with laboratory SOP ENV-SOP-GBAY-0006 *Sample Management* (current revision or replacement). Chemical preservation is checked and recorded at time of receipt or prior to sample preparation.

After receipt, samples are stored at ambient temperature until sample preparation. Prepared samples (extracts, digestates, distillates, other) are stored at ambient temperature 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.

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TEST METHOD STANDARD OPERATING PROCEDURE
TITLE: Soil Sieve
TEST METHOD ENV-SOP-GBAY-0164
ISSUER: Pace ENV – Green Bay Quality – GBAY

7.0 EQUIPMENT AND SUPPLIES

7.1 Equipment

Equipment*	Manufacturer / Vendor*	Catalog #*
Sieve Shaker	RO-TAP®	RX-29
Sieve Shaker	Gilson	SS-15
Sieve Shaker	Endecotts	Minor 200
Sieve Shaker	Endecotts	Octagon 200
Sieve	Gilson or equivalent	Stainless steel, #10, #60, or other as needed
Sieve catch pans and lids	Gilson or equivalent	Stainless steel
Bakers' racks	Restaurant Supply	To hold 18" x 26" trays
Drying fan	Various	Local Store
Mortar ceramic/porcelain	Cole-Parmer	60322
Pestle ceramic/porcelain	Cole-Parmer	60323

*Or Equivalent

7.2 Supplies

Supplies	Vendor	Model/Version
Aluminum Foil Cake Pan	Durable Packaging / Webstaurant	612604245
8x8 Ziploc Bags	Fisher Scientific	23700218
12x12 Ziploc Bags	Uline	S-14416
Freezer Paper	Fisher Scientific or equivalent	50-200-5215
Wooden Rolling Pin	Restaurant Supply	Local Store
Rubber Mallet	Various	Local Store
Scissors	Various	Local Store

*Or Equivalent

8.0 REAGENTS AND STANDARDS

Not applicable to this SOP.

9.0 PROCEDURE

9.1 Balance calibration must be verified daily prior to use. Refer to SOP ENV-SOP-GBAY-0115 *Support Equipment* (current revision or replacement).

9.2 For any USDA marked samples, refer to SOP ENV-SOP-GBAY-0121 *Regulated Soil Handling* (current revision or replacement). Containers will be labeled with a pink Regulated Soil sticker.

9.3 Pulling Samples

9.3.1 Batch the samples in the LIMS.

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TEST METHOD STANDARD OPERATING PROCEDURE
TITLE: Soil Sieve
TEST METHOD ENV-SOP-GBAY-0164
ISSUER: Pace ENV – Green Bay Quality – GBAY

9.3.2 Pull the samples from either the soil Walk-In Cooler or from the ambient storage area in the Physical Testing Lab and organize them in the order to be processed. Their location will be dependent on the analytical work, if any, that will be done after the sieving.

9.4 Create a new Dry Sieve Worksheet File.

9.4.1 Use the Dry Sieve Template in the Dry Sieve folder, and make sure to "Save As", using the Horizon Batch Number (HBN).

9.4.2 Fill in the drying information for each sample on the Worksheet.

9.5 Air Dry Samples

9.5.1 Wearing gloves, line a tray with freezer paper wax side down. Fold the sides of freezer paper up about 1- 1 1/2" on each side to form a "boat".

9.5.2 Label the freezer paper with the sample number. Place the entire sample on the freezer paper. Multiple trays may be used for drying if a large sample volume was received.

9.5.3 Entire sample does not need to be dried if excess volume was received. Sample must be homogenized before splitting. Return undried portion to original container.

9.5.4 Weigh and document remaining sample mass. Some projects may require this to be labeled as "Archive".

9.5.5 Spread the soil evenly. Break up all clumps into about 1/2" or less size pieces. This will speed the drying process and ease the disaggregation process prior to sieving. Continue this process for all samples in the set. Change gloves between each sample.

9.5.6 In the drying logbook record the sample numbers, date, time, temperature, and humidity when the samples are placed in the drying cabinet. Place the entire set in a drying cabinet to air dry overnight. Longer drying may be required for wetter samples.

9.6 Soil Disaggregation

9.6.1 After the samples are dried remove them from the drying cabinets. Record the date, time, temperature, and humidity in the drying logbook.

9.6.2 Place a tray on the counter. Pick any rocks, twigs or other foreign matter and set to the side of the freezer paper boat.

9.6.3 Disaggregate the soil. Disaggregation is the process of loosening the clumped soil. It is not meant to crush or reduce the natural particle size of the soil. Place a sheet of paper, wax side up, over the sample. Using a rolling pin, roll over the dried soil for 1-2 minutes. A rubber mallet or pestle may be used to disaggregate soil clumps. Take care that the sample remains on the freezer paper. If sample is hard clay, a porcelain pestle may be used to break up chunks, being careful not to crush rocks.

9.7 Soil Sieve Procedure Using #10 Sieve

9.7.1 Place sieve on catch pan or clean freezer paper, wax side down. Pour sample into #10 sieve and sift. Gently rub the sample remaining on the sieve to break up clumps. When no more sample passes through sieve, dump all remaining sample on top of sieve onto

Pace Analytical®

TEST METHOD STANDARD OPERATING PROCEDURE
TITLE: Soil Sieve
TEST METHOD ENV-SOP-GBAY-0164
ISSUER: Pace ENV – Green Bay Quality – GBAY

a separate sheet of freezer paper. If large clumps are still present, repeat disaggregation and sieve until no clumps remain.

9.7.2 The sample portion remaining in the #10 sieve is then weighed, documented, and bagged with the sample number and a "Coarse Fragments" label on it.

9.7.3 Weigh, document, and place all the sample passing through the #10 sieve into a labeled Ziploc bag with the sample number and a "Fines" label on it. Add any organic matter that had been removed previously. This Organic matter may need to be cut up into smaller pieces using clean scissors.

9.7.4 Change gloves between samples.

9.8 Soil Sieving Procedure using sieves other than #10

9.8.1 Determine the sieve sizes and process to be used to meet project specifications.

9.8.1.1 Check with the project manager or lab manager for project specifications.

9.8.1.2 If multiple sieve portions are to be obtained, stack the set of sieves in the with the largest size openings on top to the smallest on the bottom, with a catch pan at the base.

9.8.1.3 If sieve sizes smaller than a #10 sieve are being used, the #10 sieve can be used to not plug up the smaller sieve. Anything retained by the #10 sieve must be considered part of the biggest sieve portion.

9.8.2 Pour the dried and disaggregated soil onto top sieve.

9.8.3 Record the sample number on the side of the catch pan. An abbreviated number may be used such as 407-1.

9.8.4 All dried contents are poured onto the sieve including the rocks and foreign matter that had been set to the side. The organic foreign matter may need to be cut up into smaller pieces using clean scissors.

9.8.5 Place the set of sieves on a mechanical shaker. Tighten the mechanical shaker adjustments so that the sieves fit tightly and securely in the mechanical shaker. Set the timer for 10 minutes and begin the sieve shaking.

9.8.6 After 10 minutes remove the sieves from the mechanical shaker.

9.8.7 Weigh, document, and place all the sample contents in the catch pan into a labeled Ziploc bag with the sample number and a "Fines" label on it.

9.8.8 Great care should be taken in matching the sample number written on the catch pans to the sample numbers on the labeled container.

9.8.9 Certain projects may require that the portion of sample above the sieve be retained. If this is required pour the sample remaining on top of the sieve(s) into a second bag, label with the lab number and mark "Coarse Fragments". Zero the balance with the same bags used, then weigh and document the mass of this portion.

9.8.10 Record the sieve date, analyst, shaker ID, and sieve size used on the Soil Sieve Prep Log. Note if coarse fragments were retained.

Pace Analytical®

TEST METHOD STANDARD OPERATING PROCEDURE
TITLE: Soil Sieve
TEST METHOD ENV-SOP-GBAY-0164
ISSUER: Pace ENV – Green Bay Quality – GBAY

9.9 Pulverization - Some projects or methods may require that the sieved sample be further pulverized prior to analysis. The sample may be pulverized with a motor and pestle or other method.

9.10 Cleaning Sieves – the sieves must be washed and dried between each use.

9.10.1 Place the sieves in the sink and scrub with a brush or green scrubbie and running hot water to remove any soil particles embedded in the mesh. Rinse well with tap water then rinse with deionized water. Soap is not used as it is very difficult to rinse from the sieves.

9.10.2 Place the sieves and catch pans in an oven to dry. Alternatively allow to air dry overnight on the counter.

10.0 Data Analysis And Calculations

Not applicable to this SOP.

11.0 Quality Control and Method Performance

11.1 Quality Control - Not applicable to this SOP.

11.2 Instrument QC - Not applicable to this SOP.

11.3 Method Performance

11.3.1 Method Validation

11.3.1.1 Detection Limits - Not applicable to this SOP.

12.0 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-GBAY-0094 *Training and Employee Orientation* (current revision or replacement) for more information.

13.0 Data Review And Corrective Action

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.

Pace Analytical®

TEST METHOD STANDARD OPERATING PROCEDURE
TITLE: Soil Sieve
TEST METHOD ENV-SOP-GBAY-0164
ISSUER: Pace ENV – Green Bay Quality – GBAY

The review steps and checks that occur as employees 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-GBAY-0120 *Data Review and Final Report Processes* (current revision or replacement) for specific instructions and requirements for each step of the data review process.

13.1 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.

There is no QC performed with this analysis.

Pace Analytical®

TEST METHOD STANDARD OPERATING PROCEDURE
TITLE: Soil Sieve
TEST METHOD ENV-SOP-GBAY-0164
ISSUER: Pace ENV – Green Bay Quality – GBAY

14.0 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 cycletainers 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.

15.0 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.

16.0 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.

17.0 Attachments

Attachment I: Sieve prep log (Example)

Attachment II: Dry Sieve Flow Chart

18.0 References

18.1 Pace Quality Assurance Manual - most current version.

18.2 The NELAC Institute (TNI); Volume 1, "Management and Technical Requirement for Laboratories Performing Environmental Analysis" - most current version.

Pace Analytical®

TEST METHOD STANDARD OPERATING PROCEDURE
TITLE: Soil Sieve
TEST METHOD ENV-SOP-GBAY-0164
ISSUER: Pace ENV – Green Bay Quality – GBAY

19.0 REVISION HISTORY

This Version: ENV-SOP-GBAY-0164-Rev.00

Section	Description of Change
All	First Issue of SOP.

This document supersedes the following document(s):

Document Number	Title	Version

Pace Analytical®

TEST METHOD STANDARD OPERATING PROCEDURE
TITLE: Soil Sieve
TEST METHOD ENV-SOP-GBAY-0164
ISSUER: Pace ENV – Green Bay Quality – GBAY

Attachment I: Sieve Prep Log

Work Order		Date/Time In		Humidity In (%)		Temp In (°C)		Reviewed by	
Batch		Date/Time Out		Humidity Out (%)		Temp Out (°C)			

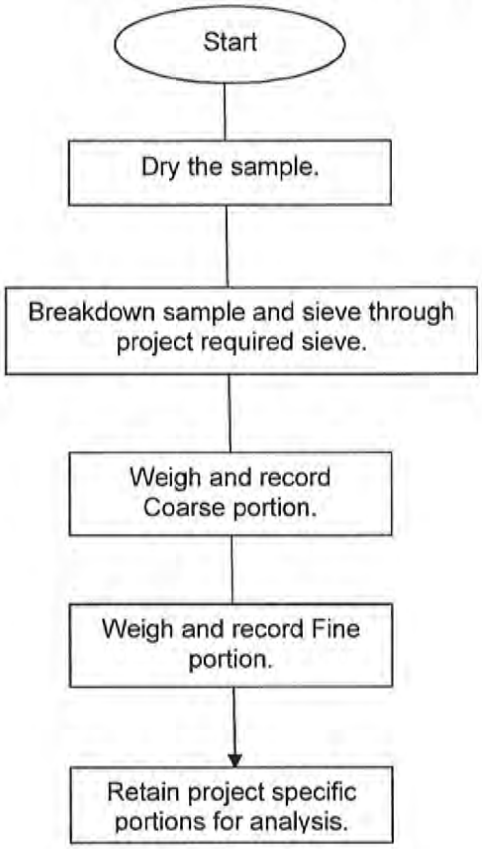
Samples		Sieve Date	Shaker ID	Analyst	Archive Weight (g) Balance ID: 40BALW	Analyst	Weight of >60 Mesh (g) Balance ID: 40BALX	Analyst	Weight of <60 Mesh (g) Balance ID: 40BALX	Analyst
	-001		40SKR3							
	-002		40SKR4							
	-003		40SKR5							
	-004		40SKR3							
	-005		40SKR4							
	-006		40SKR5							
	-007		40SKR4							
	-008		40SKR6							
	-009		40SKR3							
	-010		40SKR4							
	-011		40SKR6							
	-012		40SKR7							
	-013		40SKR8							
	-014		40SKR3							
	-015		40SKR4							
	-016		40SKR5							
	-017		40SKR6							
	-018		40SKR7							
	-019		40SKR8							
	-020		40SKR4							

A similar version including the same information may be used.

Pace Analytical®

TEST METHOD STANDARD OPERATING PROCEDURE
TITLE: Soil Sieve
TEST METHOD ENV-SOP-GBAY-0164
ISSUER: Pace ENV – Green Bay Quality – GBAY

Attachment II: Dry Sieve Flow Chart



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ENVIRONMENTAL SCIENCES

Document Information

Document Number: ENV-SOP-MIN4-0043 **Revision:** 04

Document Title: Metals Analysis by ICP/MS - Method 6020 and 200.8

Department(s): Metals

Date Information

Effective Date: 22 Feb 2021

Notes

Document Notes:

All Dates and Times are listed in: Central Time Zone

Signature Manifest

Document Number: ENV-SOP-MIN4-0043 **Revision:** 04

Title: Metals Analysis by ICP/MS - Method 6020 and 200.8

All dates and times are in Central Time Zone.

ENV-SOP-MIN4-0043

QM Approval

Name/Signature	Title	Date	Meaning/Reason
Janielle Ward (007319)	Manager - Quality	22 Feb 2021, 11:06:56 AM	Approved

Management Approval

Name/Signature	Title	Date	Meaning/Reason
Adam Haugerud (005828)	General Manager 2	17 Feb 2021, 04:18:39 PM	Approved
Andrew Mickelson (009792)	Manager	18 Feb 2021, 08:49:25 AM	Approved
Krista Carlson (004514)	Project Manager 1	18 Feb 2021, 10:54:23 AM	Approved

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 – Mass Spectrometry (ICP-MS).

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 the lowest point of the calibration curve used for each target analyte.

1.2 Applicable Matrices

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

Dissolved elements are determined after suitable filtration and acid preservation. In order to reduce potential interferences, dissolved solids should not exceed 0.2 % (w/v).

For the determination of total recoverable analytes in aqueous samples containing particulate and suspended solids a digestion step is required prior to analysis.

2.0 SUMMARY OF METHOD

Prior to analysis, samples must be solubilized or digested using appropriate sample preparation methods. For the total recoverable determination of analytes in drinking water by 200.8 where sample turbidity is < 1 NTU, the sample is made ready for analysis by the appropriate addition of nitric acid, mixed, and allowed to equilibrate for the required time prior to analysis.

Sample solutions are introduced by pneumatic nebulization into a plasma, in which desolvation, atomization and ionization occurs. Ions are extracted from the plasma through a differentially pumped vacuum interface and sorted on the basis of their mass-to-charge ratio. The ions transmitted through the quadrupole are detected by an electron multiplier. Ion intensities at each mass are recorded and compared to those obtained from external calibration standards to generate concentration values for the samples. Results are corrected for instrument drift and matrix effects using internal standards.

3.0 INTERFERENCES

Isobaric Elemental Interferences – Isobaric elemental interferences result when isotopes of different elements have the same nominal mass-to-charge ratio and cannot be resolved with the instruments spectrometer. One way to solve this problem is to measure a different isotope for which there is no

interference. Alternatively, one can monitor another isotope of the element and subtract an appropriate amount from the element being analyzed, using known isotope ratio information. Corrections for most of the common elemental interferences are programmed into the software.

Isobaric Polyatomic Interferences – Isobaric polyatomic interferences result when ions containing more than one atom have the same nominal mass-to-charge ratio as an analyte of interest and cannot be resolved by the instrument's spectrometer. An example includes ClO+ (mass 51), which interferes with V, and must be corrected by measuring ClO+ at mass 53. When possible an interference free isotope should be chosen for measurement.

Physical interferences are associated with the sample nebulization and transport processes as well as with ion-transmission efficiencies. Nebulization and transport processes can be affected if a matrix component causes a change in surface tension or viscosity. Changes in matrix composition can cause significant signal suppression or enhancement. Dissolved solids can deposit on the nebulizer tip of a pneumatic nebulizer and on the interface skimmers (reducing the orifice size and the instrument performance). Total solid levels below 0.2% (2,000 mg/L) have been currently recommended to minimize solid deposition. An internal standard can be used to correct for physical interferences, if it is carefully matched to the analyte so that the two elements are similarly affected by matrix changes.

Memory interferences can occur when there are large concentration differences between samples or standards, which are analyzed sequentially. Sample deposition on the sampler and skimmer cones, spray chamber design, and the type of nebulizer affects the extent of the memory interferences, which are observed. The rinse period between samples must be long enough to eliminate significant memory interference.

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	25 mL	Acidified[2] with nitric acid to pH<2, stored ambient	Must be analyzed within 180 days of collection. If mercury is requested, analysis must occur within 28 days of sample 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 following acidification. Lead and Copper Rule Monitoring and Reporting Guidance for Public Water Systems, EPA 816-R-10-004, March 2010, Exhibit II-9, Samples must stand in the original container used for sampling for at least 28 hours after acidification.*

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 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
ICPMS (Inductively Coupled Plasma Mass Spectrometer)	Agilent 7700, 7800 7900 ICPMS instrumentation equipped with interference reduction technology. Each instrument has an associated auto-sampler, rough pump 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 volumetric flasks and graduated cylinders of various sizes

7.2 Supplies

Supply	Description
Argon gas	Praxair or equivalent, High purity grade, 99.99%
Collision Gas	Praxair or equivalent, Ultra high purity He, Ultra high purity H_2,
Analytical Balance	Sartoriius or equivalent, capable of weighing to 0.01g
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 II
Nitric Acid (HNO_3)	Fisher Scientific, A-509-P212 or equivalent replacement
Hydrochloric acid (HCl)	Fisher Scientific, A-508-P212 or equivalent replacement
2% (v/v) Nitric Acid/1% (v/v) Hydrochloric Acid Solution	Used for instrument blanks, standards and dilutions. Prepared in 1 L increments utilizing a volumetric flask and transferring into a C&G narrow mouth storage bottle. This is measured by mixing 20 mL of HNO_3 trace metals grade acid and 10 mL of HCl trace metals grade acid and DI H2O, and bringing to volume of 1 L.
Rinse Blank	2-5% (v/v) Nitric Acid solution for rinsing between runs. Combine76 mL of HNO_3 trace metals grade acid and 38 mL of HCl trace metals grade and DI H2O, and bringing to volume of 1 G.

8.2 Standards

Reagent	Description
Calibration Stock Standards	Custom blend of elements. See Appendix D for the standard information
Agilent Tune Solution	Purchased multi-element standard from a qualified vendor, 10ug/mL.
EPA Tune solution	Purchased multi-element standard from a qualified vendor, 10ug/mL.
Internal Standard Stock Solution	Various suppliers; single element standards to be mixed prior to use with concentrations of 1,000 and 10,000 ug/mL
Working Standards	See Appendix C

9.0 PROCEDURE

9.1 Equipment Preparation

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 Support Equipment

- Vacuum pump oil - Examine the sight glasses of the vacuum pump. Oil should be no darker than a light brown color. If it is, change the oil in the pump according to the directions in the manufacturer's guide.
- Chiller temperature, pressure and water level - The temperature should be regulated at 17 ± 1ºC. Check the current temperature on the chiller to ensure it is within this range. Check the inlet cooling water pressure that must be between 55 and 60psi. Check to ensure that chiller water level is full. If it is not, fill with Polyclear 30.
- Verify the level of nebulizer waste and rinse waste, if more than half full, empty it into the acid waste stream.
- Ar/O pressure - The argon supply pressure should be set at about 80psi. If the supply argon pressure falls below about 45psi, a safety interlock automatically shuts off the torch.
- Helium / Hydrogen pressure - The helium and hydrogen supply pressure should be set at about 15 and 9 psi respectively.
- 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.
- 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.
- Interface cones - Remove and inspect the outside of the sampling and skimmer cones around the orifice. Install a new set of cones if needed or clean the existing cones using the following procedure: Carefully polish each cone with silver polish and cotton swabs dampened with deionized water. Rinse cones with deionized water and blow-dry with house air supply, being careful not to damage the cones. After the cones are fully dry, replace them in the instrument. Allow for conditioning of the cones with a solution containing sufficient concentrations of major cations. The orifice should be circular and about 1mm in diameter. Examine the orifice periodically with a magnifier to determine if there are irregularities that may impair instrument performance. DO NOT use a cone with a significantly degraded tip.

9.1.2 Instrument

Lighting Torch and Warm-Up: After all pre-start checks pass inspection, perform the following steps:

- Torch Ignition - Click on the Plasma icon to open the Instrument window, and then click on the plasma on button to light the plasma. This takes a little over a minute to complete. (See instrument software guide.)
- Warm-up- Instrument is allowed to warm-up 30 minutes. Instrument has a timer to let you know when it is ready to move on to the next step.
- Check peristaltic pump flow by monitoring bubble movement in the pump tubing. Adjust tension as needed to achieve a smooth flow.

- Start-up Configuration - Once the analysis tubing is placed in the Agilent tune solution and stable signal is achieved, the start-up configuration can be initiated. See section 9.1.2.1 for Agilent tune performance monitoring and criteria.
- Create New Experiment File – Open template from the drive. Apply the proper run name for the day (MMDDYYICPMS#). Introduce EPA tune solution and allow signal to stabilize. Initiate performance verification for each mode of analysis. Save each performance report to the network drive. See section 9.1.2.1 for EPA tune acceptance criteria.

9.1.2.1 Routine Instrument Operating Conditions

The instrument is configured to go through the manufacturer recommended startup tune procedure which includes; Torch Alignment, Axis/Resolution, EM settings, Plasma Correction, Standard Lenses tune, and standard mode performance verification. The measured ratios of oxides 156/140 and doubly charged 70/140 should be <3%. The measured masses of ^{7}Li, ^{89}Y, ^{205}Tl are monitored for initial resolution/axis tuning. EPA Performance verification is later performed for each cell condition used for sample analysis.

EPA Tune Verification - The EPA tuning standard must be analyzed in each mode of analysis to verify resolution and mass calibration are within the required specifications. The tuning standard is analyzed in each mode of analysis at least five times and the relative standard deviation (RSD) must be <5% for all analytes contained in the tuning standard. Conduct mass calibration and resolution checks in the mass regions of interest. If the mass calibration differs more than 0.1 amu from the true value, then the mass calibration must be adjusted to the correct value. The resolution must also be verified to be <0.9 amu full width at 5% peak height.

Pace Minneapolis maintains approval for the analysis of up to 35 elements by the EPA Methods 200.8, 6020, 6020A, 6020B for water and soil matrices. All target analytes are analyzed either in a Helium mode (Collision Cell), hydrogen (Collision Cell), or No gas mode on the Agilent instruments depending on the sample matrix type. The use of interference reduction technologies (Collision Cell) is not allowed for drinking water analysis. Separate calibrations are performed for samples reporting by regulation of the SDWA.

9.2 Initial Calibration

9.2.1 Calibration Design

The calibration curve must consist of a minimum of a calibration blank and five non-zero standards for each mode of analysis. Use the average of at least three integrations for both calibration and sample analyses. Using the instrumentation software, prepare a standard curve for each element by plotting absorbance versus concentration. The working range varies with each analyte, see appendix C for summary. 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. Additional calibration specifications may be referenced in ENV-POL-CORQ-0005 *Acceptable Calibration Practices for Instrument Testing*, or equivalent replacement.

9.2.2 Calibration Sequence

Calibration Blank (CAL0)

CAL1
CAL2
CAL3
CAL4
CAL5
CAL6 (optional)
CAL7 (optional)
ICV
ICB
CRDL
ICSA
ICSAB
CCV
CCB
Client samples
CCV
CCB
CRDL (Optional)

9.2.3 ICAL Evaluation

9.2.3.1 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 > 0.998.

9.2.3.2 Relative Standard Error (RSE)

%RE is measured at the lowest calibration level and at a point near the mid-level of the calibration (the continuing calibration verification level is recommended). In order for a standard curve to be acceptable, the correlation coefficient/coefficient of determination criterion specified in the method must be met **and** both the low-level and mid-level %RE measures must meet the acceptance criteria. The low-level %RE acceptance criteria is 60%-140% and the mid-level is 90-110%.

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 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 Digestate Preparation

9.3.1 Homogenization and Subsampling

All solid matrices are subject to centrifuge at a rate of 1000 rpm for 15 minutes or allowed to settle overnight prior to analysis. Once samples have been centrifuged or allowed to settle, an initial dilution of 20 fold is performed on each sample. This is completed by taking 4.75mL of 2% HNO3 / 1% HCL diluent and mixing with a 0.25mL aliquot of sample by means of vortex.

Aqueous samples are inverted multiple times and poured without initial dilution unless historical data demonstrates otherwise.

9.4 Analysis

The instrument performs sample analysis by executing 100 mass sweeps per replicate. Three replicates are utilized for an average result which must fall within a 20% RSD for the replicate values. If any sample or QC is found to have a concentration of >5x the RL and >20% RSD it must be evaluated for interference. If a matrix interferent is determined to be the cause, dilute the sample by 5x and re-analyze. Perform further dilutions if necessary.

The instrument(s) have been setup and configured in conjunction with manufacturer specifications. Masses were carefully selected to avoid and/or minimize interferences. Internal standard selection was based on performance for the appropriate mass range. Internal standard association must remain within 50 amu of targeted analyte.

The total recoverable sample digestion procedure is suitable for the determination of silver in aqueous samples containing concentrations up to 0.1 mg/L. For the analysis of wastewater samples containing higher concentrations of silver, succeeding smaller volumes of well mixed sample aliquots must be prepared until the analysis solution contains < 0.1 mg/L silver.

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 Hardness as CaCO3 in mg/L = 2.497 * [Ca in mg/L] + 4.118 * [Mg in mg/L]

10.2 Concentration of lead = summation of signals at 206, 207, and 208 m/z.

10.3 Silica (SiO2) (µg/L) = Silicon (Si) (µg/L) * DF * 60.09 amu (SiO2 molecular weight) / 28.09 amu (Si atomic weight)

Where: DF is the sample Dilution Factor

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

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

10.5 Calculate the Relative Percent Difference (RPD) between the matrix spike and matrix spike duplicate using Equation 1:

Equation 1

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

Where, S = Sample result, mg/L or mg/kg

D = Duplicate sample result, mg/L or mg/kg

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 6020 (A)(B). 1 per batch of 10 or fewer samples for 200.8
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.
Post Digestion Spike	1 per batch of 20 or fewer samples for method 6020(A)(B).
Internal Standard	An appropriate internal standard is required for each analyte and sample determined by ICP-MS.

Internal Standard	Associated element
Scandium 45	Li, Be, B, Na, Mg, Al, Si, K, Ca, Ti, V, Cr, Mn, Fe, Se
Germanium 72	Co, Ni, Cu, Zn, As, Sr
Indium 115	Mo, Pd, Ag, Cd, Sn, Sb
Terbium 159	Ba, Pt, Hg, Tl, Pb, Bi
Iridium 193	U Th

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
Tune	Daily prior to any calibration
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 for 6020/6020B/200.8 and must be analyzed at the beginning of each run, and once at the end of each analytical batch for 6020A.
ICSA verification	At the beginning of each sample run sequence after the CRDL. 6020A and 6020B requires the ICSA/AB be analyzed every 12 hours thereafter.
ICSAB verification	At the beginning of each sample run sequence after the ICSA. 6020A and 6020B requires the ICSA/AB be analyzed every 12 hours thereafter.

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 near the midpoint of the calibration range. 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* for the conditions under which the procedures in test method SOPs may be modified and for the procedure and document requirements.

14.1 Tuning criteria observed is more stringent than required by the SW846 methods so that the same criteria can be used for both methods 6020 and 200.8.

14.2 The following elements are not listed in the method 6020A recommended analyte list; bismuth, boron, lithium, molybdenum, palladium, platinum, silica, silicon, strontium, tin, titanium, thorium, and uranium. The accuracy and precision for the analysis of these analytes have been demonstrated in the matrices of interest, at the concentration of interest, and in the same manner as the elements recommended in the method.

14.3 The following elements are not listed in the method 200.8 recommended analyte list: bismuth, boron, calcium, iron, lithium, magnesium, palladium, platinum, potassium, silica, silicon, sodium, strontium, tin, and titanium. The accuracy and precision for the analysis of these analytes have been demonstrated in the matrices of interest, at the concentration of interest, and in the same manner as the elements recommended in the method.

14.4 The following elements are not listed in the method 6020B recommended analyte list: bismuth, boron, lithium, molybdenum, palladium, platinum, silica, silicon, strontium, tin, titanium and uranium. The accuracy and precision for the analysis of these analytes have been demonstrated in the matrices of interest, at the concentration of interest, and in the same manner as the elements recommended in the method.

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 – Working Standard Summary

Appendix D – 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.

U.S. Environmental Protection Agency. Method 200.8, Determination of Trace Elements in Waters and Wastes by Inductively Coupled Plasma – Mass Spectrometer, Revision 5.4, EMMC Version, May 1994.

U.S. Environmental Protection Agency. SW846 Method 6020, Inductively Coupled Plasma – Mass Spectrometry, Revision 0, 9/94.

U.S. Environmental Protection Agency. SW846 Method 6020A, Inductively Coupled Plasma – Mass Spectrometry, Revision 1, 02/2007.

U.S. Environmental Protection Agency. SW846 Method 6020B, Inductively Coupled Plasma – Mass Spectrometry, Revision 2, 7/2014.

Test Methods for Evaluating Solid Waste Physical/Chemical Methods, SW-846, Third Edition. Method 3020A.

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
6.0	Updated sample retention from 45 to 21 days.
8.2	Internal Standard Stock Solution – added “1,000 and”
9.2.1	Updated 3 to 5 non-zero standards. Added “The working range…C for summary.”
9.2.2	Added “(optional)” to CAL6. Added “CAL7 (optional)”.
10.0	Added sections 10.4 and 10.5.
11.1	Updated Thoridium 232 to Iridium 193.
14.0	14.2 & 14.4: removed “-238” from uranium. 14.2: added thorium.
17.0	Removed references for Fisions and Region 9 Laboratory SOP.
Appendix A	Added Thorium. Updated Silica and Silicon entries. Removed Mercury NPW and potable water entries.
Appendix B	Updated ICAL Acceptance Criteria. Updated methods referenced in MB Acceptance Criteria. Added LDR acronym to QC Item.
Appendix C & D	Re-formatted tables.

This document supersedes the following document(s):

Document Number	Title	Version
ENV-SOP-MIN4-0043	Metals Analysis by ICP/MS – Method 6020 and 200.8	03

Appendix A: Target Analyte List and Routine LOQ[1]

Analyte	Non-Potable Water (ug/L)	Potable Water (ug/L)	Soil (mg/kg)
Aluminum	20.00	20.0	20.00
Antimony	0.50	0.50	0.50
Arsenic	0.50	0.50	0.50
Barium	0.30	0.30	0.30
Beryllium	0.20	0.20	0.20
Bismuth	0.50	-	0.50
Boron	10.00	-	10.00
Cadmium	0.08	0.08	0.08
Calcium	40.00	-	40.00
Chromium	0.50	0.50	0.50
Cobalt	0.50	-	0.50
Copper	1.00	1.00	1.00
Iron	50.00	-	50.00
Lead	0.10	0.10	0.20
Lithium	0.50	-	0.50
Magnesium	10.00	-	10.00
Manganese	0.50	0.50	0.50
Mercury	-	-	0.20
Molybdenum	0.50	-	0.50
Nickel	0.50	0.50	0.50
Palladium	0.50	-	-
Platinum	0.50	-	-
Potassium	100.00	-	100.00
Selenium	0.50	0.50	0.50
Silica	214.00	-	214.0
Silicon	100.00	-	100.00
Silver	0.50	0.50	0.50
Sodium	50.00	-	50.00
Strontium	0.50	-	0.50
Thallium	0.10	0.10	0.10
Thorium	0.50	-	0.50
Tin	0.50	-	2.000
Titanium	1.00	-	1.00
Vanadium	1.00	1.00	1.00
Uranium-238	0.50	0.50	0.50
Zinc	5.00	5.00	5.00

[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.

Appendix B: QC Summary

QC Item	Frequency	Acceptance Criteria	Corrective Action	Qualification
Tune	Daily prior to any calibration	Adjust spectrometer resolution to produce a peak width of approximately 0.75 amu at 5% peak height. This must be completed using 5 replicates with a resulting RSD of <5%.	Adjust mass calibration if it has shifted by more than 0.1 amu from unit mass. Identify and correct source of problem, repeat performance verification(s).	None. Do not proceed with analysis.
ICAL	Daily	r ≥ 0.998 a Midlevel (recommended near ICV/CCV concentrations) %RE 90-110% Low-Level (Cal1) %RE 60-140%	Identify and correct source of problem, repeat.	None. Do not proceed with analysis.
ICV	After Each ICAL	All analytes must be within ± 10% of the true value. (%R)	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 criterion of +/- ½ of the RL for method 6020 (A)(B) and samples originating from NC. All elements of interest must be evaluated to +/- the RL for method 200.8, and 6020. 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. 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	At the beginning of each run for 6020/6020B/200.8 and must be analyzed at the beginning of each run, and once at the end of each analytical batch for 6020A.	For 6020/200.8: The acceptance criteria are ± 40% (or specified by the client). For 6020A: The acceptance criteria are ± 30% (or specified by the client). 6020B: The acceptance criteria is ± 20% (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).	Qualify outages and explain in case narrative.

			If the CRDL is biased low, no data can be reported for the target elements failing criteria.	
CCV	Daily, before sample analysis, after every 10, and at end of analytical window.	All analytes must be within ± 10% of the true value. (%R): %RSD between multiple integrations must be ≤ 5%	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	All elements of interest must be evaluated to a criterion of +/- ½ of the RL for method 6020 (A) and samples originating from NC. All elements of interest must be evaluated to +/- the RL for method 200.8, and 6020 (B). 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. 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	For method 6020, the intensity of internal standard in the ICB/CCB and ICS (ICSA/AB) standards must not deviate more than 80-120% from its original intensity in the associated calibration blank. The intensity of internal standard in the samples and remaining QC must not deviate more than 30-120%. For method 6020A/B, the intensity of the internal standard must not fall below 70% and not exceed 130% from its original intensity in the associated calibration blank. For Method 200.8 the intensity of internal standard in the samples and QC must not deviate more than 60-125% from its original intensity in the associated calibration blank.	Troubleshoot instrument performance. Reanalyze samples and dilute if needed.	Qualify outages and explain in case narrative.
Interference check solutions	ICSA containing high concentrations of C, Cl, Al, Ca, Fe, K, Mg, Mo, Na, P, S and Ti is analyzed at the beginning of each sample run sequence after the CRDL. ICSAB containing high concentrations of	ICSA all spiked elements are to be within 20% of the expected true value. The non-spiked elements are to be below the RL. ICSAB all spiked elements are to be within 20% of the expected true value.	Identify and correct source of problem, repeat performance verification(s).	None. Do not proceed with analysis for elements that cannot be verified.

	C, Cl, Al, Ca, Fe, K, Mg, Mo, Na, P, S and Ti and mid-range concentrations of the remaining elements is analyzed at the beginning of each sample run sequence following the ICSA. 6020A and 6020B requires the ICSA/AB be analyzed every 12 hours thereafter.			
Method Blank (MB)	One per 20 samples	Method 200.8: 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 6020, 6020A and 6020B: The method blank is considered to be acceptable if it does not contain the target analytes that exceed the LLOQ or project-specific DQOs.	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	6020/6020A/6020B: 80-120% 200.8: 85-115%	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.	6020/6020A/6020B: 80-120% 200.8: 85-115% %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 6020 / 6020A / 6020B One per 10 samples for 200.8	6020/6020A/6020B: 75-125% 200.8: 70-130%	Perform a SD and PDS on any elements that fail to meet criteria for method 6020(A)(B).	Qualify analytes with MS out of criteria.
Sample Duplicate	Per client request	%Diff ≤ 20%	Qualify outages	Qualify outages.
Serial Dilution[1]	One per batch of 20 samples or less		If criteria is not met, original sample and dilution shall be	Qualify outages.

		6020/6020A fivefold dilution must agree within ± 10% of the original determination if analyte concentration is >50x MDL. 6020B 1:5 dilution of sample 25x > LLOQ or 1:5 dilution of MS since reasonable concentrations are present, results to agree to ± 20%.	reanalyzed. If reanalysis fails, it is determined to be matrix interference.	
Post Digestion Spike[2]	One per batch if there is a MS failure.	6020/ 6020A 80-120% 6020B applicable to elements failing MS, results to agree to +/- 25%. Recommended if high concentration sample not available for dilution test.	If the element fails to meet the recovery criteria, reanalyze. If reanalysis fails, it is determined to be matrix interference.	Qualify outages.
Laboratory Filter Blank (FB)	Analyzed only with batches of lab filtered dissolved metals, one per batch of 20 or less.	Target analytes must be less than reporting limit. NC samples are required to be < ½ RL for target analytes. 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 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 (LDR)	For method 6020B: Following calibration, the laboratory may choose to analyze a standard at a higher concentration than the high standard in the calibration. If a linear range standard is not analyzed for any specific element, the highest standard in the calibration becomes the linear range.	The standard must recover within 10% of the true value, and if successful, establishes the linear range. In each scenario, the linear 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 as well as maintaining the minimum number of calibration standard requirements.	N/A

[1]To prepare a 5-fold dilution: take a 1 mL aliquot from the sample and add to 4 mL of diluent. Note: this is a typical process for 200.8 and 6020W. It can be replicated for the preparation of highly concentrated samples by starting with a diluted "parent" sample and then performing the stepwise dilution process.

[2]To Prepare a Post Digestion Spike: An aliquot of the parent sample used for the MS, prepared at the same dilution as the parent sample. The spike addition should produce a minimum level of 10 times the lower limit of quantitation; routine spike volume is 0.020 mL of 20/250 mg/L and 1mg/L mercury stock concentration(s).

Appendix C: Working Standard Summary

Standard	Standard(s) Used	Standard(s) Amount (mL)	Diluent	Diluent Volume (mL)	Final Total Volume[1] (mL)	Final Concentration (ug/L)
Internal Standard	6020-Ge	1	See table 8.1	495	500	2000
	6020-Sc	1				
	6020-Tb	1				
	6020-In	1				
	6020-Ir	1				
Bi/Th primary (Intermediate)	6020-Th	0.5		49.5	50	1,000
	6020-Bi	0.5				
Bi/Th secondary (Intermediate)	6020-Th	0.5		49.5	50	1,000
	6020-Bi	0.5				
Hg 10ppb (intermediate)	HG-LL Stock	0.05		49.95	50	10
6020 Hg-SPK	MERC-STK1	0.05		49.95	50	1000
Hg (Intermediate) C	MERC-STK2	0.25		249.75	250	1000
6020-SPK (intermediate)	Bi-STK	0.2		4.6	10	20,000 / 250,000 / 500,000
	Th-STK	0.2				
	HP7375	5				
6020-SPK2 (intermediate)	HP7376	1		9	10	20,000
6020-SPK3 (intermediate)	HP7379	1		9	10	20,000 / 10,000
CAL-SPK1 (intermediate)	HP7375	0.25		9.5	10	25000/12500/1000/500/10
	HP7379	0.05				
	HP7376	0.05				
	6020Hg-SPK	0.1				
	Bi/Th Intermediate	0.05				
Cal 0	N/A	N/A		50	50	0
Cal 1	ZPACEMN103	0.1		9.7	10	Varied
	ZPACEMN104	0.1				
	Hg 10ppb (intermediate)	0.1				0.1
Cal 2	CAL-SPK1	0.1		9.9	10	250/125/10/5/0.1
Cal 3	CA:L-SPK1	0.5		9.5	10	1250/625/50/25/0.5
Cal 4	CAL-SPK1	1		9	10	2500/1250/100/50/1

Cal 5	CAL-SPK1	2.5		7.5	10	6250/3120/250/125/2.5
Cal 6	CAL-SPK1 (intermediate)	5		-	5	25000/12500/1000/500/10
CRDL	ZPACEMN-103	0.1		9.6	10	varied
	ZPACEMN-104	0.1				
	6020 Hg-SPK	0.2				0.2
ICS-A	ICS-ICPMS	0.25		9.75	10	25000/500
ICS-AB	ICS-ICPMS	0.25		9.56	10	27500/26200/1250/600/100/50/4
	6020-SPK	0.05				
	6020-SPK2	0.05				
	6020-SPK3	0.05				
	6020Hg-SPK	0.04				
ICV / CCV add Hg	XPACEMN-75	0.05		49.31	50	4/80/1000
	XPACEMN-76	0.02				
	Bi/Th Intermediate	0.4				
	XPACEMN-77	0.02				
	Hg Intermediate C	0.2				

[1]Alternate final volumes may be prepared at the discretion of the scientist, so long as the concentrations specified above are maintained.

Appendix D: Stock Standard Summary

Stock Standard Concentrations

	HP7379	HP7376	HP7375	XPACEMN 77	XPACEMN 76	XPACEMN 75	ZPACEMN 103	ZPACEMN 104	ICS-ICPMS	Agilent Tune	EPA Tune
Analyte	(ug/mL)	(ug/mL)	(ug/mL)	(ug/mL)	(ug/mL)	(ug/mL)	(ug/mL)	(ug/mL)	(ug/mL)	(ug/mL)	(ug/mL)
Aluminum	-		1000			1000	2		1,000		
Antimony		200		200				0.005			
Arsenic	200				200			0.05			
Barium	200				200		0.03				10
Beryllium	200				200		0.02				10
Bismuth							0.05				
Boron		200		200			1				
Cadmium	200				200		0.008				
Calcium			1000			1000	4		1,000		
Chromium	200				200		0.05				
Cobalt	200				200		0.05			10	10
Copper	200				200		0.1				
Iron			500			500	5		1,000		
Lead	200				200		0.01				
Lithium	200				200		0.05			10	10
Magnesium			1000			1000	1		1,000		10
Manganese	200				200		0.05				
Molybdenum		200		200				0.05	20		
Nickel	200				200		0.05				
Palladium		200		200				0.05			
Platinum		200		200				0.05			
Potassium			1000			1000	10		1,000		
Selenium	200				200			0.05			
Silicon			500			500		10			
Silver	100				100		0.05				
Sodium			1000			1000	5		1,000		
Strontium	200				200		0.05				
Thallium					100		0.01			10	10

Tin		200		200		20		0.05			
Titanium		200		200		20		0.1	20		
Vanadium	200				200		0.1				
Zinc	200				200		0.5				
Uranium	200						0.05				10
Indium											10
Cesium					200						10
Cerium										10	
Yttrium										10	10
Rhodium											10
Thorium							0.05				

Single Element Stock Standard Concentrations

	Bi-STK (Spex)	Bi-STK (Agilent)	6020-Th (Spex)	6020-Th (Agilent)	MERC-STK1	MERC-STK2	HG-LL Stock	6020-Ge	6020-Sc	6020-Tb	6020-In	6020-Ir
Analyte	(ug/mL)	(ug/mL)	(ug/mL)	(ug/mL)	(ug/mL)	(ug/mL)	(ug/mL)	(ug/mL)	(ug/mL)	(ug/mL)	(ug/mL)	(ug/mL)
Bismuth	1000											
Bismuth		1000										
Thorium			1000									
thorium				10000								
Mercury					1000							
Mercury						1000						
Mercury							10					
Germanium								1000				
Scandium									10000			
Terbium										1000		
Indium											1000	
Iridium												1000

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ENVIRONMENTAL SCIENCES

Document Information

Document Number: ENV-SOP-MIN4-0054 **Revision:** 04

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

Department(s): Metals

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Document Notes:

All Dates and Times are listed in: Central Time Zone

Signature Manifest

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All dates and times are in Central Time Zone.

ENV-SOP-MIN4-0054 - Mercury

QM Approval

Name/Signature	Title	Date	Meaning/Reason
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Management Approval

Name/Signature	Title	Date	Meaning/Reason
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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 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 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 45 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 equivalent. 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
Auto-sampler tubes	Moldpro or equivalent, 15 mL metals free auto-sampler tubes
Digestion cups	Moldpro or equivalent, 50 mL disposable digestion cups

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. 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. 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 ± 2°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 MB by weighing 0.3 g of resin pellets in a 50 mL cup.

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.

9.2.1.3 Weigh a representative 0.3-0.36 g portion of sample in a 50 mL 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 ± 2°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. 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 ± 2°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. 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-SOP-NW-0027 *Calibration Procedures*, 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

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

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

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 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 B 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-NW-0018 *Determination of LOD and LOQ* 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-NW-0025 *Training and Orientation Procedures* 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* 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 cycletainers 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.

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
Appendix A	Updated MB Acceptance Criteria and Corrective Action.

This document supersedes the following document(s):

Document Number	Title	Version
ENV-SOP-MIN4-0054	Mercury in Liquid and Solid/Semi-Solid Waste by 7470A, 7471, 7471B, and 245.1	03

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 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	Result must be less than the absolute value of the Reporting Limit (LOQ). NC requires blanks to be clean to ½ RL. WIDNR and West Virginia 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 and West Virginia 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 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.	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 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,	Qualify analytes with MS out of 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.	
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.
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.

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:** 03

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

Department(s): Metals

Date Information

Effective Date: 22 Feb 2021

Notes

Document Notes:

All Dates and Times are listed in: Central Time Zone

Signature Manifest

Document Number: ENV-SOP-MIN4-0056 **Revision:** 03

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	22 Feb 2021, 11:06:07 AM	Approved

Management Approval

Name/Signature	Title	Date	Meaning/Reason
Andrew Mickelson (009792)	Manager	18 Feb 2021, 08:49:18 AM	Approved
Krista Carlson (004514)	Project Manager 1	18 Feb 2021, 10:35:56 AM	Approved
Adam Haugerud (005828)	General Manager 2	22 Feb 2021, 09:08:26 AM	Approved

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 PACE-67Aand Pace-67B. 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

Calibrate 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.

Calibrate 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.

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 using 0.25 mL of each PACE-67A and PACE-67B. If mercury is requested spike 0.40 mL of Hg-STK 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.

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 cycletainers for solvent management, and real-time purchasing.

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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
All	Updated SOP references.
6.0	Updated from 45 to 21 days for sample retention.
9.2.3	Updated DI addition from "Add 10 mL DI.." to "Add DI to the 10 mL marking…".
Appendix A	Updated standard composition – to ZPACEMN-105 from PACE-67B and to ZPACEMN-106 from PACE-67A. Updated elements and concentrations accordingly.

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	02

Appendix A: Metals Standard Reference

Stock standards used for solid sample preparation

ZPACEMN-105		ZPACEMN-106		Hg-STK 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				

Pace Analytical®
ENVIRONMENTAL SCIENCES

Document Information

Document Number: ENV-SOP-MIN4-0059 **Revision:** 01

Document Title: Preparation of Attic Dust and Filter Cartridge Samples by 3050B for Analysis by ICPMS

Department(s): Metals

Date Information

Effective Date: 23 Dec 2019

Notes

Document Notes:

All Dates and Times are listed in: Central Time Zone

Signature Manifest

Document Number: ENV-SOP-MIN4-0059 **Revision:** 01

Title: Preparation of Attic Dust and Filter Cartridge Samples by 3050B for Analysis by ICPMS

All dates and times are in Central Time Zone.

ENV-SOP-MIN4-0059 - 3050B

QM Approval

Name/Signature	Title	Date	Meaning/Reason
Janielle Ward (007319)	Quality Manager	05 Dec 2019, 12:46:47 PM	Approved

Management Approval

Name/Signature	Title	Date	Meaning/Reason
Christina Schmitt (005842)	Support Coordinator II	27 Nov 2019, 09:52:00 AM	Approved
Andrew Mickelson (009792)	Manager - Lab Services	27 Nov 2019, 03:24:30 PM	Approved
Adam Haugerud (005828)	Assistant General Manager	23 Dec 2019, 10:51:24 AM	Approved

1. Purpose/Identification of Method

1.1. The purpose of this SOP is to establish a procedure for the digestion of attic dust and filter cartridge samples to be analyzed by ICP-MS as described in EPA Method 3050B.

2. Summary of Method

2.1. Filter Cartridge samples are opened and a total weight is recorded.

2.2. The samples are digested in concentrated nitric acid, hydrochloric acid and hydrogen peroxide. After digestion samples are brought to volume of 50 mL.

3. Scope and Application

3.1. **Personnel**: The policies and procedures contained in this SOP are applicable to all personnel involved in the analytical method or non-analytical process.

3.2. **Parameter**s: Not applicable to this SOP.

4. Applicable Matrices

4.1. This SOP is applicable to solid samples.

5. Limits of Detection and Quantitation

5.1. Not applicable to this SOP.

6. Interferences

6.1. Not applicable to this SOP.

7. Sample Collection, Preservation, Shipment and Storage

7.1. Table 7.1 – Sample Collection, Preservation, Shipment and Storage

Sample type	Collection per sample	Preservation	Storage	Hold time
Solid	Pre-cleaned vacuum filter cartridges supplied by client.	N/A	Ambient temperature.	Must be analyzed within 6 months of collection.

8. Definitions

8.1. Definitions of terms found in this SOP are described in the Pace Analytical Services Quality Manual, Glossary Section.

9. Equipment and Supplies (Including Computer Hardware and Software)

9.1. Table 9.1 – Equipment and Supplies

Supply	Description	Vendor/Item #/Description
Mechanical pipettes	Various sizes	Fisher Scientific or equivalent
Digestion Cups	50 mL	Environmental Express
Filtermate Plunge filters	2 um PTFE SC0401	Environmental Express
Hot Block™	54 Place Hot Block™	Environmental Express
Reflux Caps	Caps with a center hole	Environmental Express
Analytical Balance	Ability to weigh to the nearest 0.01 g	Fisher Scientific or equivalent
Resin beads	For solid matrix QC	Environmental Express or equivalent

10. Reagents and Standards

10.1. Table 10.1 – Reagents and Standards

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
ICP-MS Spike - Stock solution standards for LCS and MS/MSD	The solution identifications are Pace-67A, Pace-67B. See 10.1.1.	Purchased from Inorganic Ventures (or equivalent). Store at room temperature. Expires as specified by manufacturer.

10.1.1. Table 10.1.1. - ICPMS Stock Standards Table

PACE-67B (Metals-STK2)			PACE-67A (Metals-STK1 Stock)		
Element	(mg/L)	Spike amount (mL)	Element	(mg/L)	Spike amount (mL)
Ca	4000	.25	Si	1000	.25
Fe	4000	.25	Mo	200	.25
Mg	4000	.25	Sb	200	.25
K	4000	.25	Sn	200	.25
Na	4000	.25	Ti	200	.25
Al	4000	.25			
Se	200	.25			
Ba	200	.25			
Be	200	.25			
Bi	200	.25			
B	200	.25			
Cd	200	.25			
Cs	200	.25			
Cr	200	.25			
Co	200	.25			
Cu	200	.25			
Li	200	.25			
Mo	200	.25			
Mn	200	.25			
Pb	200	.25			
Ni	200	.25			
Ag	100	.25			
Sr	200	.25			
Tl	200	.25			
V	200	.25			
Zn	200	.25			
U	200	.25			

11. Calibration and Standardization

11.1. Calibrate variable and fixed volume pipettes as specified in SOP ENV-SOP-NW-0016 *Support Equipment* (or equivalent replacement). Calibration records are kept in the QA Office.

11.2. Calibrate the thermometer as specified in SOP ENV-SOP-NW-0016 *Support Equipment* (or equivalent replacement). Calibration records are kept in the QA Office.

12. Procedure

12.1. Sample Preparation

12.1.1. Record the total weight of the attic dust cartridge and filter to the nearest 0.001 g. Empty contents of the cartridge including the filter into tared 50 mL digestion vessel. Subtract the average filter weight to obtain true sample weight. Please note the procedure for determining the exact weight

of the sample collected on the filter will depend on the type of cartridge used for collection. Some filters will come with a filter weight or a blank filter to weight for correction. If not, a blank must be provided, weight of the entire cartridge must be documented prior to use and prior to opening.

12.1.1.1. Create a method blank (MB), laboratory control sample (LCS), and laboratory control sample duplicate (LCSD) by weighing out 1 g of resin beads for each. Spike the LCS and LCSD with 0.25 mL spike solution. If sample volume permits, preparation of a matrix spike (MS) and matrix spike duplicate (MSD) will be employed using same volume of spike solution.

12.1.2. Add 10 mL of DI water to each sample.

12.1.3. Add 7.5 mL of concentrated HNO_3, mix the slurry, and cover with a reflux cap. Heat the sample to 95 +/- 2°C and reflux for 70 minutes without boiling. Observe the sample during heating for brown fumes indicating oxidation of the sample. If this occurs, add up to an additional 5 mL HNO_3 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: Record initial hot block temperature in the digestion log.

12.1.4. Cool the sample 10 minutes. Add 2.5 mL 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.

12.1.4.1. If effervescence does not subside, continue to add 30% hydrogen peroxide in 1 mL 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 10 mL hydrogen peroxide.

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

12.1.6. 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. Invert several times for good mixing. FOR ICP-MS sample prep, cap and label samples for analysis – do not filter if analyzed by ICP-MS.

NOTE: The method modifications that have been utilized in the above process have been demonstrated effective in MDLs, DOCs, and ongoing precision and accuracy data samples.

12.2. Documentation

12.2.1. Standard Prep Logbook

12.2.1.1. Record the necessary information in the prep logbook, including source, lot numbers, volumes utilized, and expiration date.

12.2.2. Digestion Logbook

12.2.2.1. Record the necessary information in the digestion log book including sample ID, initial and final volumes, prep date, prep analyst, and lot numbers of solutions used, including spike solutions.

12.2.2.2. Also, include any additional comments if needed.

12.2.3. Temperature Logbook

12.2.3.1. Record the temperature of each hot block daily in the temperature logbook.

12.2.3.2. Use a NIST-traceable thermometer inserted into a digestion cup filled with 50 mL 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.

13. Quality Control

13.1. Table 13.1 – Quality Control

QC Sample	Components	Frequency	Acceptance Criteria	Corrective Action
Preparation Blank	A clean matrix similar to the samples. For solids, 1.0 g of resin beads.	Prepared with each batch.	See appropriate analysis SOP.	See appropriate analysis SOP.
Laboratory Control Sample (LCS) / Laboratory Control Sample Duplicate (LCSD)	For solids, weigh 1.0 g of resin beads. Spike with appropriate spiking solutions listed in Section 10.1.1.	Prepared with each batch. If there is insufficient volume for matrix spike and duplicate, an LCSD must be performed.	See appropriate analysis SOP.	See appropriate analysis SOP.
Matrix Spike (MS) / Matrix Spike Duplicate (MSD)	Weigh out similar amounts of soil as the parent sample; be sure to weigh QC sample and MS/MSD samples as close as possible. Spike with appropriate spike solutions and record in digestion log.	Prepared with each batch of samples. Client specific requirements may result in a greater number of MS or MS/MSD sets in a batch.	See appropriate analysis SOP.	See appropriate analysis SOP.
Duplicate (DUP)	In some cases, the client may request a duplicate in lieu of an MSD. This is weighed out in similar amount (as close as possible) to the background sample.	As requested.	See appropriate analysis SOP.	See appropriate analysis SOP.

14. Data Analysis and Calculations

14.1. Not applicable to this SOP.

15. Data Assessment and Acceptance Criteria for Quality Control Measures

15.1. See table in section 13.

16. Corrective Actions for Out-Of-Control Data

16.1. See table in section 13.

17. Contingencies for Handling Out-Of-Control or Unacceptable Data

17.1. If not specifically listed in the table in Section 13, the contingencies are as follows. If there is no additional sample volume to perform re-analyses, all data will be reported as final with applicable qualifiers. If necessary, an official case narrative will be prepared by the Quality Manager or Project Manager.

18. Method Performance

18.1. All applicable personnel must read and understand this SOP with documentation of SOP review maintained in their training files.

18.2. **Method Detection Limit (MDL) Study**: Method Detection Limit Studies (MDLs) will be established and analyzed at a frequency determined in ENV-SOP-NW-0018 *Method Detection Limit Studies*, or equivalent replacement and 40 CFR Part 136, Appendix B.

18.3. **Demonstration of Capability (DOC)**: Every analyst who performs this method must first document acceptable accuracy and precision by passing a demonstration of capability study (DOC) per ENV-SOP-NW-0025 *Orientation and Training Procedures* (or equivalent replacement).

18.4. **Periodic Performance Evaluation (PE):** Not available for this matrix**.**

19. Method Modifications

19.1. The preparation method has been modified in terms of the amounts of reagents used and the individual heating times. The chemistry is maintained. Part of the 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.

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

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

20. Instrument/Equipment Maintenance

20.1. Please refer to the specific manufacturer's instrument manual for maintenance procedures performed by the lab.

20.2. All maintenance activities are listed daily in maintenance logs that are assigned to each separate instrument.

20.3. Logs are kept daily for each hot block, monitoring temperature. The temperature probe is varied daily so that each individual hot block sample cell is monitored to ensure consistency across the block.

21. Troubleshooting

21.1. Not applicable to this SOP.

22. Safety

22.1. Standards and Reagents: The toxicity and carcinogenicity of standards and reagents used in this method have not been fully defined. Each chemical compound should be treated as a potential health hazard. Reduce exposure by the use of gloves, lab coats and safety glasses. Safety Data Sheets (SDSs) are on file in the laboratory and available to all personnel. Standard solutions should be prepared in a hood whenever possible.

22.2. Samples: Take precautions when handling samples. Samples should always be treated as potentially hazardous "unknowns". The use of personal protective equipment (gloves, lab coats and safety glasses) is required when handling samples. In the event a sample container must be opened, it is recommended to perform this in a hood whenever possible.

23. Waste Management

23.1. Procedures for handling waste generated during this analysis are addressed in ENV-SOP-MIN4-0098 *Waste Handling and Management* (or equivalent replacement).

23.2. In order to minimize the amount of waste generated during this procedure, analyst should prepare reagents in an amount which may be used in a reasonable amount of time (e.g., before a reagent expires).

24. Pollution Prevention

24.1. The company wide Chemical Hygiene and Safety Manual contains information on pollution prevention.

25. References

25.1. Pace Quality Assurance Manual- most current version.

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

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

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

25.5. 40 CFR Appendix B to Part 136, *Definition and Procedure for the Determination of the Method Detection Limit – Rev 2*, August 28, 2017.

26. Tables, Diagrams, Flowcharts, and Validation Data

26.1. Not applicable to this SOP

27. Revisions

Document Number	Reason for Change	Date
S-MN-I-604 Rev.01	Updated LLC Removed "uncontrolled" Added "Copies without a distribution number below are considered uncontrolled." To the statement of copyright. Edited last row of Table 10.1 – Concentration column to include Pace-67A and Pace-67B instead of XFSPA-656-250, XFSPA-221-250 and XFSPA-220-250; Requirements column to from "Inorganic Ventures" instead of "Spex CertiPrep" Added section header to ICPMS Stock Standards Table, should have read 10.1.1. but did not have title Changed to PACE-67B and PACE-67A for ICPMS Stock Standards in Table 10.1.1. Added Spike Amount column to ICPMS Stock Standard Table Changed concentrations to mg/L in Table 10.1.1. Removed Ce, La, and As elements, added Mo and Ti to Table 10.1.1. Deleted Table 10.2 and Section 10.2.1. Fixed numbering for Section 12.1.1.1. Added "with 0.25 mL spike solution" and "using same volume of spike solution" to Section 12.1.1.1. Deleted "have been defined" from Section 12.1.6.1.	11Sept2017
ENV-SOP-MIN4-0059-Rev.01	Updated to MasterControl format and numbering. Updated hot block references. Updated TNI references 25.2 & 3. 12.2.2.1 – deleted "and LCS solutions" Table 13.1, LCS row – added LCSD, added "listed in section 10.1.1" to Components, added "If there is…performed" to Frequency. 18.2 – updated MDL verbiage and added corresponding reference 25.5. 22.1 – updated MSDS to SDS.	27Nov2019

APPENDIX C ACCESS FORMS

Atlantic Richfield Company

317 Anaconda Road
Butte, MT 59701
Main: (406) 723-1822

[Insert Date]

[Insert Contact Name]
[Insert Entity/School/Daycare Name]
[Insert Number & Street Name]
Butte, MT 59701

Re: Residential Metals Abatement Program (RMAP) Indoor Dust Sampling Access Agreement

Dear [Insert Name]:

Atlantic Richfield Company as part of the Residential Metals Abatement Program (RMAP) will be conducting soil sampling of school properties located in and around Butte, Montana. These tasks are required under the Silver Bow Creek/Butte Area National Priorities List (NPL) Site, Butte Priority Soils Operable Unit (BPSOU) Unilateral Administrative Order (UAO) Amendment issued by the U.S. Environmental Protection Agency (EPA) in August 2020 (UAO Amendment). The UAO Amendment expanded the RMAP program to include schools consistent with the BPSOU Record of Decision (ROD) Amendment issued by EPA in February 2020.

By way of this letter and the enclosed Access Agreement, Atlantic Richfield is requesting access to your property to collect interior/attic dust samples. Representatives of the EPA and the Montana Department of Environmental Quality (DEQ) may also be present to provide oversight during these sampling activities.

Samples collected from your property will be sent to a laboratory and analyzed for concentrations of lead, arsenic, and mercury. The data results from these samples will be shared with you after proper data qualification is complete and used to determine whether any further action is needed to meet EPA remedy requirements. Atlantic Richfield will make every reasonable effort to schedule sampling at a time that is convenient for you and to minimize any inconvenience to you during the sampling work.

Atlantic Richfield respectfully asks that you review and sign the enclosed Access Agreement. Also, please include a phone number where you can be contacted to notify you of the proposed sampling schedule. If you have the ability to do so, please scan and email me back the signed Access Agreement. If it is more convenient for you, can also mail the signed Access Agreement to:

Environmental Resources Management
1 Ninth Street Island Drive
Livingston, MT 59047
c/o Christopher Berg

Upon receipt of the Access Agreement from you, I will countersign and provide you with a fully signed copy of the Agreement for your records. Your cooperation during this sampling effort is appreciated. If you have any questions or concerns please do not hesitate to call me at **(907) 355-3914**.

Sincerely,

Mike McAnulty

Mike Mc Anulty
Liability Manager
Remediation Management Services Company
An affiliate of Atlantic Richfield Company
(907) 355-3914

Enclosure

File: MiningSharePoint@bp.com

ACCESS AGREEMENT

Insert School/Daycare Owner's Name("OWNER"), whose mailing address is, _____, and **Atlantic Richfield Company** ("Atlantic Richfield"), whose mailing address is 317 Anaconda Road, Butte, MT 59701, enter into this Access Agreement ("Agreement") this _____ day of _____, 2021 and agree as follows:

1. GRANT OF ACCESS. OWNER hereby grants to Atlantic Richfield, including its authorized representatives (and, as may be appropriate, to EPA and/or the State of Montana and the authorized representatives of each) the right to enter OWNER's real property, as described in Exhibit A, which is attached hereto and incorporated herein by reference (the "Property"), to conduct all activities related to sampling of interior/attic dust (collectively referred to as "Sampling"). OWNER represents to Atlantic Richfield that, to the best of OWNER's knowledge, OWNER possesses ownership interests in the Property sufficient to grant access to Atlantic Richfield to conduct the Sampling.

2. ATLANTIC RICHFIELD REPRESENTATIONS. Atlantic Richfield or its representative will notify OWNER, either in writing or verbally, at least 24 hours prior to first commencing Sampling on the Property. Atlantic Richfield will make every reasonable effort to minimize any inconvenience to OWNER during its Sampling on the Property, to return the Property to the condition it was in at the time Atlantic Richfield first entered the Property under this Agreement, and to consult with OWNER to address any concerns OWNER may have about the Sampling activity.

3. SPLIT SAMPLE. Atlantic Richfield agrees to use its best efforts to provide, upon OWNER's prior written request a portion of any sample taken on OWNER's Property for subsequent laboratory analysis, provided that a sufficient quantity of the materials to be sampled are available on the day of sampling, and provided further that the sampling requirements of Atlantic Richfield are satisfied.

4. TERMINATION. This Access Agreement will terminate thirty (30) days following receipt of the written notice from Atlantic Richfield stating the Sampling activities on your Property have been completed.

IN WITNESS WHEREOF, OWNER and Atlantic Richfield Company have executed this Agreement effective as of the date first written above.

OWNER:

By: _____

Title:

_____

Telephone Contact No. _____

ATLANTIC RICHFIELD COMPANY

By: _____

Title: Liability Manager

EXHIBIT A

For the purposes of this Access Agreement, the term Property refers to the following described real estate, situated in the County of Silver Bow, State of Montana:

Name	Geocode	Legal Description

APPENDIX D LEVEL A/B FIELD DATA VERIFICATION CHECKLIST

ENVIRONMENTAL STANDARDS
Setting the Standards for Innovative Environmental Solutions

LEVEL A/B FIELD DOCUMENTATION SCREENING REVIEW

SILVER BOW CREEK/BUTTE AREA NATIONAL PRIORITIES LIST SITE, BUTTE PRIORITY SOILS OPERABLE UNIT, RESIDENTIAL METALS ABATEMENT PROGRAM PROJECT

DUST AND DIRT SAMPLES COLLECTED ON

November __, 2021

RESIDENT IDENTIFICATION: S-00XX

SAMPLE DELIVERY GROUPS:

DATE

Prepared for:

ATLANTIC RICHFIELD COMPANY
317 Anaconda Road
Butte, MT 59701

Prepared by:

ENVIRONMENTAL STANDARDS, INC.
1140 Valley Forge Road
P.O. Box 810
Valley Forge, PA 19482-0810

INTRODUCTION

This quality assurance (QA) review of field documents is based upon an examination of the data generated during the collection of the field samples on DATE, as part of the Silver Bow Creek/Butte Area National Priorities List (NPL) Site, Butte Priority Soils Operable Unit, Residential Metals Abatement Program (RMAP) sampling event. This review was performed using guidance from Section 5 of the Draft Residential Metals Abatement Program Quality Assurance Project Plan (Non-Residential Parcels) Addendum, _____, 2021

The Level A/B review is documented on the checklist below as described in the CFRSSI Data Management/Data Validation (DV/DM) Plan (ARCO, 1992a) and the CFRSSI DM/DV Plan Addendum (AERL, 2000), and will be used in the verification process for field documentation related to samples collected for laboratory analyses.

Data that meet the Level A and Level B criteria and are not qualified as estimated or rejected during the analytical 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 determination of enforcement quality data and screening quality data will be made in conjunction with the data validation report and qualified based on the requirements of Section 5.1.2.1 of the QAPP. Identification of enforcement, screening or unusable data will be added to the electronic data deliverables.

SECTION 1 LEVEL A/B FIELD DOCUMENTATION SCREENING REVIEW

1. General Information

Site:
Project:
Client:
Sample Matrix:

2. Screening Result

Data are:

- Unusable ☐
- Level A ☐
- Level B ☐

3. Level A Criteria: The following must be fully documented

Criteria		Comments
Sampling date	Yes ☐ No ☐	Recorded in Logbook ☐ COC ☐ Bottle Labels ☐
Sampling team or leader name	Yes ☐ No ☐	Recorded in Logbook ☐ COC ☐
Physical description of sampling location	Yes ☐ No ☐	Recorded in Logbook ☐ Field Forms ☐ Photo Log ☐
Sample collection depth (soils)	Yes ☐ No ☐	Recorded in Logbook ☐ Field Forms ☐
Sample collection technique	Yes ☐ No ☐	Collected in accordance with the SOPs in attachment C-1 of QAPP Yes ☐ No ☐
Field preparation technique	Yes ☐ No ☐	Collected in accordance with the SOPs in attachment C-1 of QAPP Yes ☐ No ☐
Sample preservation technique	Yes ☐ No ☐	Soils for mercury analysis submitted on ice? Yes ☐ No ☐ Soils for lead and arsenic submitted at ambient temperature? Yes ☐ No ☐
Sample shipping records	Yes ☐ No ☐	Did sample arrive at < 6°C but not frozen (mercury analysis)? Yes ☐ No ☐ _____ Reported (corrected) temperature

4. Level B Criteria – The following must be fully documented.

Criteria		Comments
Field instrumentation methods and standardization complete.	Yes ☐ No ☐	Field equipment calibrated if used? Yes ☐ No ☐
Sample container preparation	Yes ☐ No ☐	Unpreserved bottles provided by laboratory and lot number tracked? Yes ☐ No ☐
Collection of field duplicates (1/20 minimum)	Yes ☐ No ☐	
Sampling equipment decontamination	Yes ☐ No ☐	Dedicated sampling equipment decontaminated per QAPP Yes ☐ No ☐
Field custody documentation	Yes ☐ No ☐	COC complete and signed (performed during SCUR review) Yes ☐ No ☐
Shipping custody documentation	Yes ☐ No ☐	Custody Seals applied to sample shipment cooler (performed during SCUR review) Yes ☐ No ☐ Custody Seals intact (performed during SCUR review) Yes ☐ No ☐
Traceable sample designation number	Yes ☐ No ☐	Sample IDs in Logbook match COC? Yes ☐ No ☐
Field logbook(s), custody records in secure repository	Yes ☐ No ☐	All notes are complete in a PDF Yes ☐ No ☐ Secure repository under RMAP protocols
Completed field forms	Yes ☐ No ☐	Are field forms, complete, legible, and signed? Yes ☐ No ☐

5. Authorization of Field Documentation Screening Review

Report prepared by: NAME, Senior Consulting Geoscientist
Report reviewed by: Lester J. Dupes, CEAC, CQA, Senior Quality Assurance Chemist
Report approved by: Rock J. Vitale, CEAC, Technical Director of Chemistry/Principal
Date: DATE

SECTION 2 ENFORCEMENT/SCREENING DEFINITIONS

E Enforcement quality. No qualifiers, U qualifier or J qualifier (see note below) and meets Level A and B criteria.

S Screening quality. 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.

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 MDL and 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 is between the MDL and RL, values are considered enforcement data if no other qualifiers were required during validation.

SECTION 3

ERM FIELD DATA SUPPORT DOCUMENTATION

APPENDIX E EXAMPLE RESULT LETTER TEMPLATES

ATTACHMENT E

EXAMPLE RESULT LETTER TEMPLATES

ATTACHMENT E1

EXAMPLE NO ACTION RESULT LETTER

Atlantic Richfield Company

317 Anaconda Road
Butte, MT 59701
Main: (406) 723-1822

[Insert Date]

[Insert Contact Name]
[Insert Entity/School/Daycare Name]
[Insert Number & Street Name]
Butte, MT 59701

Dear [Insert Contact Name]:

This letter is in response to Residential Metals Abatement Program (RMAP) interior dust and soil sampling activities conducted by Atlantic Richfield Company on your property. Dust and soil sampling was conducted pursuant to the Silver Bow Creek/Butte Area National Priorities List (NPL) Site, Butte Priority Soils Operable Unit (BPSOU) Unilateral Administrative Order(UAO) Amendment issued by the U.S. Environmental Protection Agency (EPA) in August 2020 (UAO Amendment) and under the direct supervision of the EPA. On behalf of the EPA and Atlantic Richfield Company, we would like to provide you the results from the sampling that was conducted on your property.

The arsenic, lead, and mercury concentrations for interior dust samples and soil collected from your property are attached to this letter. Your results are below the action levels established by the EPA for RMAP soils within the Silver Bow Creek/Butte AreaNPL Site. Therefore, further sampling or remediation is not required on your property.

We would like to thank you for your cooperation during this effort. If you have any questions or require further explanation concerning the above information, please give me a call at the number listed below. Alternatively, you may also call Nikia Greene with the EPA (406-457-5019) or Daryl Reed with the MDEQ (406-444-6433) with any questions or concerns.

Sincerely,

Mike McAnulty

Mike Mc Anulty
Liability Manager
Remediation Management Services Company
An affiliate of Atlantic Richfield Company
(406) 723-1822

Attachment: Analytical Soil Sampling Results

cc: Nikia Greene/EPA
Daryl Reed/MDEQ

File: MiningSharePoint@bp.com

ANALYTICAL RESULTS FROM SOIL SAMPLING
CONDUCTED ON YOUR PROPERTY

Geocode:

Physical Address: No Physical Address

Legal Description:

School ID: S-0001

[Insert Sampling Summary Table]

Component Arsenic Concentration is ≥ 250mg/kg.
Component Lead Concentration is ≥ 1,200mg/kg.
Component Mercury Concentration is ≥ 147mg/kg.
N/A = Not applicable per 2021 RMAP Quality Assurance Project Plan.

EPA Action Levels to Determine the Need for Additional Testing or Remediation in RMAP Soils:

Arsenic: Any Component ≥ 250 ppm

Lead: Any Component ≥ 1,200 ppm

Mercury: Any Component ≥ 147 ppm

Definitions of words and abbreviations used above:

COMPONENT CONCENTRATION - The concentration of arsenic, lead, or mercury within a sampling component at a given location.

PARTS PER MILLION (PPM) – Parts per million, an expression of concentration. A good analogy: If you had 20ppm, it would be like having 20 white marbles and 999,980 black marbles in a group of 1,000,000 total marbles.

N/A – Not applicable per the 2021 RMAP Quality Assurance Project Plan (QAPP) Addendum

ATTACHMENT E2

EXAMPLE REMEDIAL ACTION RESULT LETTER

317 Anaconda Road
Butte, MT 59701
Main: (406) 723-1822

Atlantic Richfield Company

[Insert Date]

[Insert Contact Name]
[Insert Entity/School/Daycare Name]
[Insert Number & Street Name]
Butte, MT 59701

Dear [Insert Contact Name]:

This letter is in response to Residential Metals Abatement Program (RMAP) interior dust and soil sampling activities conducted by Atlantic Richfield Company on your property. Dust and soil sampling was conducted pursuant to the Silver Bow Creek/Butte Area National Priorities List (NPL) Site, Butte Priority Soils Operable Unit (BPSOU) Unilateral Administrative Order (UAO) Amendment issued by the U.S. Environmental Protection Agency (EPA) in August 2020 (UAOAmendment) and under the direct supervision of the EPA. On behalf of the EPA and Atlantic Richfield Company, we would like to provide you the results from the sampling that was conducted on your property.

You will see that one or more of the samples contained arsenic, lead, or mercury above the Residential Metals Abatement Program (RMAP) soil action levels established by the U.S. Environmental Protection Agency (EPA) for this area. EPA has determined that such dust or soil should be removed from the surface of your property.

This letter describes the work that is proposed for your property and asks you for permission to complete that work at Atlantic Richfield Company's expense. The proposal is described in more detail below, and in the proposed access agreement and work plan attached to this letter.

Sample Results

Indoor dust sampling was conducted pursuant to the Silver Bow Creek/Butte Area National Priorities List (NPL) Site, Butte Priority Soils Operable Unit (BPSOU) Unilateral Administrative Order (UAO) Amendment issued by the U.S. Environmental Protection Agency (EPA) in August 2020(UAO Amendment) and under the direct supervision of the EPA.

The arsenic, lead, and mercury concentrations for interior dust and soil samples collected from your property are attached to this letter. Your sample results, which have been reviewed and approved by EPA, indicate that the concentrations of arsenic, lead, and/or mercury detected within your property exceed the RMAP soil action level(s) established by EPA within the Silver Bow Creek/Butte Area National Priorities List (NPL) Site. Therefore, some or all of your property is eligible for interior dust remediation.

Proposed Remedy and Access Agreement

Atlantic Richfield Company requests your permission to complete the interior dust remedy that EPA has selected for your property, at Atlantic Richfield's own expense. In order to move forward with dust remediation on your property, you will need to provide us with an access agreement that allows us to complete that work.

An Individual Site Work Plan (ISWP) for your property is attached as Exhibit B to the Access Agreement. The ISWP, which also has been approved by EPA, describes the details of the dust remediation work proposed for your property.

Next Steps

Atlantic Richfield respectfully asks that you review the attached Access Agreement and ISWP. If you concur with these documents and would like to proceed with the proposed dust remediation, please sign the Access Agreement. If you return the fully executed Access Agreement to me in the enclosed self-addressed stamped envelope, I will countersign the Access Agreement and provide you with a copy for your records. Once we receive your executed Access Agreement, we will contact you to schedule the remediation work.

We would like to thank you for your cooperation during this effort. If you have any questions or would like further explanation concerning the above, please call me at **406-723-1822**.

Sincerely,

Mike McAnulty

Mike Mc Anulty
Liability Manager
Remediation Management Services Company
An affiliate of Atlantic Richfield Company

Attachments: Analytical Soil Sampling Results
Construction Access Agreement
Individual Site Work Plan (ISWP)

cc: Nikia Greene/EPA
Daryl Reed/MDEQ

File: MiningSharePoint@bp.com

ANALYTICAL RESULTS FROM DUSTS
SAMPLING CONDUCTED ON YOUR PROPERTY

Geocode:

Physical Address:

Legal Description:

School ID: S-0001

[Insert Interior Dust Sample Result Summary Table]

Component Arsenic Concentration is ≥ 250 mg/kg.

Component Lead Concentration is ≥ 1,200 mg/kg.

Component Mercury Concentration is ≥ 147 mg/kg.

N/A = Not applicable per 2021 RMAP Quality Assurance Project Plan.

EPA Action Levels to Determine the Need for Additional Testing or Remediation in RMAP Soils:

Arsenic: Any Component ≥ 250 ppm

Lead: Any Component ≥ 1,200 ppm

Mercury: Any Component ≥ 147 ppm

Definitions of words and abbreviations used above:

COMPONENT CONCENTRATION - The concentration of arsenic, lead, or mercury within a sampling component at a given depth interval.

PARTS PER MILLION (PPM) – Parts per million, an expression of concentration. A good analogy: If you had 20ppm, it would be like having 20 white marbles and 999,980 black marbles in a group of 1,000,000 total marbles.

N/A – Not applicable per the 2021 RMAP Quality Assurance Project Plan (QAPP) Addendum

ACCESS AGREEMENT

Entity/School/Daycare Owner ("Owner") and **Atlantic Richfield Company** ("Atlantic Richfield") enter into this Access Agreement ("Agreement") this _____day of_____, 2021.

1. Atlantic Richfield is conducting certain remedial activities on properties in and near Butte.

2. Access to property owned by Owner and as described in Exhibit A is needed to conduct this remedial work.

3. Owner agrees to permit Atlantic Richfield to conduct such work on Owner's property.

Therefore, in the mutual interest of Owner and Atlantic Richfield, Owner and Atlantic Richfield further agree as follows:

1. GRANT OF ACCESS. Owner hereby grants to Atlantic Richfield, Environmental Protection Agency ("EPA") and the State of Montana ("State"), including the authorized representatives of each, the right to enter Owner's real property described in Exhibit A hereto (the "Property"), to conduct all activities described in the Individual Site Work Plan attached as Exhibit B hereto, including without limitation, removal of interior/attic dust, monitoring and sampling (or to receive split samples) of environmental media, ingress and egressof equipment, machinery and personnel, staging and temporary storage of equipment, and conducting other information gathering activities such as investigation, data collection, surveys and testing (collectively referred to as "Work"). Owner warrants and represents to AtlanticRichfield that, to the best of Owner's knowledge, Owner possesses ownership interests in the Property sufficient to grant access to Atlantic Richfield to conduct the Work. Atlantic Richfield shall provide Owner, either in writing or verbally, with at least 24 hours notice prior to first commencing the Work on the Property. Atlantic Richfield will make every reasonable effort to minimize any inconvenience to Owner during its Work on the Property, and will work closely withOwner to address any concerns Owner may have about the Work.

2. INDEMNIFICATION OF OWNER. Atlantic Richfield agrees to indemnify and hold harmless Owner from any and all actions, claims, damages, losses, liabilities, or expenses, including damage to property or for loss of use of property ("Liabilities"), which may be imposed on or incurred by Owner as a result of Atlantic Richfield's negligent, wrongful acts or omissions while on the Property to conduct the Work, except to the extent that such liabilities result from the acts or omissions of Owner. Provided that the Work is conducted without negligence or wrongful acts or omissions by Atlantic Richfield, Owner and Atlantic Richfield agree that the Work conducted pursuant to this Agreement shall not give rise to a claim for indemnification under this provision.

3. NOTICE. All written notices pertaining to this Agreement shall be sent to Owner and Atlantic Richfield at the respective addresses below. Either Owner or Atlantic Richfield may

designate a different address for receipt of notice by providing written notice of such change to the other.

TO Atlantic Richfield: Mike Mc Anulty
317 Anaconda Road
Butte, MT 59701
(406) 723-1822

TO OWNER: [Insert Entity/School/Daycare Name]
[Insert Number & Street]
BUTTE, MT 59701

4. CONDITION OF THE PROPERTY. If the Work entails the removal of earthen basement soil and/or the removal of interior dust, Atlantic Richfield may photograph the Property prior to and upon completion of the removal of soil or dust to document and obtain a fair and accurate representation of the condition of the Property.

5. RESTORATION OF PROPERTY. Upon completion of the Work, Atlantic Richfield will use its best efforts to return the Property to the condition it was in at the time Atlantic Richfield first entered the Property under this Agreement, provided such restoration is not inconsistent with the Work conducted pursuant to this Agreement.

6. MISCELLANEOUS.

a. Effect of Agreement. This Agreement and the rights and obligations created hereby shall be binding upon and inure to the benefit of Owner and Atlantic Richfield and their respective assigns and successors in interest.

b. Negation of agency relationship. This Agreement shall not be construed to create, either expressly or by implication, the relationship of agency or partnership between Owner and Atlantic Richfield. Neither Owner nor Atlantic Richfield is authorized to act on behalf of the other in any manner relating to the subject matter of this Agreement.

c. Termination. Except with respect to paragraphs 2, 3 and 6.a of this Agreement, this Agreement will terminate thirty (30) days following Atlantic Richfield's written notification to Owner that the Work is complete.

d. Governing Law. This Agreement shall be governed by and construed in accordance with the laws of the State of Montana.

e. Construction. The invalidity or unenforceability of any provision of this Agreement shall not affect the validity or enforceability of any other provision.

f. Entire Agreement. This Agreement embodies the entire agreement of Owner and Atlantic Richfield with respect to the subject matter hereof, and no prior oral or written representation shall serve to modify or amend this Agreement. This Agreement may be modified only by a written agreement signed by Owner and Atlantic Richfield.

IN WITNESS WHEREOF, Owner and Atlantic Richfield have executed this Agreement effective as of the date first written above.

OWNER	Atlantic Richfield Company
[Insert Entity/School/Daycare Owner]	
By:_____	By: _____
Title (If other than Owner): _____	Title: Liability Manager_____
Telephone Contact No. _____	

EXHIBIT A
(Legal Description of the Property)

For the purposes of this Access Agreement, the term Property refers to the following described real estate, situated in the County of Silver Bow, State of Montana:

Name	Geocode	Legal Description

EXHIBIT B
(Individual Site Work Plan)

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FOR THIS TEMPLATE

APPENDIX F CORRECTIVE ACTION REPORT

RMAP CORRECTIVE ACTION REQUEST			
Number:		**Date:**	
To: _____ You are hereby requested to take corrective actions indicated below and as otherwise determined by you to resolve the noted conditions and to prevent recurrence. Your written response is to be returned to the QAM by _____.			
Condition:			
Reference Documents:			
_____ Originator	_____ Date	_____ QAM	_____ Approval Date

RMAP CORRECTIVE ACTION REQUEST - RESPONSE

Number: **Date:**

Response

Cause of Condition:

Corrective Action

(A) Resolution:

(B) Prevention:

(C) Affected Documents:

Signature:_____ Date_____

CA Follow-up:

Corrective Action verified by:_____ Date _____

CA Approval and Closure:

Corrective Action approved and closed by QAM:_____Date _____

APPENDIX G DATA VALIDATION CHECKLIST

ENVIRONMENTAL STANDARDS
Setting the Standards for Innovative Environmental Solutions

STAGE 2B/4 QUALITY ASSURANCE REVIEW

SILVER BOW CREEK/BUTTE AREA NATIONAL PRIORITIES LIST SITE, BUTTE PRIORITY SOILS OPERABLE UNIT, RESIDENTIAL METALS ABATEMENT PROGRAM PROJECT

DUST AND DIRT SAMPLES COLLECTED ON

XXXX __, 2021

RESIDENT IDENTIFICATION: S-00XX

SAMPLE DELIVERY GROUP: XXXXXXXX

DATE

Prepared for:

ATLANTIC RICHFIELD COMPANY
317 Anaconda Road
Butte, MT 59701

Prepared by:

ENVIRONMENTAL STANDARDS, INC.
1140 Valley Forge Road
P.O. Box 810
Valley Forge, PA 19482-0810

TABLE OF CONTENTS

INTRODUCTION

This quality assurance (QA) review is based upon an examination of the data generated from the analyses of the samples collected on DATE, as part of the Silver Bow Creek/Butte Area National Priorities List (NPL) Site, Butte Priority Soils Operable Unit, Residential Metals Abatement Program (RMAP) sampling event. The samples that have undergone a rigorous QA review are listed on Table 1. Table 1 also presents the laboratory sample number, collection date, matrix, parameter(s) examined, and the review level for each sample. Stage 2B review includes an evaluation of data package completeness and review of the summary forms provided (raw data are not reviewed). In addition to all the elements included in a Stage 2B review, a Stage 4 review includes the evaluation of raw data and the verification of calculated results.

This review was performed with guidance from the RMAP Quality Assurance Project Plan (QAPP; June 2021); the "Guidance for Labeling Externally Validated Laboratory Analytical Data for Superfund Use," (US EPA, January 2009); and the "National Functional Guidelines for Inorganic Superfund Methods Data Review," (US EPA, January 2020). The National Functional Guidelines validation guidance documents specifically address analyses performed in accordance with the Contract Laboratory Program (CLP) analytical methods and are not completely applicable to the type of analyses and analytical protocols performed for the SW-846 methods utilized by the laboratory for these samples. Environmental Standards, Inc. (Environmental Standards) used professional judgment to determine the usability of the analytical results and compliance relative to the methods utilized by the laboratory.

The reported analytical results are presented as qualified electronic data deliverables (EDDs). Any required data validation qualifications have been annotated on the associated EDDs. Data were examined to determine the usability of the analytical results and compliance relative to the method requirements specified in "Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, 3rd Edition" (SW-846) Method 6020A and 7471B. This report was prepared to provide a critical review of the laboratory analyses and reported analytical results. Rigorous QA reviews of laboratory-generated data routinely identify problems associated with analytical measurements, even from the most experienced and capable laboratories. The data qualifications allow the data end-user to best understand the usability of the analytical results. Data not qualified in this report should be considered valid based on the quality control (QC) criteria that have been reviewed and be considered enforcement quality if the data also passed Level A and Level B field documentation quality assessment as detailed in the QAPP. Details of this QA review are presented in Section 1 of this report.

TABLE 1

SAMPLES INCLUDED IN THIS QUALITY ASSURANCE REVIEW

Field Sample Name	Laboratory Sample Number(s)	Sample Delivery Group	Collection Date	Parameter(s) Examined

TABLE 1 (Cont.)

NOTES:

M - Total Lead and Arsenic by SW-846 Method 6020A.
Hg - Total Mercury by SW-846 Method 7471B.

SECTION 1 QUALITY ASSURANCE REVIEW

The dust and dirt samples were collected on DATE, as part of the Silver Bow Creek/Butte Area NPL Site, Butte Priority Soils Operable Unit, RMAP sampling event. The samples for the analysis of lead, arsenic and mercury were shipped to Pace Analytical Services, LLC (Pace) of Minneapolis, Minnesota, for digestion and analysis by inductively coupled plasma/mass spectrometry (ICP/MS) SW-846 Method 6020A and Cold Vapor Atomic Absorption (CVAA) SW-846 Method 7471B. The specific samples and analyses reviewed are identified on Table 1.

The findings in this QA review are based upon a review of sample holding times, condition of samples upon laboratory receipt, blank analysis results, laboratory matrix spike sample (LMS) results, laboratory control sample (LCS) results, laboratory and field duplicate results, initial and continuing calibrations, sample preparation, reporting limit (RL) standard results, interference check sample results, post-digestion spike results, serial dilution results, internal standard performance, instrument sensitivity, analytical sequence, the quantitation of positive results, and a critical evaluation of instrumental raw data. Any required data validation qualifications are annotated in the qualified EDD as defined in Section 3.

Issues are typically presented in two categories – deliverable issues and procedural issues. Deliverable issues are data issues that can easily be corrected and that may or may not impact the usability of the reported results. Procedural issues are issues that cannot be corrected and address method compliance issues; these issues may or may not impact the usability of the reported results. Comments address issues for which the data reviewer has provided information in order to clarify issues relating to the data; comments do not typically impact the usability of the reported results. The data reviewer has edited the laboratory-reported data and QC summary forms based on the issues and comments in this QA review. Furthermore, the data reviewer has included copies of all relevant raw data, QC forms, and other documentation needed to support these edits in the Inorganic Data Support Documentation (Section 4) of this report.

Deliverable Review

- Deliverable issues were not observed for the data in this QA review.

Procedural Review

- Procedural issues were not observed for the data in this QA review.

Comments

With regard to data usability, the principal areas of concern are LIST. Based upon a complete review of the data package provided, the following qualifiers are offered. The following data usability issues represent an interpretation of the QC results obtained for the project samples. Quite often, data qualifications address issues relating to sample matrix problems. Similarly, the data validation guidelines routinely specify areas of the data that require qualification, yet the

methods used for analysis may not require corrective action by the laboratory. Accordingly, the following data usability issues should not be construed as an indication of laboratory performance.

OR

Based upon a complete review of the data package provided, qualification of data was not warranted. Accordingly, the lack of data usability issues should not be construed as an indication of laboratory performance.

SECTION 2 DATA VALIDATION CHECKLIST FOR METALS SAMPLE ANALYSIS

1. Holding Times

Analyte	Laboratory	Matrix	Method	Holding Times*	Collection Date(s)	Batch(es)	Analysis Date(s)	Holding Time Met (Y/N)	Affected Data Flagged (Y/N)
Lead and Arsenic	Pace – Minneapolis, MN	Dust and Dirt	SW-846 Method 6020A	6 months from sample collection				Y	N/A
Mercury	Pace – Minneapolis, MN	Dust and Dirt	SW-846 Method 7471B	28 days from sample collection				Y	N/A

*Reference for Holding Times – Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, 3rd Edition" (SW-846) Methods 6020A and 7471B and Chapter 3

Were any data flagged because of holding time? Yes ☐ No ☐
Were any data flagged because of preservation problems? Yes ☐ No ☐

Describe Any Actions Taken: No actions were required.

Comments: Qualification of data was not warranted.

2. Instrument Calibration

Was the Tune analysis performed? Yes ☐ No ☐
Were the peak widths and resolution of the masses within the required control limits?
Yes ☐ No ☐
Was the percent relative standard deviation ≤ 5% for all analytes in the Tune solutions?
Yes ☐ No ☐
Was the Instrument successfully calibrated at the correct frequency? Yes ☐ No ☐
Was the Instrument calibrated with appropriate standards and blanks? Yes ☐ No ☐
Were Initial Calibration Verification (ICV) and Continuing Calibration Verification (CCV) samples analyzed? Yes ☐ No ☐
Were ICV and CCV results within the control window? Yes ☐ No ☐
Were any data flagged because of calibration problems? Yes ☐ No ☐

Describe Any Actions Taken: No actions were required.

Comments: Qualification of data was not warranted.

OR

Samples were reanalyzed under a valid ICV/CCV.

3. Blanks

Were Initial and Continuing Calibration Blanks (ICB and CCBs) analyzed? Yes ☐ No ☐
Were ICBs and CCBs within the control window? Yes ☐ No ☐
Were Method Blanks (MBs) analyzed at the frequency of 1 per analytical batch? Yes ☐ No ☐
Were MBs within the control window? Yes ☐ No ☐
Were any data flagged because of blank problems? Yes ☐ No ☐

Describe Any Actions Taken: No actions were required.

OR

Analyte	SDG	Sample(s) with Blank Qualified Results ("U")

Comments: Qualification of data was not warranted.

.

4. Interference Check Samples

Were ICP/MS Interference Check Samples (ICS) within the control limits? Yes ☐ No ☐
Were any data flagged because of ICS problems? Yes ☐ No ☐

Describe Any Actions Taken: No actions were required.

Comments: Information provided in the data package(s) was insufficient to permit assessment of the potential for molecular or other interferences or the adequacy of corrections for such interferences. The fact that the analysis was performed with an instrument that includes collision cell technology reduces the likelihood of significant interference if one or more of the potentially interfering elements were present. The data user should consider this information when determining the ultimate use of the reported results.

5. Laboratory Control Samples

Were Laboratory Control Samples (LCS) analyzed at the frequency of 1 per batch?
Yes ☐ No ☐
What was the source of the LCS?
Metals: Lot Number
Mercury: Lot Number
Were LCS results within the control window? Yes ☐ No ☐
Were any data flagged because of LCS problems? Yes ☐ No ☐

Describe Any Actions Taken: No actions were required.

OR

Analyte	SDG	Sample(s) with Estimated Results ("J-")

Analyte	SDG	Sample(s) with Estimated Results ("J+")

Analyte	SDG	Sample(s) with Estimated Results ("J-/UJ")

Comments: Qualification of data was not warranted.

OR

Qualification

The RLs for the analytes in the samples listed above may be higher than reported, and the "not-detected" results have been flagged "UJ" in the qualified EDD. In addition, the reported positive results for the analytes in the samples listed above should be considered estimated, biased low, and have been flagged "J-" in the qualified EDD. Low recoveries (< 80%) were observed in the associated LCS analyses.

6. Duplicate Sample Results

Were Laboratory Duplicate Samples (LDS) analyzed at the frequency of 1 per batch?
Yes ☐ No ☐
Were LDS results within the control window? Yes ☐ No ☐
Were any data flagged because of LDS problems? Yes ☐ No ☐

Describe Any Actions Taken: No actions were required.

OR

Analyte	SDG	Sample(s) with Estimated Results ("J")

Comments: Qualification of data was not warranted.

OR

7. Matrix Spike/Matrix Spike Duplicate/Post Digestion Spike Sample Results

Were LMS analyzed at the frequency of 1 per batch? Yes ☐ No ☐
Were LMS percent recovery (%R) results within the control window? Yes ☐ No ☐
Were any data flagged because of LMS problems? Yes ☐ No ☐
Was a Post Digestion Spike (PDS) performed? Yes ☐ No ☐
Were PDS percent recovery (%R) results within the control window? Yes ☐ No ☐
Were any data flagged because of PDS problems? Yes ☐ No ☐

Describe Any Actions Taken: No actions were required.

OR

Analyte	SDG	Sample(s) with Estimated Results ("J-")

Analyte	SDG	Sample(s) with Estimated Results ("J+")

Analyte	SDG	Sample(s) with Estimated Results (“J+”)

Analyte	SDG	Sample(s) with Estimated Results (“J-/UJ”)

Analyte	SDG	Sample(s) with Estimated Results (“J”)

Comments: Qualification of data was not warranted.

OR

8. ICP/MS Serial Dilutions

Were ICP/MS Serial Dilutions (SD) analyzed at the frequency of 1 per batch? Yes ☐ No ☐
Were SD percent differences (%D) results within the control window? Yes ☐ No ☐
Were any data flagged because of SD problems? Yes ☐ No ☐

Describe Any Actions Taken: No actions were required.

OR

Analyte	SDG	Sample(s) with Estimated Results (“J”)

Comments: Qualification of data was not warranted.

OR

9. Internal Standards

Were internal standards added to each sample in the analytical batch? Yes ☐ No ☐
Were the percent relative recoveries (%RI) within the control window? Yes ☐ No ☐
Were any data flagged because of internal standard problems? Yes ☐ No ☐

Describe Any Actions Taken: No actions were required.

Comments: Qualification of data was not warranted.

10. Field Blanks

Were field blanks submitted as specified in the Sampling Analysis Plan (SAP)?
Yes ☐ No ☐ N/A ☒
Were field blanks within the control window? Yes ☐ No ☐ N/A ☒
Were any data qualified because of field blank problems? Yes ☐ No ☐ N/A ☒

Describe Any Actions Taken: No actions were required.

Comments: Qualification of data was not warranted.

11. Field Duplicates

Were field duplicates submitted as specified in the Sampling Analysis Plan (SAP)?
Yes ☐ No ☐ N/A ☐
Were the field duplicates within the control window? Yes ☐ No ☐ N/A ☐
Were any data qualified because of field duplicate problems? Yes ☐ No ☐ N/A ☐

Describe Any Actions Taken: No actions were required.

OR

Analyte	SDG	Sample(s) with Estimated Results ("J")

Comments: Qualification of data was not warranted.

OR

12. Overall Assessment

Are there analytical limitations of the data that users should be aware of? Yes ☐ No ☐

If so, explain:

Comments:

- Data that meet the Level A and Level B criteria in the field documentation quality assessment as detailed in the QAPP, and not qualified as estimated or rejected during the data validation process, are considered 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 considered screening-quality data in accordance with Section 5.3 of the QAPP. Level A and Level B acceptance of these data are documented in a separate report.

- Reported positive results between the MDL and the RL should be considered estimated and have been flagged "J" in the qualified EDD. It is appropriate to note that sample results qualified as estimated "J" by the laboratory because the reported result is between the MDL and RL, values are considered enforcement-quality data if no other qualifiers were required during validation.

- When sample results were qualified both as estimated with a direction of bias ("J+" or "J-") and as estimated with unknown bias ("J") or the opposite bias, only the unknown bias qualifier has been included in the qualified EDD.

Complete support documentation for this inorganic QA review is presented in Section 4 of this report. The cover sheet for this section is a checklist of all QA procedures required by the protocol and examined in this data review.

The analytical data completeness (defined as the percentage of usable data) for the samples included in this QA review is XX%.

13. Authorization of Data Validation

Report prepared by: NAME, Quality Assurance Chemist
Report reviewed by: NAME, Senior Quality Assurance Chemist
Report approved by: Lester J. Dupes, CEAC, CQA, Senior Quality Assurance Chemist
Report approved by: Rock J. Vitale, CEAC, Technical Director of Chemistry/Principal
Date: DATE

SECTION 3 DATA VALIDATION QUALIFIER DEFINITIONS

U	The result is qualified as non-detect due to the detection of the analyte in an associated QC blank.
J	The analyte was positively identified; the associated numerical value is an estimate of the concentration of the analyte in the sample. This will also include results reported between the MDL and RL.
J+	The result is an estimated quantity, but the result may be biased high.
J-	The result is an estimated quantity, but the result may be biased low.
UJ	The analyte was not detected above the sample reporting limit. However, the reporting limit is approximate and may or may not represent the actual limit of quantitation necessary to accurately and precisely measure the analyte in the sample.
R	The sample results are rejected due to serious deficiencies in the ability to analyze the sample and meet quality control criteria. The presence or absence of the analyte cannot be verified.
No Flag	Result accepted without qualification.

RMAP REASON CODES

1	Holding time violation
2	Method blank contamination
3	Surrogate recovery
4	Matrix spike/matrix spike duplicate recovery
5	Matrix spike/matrix spike duplicate precision outside limits
6	Laboratory control sample recovery
7	Field blank contamination
8	Field duplicate precision outside limits
9	Other deficiencies (including cooler temperature)
A	Absence of supporting QC
S	ICV, CCV, or column performance check problem
Y	Initial and continuing calibration blank problem
M	Interference check samples problem
O	Post-digestion spike outside of 75-125%
F	MSA correlation coefficient < 0.995, or MSA not done
G	Serial dilution problem
K	DFTPP or BFB tuning problem
Q	Initial calibration problem
X	Internal standard recovery problem
V	Second-source standard calibration verification problem
L	Low bias
Z	Retention time problem
N	Counting time error (radionuclide chemistry)
W	Detector instability (radionuclide chemistry)
C	Co-elution of compounds
E	Value exceeds linear calibration range
I	Interferences present during analysis
T	Trace-level compound, poor quantitation
P	1C/2C precision outside of limits
B	LCS/LCSD precision outside limits
D	Lab Dup/Rep precision outside limits
H	High Bias

SECTION 4

INORGANIC DATA SUPPORT DOCUMENTATION

SECTION 5

LABORATORY CASE NARRATIVE AND

CHAIN-OF-CUSTODY RECORD

SECTION 6

PROJECT CORRESPONDENCE

ERM has over 160 offices across the following countries and territories worldwide

Argentina
Australia
Belgium
Brazil
Canada
Chile
China
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