Atlantic Richfield Company

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February 23, 2016

CERTIFIED – RECEIPT REQUESTED

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RE: ***Community Soils Operable Unit (CS OU) Final Residential Soils/Dust Quality Assurance Project Plan (QAPP)*** **and** ***Final Residential Soils/Dust Quality Assurance Project Plan (QAPP) Crosswalk***

Dear Gentlemen:

On behalf of the Atlantic Richfield Company, please find 2 copies and 2 CDs of the *Final Residential Soils/Dust Quality Assurance Project Plan (QAPP)* with the signed approval page and *Final Residential Soils/Dust Quality Assurance Project Plan (QAPP) Crosswalk* (provided in Attachment G of the QAPP). If you have questions or concerns, please do not hesitate to call me at (406) 723-1832.

Sincerely,

Luke Pokorny
Project Manager
Remediation Management Services Company
An affiliate of Atlantic Richfield Company

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ANACONDA SMELTER NPL SITE
COMMUNITY SOILS OPERABLE UNIT

Final

Residential Soils/Dust
Quality Assurance Project Plan (QAPP)

Atlantic Richfield Company

November 24, 2015

ANACONDA SMELTER NPL SITE
COMMUNITY SOILS OPERABLE UNIT

Final

Residential Soils/Dust
Quality Assurance Project Plan (QAPP)

Prepared for:

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Prepared by:

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November 24, 2015

APPROVAL PAGE

Quality Assurance Project Plan for
CS OU Residential Soils/Dust Remedial Action
Anaconda Smelter NPL Site

Approved: _____ Date: 1/15/16
Charles Coleman, Site Project Manager, EPA, Region 8

Approved: _____ Date: 1-15-16
Joel Chavez, Project Officer, Montana DEQ

Approved: _____ Date: 1/8/2016
Luke Pokorny, Project Manager
Atlantic Richfield Company

Approved: _____ Date: 1/12/2016
Terry Moore, Quality Assurance Manager
Atlantic Richfield Company

Plan is effective on date of approval.

DISTRIBUTION LIST

Anaconda Smelter NPL Site
Quality Assurance Project Plan (QAPP)
Anaconda-Deer Lodge County, Montana

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ED_014362_00000173-00006

TABLE OF CONTENTS

Page

APPROVAL PAGE i

DISTRIBUTION LIST ii

LIST OF FIGURES v

LIST OF TABLES v

LIST OF ATTACHMENTS v

1.0 INTRODUCTION 1

2.0 PROJECT MANAGEMENT 1
- 2.1 Project Organization and Responsibilities 1
- 2.2 Problem Definition and Background 3
- 2.3 Project Description and Schedule 3
- 2.4 Quality Objectives and Criteria 4
  - 2.4.1 Data Quality Objectives 4
  - Step 1: State the Problem 4
  - Step 2: Identify the Decision 6
  - Step 3: Identify the Inputs to the Decision 6
  - Step 4: Define the Study Boundaries 7
  - Step 5: Develop a Decision Rule 7
  - Step 6: Specify Tolerable Limits on Decision Error 8
  - Step 7: Optimize the Design 8
  - 2.4.2 Measurement Performance Criteria for Data 9
- 2.5 Special Training 10
- 2.6 Documents and Records 11
  - 2.6.1 Property Access Agreements 11
  - 2.6.2 Field Logbooks/Data Sheets 11
  - 2.6.3 Field Photographs 13
  - 2.6.4 Chain of Custody Records 13
  - 2.6.5 Analytical Laboratory Records 13
  - 2.6.6 Project Data Reports 13
  - 2.6.7 Quality Records 14

3.0 MEASUREMENT AND DATA ACQUISITION 15
- 3.1 Sampling Process and Design 15
  - 3.1.1 Residential Soils and Alleys Field Sampling Plan 15
    - 3.1.1.1 Standard Operating Procedures for Sampling Activities 16
    - 3.1.1.2 Yard/Lot Maps 16
    - 3.1.1.3 Sample Density, Location and Compositing 17
    - 3.1.1.4 Sample Depths 18

3.1.1.4.1 Residential Soil Samples 18
3.1.1.4.2 Unpaved Alley Samples 19
3.1.1.5 Sample Identification 20
3.1.1.5.1 Residential Soil Sample Identification 20
3.1.1.5.2 Unpaved Alley Soil Sample Identification 21
3.1.2 Attic Dust Field Sampling Plan 21
3.1.2.1 Attic Dust Sampling Eligibility 21
3.1.2.2 Attic Dust Sampling Equipment and Sample Collection 22
3.1.2.3 Attic Dust Sample Identification 22
3.1.2.4 Attic Dust Sample Handling 23
3.1.2.5 Attic Dust Confirmation Sampling 23
3.1.2.5.1 Attic Dust Confirmation Sampling Sample Identification 24
3.2 Laboratory Methods 25
3.2.1 Soil Preparation Methods 25
3.2.2 Soil Metals Analyses Methods 26
3.2.3 Attic Dust Preparation Methods 26
3.2.4 Attic Dust Analyses Methods 26
3.3 Quality Control 26
3.3.1 Field Quality Control Samples 27
3.3.2 Laboratory Quality Control Samples 27
3.4 Instrument/Equipment Testing, Inspection and Maintenance 29
3.4.1 Field Equipment 29
3.4.2 Laboratory Equipment 29
3.5 Inspection/Acceptance of Supplies and Consumables 30
3.6 Data Management Procedures 30

4.0 ASSESSMENT AND OVERSIGHT 31
4.1 Corrective Actions 31
4.2 Corrective Action during Data Assessment 32
4.3 Quality Assurance Reports to Management 32

5.0 DATA REVIEW AND USABILITY 33
5.1 Data Review and Verification 33
5.1.1 Field Data Review 33
5.1.2 Laboratory Data Review 34
5.1.3 Laboratory Data Reporting Requirements 34
5.1.4 Laboratory Electronic Data Deliverable 34
5.1.5 Specific Quality Control/Assessment Procedures 34
5.2 Internal Data Review 35
5.2.1 Field Quality Control Data 35
5.2.2 Laboratory Chemistry Data 35

6.0 REFERENCES 37

ED_014362_00000173-00008

LIST OF FIGURES

Figure 1 CS OU Project Organization Chart
Figure 2 Anaconda-Deer Lodge County (ADLC) Superfund Planning Area Overlay District (SPAOD)
Figure 3 Example Residential Soil Sample Location Map
Figure 4 Sampling and Remedial Action Flow Chart for Residential Soils Not Previously Sampled
Figure 5 Yard Sampling Flow Chart for Component Previously Sampled but not Subject to RA
Figure 6 Yard Sampling Flow Chart for Component Previously Remediated at 0" – 2" Depth
Figure 7 Yard Sampling Flow Chart for Component Previously Remediated at 0" – 6" Depth
Figure 8 Yard Sampling Flow Chart for Component Previously Remediated at 0" – 12" Depth
Figure 9 Sampling and Remedial Action Flow Chart for Attic Dust

LIST OF TABLES

Table 1 CS OU Project Schedule
Table 2 Precision, Accuracy, and Completeness Calculation Equations
Table 3 Example Arsenic Area Weighted Average Calculation

LIST OF ATTACHMENTS

Attachment A Standard Operating Procedures
- Attachment A-1 CS OU Field Standard Operating Procedures
- Attachment A-2 Soil Laboratory Standard Operating Procedures
- Attachment A-3 Attic Dust Laboratory Standard Operating Procedures

Attachment B Technical Memoranda
- Attachment B-1 Technical Memorandum #2 – Modifying the Sample Depth Intervals Used to Make Remedial Decisions for Arsenic and Lead in Yard Soils
- Attachment B-2 Technical Memorandum #1 – Basis for Excluding Living Space Dust and Addressing Only Accessible Attic Dust Through Analysis of the Existing Data
- Attachment B-3 Agency Approval Letter for Technical Memoranda #1 and #2

ED_014362_00000173-00009

Attachment C Sampling Field Data Sheets
Attachment C-1 Residential Yard Sampling Field Data Sheet
Attachment C-2 Unpaved Roads and Alleys Sampling Field Data Sheet
Attachment C-3 Attic Dust In-Home Sampling Data Sheet

Attachment D Lead Dust Sampling Technician Field Guide

Attachment E Corrective Action Report

Attachment F Level A/B Screening Checklist

Attachment G QAPP Crosswalk

REVISION SUMMARY

Revision No.	Author	Version	Description	Date
Rev 0	Jesse Schwarzrock	Draft	Issued for Atlantic Richfield Review	April 1, 2015
Rev 1	Jesse Schwarzrock	Draft Final	Issued for Agency Review	May 1, 2015
Rev 2	Jesse Schwarzrock	Final	Issued for Agency Review	August 7, 2015
Rev 3	Jesse Schwarzrock	Final	Issued for Agency Review	November, 24, 2015

ED_014362_00000173-00010

LIST OF ACRONYMS AND ABBREVIATIONS

ADLC	Anaconda-Deer Lodge County
Atlantic Richfield	Atlantic Richfield Company
AWA	Area-Weighted Average
Bgs	Below Ground Surface
CAR	Corrective Action Report
CCB	Continuing Calibration Blank
CCV	Continuing Calibration Verification
CFRSSI	Clark Fork River Superfund Site Investigations
CLP	Contract Laboratory Program
COC	Constituent of Concern
CPM	Contractor Project Manager
CPMP	Community Protective Measures Program
CS	Community Soils
DEQ	Montana Department of Environmental Quality
DIAR	Data Interpretation and Analysis Report
DPS	Development Permit System
DQA	Data Quality Assessment
DQO	Data Quality Objectives
DSR	Data Summary Report
EDD	Electronic Data Deliverable
EPA	U.S. Environmental Protection Agency
FDR	Final Design Report
GIS	Geographical Information System
GPS	Global Positioning System
GUS	General Utility and Service
HHRA	Human Health Risk Assessment
ICP-AES	Inducted Coupled Plasma Atomic Emission Spectroscopy
ICs	Institutional Controls
ICB	Initial Calibration Blank
ICV	Initial Calibration Verification
IM	Integrity Management
ISWP	Individual Site Work Plan
LaMP	Laboratory Management Program
LCS	Laboratory Control Sample
LCSD	Laboratory Control Sample Duplicate
MDL	Method Detection Limit
mg/kg	milligram per kilogram
MS	Matrix Spike
MSD	Matrix Spike Duplicate
NLLAP	National Lead Laboratory Accreditation Program
NPL	National Priorities List

ED_014362_00000173-00011

OU	Operable Unit
Ppm	parts per million
QA	Quality Assurance
QAPP	Quality Assurance Project Plan
QC	Quality Control
QMP	Quality Management Plan
RA	Remedial Action
RAWP	Remedial Action Work Plan
RD	Remedial Design
RL	Reporting Limit
ROD	Record of Decision
RPD	Relative Percent Difference
RSD	Relative Standard Deviation
SAP	Sampling Analysis Plan
SOP	Standard Operating Procedure
SPAOD	Superfund Planning Area Overlay District
SSHASP	Site-Specific Health and Safety Plan
µm	Micron
$\mu g/ft^2$	micrograms per square foot
°C	degrees Celsius

ED_014362_00000173-00012

1.0 INTRODUCTION

The purpose of this Quality Assurance Project Plan (QAPP) is to provide guidance for residential soil and attic dust sampling and analysis withinthe Community Soils Operable Unit (CS OU) of the Anaconda Smelter National Priorities List (NPL) Site and to describe the Quality Assurance/Quality Control (QA/QC) policies and procedures to be used during data collection and analyses. This QAPP was prepared in a manner consistent with the *EPA Requirements for Quality Assurance Project Plans (QA/R-5)*. (EPA 2001), the *Uniform Federal Policy for Quality Assurance Project Plans: Evaluating, Assessing, and Documenting Environmental Data Collection and Use Programs* (EPA, 2005) and the forthcoming Anaconda Smelter National Priorities List (NPL) Site Quality Management Plan (QMP). This QAPP includes the following four key elements:

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

The sections below provide the project elements and include the appropriate content needed for planning, sampling, and analyses within the site. The sections in this QAPP expand or reference information in other site-wide documents to comply with the *Uniform Federal Policy for Quality Assurance Project Plans: Evaluating, Assessing, and Documenting Environmental Data Collection and Use Programs* (EPA, 2005) and to present project-specific requirements.

2.0 PROJECT MANAGEMENT

This section addresses project administrative functions as well as project background, objectives, and documentation requirements for sampling and analyses activities on the site.

2.1 Project Organization and Responsibilities

An organizational chart showing the overall organization of the project team is provided as Figure 1. Responsibilities of key individuals comprising the project team and their detailed responsibilities are listed below.

<u>Atlantic Richfield Project Manager (Atlantic Richfield PM)</u>
The Atlantic Richfield PM monitors the performance of the contractor(s), consults with the Contractor Project Manager and Contractor QA Officer on deficiencies, and aids in finalizing resolution actions.

<u>Atlantic Richfield Quality Assurance Manager (Atlantic Richfield QA Manager)</u>
The Atlantic Richfield QA Manager interfaces with the Atlantic Richfield PM for company policies regarding quality and has the authority and responsibility to approve QA documents specific to the project including this QAPP. The QA Manager functions independently from the project team generating the data.

Contractor Project Manager (CPM)
The CPM is responsible for scheduling all sampling work to be completed and ensures that the work is performed in accordance with the requirements contained herein. The CPM is also responsible for consulting with the Contractor QA Officer regarding any project deficiencies and resolutions.

Contractor Quality Assurance Officer
The Contractor QA Officer is responsible for verifying effective implementation of QAPP requirements and procedures including reviewing field and laboratory data and evaluating data quality.

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

The Field Team Leader is responsible for facilitating field activities, managing equipment, and problem solving and decision making in the field. The Field Team Leader is responsible for technical aspects of the project and provides "on-the-ground" overview of project implementation by observing site activities to ensure compliance with technical project requirements, and the Site-Specific Health and Safety Plan (SSHASP). The Field Team Leader is responsible for identifying potential Integrity Management (IM) issues during field activities and reporting any and all issues to the Contractor QA Officer.

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

Contract Laboratory
As detailed in the QMP, Atlantic Richfield maintains a Laboratory Management Program (LaMP) that ensures analytical testing service needs are consistently met. The LaMP consists of a network of approved contract laboratories that supply analytical testing services for Atlantic Richfield's environmental and remediation programs. Organizations performing environmental analyses for Atlantic Richfield will demonstrate their qualifications per the LaMP prior to acceptance as a LaMP laboratory. If a laboratory's participation in the LaMP is suspended or revoked at any time during the period of performance, the Atlantic Richfield PM will be notified immediately to ensure any potential impact to the scope of work being performed is addressed accordingly. The contract laboratory's responsibility is to ensure its QA Personnel are familiar with the approved QAPP and are performing the analytical and QC work as specified per laboratory methods and this QAPP. Contract 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.

ED_014362_00000173-00014

2.2 Problem Definition and Background

The CS OU is located in southwest Montana in and around the city of Anaconda. Mining and smelting activities were conducted for nearly 100 years in Anaconda and the surrounding areas, resulting in the contamination of soils, surface water and groundwater. This contamination was spread primarily through airborne emissions and waste disposal practices from smelting operations.

This QAPP was developed in response to the U. S. Environmental Protection Agency's (EPA) and Montana Department of Environmental Quality (DEQ) (the Agencies) 2013 CS OU *Record of Decision (ROD) Amendment* (CS OU ROD Amendment), which amended the original 1996 CS OU ROD (EPA, 1996). The CS OU ROD Amendment (EPA/DEQ, 2013) added a lead action level for residential soils (in addition to the pre-existing arsenic action level) as well as lead and arsenic action levels for attic dust.

This QAPP details both lead and arsenic sampling procedures applicable to residential soils and attic dust within the Anaconda-Deer Lodge County (ADLC) Superfund Planning Area Overlay District (SPAOD– [Figure 2]). For residential soils, this QAPP addresses lead remedial action (RA) on a yard component-specific (e.g., front yard, back yard, garden, etc.) basis and arsenic based primarily on area-weighted average (AWA) calculations.

Due to the additional requirements of the CS OU ROD Amendment (EPA/DEQ, 2013), an updated CS OU Sampling and Analysis Plan (SAP) is needed. This QAPP will function as the CS OU SAP for all future CS OU sampling activities.

2.3 Project Description and Schedule

The purpose of this QAPP is to address arsenic and lead impacts in residential soils and attic dust within the CS OU area of concern (ADLC SPAOD). This investigation will supplement the original CS OU sampling investigation and RA activities that focused on arsenic in residential soils by adding a lead action level for residential soils as well as lead and arsenic action levels for attic dust. Based on the results of sampling completed under this QAPP, additional RA will be applied, as appropriate.

The objectives of the QAPP are as follows:

1. Provide a sampling and analysis program for CS OU sampling activities (soil and attic dust) and ensure compliance with performance standards; and
2. Provide data to determine the scope of future CS OU RA.

Implementation of this QAPP will begin with CS OU sampling activities in the fall of 2015 in anticipation of RA activities beginning in 2016. The CS OU project schedule information is located in Table 1.

ED_014362_00000173-00015

2.4 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.

Development of Data Quality Objectives (DQOs) was completed in accordance with the EPA's *Guidance on Systematic Planning Using the Data Quality Objectives Process* (EPA, 2006). The DQOs are statements that define the type, quality, quantity, purpose, and use of data to be collected. The EPA has developed a seven-step process for establishing DQOs to help ensure that data collected during a field sampling program are adequate to support reliable site-specific decision making (EPA, 2001 and 2006). The following section outlines and establishes the QAPP DQOs.

2.4.1 Data Quality Objectives

The DQOs 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 that the resources required to generate the data are justified. 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. These steps include:

- Step 1: State the Problem;
- Step 2: Identify the Decision;
- Step 3: Identify the Inputs to the Decision;
- Step 4: Define the Study Boundaries;
- Step 5: Develop a Decision Rule;
- Step 6: Specify Tolerable Limits on Decision Errors; and
- Step 7: Optimize the Design.

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 DQOs. A brief discussion of these steps and their application to this sampling effort are provided in the following subsections.

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.

Past sampling efforts have demonstrated that elevated concentrations of metals/metalloids are present in attic dust, residential soils and alleys within the CS OU. From a potential risk to human health standpoint, arsenic and lead are considered the Constituents of Concern (COCs) within these areas. Sources of metals contamination may be from industrial impacts including smelter emissions, use of imported backfill materials, historic use of lead arsenate and arsenical pesticides, batteries, leaded gasoline and/or lead-based paint.

Comprehensive sampling of residential soils and alleys within the CS OU was conducted between 2002 and 2010. This remedial design (RD) sampling program was conducted to determine those residential yards and individual yard components (front yard, back yard, garden, etc.) subject to RA based on exceedance of the residential soil action level for arsenic (250 parts per million [ppm]) at targeted depth intervals. In 2006, available "screening level" lead data from these samples were evaluated, summarized and provided to the EPA in 2007 in the *Analysis of Lead in Anaconda Community Soils* (Atlantic Richfield Company, 2007). In the fall of 2007, the EPA conducted additional sampling and evaluation of lead in residential soils in Anaconda. The results of the EPA sampling effort are reported in the *Community Soils OU Residential Subsurface Soil Characterization Data Summary Report* (CDM, 2007).

The Atlantic Richfield Company (Atlantic Richfield) conducted a residential dust study within the CS OU in 2007. This study focused on collecting samples of interior, exterior and attic dust from newer and older homes spatially separated throughout the entire town of Anaconda, both inside and outside the CS OU focus area (residences located east of Main Street in Anaconda). Data generated from this study are presented in the *Draft Final Community Soils Interior and Attic Dust Characterization Study Data Summary Report* (Atlantic Richfield Company, 2008).

The Agencies evaluated these data sets (screening level lead data in residential soil, and subsurface lead and arsenic data in residential soil as well as residential dust data) generated in 2007, and reported the following conclusions in the *Residential Soils Data Interpretation and Analysis Report (DIAR)* (CDM, 2008).

- *"Lead in soils. 95 of the 142 yards (67 percent) that were sampled but not cleaned up had area weighted average lead concentrations above 400 ppm. 125 of the 142 yards (88 percent) evaluated had surface soil lead concentrations above 400 ppm in at least one yard component. 33 of the 142 yards (23 percent) had surface soil lead concentrations in at least one yard component greater than 1,200 ppm. The actual average concentration of lead in surface soils was 507 ppm, which is much higher than the calculated 290 ppm average from the 1996 human health risk assessment (HHRA).*

- *Arsenic in subsurface soils. Some portions of about one third of yards sampled but not cleaned up exceed 250 ppm arsenic in the subsurface. Soils with elevated arsenic generally also have elevated lead and vice versa. Correlations are weaker in subsurface soils than surface soils.*

- *Attic dust. Attic dust concentrations in the Anaconda focus area are significantly higher than interior dust concentrations and show no correlation, suggesting that attic dust does not influence interior dust.*

- *Rural areas. In most rural areas, smelter emissions appear to be the only site-related source of contamination. The exception is part of South Opportunity/Crackerville where tailings were deposited by Silver Bow Creek flooding events and irrigation practices."*

These previous studies have demonstrated the potential for arsenic or lead concentrations above action levels and therefore further sampling is required to define where additional RA is required.

Step 2: Identify the Decision

This step identifies what questions the study will attempt to resolve and what actions may result. The key questions may be stated as follows:

For residential yards to be sampled, does the weighted average composite arsenic concentration in residential soils exceed the 250-ppm residential soil action level at the 0 to 2-inch, 2 to 6-inch and/or 6 to 12-inch depth intervals?

For residential gardens to be sampled, does the garden component arsenic concentration exceed the 250-ppm residential soil action level at the 0 to 2-inch, 2 to 6-inch, 6 to 12-inch 12 to 18-inch, and/or 18 to 24-inch depth intervals?

For residential yards to be sampled, does the lead concentration for an individual yard component (front yard, back yard, etc.) exceed the 400-ppm residential soil action level at the 0 to 2-inch, 2 to 6-inch and/or 6 to 12-inch depth intervals?

For residential gardens to be sampled, does the garden component lead concentration exceed the 400-ppm residential soil action level at the 0 to 2-inch, 2 to 6-inch, 6 to 12-inch 12 to 18-inch, and/or 18 to 24-inch depth intervals?

For Anaconda alleys to be sampled, does the composite lead concentration exceed the residential soil action level of 400 ppm at the 0 to 6-inch depth interval (Anaconda alleys have been evaluated for arsenic previously)?

For residential attic dust meeting the accessible attic dust sampling criteria, does the arsenic and/or lead concentration exceed the residential attic dust action level of 250 ppm or 400 ppm, respectively?

Step 3: Identify the Inputs to the Decision

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.

The following data and information are required to satisfy or resolve the decision statements:

- *"Arsenic and lead analytical results applicable to residential soil and attic dust samples collected within each residential location;*
- *Lead analytical results applicable to composite soil samples collected from alleys;*
- *Dimensions of residential yards and individual yard components (i.e., front yard, back yard, garden, earthen driveway, etc.);*
- *Weighted-average composite arsenic concentrations within each residential yard at each target depth interval* (0- to 2 inch, 2- to 6-inch and/or 6- to 12-inch depth*);*
- *Composite lead concentrations within each residential yard component at each target depth interval; and*
- *Risk-based action levels for arsenic and lead in soil and attic dust."*

Step 4: Define the Study Boundaries

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

The study area will include residential properties (yard soil and attic dust) and alleys contained within the ADLC SPAOD.

Step 5: Develop a Decision Rule

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.

Atlantic Richfield will pursue access agreements for soil and attic dust sampling from those residences with available screening level lead data in soil exceeding the 400 ppm action level. All other residences (those with screening level lead data below the 400 ppm action level, those that have been sampled previously but have no lead data available, and those within the ADLC Superfund Planning District that have not been sampled previously) may request sampling of their soil and attic dust.

Composite soil samples and/or attic dust samples will be systematically collected from alleys and residential properties as described in this QAPP. If the composite sample result in an alley is less than the lead residential action level, then no RA will be performed in that alley. If the composite sample result in an alley is greater than or equal to the lead residential action level, then the alley will be remediated to a depth of 6 inches. Specific removal requirements will be identified in an Individual Site Work Plan (ISWP) for each alley subject to RA.

An area-weighted average arsenic concentration will be calculated for each of 3 soil depth intervals (0 to 2-inch, 2 to 6-inch and 6 to 12-inch depths) within each residential yard. However, for residences that were previously sampled under the original CS OU design, area-

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weighted average arsenic concentrations were already calculated for the 0 to 2-inch depth interval; consequently, for these residences that were previously sampled, samples collected/evaluated for the 0 to 2-inch depth interval under this design will be analyzed for lead only (not to include arsenic) and an area-weighted average arsenic concentration will not be recalculated for this depth interval. If the area-weighted average arsenic concentration is less than the residential action level in all layers, then no arsenic-specific RA will be performed in that residential yard; however, individual components within the yard may still be subject to RA for lead. If the area-weighted average arsenic concentration is greater than or equal to the residential action level within any layer, then each individual yard component (front yard, back yard, earthen driveway, etc.) within the affected layer that exceeds the residential action level for arsenic will be subject to RA. Specific removal requirements will be identified in an ISWP.

Each of the samples described above for arsenic analysis (component and depth interval) will also be analyzed for lead; however, an area-weighted average soil concentration will not be calculated for lead. If an individual yard component and depth interval is less than the residential action level for lead, then that component is not subject to RA at that particular depth interval; however, individual components within the yard may still be subject to RA for arsenic. If an individual yard component and depth interval is greater than or equal to the residential action level for lead, then that component is subject to RA for lead at the affected depth interval. Specific removal requirements will be identified in an ISWP.

Note that any clean soil layers within residential yards located above layers subject to removal will also be removed and not salvaged for replacement.

If a composite attic dust sample result is less than both the arsenic and lead residential dust action levels, then no RA will be performed in that attic. If a composite attic dust sample result is greater than or equal to either the arsenic or lead residential dust action levels, then the attic is subject to RA. Specific removal requirements will be identified in an ISWP.

Step 6: Specify Tolerable Limits on Decision Error

Decision errors occur when data mislead the site managers into choosing an inappropriate response, including no action. The potential for decision errors exists because all analytical measurements inherently contain sampling and measurement errors. Sampling design error occurs when the data collection scheme does not adequately address the inherent variability of the matrix being sampled (e.g., discrete versus composite samples). Measurement error occurs from the inherent variability in the collection, preparation, and analysis of an environmental sample. Sampling design and measurement errors will be minimized by following the procedures outlined in this QAPP and the attached Standard Operating Procedures (SOPs) presented in Attachment A.

Step 7: Optimize the Design

The data collection scheme is designed to ensure that the data will be of sufficient quality and quantity to determine the component(s) of individual residential yards, alleys and attics requiring RA (and the depth to which RA is required in residential yards and alleys). The sampling will be

completed in accordance with the recommendations provided in the EPA's *Superfund Lead-Contaminated Residential Sites Handbook* (EPA, 2003).

2.4.2 Measurement Performance Criteria for Data

Measurement performance criteria are established by defining acceptance criteria and quantitative or qualitative goals (e.g., control limits) for accuracy, precision, representativeness, comparability and completeness of measurement data. The definitions of precision, accuracy, representativeness, comparability and completeness are provided below along with the acceptance criteria for data collected. Equations for calculation of precision, accuracy and completeness are provided in Table 2. Information pertaining to the analytical methods that will be employed and the project's target quantitation limits can be found in Section 3.2.

Precision

Data precision is assessed by determining the agreement between replicate measurements of the same sample and/or measurements of duplicate samples. The overall random error component of precision is a function of sampling. The analytical precision is determined by the analyses of field duplicates by laboratories and by replicate analyses of the same sample. An analytical duplicate is the preferred measure of analytical method precision. When analytes are present in samples at concentrations below or near the quantitation limit, precision may be evaluated using duplicate analyses of laboratory prepared samples such as duplicate laboratory control samples (LCS/LCSD) and duplicate laboratory matrix spike samples (MS/MSD). Precision can be measured as relative percent difference (RPD) or as relative standard deviation (RSD; also known as a coefficient of variation). Formulae for both are presented in Table 2.

For this QAPP, precision will be determined by the analyses of field duplicates, laboratory (analytical) duplicates, and the evaluation of the RPD for these various paired measurements. The RPD goals for measures of laboratory (analytical) precision are provided in the laboratory SOPs (Attachments A-2 and A-3). The RPD field precision goal for soil field duplicates will be 35 percent for sample pairs with both sample results being greater than 5 times the reporting limit (RL). For soil field duplicate pairs with one or both sample results being less than five times the RL, a difference of less than or equal to two times the RL (difference $\leq$ 2xRL) will be used as the precision goal.

Accuracy/Bias

Accuracy is the degree of difference between the measured or calculated value and the true value. It is a measure of the bias or systematic error of the entire data collection process. Potential sources of systematic errors include:

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

ED_014362_00000173-00021

Laboratory blanks will be analyzed to assess artifacts introduced during sampling, transport and/or analyses that may affect the accuracy of the data. In addition, initial and continuing calibration verification samples (ICV and CCVs) and initial and continuing calibration blanks (ICB and CCBs) will be used to verify that the sample concentrations are accurately measured by the analytical instrument throughout the analytical run.

Laboratory accuracy will be determined by calibration verification and laboratory control samples. Accuracy/Bias goals for the specific analyses methods are summarized in the laboratory SOPs (Attachments A-2 and A-3).

Representativeness

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

Comparability

Data comparability is defined as the measure of the confidence with which one data set can be compared to another. Comparability is a qualitative parameter but must be considered in the design of the sampling plan and selection of analytical methods, QC protocols and data reporting requirements. Comparability will be ensured by analyzing samples obtained in accordance with this QAPP as well as the appropriate SOPs, which are comparable to the sampling methods used during previous investigations at the site. All data will be reported in units consistent with standard reporting procedures so that the results of the analyses can be compared with results from previous investigations. Soil and dust data will be reported in units of milligrams per kilogram (mg/kg). Attic dust confirmation samples will be reported in units of micrograms per square foot ($\mu g/ft^2$).

Completeness

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

2.5 Special Training

All field personnel will review and be trained in the requirements of this QAPP in a project meeting held prior to fieldwork. A review of sampling procedures and requirements will be

ED_014362_00000173-00022

completed prior to field activities to ensure sample collection and handling methods are according to QAPP requirements. Field personnel will be trained in proper use of field equipment and procedures according to field data collection SOPs. One hard copy of the current approved version of this QAPP will be maintained for ready reference purposes in the field vehicle and/or field office. All field team personnel will have access to PDF format files of the complete QAPP. The Field Team Leader will assure that each member of the sampling team is familiar with the QAPP and will maintain signatures of each team member who has read the QAPP (including reviews and addenda, as necessary) and has been trained in the appropriate sample collection methods per the SOPs. A copy of the field team signature page will be attached to the copy(ies) of the QAPP maintained in the field.

A review of the SSHASP will be conducted with all field personnel prior to fieldwork to assess the site's specific hazards and the control measurements that have been put in place to mitigate these hazards. The SSHASP review will cover all other safety aspects of the site including, site personnel responsibilities and contact information, additional site-specific safety requirements and procedures, and the emergency response plan.

Laboratories providing analytical services will have a documented QC program that complies with EPA *Requirements for Quality Assurance Project Plans* (QA/R-5) (EPA, 2001) and Atlantic Richfield's LaMP. The Contractor QA Officer will be responsible for ensuring that all personnel have been properly trained and are qualified to perform assigned tasks.

2.6 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.

2.6.1 Property Access Agreements

Atlantic Richfield will request that all private property owners grant access to their properties for all RA-related activities, including sampling. The Field Team Leader will manage requests for access, track the status of access requests and maintain copies of completed agreements received from property owners.

Completed agreements will be photocopied and scanned with the electronic version stored on a hard drive. Photocopied access agreements will also be copied to the project record files.

2.6.2 Field Logbooks/Data Sheets

Documentation in field logbooks provides a description of site conditions during sampling activities, and provides a permanent record of all field activities. A field logbook and/or appropriate field data sheets (refer to field SOPs) will be used for field activities. When field logbooks are used, each logbook will have a unique document control number, and will be bound and have consecutively numbered pages. The information recorded in these logbooks/field data sheets will be written in ink. Whenever a sample is collected or a measurement is made, a detailed description of the sample location and any additional observations will be recorded.

Resource-grade global positioning system (GPS) coordinates will be available for all surveyed residential yard maps.

Field logbooks/field data sheets 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.);
- Meteorological conditions at the beginning of fieldwork and any ensuing changes in the weather conditions;
- Details of the fieldwork performed and the field data sheets used with special attention to any deviation from the QAPP or applicable field SOPs;
- All field measurements made;
- Any field analyses results; and
- Personnel and equipment decontamination procedures.

For any field sampling work the following entries will be made:

- Sample location and ID number;
- Sample type collected;
- Date and time of sample collection;
- Split samples taken by other parties (note the type of sample, sample location, time/date, name of person, person's company and any other pertinent information);
- Sampling method, particularly any deviations from the field SOPs;
- 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), specifically if sample bottles/preservatives are not provided by the laboratory and certified as cleaned; and
- Sample preservation (if used).

Changes in the field logbook or on the field data sheets will be recorded with a single strike mark through the changed entry, with the sampler's initials and the date recording the new entry. All entries must remain legible. Sufficient information should be recorded to allow the sampling event to be reconstructed without having to rely on the sampler's memory.

Completed field data sheets and logbooks will be photocopied and scanned with the electronic version stored on a hard drive. Photocopied field records will also be copied to the project record files (refer to Section 3.6). No bound field logbooks will be destroyed or thrown away even if they are illegible or contain inaccuracies that require a replacement document.

ED_014362_00000173-00024

2.6.3 Field Photographs

Photographs may be taken of sampling locations and field activities using a digital camera. Photographs should include a scale in the picture when practical. Additional photographs documenting site conditions will be taken, as necessary. Documentation of all photographs taken during sampling activities will be recorded in the bound field logbook or appropriate field data sheets (refer to field SOPs), and will specifically include the following for each photograph taken:

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

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

2.6.4 Chain of Custody Records

After samples have been collected, they will be maintained under strict chain of custody protocols in accordance with the CS OU Field SOPs provided in Attachment A-1. 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 3.6).

2.6.5 Analytical Laboratory Records

Results received from the laboratories will be documented both in report form and in an electronic format. Laboratory documentation includes copies of the signed chain of custody forms, laboratory confirmation reports including information on how samples have been batched and the analyses requested, data packages including the laboratory report and the electronic data deliverable (EDD), and any change requests or corrective action requests. Section 5.1.3 presents the project's laboratory reporting requirements in detail. The deliverable ("data package" or "report") issued by the laboratories will include data necessary to complete validation of laboratory results in accordance with specifications included in Section 5.2.2.

Original reports and electronic files received from laboratories will be maintained with the project quality records (refer to Section 3.6).

2.6.6 Project Data Reports

Upon receipt of laboratory results and completion of the data review/validation process, all analytical data will be uploaded into a project database. The database will summarize sample data from yards and unpaved alleys as well as attic dust.

The sample results (for all analytes) will be reported to individual landowners along with a letter explaining what the results indicate. The action levels for arsenic and lead will be reported,

ED_014362_00000173-00025

along with sample results. The content of the letter explaining sample results to landowners will be determined in consultation with EPA.

Following landowner notification, sample results will be used to develop ISWPs for each residence where sample results exceed CS OU action levels (≥250 ppm arsenic and ≥400 ppm lead). The ISWPs will summarize the number of individual yard components associated with each property, depth of each sample and corresponding surface area of each yard component.

All sampling data will be forwarded to the Agencies for review and approval in the form of an annual Data Summary Report (DSR). This DSR will include figures displaying location of homes sampled, analytical results, data review/validation reports, and copies of all field data and all daily logbook entries. As described above, all sampling data will reside in the project database.

2.6.7 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 Contractor QA Officer (or designee) and will include, at a minimum:

- This QAPP and any approved revisions or addenda;
- Approved versions of the SSHASP 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, faxes and e-mail messages);
- Copies of completed access agreements for the individual properties sampled;
- Individual property maps, including any field drawings and field photographs;
- Field documentation forms;
- Copies of all bound field logbooks;
- Copies of all field data sheets;
- Copies of all sample chain of custody forms;
- Copies of all laboratory agreements and amendments;
- Laboratory data packages (hard copy report and electronic);
- Documentation of field and/or laboratory audit findings and any corrective actions; and
- Draft and final delivered versions of all reports and supporting procedures such as statistical analyses, numerical models, etc.

All project data will be maintained in the CS OU Residential Soils and Attic Dust Global Information System (GIS) Database. This is a long-term project with access to the database provided to many interested parties. The data will be maintained indefinitely.

ED_014362_00000173-00026

3.0 MEASUREMENT AND DATA ACQUISITION

This section addresses all aspects of project design and implementation for generating and acquiring data. Implementing these elements ensures that appropriate methods for sampling, sample handling, laboratory analyses, field and laboratory QC, instrument/equipment testing, inspection, maintenance, instrument/equipment calibration, data management and data security are used when implementing this QAPP.

3.1 Sampling Process and Design

The residential soil and attic dust sampling procedures detailed in this plan focus on those properties within the CS OU with existing screening level data in soil showing lead concentrations equal to or greater than 400 ppm. A soil and attic dust sampling access agreement letter with a questionnaire will be mailed to each affected resident. The questionnaire will request information regarding the presence of pregnant women and/or children age 12 or younger living within or frequenting the residence as well as information regarding age of the home and activities/circumstances that may result in exposure to attic dust. This feedback (presence of sensitive population) will be used in conjunction with existing lead data to prioritize the sampling schedule. Residences with the highest lead concentrations and presence of sensitive population will be scheduled to be sampled earliest. All other properties may be sampled per request by the landowner.

3.1.1 Residential Soils and Alleys Field Sampling Plan

Residential yards will be sampled according to the procedures outlined in this QAPP, which is based on both the *Superfund Lead-Contaminated Residential Sites Handbook* (EPA, 2003) and the *Final Community Soils Operable Unit (CS OU) Residential Soils Remedial Action Work Plan/Final Design Report (RAWP/FDR)* (Atlantic Richfield Company, 2002). To the extent practical, the previously delineated and approved CS OU residential yard components within each yard will continue to be used under this project.

Residential soil samples for each yard component will be collected from three depth intervals (except for those components that were previously remediated to a depth of 12 inches, or deeper). The first composite sample will consist of subsamples from the 0 to 2-inch depth interval, the second composite sample will consist of subsamples from the 2 to 6-inch depth interval, and the third composite sample will consist of subsamples from the 6 to 12-inch depth interval. The rationale for the 12-inch maximum sampling depth is detailed further in Attachment B-1: Technical Memorandum #2 – *Modifying the Sample Depth Intervals Used to Make Remedial Decisions for Arsenic and Lead in Yard Soils* (Atlantic Richfield Company, 2014a). The Agencies approved this change in the September 8, 2014 letter provided in Attachment B-3.

Consistent with the *Superfund Lead-Contaminated Residential Sites Handbook* (EPA, 2003), RA decisions applicable to removing soils with lead concentrations equal to or greater than the 400 ppm action level will be based on a component-specific basis. Consistent with the original CS

ED_014362_00000173-00027

OU design, RA decisions applicable to removing soils with arsenic concentrations greater than or equal to the 250 ppm action level will be based on an area-weighted average basis. However, for residences that were previously sampled under the original CS OU design, area-weighted average arsenic concentrations were already calculated for the 0 to 2-inch depth interval; consequently, for these residences that were previously sampled, samples collected/evaluated for the 0 to 2-inch depth interval under this design will be analyzed for lead only (not to include arsenic) and an area-weighted average arsenic concentration will not be recalculated for this depth interval. However, area-weighted average arsenic concentrations will be calculated for the 2 remaining depth intervals (2 to 6 inches and 6 to 12 inches). Any clean cover soil in previously remediated components will not be resampled under this design, but will be assigned an average arsenic concentration of 19.1 ppm (average arsenic concentration measured in the Type A borrow soil samples collected during the previous CS OU RA) when calculating the AWA arsenic concentration. For residences that were not previously sampled under the original CS OU design for arsenic, area-weighted average concentrations will be calculated for 3 depth intervals (0 to 2 inches, 2 to 6 inches, and 6 to 12 inches).

The number of residential yards ultimately sampled will depend on the number of access agreements secured by Atlantic Richfield from the residential property owners within the ADLC SPAOD. In addition to residential yards, unpaved alleys in Anaconda with existing screening level data showing lead concentrations equal to or greater than 400 ppm will be sampled to a depth of 6 inches below ground surface (bgs). Alley samples will be analyzed for lead only because alleys were previously evaluated for arsenic under the original design.

Alleys are well compacted with infrequent exposure by residents and are unlikely to be excavated via hand tools by residents to a depth beyond 6 inches. Additionally, a General Utility and Service (GUS) Permit is required for excavation conducted in alleys by ADLC personnel and contractors; consequently, such exposure to soils deeper than 6 inches in alleys will be addressed through Institutional Controls (ICs).

3.1.1.1 Standard Operating Procedures for Sampling Activities

The CS OU Field SOPs located in Attachment A-1 describe the procedures involved with CS OU RD soil and attic dust sample collection, handling, and shipment. Unless otherwise described in this QAPP, the procedures outlined in the CS OU SOPs will be followed for all CS OU soil and attic dust sampling activities.

3.1.1.2 Yard/Lot Maps

A surveyed map was created for each individual residential property sampled during the original CS OU RD sampling effort (Figure 3). These maps identify how each property was divided into individual components (i.e., front yard, back yard, earthen driveway, etc.) and will be used for this CS OU RA sampling effort. In the event that a map does not exist or the existing map is inadequate, a new surveyed map will be created. Each map includes the following information:

- Surface area applicable to each individual yard component (e.g., front yard, back yard, earthen driveway, etc.);

- Surface area applicable to the "occupation" boundary of each residence (approximately corresponding with the property boundary);
- House location;
- Garage location;
- Location of miscellaneous structures (e.g., patios, concrete pads, sidewalks, decks, etc.); and
- 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.).

All subsample locations will be plotted on the map by sampling crews in the field. Photographs will be taken of yard components as well as any unusual features, as deemed necessary by field personnel. All information will be recorded in the field logbook.

3.1.1.3 Sample Density, Location and Compositing

Composite soil samples will be collected from each individual residential yard component when sampling each residential yard. If the component was previously remediated to 12 inches bgs or deeper, samples from the 2 to 6-inch and 6 to 12-inch depth intervals will not be collected, but will be assigned an average arsenic concentration of 19.1 ppm when calculating the AWA arsenic concentration. If the component was previously remediated to 6 inches bgs, the sample from the 2 to 6-inch depth interval will not be collected, but will be assigned an average arsenic concentration of 19.1 ppm when calculating the AWA arsenic concentration. The sample from the 6 to 12-inch depth interval will be analyzed for both lead and arsenic (to be included in the area-weighted average calculation). The CS OU Residential Yard Remedial Action Decision Flow Charts (which are used to develop sampling rationale for individual components) are located on Figures 4 through 9. All samples collected, as well as other relevant observations will be documented on the Residential Yard Sampling Field Data Sheet located in Attachment C-1. Subsamples collected from each component will be composited in the field, and a single composite sample applicable to each identified depth interval for each component will be submitted to the laboratory and analyzed for arsenic and/or lead, as appropriate.

Sample densities and locations within the yard components will be determined based on the size of the area to be represented. Either the property boundary or a smaller natural boundary within the yard will be used to establish the extent of the sample area. The yard area will be defined as a maximum of 125 feet from the exterior of the residence, unless a property boundary or natural barrier (i.e., fence, hedge, tree line, abrupt change in grade, etc.) is encountered at a distance less than 125 feet. The 125-foot distance is considered a guideline and can be adjusted in the field by Agency personnel, as appropriate. For the majority of the residential properties likely to be sampled under this investigation, site maps were created during the previous CS OU sampling investigation. These maps detail individual components and their respective surface areas for each property. To the extent practical, these existing maps and corresponding components will

continue to be used. One subsample per individual yard component will be collected unless the component is greater than 625 square feet (ft^2) (25 feet by 25 feet) in surface area. If the yard component exceeds 625 ft^2, additional subsamples will be collected to meet the sample collection density of 1 subsample for each 625 ft^2. When multiple subsamples are collected from a single yard component (e.g., >625 ft^2), they will be composited in the field, and a single composite sample will be analyzed for arsenic and/or lead, as appropriate. Each residential yard will be evaluated for arsenic based on the area-weighted average composite concentration at each depth interval (see example shown in Table 3). Each residential yard will be evaluated for lead based on a component-specific basis. A maximum of 8 soil subsamples will be collected from any individual residential yard component regardless of the surface area of that component. Larger parcels, such as schools and parks, etc., may require multiple components to ensure adequate coverage and protectiveness.

In accordance with the CS OU Field SOPs (located in Attachment A-1), play areas, vegetable gardens, flower gardens, earthen driveways, and source areas (areas of visible smelter-related waste material), will have a minimum of two subsamples collected regardless of the surface area involved. The two subsample locations will be composited into one sample for each depth interval.

Discrete "drip zone" component samples will not be collected. Subsamples will be collected from within drip zones associated with a residence and the subsample will be included in the nearest component's composite sample. If the component's composite sample exceeds the arsenic and/or lead action level(s), the drip zone will be removed and replaced (in kind) along with the targeted component.

Soil subsamples will not be collected from yard components between neighboring/adjacent homes where the distance between the homes is less than 3 feet. Additionally, soil subsamples will not be collected from beneath a tree canopy. If the yard component surrounding the tree is subject to RA, soil excavation under the tree canopy will not be completed (Sheet 8 of Appendix D).

If the Agency representative or property owner chooses to collect split samples, an adequate quantity of soil will be made available by the sampler at the time of sample collection. However, the Agency representative or property owner will be responsible for providing sample containers and coolers, etc.

3.1.1.4 Sample Depths

The following 2 sections detail CS OU soil sampling depths for both residential soil and unpaved alley samples.

3.1.1.4.1 Residential Soil Samples

Within residential properties, three depth samples will be collected from each identified yard component. There will be 1 surface sample (0 to 2 inches bgs) along with 2 subsurface samples (2 to 6 inches bgs and 6 to 12 inches bgs). For residences that were previously sampled under

ED_014362_00000173-00030

the original CS OU design, area-weighted average arsenic concentrations were already calculated/evaluated for the 0 to 2-inch depth interval; consequently, for these residences that were previously sampled, samples collected from the 0 to 2-inch depth interval under this design will be analyzed for lead only (not to include arsenic) and an area-weighted average arsenic concentration will not be recalculated for this depth interval. The CS OU Residential Yard Remedial Action Decision Flow Charts (which are used to develop sampling rationale for individual components) are located on Figures 4 through 9. .

Because most residential yard/lot areas are expected to be covered with grass, the surface sample will be collected immediately beneath the vegetative mat (sod), or in the absence of vegetation, 0 to 2 inches bgs. If a vegetative mat is present, it will be separated from the soil surface with a stainless steel knife or equivalent. The removed vegetative mat will be shaken and scraped over the sample collection bowl to dislodge any mineral soil particles. All dislodged soil particles will be included in the composite sample.

Exceptions to this procedure will occur when the sample location falls on a graveled driveway or similar surface. If the surface material is coarse-grained and free of intermixed materials, the sample will be collected from the 0 to 2-inch soil layer immediately beneath the coarse materials. However, if the graveled driveway or similar surface contains fine soil/dust material on the surface, the sample will be collected from the surface (0 to 2-inch) layer.

Vegetable and flower gardens will be subject to additional subsurface sampling. In addition to the three depth samples described above, one subsurface sample will be collected from the 12 to 18-inch depth interval and another will be collected from the 18 to 24-inch depth interval, for a total of 5 depth samples (1 surface and 4 subsurface) within a vegetable or flower garden. Vegetable and flower gardens will be evaluated on a component-specific basis for arsenic, regardless of the area-weighted average result. If the arsenic concentration is greater than or equal to the 250 ppm action level, then that component is subject to RA for arsenic at the affected depth interval. Similarly, if the lead concentration is greater than or equal to the 400 ppm action level, then that component is subject to RA for lead at the affected depth interval. Specific removal requirements will be identified in an ISWP.

The decision to collect additional "opportunistic" samples will be made in the field by the sampling crew personnel and/or Agency personnel during the time of sampling. All CS OU residential soil samples will be shipped to an Atlantic Richfield LaMP certified laboratory, for analyses.

3.1.1.4.2 Unpaved Alley Samples

Unpaved alleys in Anaconda with existing screening level data showing lead concentrations equal to or greater than 400 ppm will be re-sampled to a depth of 6 inches bgs. All samples collected, as well as other relevant observations will be documented on the Unpaved Roads and Alley Sampling Field Data Sheet located in Attachment C-2. Alley samples will be analyzed for lead only because alleys were previously evaluated for arsenic under the original design. Alley samples will not be collected to a depth beyond 6 inches bgs. Alley subsamples will be collected from each end of the alley and at one location between the ends of the alley, for a total of three

subsamples. The three alley subsamples will be combined into one composite sample per alley and submitted to the laboratory for analyses. All CS OU alley samples will be shipped to an Atlantic Richfield LaMP certified laboratory for analyses.

3.1.1.5 Sample Identification

Sample identification procedures for both CS OU residential soil samples and unpaved alley soil samples are detailed in the sections below.

3.1.1.5.1 Residential Soil Sample Identification

An alphanumeric coding system will be used to uniquely identify each sample collected during this residential soils investigation. Sample identifiers will begin with an Anaconda or regional property code (i.e., A, R, OP). This will be followed by the CS OU Database Resident ID (i.e., A-0001, A-0002) associated with a specific residential property (address and/or geocode specific). Following the CS OU Database Resident ID will be an alpha identifier for the yard component represented by the sample (i.e., FY for front yard or BY for back yard, etc.). A four-digit numeric code will follow, indicating the sample depth interval. The following is a list of the codes to be used in sample identification:

Site Property Code: A – Anaconda Residential Properties
R – Regional Residential Properties
OP – Opportunity Residential Properties

CS OU Database Resident ID: 0001 – CS OU Database Resident ID, associated with specific address or geocode

Yard Component: FY – Front Yard
BY – Back Yard
ED – Earthen Drive
FG – Flower Garden
VG – Vegetable/Fruit Garden
PA – Play Area
BA– Bare Area
SA – Source Area
BV – Boulevard

Quality Control/ Quality Assurance: D – Field Duplicate

Depth Intervals: 0002 – 0 to 2 inches bgs
0206 – 2 to 6 inches bgs
0612 – 6 to 12 inches bgs
1218 – 12 to 18 inches bgs
1824 – 18 to 24 inches bgs

ED_014362_00000173-00032

An example sample identification would be: R-0001-BY-0206. This indicates that the sample was collected at the regional residential property R-0001 (corresponding to a physical address and/or geocode) at the 2 to 6-inch depth interval in the back yard. Sample identifiers will be documented in field notebooks, on the Residential Yard Sampling Field Data Sheet, and on the chain of custody forms, as required by the CS OU Field SOPs located in Attachment A-1.

3.1.1.5.2 Unpaved Alley Soil Sample Identification

Unpaved alley samples will be identified according to the kriging grid detailed in the *Anaconda Smelter NPL Site, Community Soils Operable Unit (CS OU), Anaconda Residential Soils, Regional Soils, and Railroad Areas, Data Interpretive Report (DIR)* (AERL, 1999) (see Appendix F for alley locations and IDs).

An example alley sample identification would be: P-35-A-0006. This indicates that the sample was collected from the Anaconda unpaved alley labeled as P-35 at the 0 to 6 inches bgs depth interval. Sample identifiers will be documented in field notebooks, on the Unpaved Roads and Alley Sampling Field Data Sheet, and on the chain of custody forms, as required by the CS OU Field SOPs located in Attachment A-1.

3.1.2 Attic Dust Field Sampling Plan

The CS OU attic dust field sampling plan detailed below outlines all aspects of the process including sampling eligibility, sampling methods and confirmation sampling. Interior dust is not a part of this CS OU investigation. The basis for excluding interior dust sampling and the method for addressing attic dust is covered in Attachment B-2: *Technical Memorandum #1 – Basis for Excluding Living Space Dust and Addressing Only Accessible Attic Dust through Analysis of the Existing Data* (Atlantic Richfield Company, 2014b). The Agencies approved this change in the September 8, 2014 letter provided in Attachment B-3.

Abatement of asbestos-containing materials is not intended to be included as part of the attic dust program. If materials suspected to contain asbestos are encountered in an attic and would be disturbed by the RA, an inspection and sampling of these materials will be conducted. The samples will be collected by an accredited asbestos inspector and analyzed by an accredited asbestos laboratory. The quantity and location of the samples will be determined by site conditions. If the materials in question are determined to be a non-asbestos containing material (i.e., less than 1% asbestos), then the dust abatement activities may proceed. If the materials in question are determined to be asbestos containing material (i.e., greater than 1% asbestos), then an alternative plan will be developed, in coordination with the property owner and the Agencies.

3.1.2.1 Attic Dust Sampling Eligibility

The CS OU attic dust sampling design focuses on those properties with existing screening level data in soil showing lead concentrations equal to or greater than 400 ppm. A soil and attic dust sampling access agreement letter with a questionnaire will be forwarded to each affected resident. The questionnaire will request information regarding the presence of pregnant women and/or children age 12 or younger living within or frequenting the residence as well as

information regarding age of the home and activities/circumstances that may result in exposure to attic dust. This feedback (presence of sensitive population) will be used in conjunction with existing lead data to prioritize the sampling schedule. Residences with the highest lead concentrations and presence of sensitive population will be scheduled to be sampled earliest. All other residences within the SPAOD meeting the eligibility criteria will need to request attic dust sampling.

In order to be eligible for attic dust sampling, the home must have been constructed prior to 1980 and meet at least 1 of the following conditions:

1. The attic is used as living space.

2. On average, the resident(s) enters the attic more than once per week.

3. The ceilings in the living space immediately below the attic are in a condition of disrepair with obvious exposure to the attic.

4. The resident has contacted ADLC (per Community Protective Measures Program [CPMP] public outreach programs) regarding concerns about attic dust, which may result from a home remodeling project.

If the above qualifications are met, attic dust samples will be collected. If the criteria are not met, then no exposure pathway to the attic exists and the attic does not pose an unacceptable risk to the residents.

3.1.2.2 Attic Dust Sampling Equipment and Sample Collection

Attic dust samples will be collected using the Micro-Vacuum Surface Dust Sampler. Attic dust sampling will be performed in accordance with the CS OU Field SOPs, located in Attachment A-1. As detailed in the SOP, attic dust sampling procedures will follow the *American Society for Testing and Materials (ASTM) D7144-05a: Standard Practice for Collection of Surface Dust by Micro-vacuum Sampling for Subsequent Metals Determination* (ASTM, 2006). The Micro-Vacuum Sampler is designed to collect dust using a nozzle attached to a filter holder (sampling cassette) that is connected to an air sampling pump. Depending on accessibility, samples will be collected from a minimum of two locations within the attic and composited into one representative sample. All attic dust sampling activities will be documented on the Attic Dust In-Home Sampling Data Sheet located in Attachment C-3.

3.1.2.3 Attic Dust Sample Identification

An alphanumeric coding system will be used to uniquely identify each attic dust sample collected during this investigation. Sample identifiers will begin with an Anaconda, regional or Opportunity residential property code (i.e., A, R, OP). This will be followed by the CS OU Database Resident ID (i.e., A-0001, A-0002) associated with a specific residential property (address and/or geocode specific). Following the CS OU Database Resident ID will be an alpha

ED_014362_00000173-00034

identifier indicating attic dust sample (i.e., AD for attic dust). The following is a list of the codes used for attic dust sample identification:

Site Property Code: A – Anaconda Residential Properties
R – Regional Properties
OP – Opportunity Residential Properties

CS OU Database Resident ID: 0001 – CS OU Database Resident ID, associated with specific address or geocode

Sample Location: AD – denotes attic dust sample;

QA/QC D – Field Duplicate

An example sample identification would be: A-1250-AD. This indicates that an attic dust sample was collected at the Anaconda residential property A-1250 (corresponding to a physical address and/or geocode). Sample identifiers will be documented in the field notebooks, the Attic Dust In-Home Sampling Data Sheet and on the chain of custody forms, per the CS OU Field SOPs (Attachment A-1).

Sample filters are delivered by the supplier using a unique and consecutive numbering system. The numbers are assigned during the filter preparation (pre-weighing) process. The filter number provided by the supplier will also be documented in the field notebooks and the Attic Dust In-Home Sampling Data Sheet.

3.1.2.4 Attic Dust Sample Handling

All filters collected during attic dust sampling operations with the Micro Vacuum Surface Dust Sampler will be placed in a ziplock bag and delivered to an Atlantic Richfield LaMP certified laboratory, to be analyzed for arsenic and lead. All samples will be transferred and handled using established chain of custody procedures which are detailed in the CS OU Field SOPs located in Attachment A-1.

3.1.2.5 Attic Dust Confirmation Sampling

Following cleanup of an attic, confirmation sampling will be performed within the attic in accordance with the *Lead Dust Sampling Technician Field Guide, EPA-W-04-022* (EPA, 2009) located in Attachment D. Confirmation sampling will consist of wiping the encapsulation material on the attic floor following encapsulation material placement. If confirmation sampling provides a lead result greater than or equal to 40 micrograms per square foot ($\mu g/ft^2$), encapsulation material will be re-applied, followed by another round of confirmation sampling. All confirmation wipe samples will be placed in a ziplock bag and delivered to an Atlantic Richfield LaMP certified laboratory, to be analyzed for lead. The following steps will be used during confirmation sampling:

1. Sampler will don disposable boot covers and layout the confirmation sampling area. The sampling area will be designated using a clean template or tape. The sampling area will be as close to square, in shape, as possible.

2. Sampler will prepare the sample container/tube by recording the sample identification code on a clean tube, field logbook, sample form, and chain of custody form.

3. Sampler will don a clean/new set of disposable sampling gloves, being careful not to touch anything except the sampling wipe.

4. Sampler will wipe the sample area and place the wipe in the labeled sample tube. To begin sampling, press the wipe down firmly in one of the upper corners of the sample area. Making an "S" motion wipe the entire sample area moving from side to side. Fold the wipe in half making sure to fold the used side in. Starting in the same corner wipe the entire sample area in a forward and back motion. Fold the wipe again. Wipe the area a third time focusing on the corners and edges of the sample area. Fold the sample side of the wipe in again a third time and place the wipe into the labeled sample tube. Seal the container and discard the disposable gloves.

5. Sampler will measure the width and length of the sample area (unless a template of known area is used) to the nearest 1/8 inch. Record the dimensions of the sample area on the sample container and the sample form.

6. Using the sample area measurements, sampler will calculate the sample surface area and record in the field logbook as well as on the sample collection form and chain of custody form.

7. If a template was used, sampler will clean it with a clean wipe and place the template in a plastic bag for storage.

8. Sampler will ship samples to a laboratory recognized by the National Lead Laboratory Accreditation Program (NLLAP) as being proficient in lead in dust analysis. All CS OU attic dust confirmation samples will be delivered to an Atlantic Richfield LaMP certified laboratory, to be analyzed for lead.

Following confirmation sampling and verification that the lead dust level is below 40 $\mu g/ft^2$, the residential property sampled will be updated in the CS OU database as RA Complete for attic dust.

3.1.2.5.1 Attic Dust Confirmation Sampling Sample Identification

An alphanumeric coding system will be used to uniquely identify each sample collected during attic dust confirmation sampling. Sample identifiers will begin with a residential property code (i.e., A, R, OP). This will be followed by the CS OU Database Resident ID (i.e., A-0001, A-0002) associated with a specific residential property (address and/or geocode specific).

ED_014362_00000173-00036

Following the CS OU Database Resident ID will be an alpha identifier indicating sample matrix (i.e., CWS for attic dust confirmation wipe sample). The following is a list of the codes to be used in sample identification:

Site Property Code: A – Anaconda Residential Properties
R – Regional Properties
OP – Opportunity Residential Properties

CS OU Database Resident ID: 0001 – CS OU Database Resident ID, associated with specific address or geocode

Sample Location: CWS – denotes attic dust confirmation sample (confirmation wipe sample)

An example sample identification would be: A-1250-CWS. This indicates that an attic dust confirmation wipe sample was collected at the Anaconda residential property A-1250 (corresponding to a physical address and/or geocode). Sample identifiers will be documented in the field notebook, the Attic Dust In-Home Sampling Data Sheet and on the chain of custody forms, per the CS OU Field SOPs located in Attachment A-1.

3.2 Laboratory Methods

All soil samples collected for this investigation will be analyzed for arsenic and lead. All attic dust samples, including confirmation wipe samples, will be analyzed for arsenic and/or lead, as appropriate. All samples for this investigation will be analyzed by an Atlantic Richfield LaMP certified laboratory. The sample preparation and analytical methods to be used for this investigation are described in Sections 3.2.1 and 3.2.2.

3.2.1 Soil Preparation Methods

Sample preparations and analyses will be in accordance with the EPA analytical method specifications as well as standard laboratory practices. The soil samples will be prepared for metals analyses by air drying and then sieving the sample to obtain the fine-grained fraction for analyses. Each as-received soil sample will be sieved to homogenize the sample and obtain fine-grained material for analyses. The sample will first be air dried at room temperature (<40 degrees Celsius [^{o}C]) and then sieved using a No. 60 sieve to obtain the fine fraction, <250 micrometers or microns (μm), for metals analyses. The remaining coarse fraction will be placed in a new plastic bag labeled with the original sample number, date of sieving, and "Coarse Fraction" and then archived along with the remaining fine fraction for a minimum of two years for potential future use. The weight of the original dried sample, the weight of the coarse fraction, and the weight of the fine fraction will be measured and recorded by the laboratory for each soil sample prepared in this manner. The SOP addressing soil drying and sieving is included in Attachment A-2.

ED_014362_00000173-00037

3.2.2 Soil Metals Analyses Methods

All soil samples will be analyzed for arsenic and/or lead, as appropriate. Sample preparations and analyses will be in accordance with the referenced EPA analytical method specifications as well as standard laboratory practices. The fine fraction of the sieved soil will be digested according to modified EPA Method 3050B, and analyzed by EPA Method 6010 (inductively-coupled plasma atomic emission spectroscopy [ICP-AES]) or EPA Method 6020 (inductively-coupled plasma mass spectrometry [ICP-MS]). The SOPs for EPA Methods 3050B, 6010, and 6020 are included in Attachment A-2.

3.2.3 Attic Dust Preparation Methods

Sample preparations and analyses will be in accordance with the EPA analytical-method specifications as well as standard laboratory practices. The attic dust (filter cartridge or confirmation wipe) samples will be prepared for analyses by first determining the net weight of the dust sample. The method used to determine the weight of the filter cartridge sample is dependent on the type of cartridge used and type of data provided by the manufacturer (i.e., tare weight of filter, blank filter, weight of entire cartridge, etc.). After the net weight of the dust sample (filter cartridge or confirmation wipe) is determined and recorded, the sample is ready for digestion according to modified EPA Method 3050B. The SOP addressing attic dust filter cartridge and confirmation wipe sample preparation is included in Attachment A-3.

3.2.4 Attic Dust Analyses Methods

All attic dust samples will be analyzed for arsenic and lead. Sample preparations and analyses will be in accordance with the referenced EPA analytical method specifications as well as standard laboratory practices. The attic dust samples will be digested according to EPA-modified Method 3050B, and analyzed by EPA Method 6010 (ICP-AES) or EPA Method 6020 (ICP-MS). Results of the attic dust analyses will be reported on a dry-weight basis, with percent moisture also being reported to allow for future conversion of dry weight to wet weight concentrations, if necessary. The SOPs for EPA Methods 3050B, 6010, and 6020 are included in Attachment A-3.

3.3 Quality Control

Sample QC protocols will be consistent with the CS OU Field SOPs in Attachment A-1 and will include 1 field duplicate collected for every 20 primary samples or once per sampling event, whichever is more frequent (in accordance with Level A/B field screening/data review criteria, Attachment F). A temperature blank will additionally be collected for every cooler shipped to the laboratory to ensure the samples were maintained within the temperature of <6 ºC, but above freezing. All sampling equipment is anticipated to be "one time use"; therefore, no external contamination blank/cross-contamination blank samples will be submitted unless the equipment has to be decontaminated and used between samples. Any deviation from the SOPs or this QAPP will be identified in the logbook and discussed in the annual DSR.

ED_014362_00000173-00038

All sampling activities will follow the field SOPs. All field SOPs were developed from the *Clark Fork River Superfund Site Investigations (CFRSSI) SOPs* (ARCO, 1992), and represent updated versions of those SOPs for the sampling activities conducted under this QAPP. Applicable CS OU Field SOPs are included in Attachment A-1.

3.3.1 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 shipped with field samples to the laboratory per the CS OU Field SOPs in Attachment A-1. Brief descriptions of these QC samples to be collected during sampling activities described in this QAPP are provided below along with instructions for their frequencies of collection and analyses.

Field Duplicate (Soil Samples)

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

Field Duplicate (Attic Dust Samples)

Field duplicate samples associated with attic 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 the 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.

3.3.2 Laboratory Quality Control Samples

All analyses will be governed by the appropriate calibration procedures and frequencies that are specified in the laboratory's SOPs. The project SOPs for the referenced EPA methods are included in Attachments A-2 and A-3, respectively. Although laboratories for this project have not yet been determined, these SOPs are from laboratories that have conducted similar laboratory work in the past.

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. The appropriate type and frequency of laboratory QC samples associated with each method are specified in the laboratory SOPs (Attachments A-2 and A-3, respectively).

Laboratory blanks, laboratory control samples, analytical duplicates, serial dilutions, and pairs of Matrix spike/matrix spike duplicate (MS/MSD) samples will be analyzed in each laboratory QC batch with a minimum 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 the set of less than 20 samples. A second MS sample is not necessary for all laboratory QC batches that already have one MS/MSD.

Laboratory Blanks

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

Laboratory Control Samples (LCS)

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 than that used for the calibration standards. The LCS is the primary indicator of process control used to demonstrate whether the sample preparation and analytical steps are in control, apart from sample matrix effects. If the LCS recovery falls outside the specified control limits, the samples will be re-run and/or flagged by the laboratory per the QC requirements of the analytical method.

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 matrix spike duplicates 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 (Attachments A-2 and A-3). 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.

ED_014362_00000173-00040

Serial Dilutions

Serial dilutions are performed in conjunction with EPA Method 6010 or 6020 to determine whether or not significant physical or chemical interferences exist due to sample matrix. A serial dilution is performed by analyzing a five-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 Method Detection Limit (MDL). The frequency of analyses, precision goals, and corrective action information pertaining to serial dilutions are provided in the laboratory SOPs in Attachments A-2 and A-3.

Matrix Spikes

Laboratory matrix spike samples are used to evaluate potential sample matrix effects on the accurate quantitation of an analyte using the prescribed analytical method. Matrix Spike /MSD are prepared by adding an analyte to a subsample of a field sample before sample preparation and analyses. A percent recovery is calculated from the concentrations of the analyte in the spiked and unspiked samples. 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.

3.4 Instrument/Equipment Testing, Inspection and Maintenance

In order to ensure continual quality performance of any instruments or equipment, testing, inspection and maintenance will be performed and recorded as described in this section.

3.4.1 Field Equipment

Field equipment will be examined to certify that it is in proper operating order prior to its first 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.

3.4.2 Laboratory Equipment

Instruments used by the laboratories will be maintained in accordance with each laboratory's Quality Assurance Plan and analytical method requirements. All analytical measurement instruments and equipment used by the laboratory will be controlled by a formal calibration and preventive maintenance program.

The laboratories will keep maintenance records and make them available for review, if requested, during laboratory audits. Laboratory preventive 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.

3.5 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 Field Team Leader or Field Team Members.

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

3.6 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 Atlantic Richfield. These records, either electronic or hard copy in form, may include:

- Project work plans with any approved modifications, updates, and addenda;
- Individual property maps (hard copy or scanned field drawings and electronic files);
- Project QAPP, including this QAPP, with any approved modifications, updates, addenda, and any approved corrective or preventative actions;
- Access agreements from property owners;
- 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); and
- DSR.

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 on a daily basis.

Before field and laboratory data are incorporated into the project database, the data and supporting documentation will be subject to appropriate review to ensure 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 project database. All manual data entry into an electronic format will be reviewed by a separate party before such data are incorporated into the database. Laboratory EDDs and related data packages will be reviewed as part of the internal data review process. Following these review steps, field and laboratory electronic data files will be imported to the project database.

ED_014362_00000173-00042

Standardized data import formats and procedures will be used to upload both field and laboratory data into the electronic database. 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 database.

4.0 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 audits of field and laboratory activities include two independent parts: internal and external audits. Internal audits may be performed by Atlantic Richfield, their contractor or a contracted laboratory as necessary. External audits may be performed by the EPA as necessary.

Performance and systems 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.

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. A number of 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 Contractor QA Officer and reported on a Corrective Action Report (CAR) included in Attachment E. In the event that corrective action requests are not in complete accordance with approved project planning documents, the EPA will be consulted and concurrence will be obtained before the change is implemented.

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 Contractor 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 Contractor QA Officer 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 Contractor QA Officer and CPM 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 CPM and Contractor QA Officer. In the event that corrective action requests are not in complete accordance with approved project planning documents, the EPA will be consulted and concurrence will be obtained before the change is implemented. All corrective action records will be included in the QAPP's quality records.

4.2 Corrective Action during Data Assessment

The Contractor QA Officer may identify the need for corrective action 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, etc.). In the event that corrective action requests are not in complete accordance with approved project planning documents, the EPA will be consulted and concurrence will be obtained before the change is implemented. Corrective actions of this type will be documented by the Contractor QA Officer on a CAR and will be included in any subsequent reports.

4.3 Quality Assurance Reports to Management

After investigations are complete, Atlantic Richfield will prepare an annual DSR summarizing the sampling activities described in the QAPP. The laboratory turnaround time for providing sample results will be adequate to allow data review and completion of the annual DSR. The report will describe specific field activities performed during implementation of the QAPP and the physical characteristics of the study area. Each report will include field documentation, documentation of field QC procedures, and results of all field and laboratory audits. The report will also contain a discussion of the data quality assessment. The data quality discussions will contain, on a routine basis, the results of any associated field and laboratory audits, information generated on achieving specific DQOs and a summary of any corrective actions that were implemented and their immediate results on the project. A detailed listing of any deviations from the approved QAPP will also be provided with an explanation for each deviation and a description of the effect on data quality and usability, if any.

The CPM and Contractor QA Officer are responsible for preparing the report. The report will be submitted in draft form to the EPA for review. Upon receipt of comments, the draft report will be revised to address the comments and re-submitted to the EPA for final approval.

5.0 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. In accordance with the *CFRSSI Data Management/Data Validation Plan Addendum* (AERL, 2000), the data review/validation process under this QAPP is streamlined to support the post-ROD decision-making process. The data compiled under this QAPP will be subject to Level A/B criteria review and validation per the EPA National Functional Guidelines and the project DQOs. Data review and validation will be conducted by a qualified technical consultant who is independent from the sampling consultant.

5.1 Data Review and Verification

The process to be used for reviewing and verifying field data and the internal laboratory data reduction process are described in the following sections. Laboratory data reporting requirements, which describe how results are conveyed to data users, are also discussed.

5.1.1 Field Data Review

Raw field data will be entered in field logbooks and/or field data sheets, which will be 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 by an independent party in accordance with Level A/B criteria that apply to sample collection and documentation records. Additionally, field data precision will be assessed by the independent party to verify collection of field duplicate samples in accordance with the specified frequency and to document data precision. The Level A/B Screening Checklist is included in Attachment F.

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 and/or data sheets and that any necessary and appropriate corrective actions were implemented and recorded. Such data will be written into field logbook and/or data sheets 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 proof the field logbooks and/or data sheets 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 project database. Data entries will be made from the reviewed field data sheets or logbooks, and all data entries will be reviewed for accuracy and completeness by a separate party before the electronic file is provided to the database manager. Electronic files of field measurement data will be maintained as part of the project's quality records.

Results of the Level A/B criteria review will be included in annual DSR, which will provide a basis for meaningful interpretation of the data quality and evaluate the need for corrective actions.

5.1.2 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 to Atlantic Richfield. The laboratory is responsible for assigning appropriate flags/qualifiers in accordance with the analytical method and internal laboratory SOPs.

5.1.3 Laboratory Data Reporting Requirements

The laboratory will prepare hard copy data packages for transmittal of results and associated QC information to Atlantic Richfield or their designee. At a minimum, the data packages will include the case narrative, sample results, units and QC sample results.

The laboratory will prepare hard copy data packages for transmittal of results and associated QC information to Atlantic Richfield, or their designee, in general accordance with the *US EPA Contract Laboratory Program (CLP) Statement of Work for Inorganic Superfund Methods (Multi-Media, Multi-Concentration) ISM01.2* (EPA, 2010). Deviations from these specifications may be acceptable provided the hard-copy report presents all of the requested types of information in an organized, consistent and readily reviewable format.

5.1.4 Laboratory Electronic Data Deliverable

Each hard copy data package, as described above, will be accompanied by an EDD prepared by the laboratory. Additional laboratory QC data can be included in the EDD. The EDDs will be cross checked against corresponding hard copy data reports to confirm consistency in results reported in these two separate formats. This cross check will take place as part of the data review process.

5.1.5 Specific Quality Control/Assessment Procedures

Any qualification of the data resulting from the Level A/B criteria review or flags/qualifies identified by the laboratory will be incorporated into the project's electronic database so that all data users are aware of any uncertainties associated with individual results.

5.2 Internal Data Review

Data review is the process of verifying that information generated relative to a given sample is complete and accurate. Data review procedures will be performed for both field and laboratory operations as described below.

5.2.1 Field Quality Control Data

The results of field QC sample analyses associated with each laboratory data package will be reviewed to allow for evaluation of field duplicates and further indications of the data quality. If a problem is identified through the review of field QC data, all related field samples will be identified, and if possible, corrective actions can be instituted and documented on a CAR. In the event that corrective action requests are not in complete accordance with approved project planning documents, the EPA will be consulted and concurrence will be obtained before the change is implemented. If data are compromised due to a problem identified via field QC sample review, appropriate data qualifications will be used to identify the data for future data users.

The handling, preservation and storage of samples collected during the sampling program will be monitored on an on-going basis. The project laboratories will document sample receipt including proper containers and preservation at the time samples are logged in by the laboratory. The sample receipt records (a required data package deliverable), as well as the chain of custody documentation, will also be assessed during data review.

5.2.2 Laboratory Chemistry Data

The second level of review will be performed by the laboratory and will include a review of laboratory performance criteria. The laboratory data quality review will include verification of the following:

- Compliance with the QAPP;
- Holding times (maximum of 6 months for arsenic and lead in soil);
- Instrument calibration verification;
- Laboratory blank analysis;
- Detection limits;
- Laboratory duplicates;
- MS/MSD percent recoveries and relative percent differences;
- Surrogate percent recoveries; and
- Data qualifiers assigned by the laboratory.

Qualifiers that may be applied to the data include the following:

U The analyte was analyzed for but was not detected above the reporting limit.
J The analyte was positively identified; the associated numerical value is an estimate of the concentration of the analyte in the sample.

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.

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

Step 1: Review DQOs and sampling design.
Step 2: Conduct preliminary data review.
Step 3: Apply Statistical test(s) as described in this QAPP to the data se.t
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 limitations of the data.

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

6.0 REFERENCES

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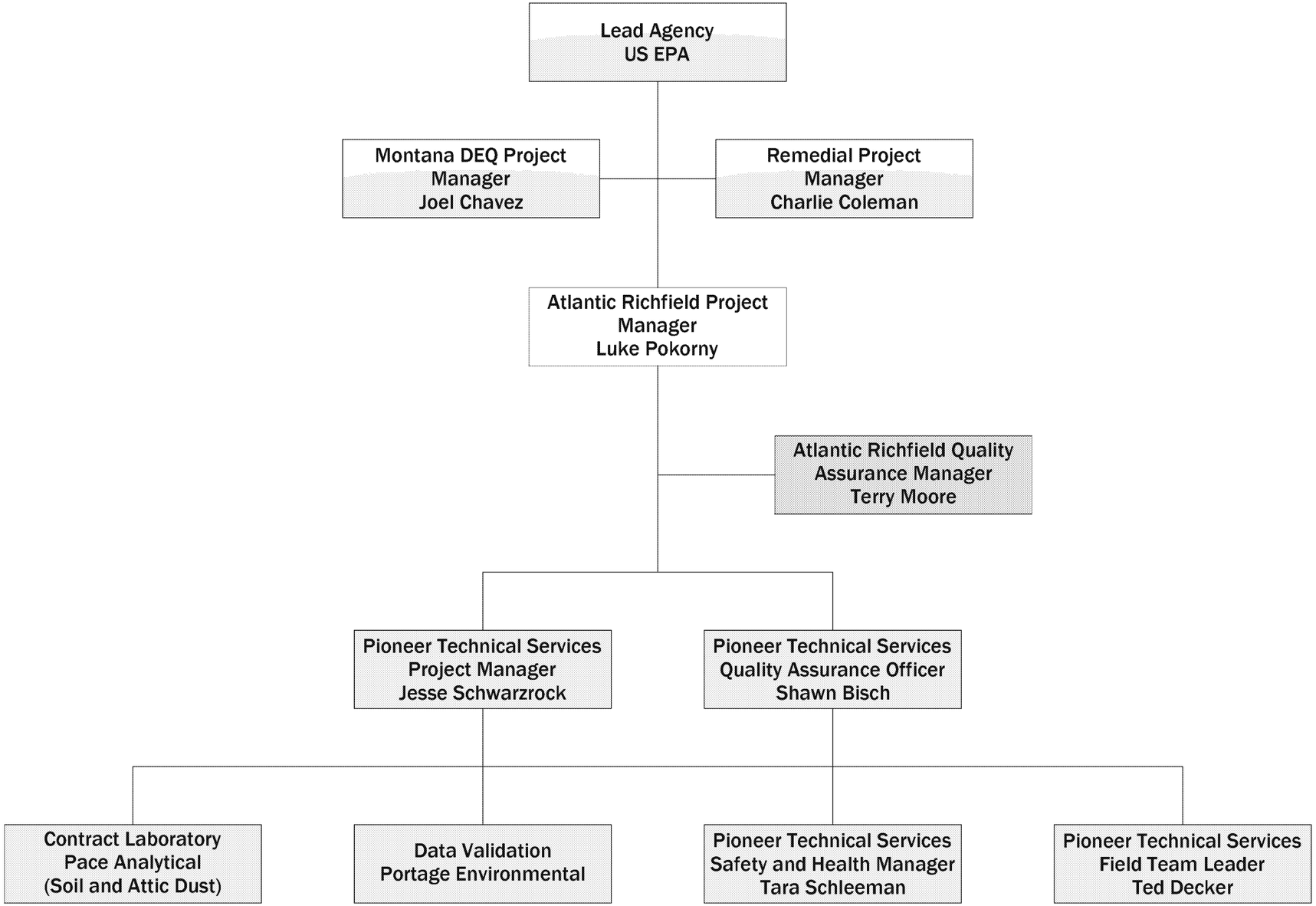
FIGURES



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COORD SYS/ZONE: NA
DATUM: NA
UNITS: NA
SOURCE: PIONEER

SCALE IN FEET
0 NTS NTS

FIGURE 1

CSOU PROJECT ORGANIZATION CHART

PIONEER TECHNICAL SERVICES, INC.
307 E. PARK AVE., SUITE 421
ANACONDA, MONTANA 59711
(406) 563-9371

DATE: 10/16/2015

10/19/2015 11:02:14 AM \\192.168.252.15\PROJECT$\ANADATA\AR\CSOU\2015\DOCUMENTS\DRAFT CS OU FDR (TO AR 4-1-15)\APPENDIX E - CS OU QAPP\FIGURES\CSOU ORGANIZATION CHART.DWG

ADLC SUPERFUND PLANNING DISTRICT

N

	DISPLAYED AS:
COORD SYS/ZONE:	MSPIF
DATUM:	NAD 83
UNITS:	FEET
SOURCE:	PIONEER- NAIP

SCALE IN FEET
0 5000 10000

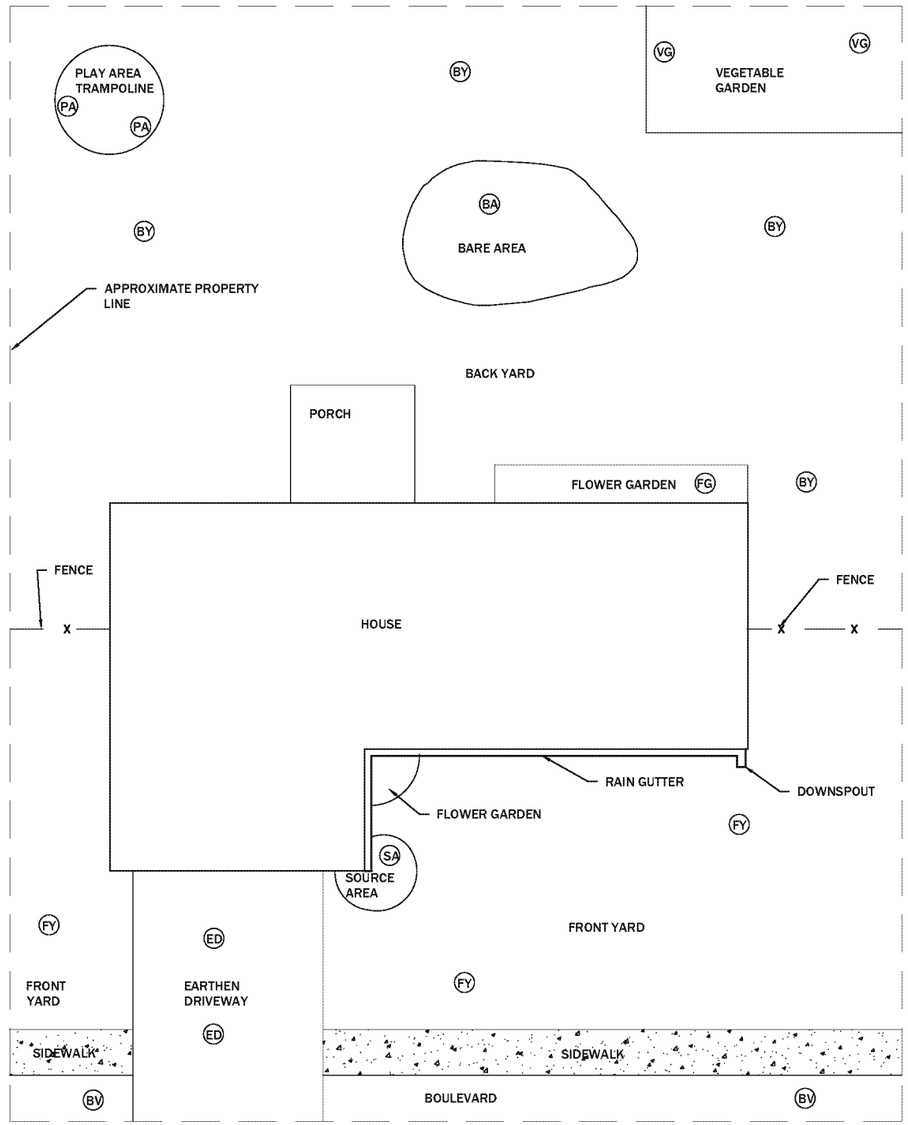
FIGURE 2

PIONEER TECHNICAL SERVICES, INC.
307 E. PARK AVE., SUITE 421
ANACONDA, MONTANA 59711
(406) 563-9371

ALDC SUPERFUND PLANNING AREA OVERLAY DISTRICT (SPAOD)

DATE: 3-12-15

3/12/2015 12:24:19 PM \\192.168.252.15\PROJECT$\ANADATA\AR\CSOU\2014\DOCUMENTS\DRAFT CSOU RAWP FDR\CS OU FDR\CS OU QAPP\FIGURES\ADLC-SPD-FIG-001-14.DWG



LEGEND:

—— ——	PROPERTY LINE		CONCRETE		
— x —	FENCE				
FY	FRONT YARD SUBSAMPLE	ED	EARTHEN DRIVEWAY SUBSAMPLE	BV	BOULEVARD AREA SUBSAMPLE
BY	BACK YARD SUBSAMPLE	BA	BARE AREA SUBSAMPLE	PA	PLAY AREA SUBSAMPLE
VG	VEGETABLE GARDEN AREA SUBSAMPLE	FG	FLOWER GARDEN SUBSAMPLE	SA	SOURCE AREA SUBSAMPLE

N

DISPLAYED AS:
COORD SYS/ZONE: NA
DATUM: NA
UNITS: NTS
SOURCE: PIONEER

SCALE IN FEET
0 NTS NTS

FIGURE 3

PIONEER TECHNICAL SERVICES, INC.
ANACONDA, MONTANA
307 EAST PARK, SUITE 421
(406) 563-9371

EXAMPLE RESIDENTIAL SOIL SAMPLE LOCATION MAP

DATE: 3-12-2015

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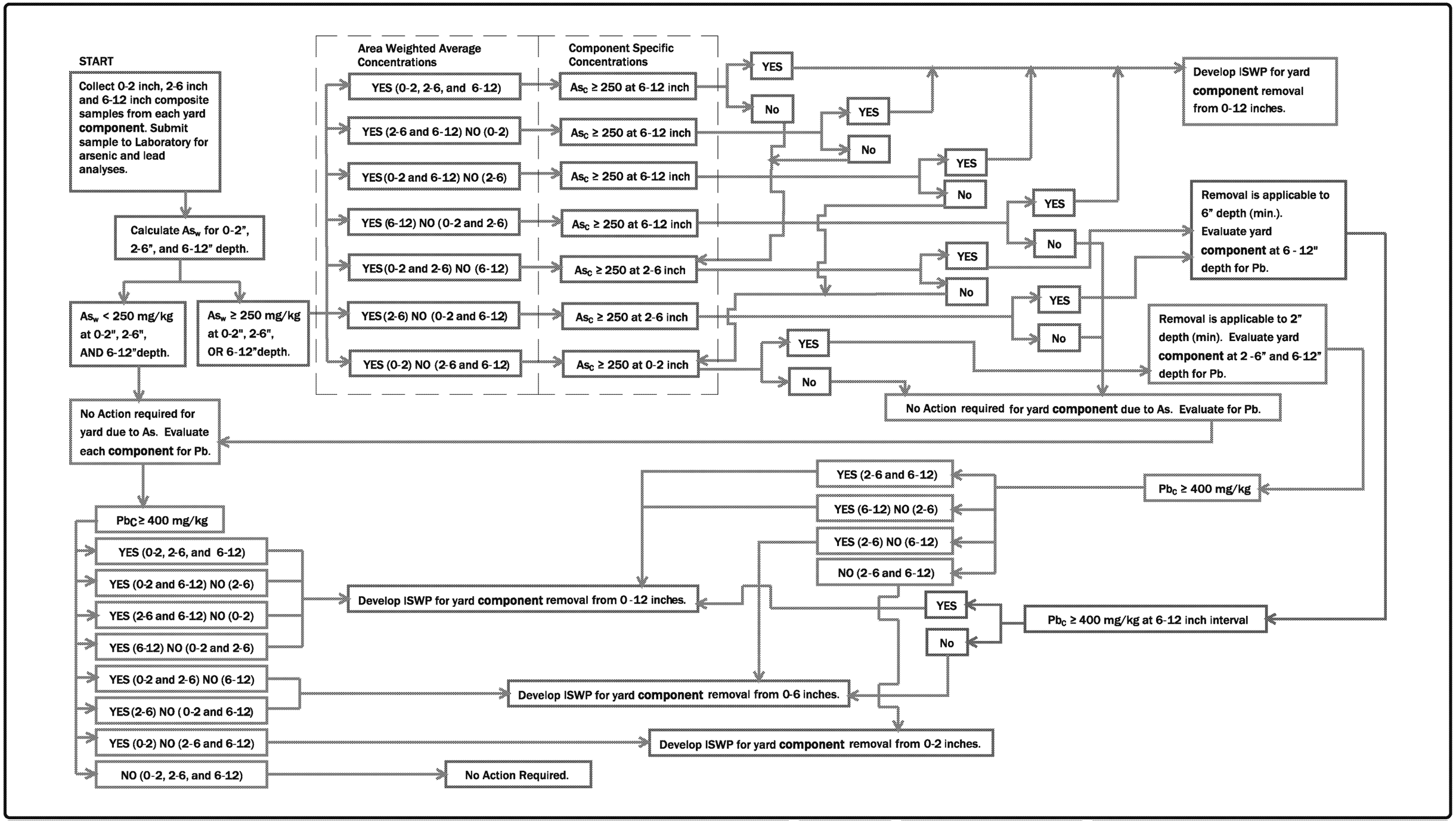


LEGEND:
Pb: Lead
As: Arsenic
As_w: Arsenic Area-Weighted Average Concentration
As_c: Arsenic Component Specific Concentration
Pb_c: Lead Component Specific Concentration
ISWP: Individual Site Work Plan

NOTES:
1. For those properties previously sampled at 0-2 inches, an arsenic area-weighted average currently exists. This area-weighted average will not be recalculated.
2. For vegetable gardens and flower gardens this flow chart will need to be followed in a similar progression, beginning with the 18-24 inch and 12-18 inch depth intervals.

DISPLAYED AS:
COORD SYS/ZONE:
DATUM:
UNITS:
SOURCE:
SCALE IN FEET
0 NTS NTS

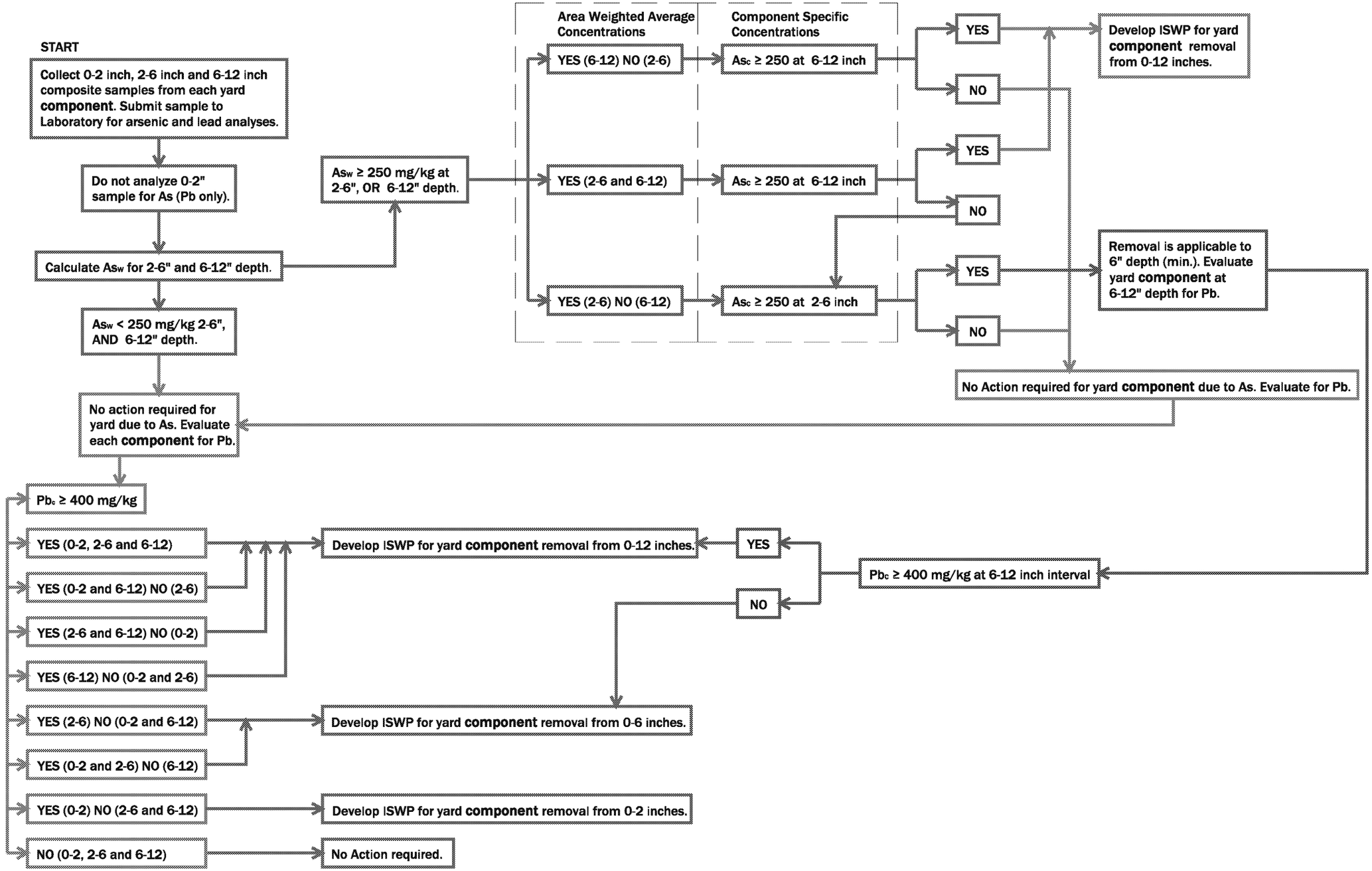
FIGURE 4

PIONEER TECHNICAL SERVICES, INC.
307 E. PARK AVE., SUITE 421
ANACONDA, MONTANA 59711
(406) 563-9371

SAMPLING AND REMEDIAL ACTION FLOW CHART FOR RESIDENTIAL SOILS NOT PREVIOUSLY SAMPLED

DATE: 4/20/2015

4/24/2015 8:09:36 AM \\192.168.252.15\PROJECTS\ANADATA\AR\CSOU\2015\DOCUMENTS\DRAFT FINAL CS OU FDR (TO EPA 4-XX-15)\APPENDIX E - QAPP\FIGURES\CSOU SAMPLING PLAN 4.DWG



LEGEND:
Pb: Lead
As: Arsenic
As_w: Arsenic Area-Weighted Average Concentration
As_c: Arsenic Component Specific Concentration
Pb_c: Lead Component Specific Concentration
ISWP: Individual Site Work Plan

NOTES:
1. For those properties previously sampled at 0-2 inches, an arsenic area-weighted average currently exists. This area-weighted average will not be recalculated.
2. For vegetable gardens and flower gardens this flow chart will need to be followed in a similar progression, beginning with the 18-24 inch and 12-18 inch depth intervals.

DISPLAYED AS:
COORD SYS/ZONE:
DATUM:
UNITS:
SOURCE:
SCALE IN FEET
0 NTS NTS

FIGURE 5

PIONEER TECHNICAL SERVICES, INC.
307 E. PARK AVE., SUITE 421
ANACONDA, MONTANA 59711
(406) 563-9371

YARD SAMPLING FLOW CHART FOR **COMPONENT** PREVIOUSLY SAMPLED BUT NOT SUBJECT TO RA

DATE: 4/20/2015

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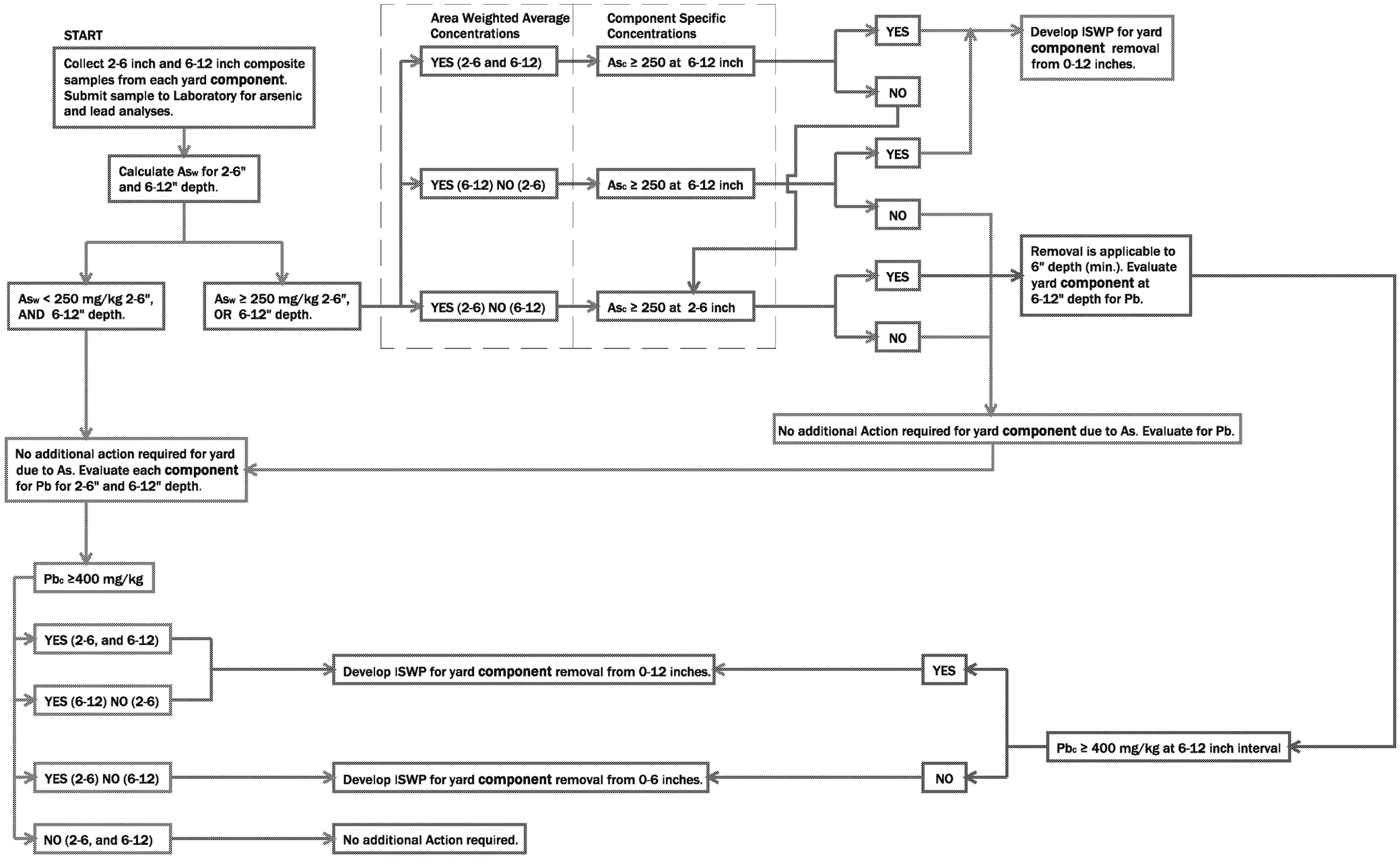

LEGEND:
Pb: Lead
As: Arsenic
As_W: Arsenic Area-Weighted Average Concentration
As_C: Arsenic Component Specific Concentration
Pb_C: Lead Component Specific Concentration
ISWP: Individual Site Work Plan

NOTES:
1. For those properties previously sampled at 0-2 inches an arsenic area-weighted average currently exists. This area-weighted average will not be recalculated.
2. For vegetable gardens and flower gardens this flow chart will need to be followed in a similar progression, beginning with the 18-24 inch and 12-18 inch depth intervals.

DISPLAYED AS:
COORD SYS/ZONE:
DATUM:
UNITS:
SOURCE:
SCALE IN FEET
0 NTS NTS

FIGURE 6

PIONEER TECHNICAL SERVICES, INC.
307 E. PARK AVE., SUITE 421
ANACONDA, MONTANA 59711
(406) 563-9371

YARD SAMPLING FLOW CHART FOR **COMPONENT** PREVIOUSLY REMEDIATED AT 0" - 2" DEPTH

DATE: 4/20/2015

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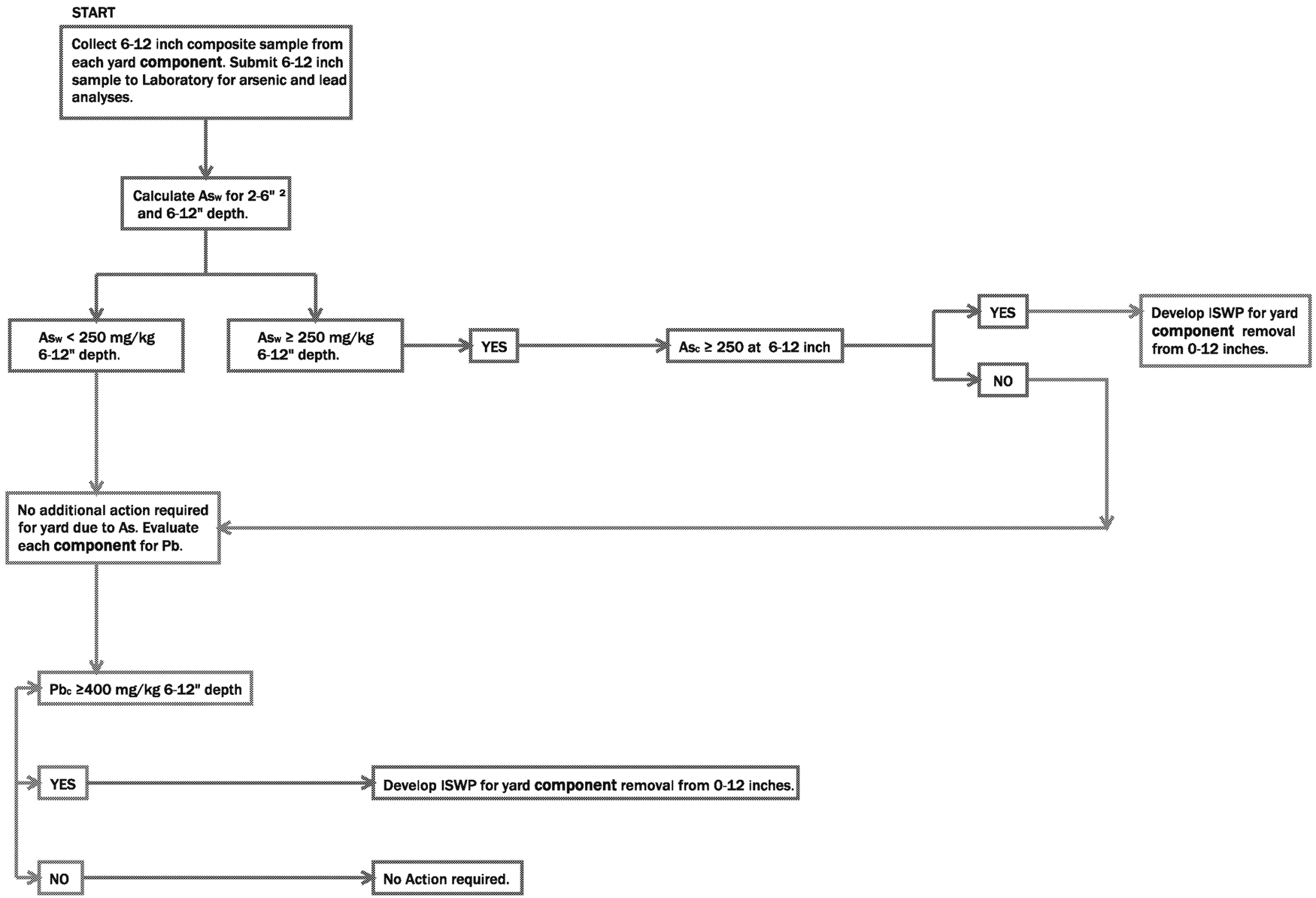

LEGEND:
Pb: Lead
As: Arsenic
As_W: Arsenic Area-Weighted Average Concentration
As_C: Arsenic Component Specific Concentration
Pb_C: Lead Component Specific Concentration
ISWP: Individual Site Work Plan

NOTES:
1. For those properties previously sampled at 0-2 inches an arsenic area-weighted average currently exists. This area-weighted average will not be recalculated.
2. When calculating the arsenic area weighted average, in previously remediated components, an average arsenic concentration of 19.1 mg/kg will be applied to all previously placed clean cover soil.
3. For vegetable gardens and flower gardens this flow chart will need to be followed in a similar progression, beginning with the 18-24 inch and 12-18 inch depth intervals.

DISPLAYED AS:
COORD SYS/ZONE:
DATUM:
UNITS:
SOURCE:
SCALE IN FEET
0 NTS NTS

FIGURE 7

PIONEER TECHNICAL SERVICES, INC.
307 E. PARK AVE., SUITE 421
ANACONDA, MONTANA 59711
(406) 563-9371

YARD SAMPLING FLOW CHART FOR **COMPONENT** PREVIOUSLY REMEDIATED AT 0" - 6" DEPTH
DATE: 8/7/2015

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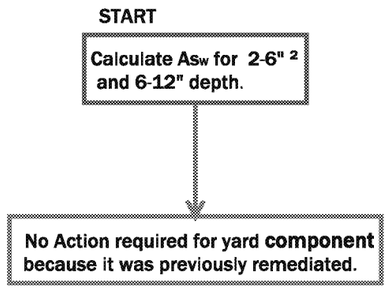

LEGEND:
Pb: Lead
As: Arsenic
As_w: Arsenic Area-Weighted Average Concentration
As_c: Arsenic Component Specific Concentration
Pb_c: Lead Component Specific Concentration
ISWP: Individual Site Work Plan

NOTES:
1. For those properties previously sampled at 0-2 inches an arsenic area-weighted average currently exists. This area-weighted average will not be recalculated.
2. When calculating the arsenic area weighted averages, in previously remediated components, an average arsenic concentration of 19.1 mg/kg will be applied to all previously placed clean cover soil.
3. For vegetable gardens and flower gardens this flow chart will need to be followed in a similar progression, beginning with the 18-24 inch and 12-18 inch depth intervals.

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

SCALE IN FEET
0 NTS NTS

FIGURE 8

PIONEER TECHNICAL SERVICES, INC.
307 E. PARK AVE., SUITE 421
ANACONDA, MONTANA 59711
(406) 563-9371

YARD SAMPLING FLOW CHART FOR **COMPONENT** PREVIOUSLY REMEDIATED AT 0" - 12" DEPTH

DATE: 8/7/2015

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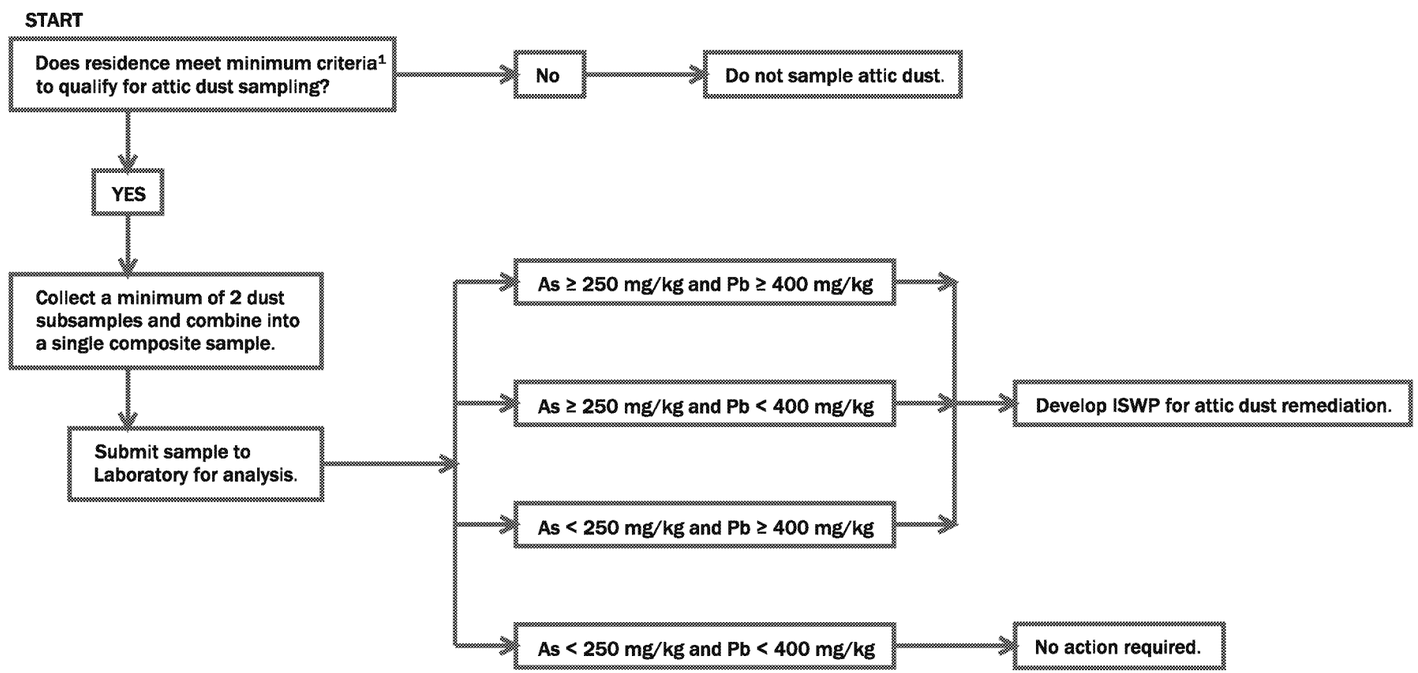

LEGEND:
Pb: Lead
As: Arsenic
ISWP: Individual Site Work Plan

[1] - Criteria to qualify for attic dust sampling is detailed in RAWP/FDR text, Section 3.2 Attic Dust.

DISPLAYED AS:
COORD SYS/ZONE:
DATUM:
UNITS:
SOURCE:
SCALE IN FEET
0 NTS NTS

FIGURE 9

SAMPLING AND REMEDIAL ACTION FLOW CHART FOR ATTIC DUST

PIONEER TECHNICAL SERVICES, INC.
307 E. PARK AVE., SUITE 421
ANACONDA, MONTANA 59711
(406) 563-9371

DATE: 4/20/2015

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TABLES

TABLE 1: CS OU PROJECT SCHEDULE

Dates:	Tasks:
2nd Quarter 2015	Submit Draft Final CS OU Residential Soils/Dust RAWP/FDR to Agencies for review and approval.
3rd Quarter 2015	Submit Agency approved Final CS OU Residential Soils/Dust RAWP/FDR.
3rd Quarter 2015	Initiate access agreement process for those residences that have soil lead data exceeding the action limit as well as those that have requested sampling[1].
4th Quarter 2015 – 2021	Perform soil and attic dust sampling activities[2] (pending receipt of completed access agreements).
2nd Quarter 2016 – 2021	Perform remedial action activities[3].

[1] – Soil and dust sampling requests will be accepted until December 31, 2020. Past this date, additional sampling requests will be addressed by Atlantic Richfield or its contractor in accordance with the requirements of the Institutional Controls Implementation and Assurance Plan (ICIAP).

[2] – After data validation activities are completed, results for all analysis (arsenic and lead) will be reported to each individual landowner as well as the Agencies.

[3] – This schedule will allow the CS OU sampling and RA program to be subject to an Agency 5-year review.

TABLE 2: PRECISION, ACCURACY AND COMPLETENESS CALCULATION EQUATIONS

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

Table 3: Example Arsenic Area Weighted Average Calculation

Residential yard sampling

	Yard Components[a]	Total Area of Component[a] (square feet)	Number of Subsamples[b]	Percentage of Yard Area	Component Concentration[a] (mg/kg)	Fraction of Yard	Composite Concentration[c] (mg/kg)
FY	Front yard	1,875	3	36%	240	0.36	85.6
BY	Back yard	2,250	4	43%	210	0.43	89.8
VG	Vegetable Garden	300	2	5.7%	83	0.057	4.7
ED	Earthen driveway	425	2	8.1%	117	0.081	9.5
SA	Source area	225	2	4.3%	630	0.043	26.9
BA	Bare area	175	1	3.3%	390	0.033	13.0
BV	Boulevard	10	1	0.19%	255	0.0019	0.48
	Total	5,260	15	100%		1.0	230

Note: [a] These are for example purposes only and are not based on site data.

[b] Number of subsamples required to achieve a sampling density of at least 1 sample per 625 square feet of area per CSOU-SOP-S-04. Multiple subsamples will be homogenized in the field and a single composite sample submitted to the laboratory for analysis.

[c] Composite concentration is the sum of the area-weighted concentrations (component concentration * fraction of yard)

ATTACHMENT A

STANDARD OPERATING PROCEDURES

Attachment A-1:	**CS OU Field Standard Operating Procedures**
Attachment A-2:	**Soil Laboratory Standard Operating Procedures**
Attachment A-3:	**Attic Dust Laboratory Standard Operating Procedures**

ATTACHMENT A-1

CS OU FIELD
STANDARD OPERATING PROCEDURES

Attachment A-1
CS OU Field SOP
Index

SOP Number	SOP Title	# Pages	Equivelant CFRSSI SOP
CSOU-SOP-S-01	Surface Soil Sampling	4	SS-4 Residential Yard/Lot Soil Sampling Procedures; SS-6 Compositing Soil Samples
CSOU-SOP-S-04	Residential Yard/Lot Soil Sampling	8	SS-4 Residential Yard/Lot Soil Sampling Procedures; SS-6 Compositing Soil Samples
CSOU-SOP-S-06	Test Pit Sampling	4	SS-4 Residential Yard/Lot Soil Sampling Procedures; SS-6 Compositing Soil Samples
CSOU-SOP-D-01	Attic Dust Sampling with a Micro-Vacuum	11	-
CSOU-SOP-SA-01	Soil Sample Packaging and Shipping	2	G-5 Sample Packaging and Shipping
CSOU-SOP-SA-03	Field Quality Control Samples	1	SS-4 Residential Yard/Lot Soil Sampling Procedures; SS-6 Compositing Soil Samples
CSOU-SOP-SA-04	Chain of Custody Forms for Environmental Samples	3	G-7 Sample Custody
CSOU-SOP-SA-05	Project Documentation	2	G-4 Field Logbook/Photographs
CSOU-SOP-DE-01	Personnel Decontamination Procedures	5	G-8 Decontamination of Equipment used to Sample Soil and Water
CSOU-SOP-DE-02	Equipment Decontamination	3	G-8 Decontamination of Equipment used to Sample Soil and Water

COMMUNITY SOILS OPERABLE UNIT

STANDARD OPERATING PROCEDURE
CSOU-SOP-S-01
SURFACE SOIL SAMPLING

A surface sample is defined as a mineral soil sample collected from immediately beneath the vegetative mat. It generally includes some interval from the upper six inches of soil. Surface sampling under biased conditions may be selected after considering factors such as type of contaminant, length of time the area has been contaminated, the type of soil and the past use of the area.

GRAB SAMPLE:

Sample collection devices include stainless steel scoops or trowels, disposable Teflon trowels or for inorganic contaminants disposable plastic scoops. The following procedure is designed to be used to collect a surface soil sample from the 0-2 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. These procedures are **not to** be used when sampling for volatile organic compounds. The procedure for collecting volatile organic samples is included in Section 3 of this SOP.

1. Locate the site as directed in the appropriate Quality Assurance Project Plan (QAPP).

2. Complete a site walk through and determine any site specific hazards associated with the sampling area. Discuss with sampling crew and note in the field logbook. During the site walk through, note possible locations for underground utilities. As an example identify where natural gas pipes enter any structures on the property or if yard lights or street lights are present with no overhead lines. Determine if an underground sprinkling system is present. If sample locations have not been assigned in the QAPP, note the probable locations of underground utilities and try to avoid those areas when choosing sample locations. If sample locations are identified in the QAPP use the appropriate survey method to locate.

3. Dig a 6 to 12-inch square pit to a depth of approximately 6 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. If the surface material is coarse-grained material free of intermixed materials (i.e., graveled driveway) the sample will be collected from the layer below the protective barrier. However, if the graveled driveway, alley or lot contains soil/dust material on the surface the sample will be collected from the appropriate interval. If the sample area is unvegetated the sample material will be collected from the designated depth intervals below ground surface.

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4. Measure the interval to be sampled (0-2 inches or 0-6 inches) with a stainless steel tape measure or a ruler and mark the appropriate interval.

5. Scrape the walls of the sample pit within the marked interval with a decontaminated stainless steel trowel or scoop, a Teflon scoop, or a disposable plastic scoop to expose a clean surface.

6. Once the wall of the test pit has been cleaned, collect the sample by scraping the appropriate interval on the cleaned face of the pit with the sampling tool and placing the material in a decontaminated stainless steel bowl, or a new cleaned foil pan.

7. Remove all coarse fragments greater than 0.5 inches from the bowl. Mix the remaining material in the bowl with the sampling tool.

8. Transfer the soil sample directly into the appropriate sample container according to Community Soils Operable Unit Standard Operating Procedure (*Soil and Water Sample Packaging and Shipping*) (CSOU-SOP-SA-01) and store in a cooler at 4°C or less.

9. Record appropriate information about the sample collection in the field logbook.

10. Decontaminate sampling tools according to procedures outlined in Community Soils Standard Operating Procedure (*Equipment Decontamination*) (CSOU-SOP-DE-02).

COMPOSITE SAMPLING

In many situations a composite sample is more appropriate for sample collection than a grab sample. Several types of composite samples can be collected. A biased composite sample can be collected by the sampler identifying specific spots within the sample area that appear to be contaminated or not contaminated and digging sample pits in those locations. Composite samples can also be collected randomly as defined in the QAPP.

Sub samples are often collected in a five-point (star) pattern. At each point, a subsample of a predetermined depth is collected. The diagonal distance between points is commonly ten feet depending on the area of soil homogeneity. Sub samples can also be collected in a three-point (triangular) pattern. At each point, a subsample of predetermined depth is collected. The diagonal distance between the points is commonly ten feet depending on the area of soil homogeneity. The precise method for compositing the sample will be discussed in the QAPP. Each subsample test hole will be prepared and sampled in the manner discussed above under Grab Samples.

1. Composite samples will consist of discrete aliquots of equal amounts of soil from each subsample location. The soil aliquots will be collected into a stainless steel bowl and thoroughly mixed. 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, labeled, stored in a cooler with ice and maintained at 4 degrees Celsius (°C) until delivered to the laboratory. Any remaining sample material will be returned to

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the sample holes. A sufficient quantity of soil will be collected in each sample container to provide for analysis with additional soil left over to be archived. An alternative method of compositing soil subsamples is with a large disposable plastic or canvas sheet. The subsamples are mixed in the center of the sheet. Each corner is pulled up and toward the diagonally opposite corner. This process is done from each corner. After the soil is mixed, it is again spread out on the cloth into a relatively flat pile. The pile is quartered. A small scoop is used to collect small samples from each quarter until the desired amount of soil is acquired. Note: High concentrations of organic chemicals in soils can react with the plastic sheet. The sampler may also "eyeball" an equal amount of sample material from each hole into a resealable plastic bag (i.e. Ziploc®). The sample material would be thoroughly mixed between each subsample pit and prior to placing in the appropriate sample containers.

2. Remove all coarse fragments greater than 0.5 inches from the bowl. Mix the remaining material in the bowl with the sampling tool.

3. Transfer the soil sample directly into the appropriate sample container according to Community Soils Operable Unit Standard Operating Procedure (*Soil and Water Sample Packaging and Shipping*) (CSOU-SOP-SA-01) and store in a cooler at 4°C or less.

4. Record appropriate information about the sample collection in the field logbook.

5. Decontaminate sampling tools according to procedures outlined in Community Soils Operable Unit Standard Operating Procedure (*Equipment Decontamination*) (CSOU-SOP-DE-02).

VOLATILE ORGANIC SAMPLING

1. Locate the site as directed in the appropriate QAPP.

2. Do a site walk through and determine any site specific hazards associated with the sampling area. Discuss with sampling crew and note in the field logbook. During the site walk through note possible locations for underground utilities. As an example identify where natural gas pipes enter any structures on the property or if yard lights or street lights are present with no overhead lines. If sample locations have not been assigned in the QAPP, note the probable locations of underground utilities and try to avoid those areas when choosing sample locations. If sample locations are identified in the QAPP use the appropriate survey method to locate.

3. Dig a 6 to 12-inch square pit to a depth of approximately 6 inches. The size and depth of the sample pit required would depend on the amount of material needed for sample analysis and the interval being 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 bottle to dislodge any mineral soil particles. All dislodged particles shall be placed in the sample. If the surface material is coarse-grained material free of intermixed materials (i.e., graveled driveway) the sample

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will be collected from the appropriate layer below the protective barrier. However, if the graveled driveway, alley or lot contains soil/dust material on the surface the sample will be collected from the appropriate interval. If the sample area is unvegetated the sample material will be collected from appropriate depth below ground surface.

4. Measure the interval to be sampled (0-2 inches or 0-6 inches) with a stainless steel tape measure or a ruler and mark the appropriate interval.

5. Scrape the walls of the sample pit within the marked interval with a decontaminated stainless steel trowel or scoop, a Teflon scoop, or a disposable plastic scoop to expose a clean surface.

6. After the face of the test pit has been cleaned either immediately place the sampling container into the sample pit and collect the sample by scraping the appropriate interval of mineral soil directly into the sample container, material should be packed in as tightly as feasible and the sampler should try to avoid getting large particles in the jar. The sampling container should be filled to the top with little to no headspace and the lid placed on the container as soon as the jar is full. The sample should be placed immediately in a cooler at 4°C or less.

7. Record appropriate information about the sample collection in the field logbook.

8. Decontaminate sampling tools according to procedures outlined in Community Soils Operable Unit Standard Operating Procedure (*Equipment Decontamination*) (CSOU-SOP-DE-02).

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COMMUNITY SOILS OPERABLE UNIT

STANDARD OPERATING PROCEDURE
CSOU-SOP-S-04
RESIDENTIAL YARD/LOT SOIL SAMPLING

The purpose of this standard operating procedure (SOP) is to ensure that a consistent sampling approach is used at Superfund Sites for the delineation of residential areas that may require remediation to protect the public health. This SOP is applicable to areas within a residential tract such as yards, vacant lots, playgrounds, parks, and other areas in and adjacent to residential yards (yards/lots).

INTRODUCTION

Prior to the use of this SOP, other less intensive sampling designs may be required to indicate the need for sampling at this scale. Sampling performed according to this SOP will supply yard/lot-specific analytical data from which remedial action decisions can be made. If remedial action is necessary, either the property boundary or a smaller natural boundary within the yard/lot will be used to establish the area of remediation. Soil sampling conducted to determine whether a remedial action is necessary in the "use areas" utilized these boundaries to delineate the sample areas.

Samples representing use areas such as half yards/lots, gardens/flowerbeds, earthen driveways, rock gardens, play areas, bare areas, earthen driveways, and opportunity areas (e.g., areas underneath a porch at a residence with children and pets), and source areas are collected in suspect residential areas. Composite sampling is used to characterize the average concentration of inorganic constituents of concern in the use areas. The number of subsamples comprising a composite sample and the total area composited is standardized to limit sampling to similar sized areas for comparative purposes.

SAMPLING APPROACH

The approach to yard/lot sampling is based on composite sampling of selected use areas of a residential yard/lot. The composite sample best represents constituent concentrations within a use area by averaging subsamples collected at locations that spatially represent the area.

COMPOSITE SAMPLE AREAS

Composite sample areas within a yard/lot will be selected prior to sampling. These sampling areas are determined by using natural boundaries which divide the use areas of a yard/lot. Boundaries may include property or fence lines, front and back yard divisions, driveways, and
vegetable and flower garden boundaries. The following use areas are considered separate composite sample areas.

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YARD/LOT COMPOSITES

Yards are divided into front and back yard composite sample areas. This is the only division for sampling and removal in yards unless a front or back yard exceeds 5,000 square feet or if it contains selected smaller use areas such as gardens, earthen driveways, play areas, bare areas, opportunity sampling areas, or source areas described below.

Lot areas (vacant lots, playgrounds, parks, and other adjacent areas) exceeding 5,000 square feet in size, will be divided up into equal sized areas for sampling and removal purposes. The sample collection density and depth intervals should be carried out in accordance with the "yard/lot composite" sections. Smaller selected use areas such as gardens, earthen driveways, play areas, bare areas, opportunity sampling areas, or source areas are applicable as described below.

VEGETABLE AND FLOWER GARDEN COMPOSITE

Subsamples are collected in the vegetable gardens (i.e., areas where vegetables are grown for the intent of consumption) and flower gardens (i.e., areas where flowers are grown for aesthetic purposes) within a yard and combined into composite samples. The garden samples are collected separately because of the potential increased exposure of residents to soil through normal gardening activities. The garden soils may also differ from the yard soils.

PLAY AREA COMPOSITE

Subsamples are collected in the play areas within a yard and combined into composite samples. The play area subsamples are collected separately because of the potential increased exposure of children to soil through play-time activities as play areas often contain minimal vegetation and areas of exposed soil.

EARTHEN DRIVEWAY COMPOSITE

The earthen driveway composite sample is separate from the yard composite. This area is sampled separately because the driveway material may differ from the yard soil in type and origin. This method will allow for separate remediation decisions based on the concentrations of constituents of concern in each composite sample.

SOURCE AREA COMPOSITE

A composite sample is collected in potential source areas (waste rock piles, mine dumps, etc.) within yards or residential lots. This composite sample characterizes the surface material in the source areas where direct exposure to residents may occur and identifies the potential effect of the source area on the yard or residential lot through runoff.

In cases where a potential source area is contained within two or more yard or lot boundaries, these property boundaries are used as sampling limits when selecting subsample sites for the source area composite. Characterization sampling of a potential source area for purposes of determining environmental risk is outside the scope of this

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SOP.

OPPORTUNITY COMPOSITE

Subsamples are collected in the areas within a yard where dissimilar materials are noted and combined into composite samples. The opportunity samples are collected separately because of the material differences between the noted materials and the yard soils.

SAMPLE COLLECTION

Samples will be collected as use area composites described previously. Subsample density and locations within the composite areas are determined based on the size of the area to be represented by the subsample, and specific locations within the composite areas that may require sampling. The depth interval from which samples are collected within the composite area is dependent on the area type. Subsample density, location, and depth intervals are discussed in the following sections.

SUBSAMPLE DENSITY AND LOCATION

A minimum of two and a maximum of eight subsamples are collected for any composite sample. The subsample density is determined by the type of composited area. The area represented by a subsample will necessarily decrease where the composite area is small or where other natural boundaries require additional subsamples to spatially represent the composite area. Subsample density and locations are discussed below.

YARD/LOT COMPOSITES

Yard/lot composite subsamples are collected at a density of approximately one for every 625 square feet (25 feet by 25 feet) of front or back yard area as determined through conventional or GPS survey. The subsamples are located within the yard/lot area to be spatially representative.

Using this density, subsamples are collected at selected locations within the yard/lot to characterize the yard/lot with respect to specific areas where constituents of concern may be concentrated or may be most exposed to residents. These areas are roof drip lines where no collection gutter exists, topographically low areas, play areas, bare areas, areas adjacent to downspouts, or areas that are near a potential source such as a waste rock dump. These subsample locations are selected to best fit the established yard/lot composite subsample density.

VEGETABLE AND FLOWER GARDEN COMPOSITE

Vegetable and flower garden composite samples include at least two subsamples per total garden area and a maximum of eight subsamples. All garden areas of reasonable size will be included in the composite. Subsample density is approximately one for every 100 square feet.

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PLAY AREA COMPOSITE

Play area composite subsamples are collected at the same approximate density as subsamples for a yard component (1 for every 625 square feet). A minimum of two and a maximum of eight subsamples will be composited. The subsample sites should be equally spaced within the play area. All play areas of reasonable size will be included in the composite.

EARTHEN DRIVEWAY COMPOSITE

Subsamples are collected in earthen driveways at the same approximate density as subsamples for a yard composite (1 for every 625 square feet). A minimum of two subsamples and a maximum of eight will be composited. The subsamples sites should be equally spaced within the earthen driveway and areas where noticeable fuel or oil spills have occurred should be avoided.

SOURCE AREA COMPOSITE

Potential source area composite subsamples will be collected at the same approximate density as yard/lot composite subsamples (1 for every 625 square feet of source area). Again, a minimum of two subsamples and a maximum of eight will comprise the composite sample. The subsample sites should be equally spaced within the source area.

OPPORTUNITY COMPOSITE

Opportunity composite subsamples will be collected at the same approximate density as yard/lot composite subsamples (1 for every 625 square feet of source area). A minimum of two and a maximum of eight subsamples will comprise the composite sample. The subsample sites should be equally spaced within the area from which the opportunity sample is being taken.

SAMPLING DEPTH INTERVALS BY COMPOSITE AREA

This SOP addresses residential yard or lot sampling to decide whether a remedial action is required in the yard or lot. The depth of sampling in a yard/lot is determined by the potential for exposure of residents to soil through everyday activities. The depth of the remediation within a yard/lot will be determined using data from other large scale sampling efforts that will characterize the depth of constituents of concern in the soil and determine their origin and mode of transportation. For example, where an area has been subjected to outfall constituents, only surficial soil removal or capping may be required to mitigate the exposure. In contrast, an area where the soil has been mixed with mine waste rock or mill tailings, a deeper removal or capping effort or both may be required.

All subsample locations will be plotted on the map representing each residential yard. Photographs will be taken of yard components and any unusual features, as deemed necessary by field personnel. All information will be recorded on field data sheets and/or in the field logbook.

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SAMPLE DEPTHS

Most residential yard/lot areas are expected to be covered with grass; consequently, a surface sample will be collected from immediately beneath the vegetative mat, or in the absence of vegetation, 0 to 2 inches bgs. If a vegetative mat (sod) is present, it will be separated from the soil surface with a stainless steel knife or equivalent. The removed vegetative mat will be shaken and scraped over the sample collection bowl to dislodge any soil particles. All dislodged particles will be placed in the sample.

Exceptions to this procedure will occur when the sample location falls on a graveled driveway or similar surface. If the surface material is coarse-grained material free of intermixed materials, the sample will be collected from the 0- to 2-inch soil layer immediately beneath the coarse-grained material. However, if the graveled driveway or similar surface contains soil/dust material on the surface, the sample will be collected from the surface, 0- to 2-inch layer.

Subsurface samples from the front yard, back yard, side yard (as applicable), play areas, unpaved driveways, gardens, drip zones and bare areas will be collected from the following depth intervals: 2 to 6 inches bgs and 6 to 12 inches bgs. Decisions regarding collection of additional "opportunistic" samples will be made in the field by sampling personnel and/or Agency personnel.

Subsurface samples from vegetable and flower gardens will be collected from the following depth intervals: 2 to 6 inches bgs; 6 to 12 inches bgs; 12 to 18 inches bgs; and 18 to 24 inches bgs.

SAMPLE DENSITY, LOCATION, AND COMPOSITING

One subsample per individual yard component (e.g., front yard, back yard, garden, etc.) will be collected unless the component is greater than 625 square feet (ft2) (25 feet by 25 feet) in surface area. If the yard component exceeds 625 ft2, additional subsamples will be collected to meet the sample collection density of 1 subsample for each 625 ft2 (in accordance with the project- specific approved SAP). For gardens, earthen driveways, and source areas (areas of visible smelter-related waste material), a minimum of two subsamples will be collected regardless of the surface area involved. A single composite sample representing each individual yard component and depth interval will be submitted to the laboratory for analysis. The sampler will record any unusual soil properties, such as bare surface conditions, presence of foreign materials, staining, etc. Additionally, for gardens, the type of garden will be noted in the field logbook during sampling. Non-drip zone samples will be located outside of the drip zone and away from influences of any other painted surfaces; otherwise, the presence of paint chips in the sample area (or presence of deteriorating paint on siding, foundations, etc.) will be noted if space is not available to avoid these areas. Each composite sample will consist of equal aliquots of soil collected from the corresponding depth interval in adjacent sample locations representing a common yard component.

Subsamples will be collected from within the drip zone of each residence if possible (i.e., at times concrete or other obstruction makes it impossible to gather a subsample within the drip

zone). The drip zone subsamples will be included in the nearest component's composite sample. Distinct drip zone samples will not be collected. The drip zone subsample locations will be situated, generally, between 6 and 30 inches from the exterior of the house and near the mid-point of each side of the house. Subsamples will be collected from within rock gardens of each residential property (if applicable). The rock garden subsamples will be included in the nearest component's composite sample. Disting rock garden samples will not be collected. Additionally, composite samples will be collected from distinct play areas, vegetable gardens, and unpaved driveways. These composite samples will consist of a minimum of two subsamples (see note above) collected from the corresponding depth intervals.

Composite samples will consist of discrete aliquots of equal amounts of soil from each depth interval for a given component of a residential yard. The soil aliquots will be collected into a stainless steel bowl and 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, labeled, stored in a cooler with ice and maintained at 4 degrees Celsius (°C) until delivered to the laboratory. Any remaining sample material will be returned to the sample holes. A sufficient quantity of soil will be collected in each sample container to provide for analysis with additional soil left over to be archived. All of the samples (100%) will be archived through the duration of the holding time.

If the EPA (or the EPA Contractor) chooses to collect split samples, an adequate quantity of soil will be made available by the sampler at the time of sample collection. However, the EPA (or EPA Contractor) will be responsible for providing sample containers and coolers, etc., for storing and shipping split samples. These items will not be provided by the samplers.

YARD/LOT COMPOSITES

The top two inches of soils is where the primary exposure to residents occurs in yard/lot areas. Most yard/lot areas are covered with grass that generally limits exposure to deeper soils. Because of this exposure buffer, front or backyard composite samples are collected as surface samples. A surface sample is defined as a mineral soil sample collected from zero to two inches immediately below the vegetative mat, or in the absence of vegetation, zero to two inches bgs. Subsample intervals for yard/lot composite areas can be taken at intervals of 0 to 2 inches, 2 to 6 inches, and 6 to 12 inches bgs.

VEGETABLE AND FLOWER GARDEN COMPOSITE

The deepest potential exposure of residents to soils in a yard occurs in the garden areas. During normal gardening activities, the soil is tilled and cultivated, and is generally in physical contact with residents. Normal tilling depths are 12 inches or less, but infrequent work may occur at greater depth so subsample intervals for garden areas can be taken at 0 to 2 inches, 2 to 6 inches, 6 to 12 inches, 12 to 18 inches, and 18 to 24 inches bgs. Garden samples are taken using sampling procedures that are best fit to the soil type and obtain and adequate volume of soil for analytical and archiving purposes.

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PLAY AREA COMPOSITE

The greatest potential risk to exposure of residents to material within a play area is at the material surface, however infrequent activities such as digging in play areas may results in contact at greater depth. Play area subsamples can be taken at depths of 0 to 2 inches, 2 to 6 inches, and 6 to 12 inches bgs.

EARTHEN DRIVEWAY COMPOSITE

Earthen driveways are generally well-compacted due to driveway use. Compaction usually reduces the exposure to the deep sections of soil beneath a driveway, however infrequent work may result in resident contact with deeper soils. Earthen driveway subsamples can be taken at 0 to 2 inches, 2 to 6 inches, and 6 to 12 inches bgs.

SOURCE AREA COMPOSITE

The greatest potential risk to exposure of residents to material within a potential source area is at the material surface. Exposure may occur through direct contact with the material, or areas affected by erosional re-deposition onto an adjacent surface of the yard/lot as well as contact with at great depth if infrequent digging activities occur. Therefore, subsamples comprising the source area composite can be taken at 0 to 2 inches, 2 to 6 inches, and 6 to 12 inches bgs.

OPPORTUNITY COMPOSITE

The greatest potential risk to exposure of residents to material within an area from which an opportunity sample is collected is at the material surface. Exposure may occur through direct contact with the material, or during infrequent digging activities. Therefore, subsamples comprising the opportunity composite can be taken at 0 to 2 inches, 2 to 6 inches, and 6 to 12 inches bgs.

SAMPLING AND COMPOSITING

All surface samples will be collected at the previously determined locations in accordance with the procedures outlined in the Community Soils Operable Unit (CS OU) Standard Operating Procedure (SOP)-S-01 (*Surface Soil Sampling*), or the site-specific SAP. Garden samples will be collected using sampling procedures that will best fit the soil type and obtain an adequate volume of soil for analytical and archiving purposes. Subsamples will be combined to form a use area composite sample in accordance with procedures outlined in the CS OU-SOP-S-06 (*Test Pit Sampling*), or the site-specific SAP. Portions of each discrete subsample and composite sample shall be archived for possible future use.

SAMPLE ANALYSIS AND DATA VALIDATION

After collection and compositing, samples will be prepared and analyzed for constituents of concern using the methods described in the site-specific QAPP. Analytical results will be

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validated according to the most current EPA direction and/or as amended by the site-specific QAPP. The validated analytical results will be used to make decisions on remedial actions.

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COMMUNITY SOILS OPERABLE UNIT

STANDARD OPERATING PROCEDURE
CSOU-SOP-S-06
TEST PIT SAMPLING

A backhoe or excavator can be used effectively to sample soils from 0 to 25 feet or more below ground surface (bgs). In particularly rocky or hard soils a back hoe may be the most effective means of sampling shallow depths.

SHALLOW TEST PIT SAMPLING:

Shallow test pits are defined as those excavated pits that are less than 4 feet deep.

1. Locate the site as directed in the appropriate Quality Assurance Project Plan (QAPP).

2. Prior to site entry have a utility locate performed. Walk through the site and determine any site specific hazards associated with the sampling area. Discuss these with sampling crew and note in the field logbook. Double check the utility locates information by identifying where natural gas pipes enter any structures on the property or if yard lights or street lights are present with no overhead lines. Determine if an underground sprinkling system is present. If sample locations have not been assigned in the QAPP, note the marked and/or probable locations of underground utilities and try to avoid those areas when choosing sample locations. Note the locations of overhead lines and overhead hazards and avoid those areas if possible.

3. If sample locations are identified in the QAPP use the appropriate survey method to locate. Stake test pit locations.

4. Most excavation companies have their own standard operating procedures (SOPs) for excavations, these machine specific SOPs and safety requirements should be consulted prior to site entry and the sampling protocols adjusted accordingly. Prior to beginning excavation, on-site safety protocol for excavation and sampling will be reviewed by the excavator operator and sampling crew.

5. The decontaminated backhoe will excavate the test pit to the desired depth. The excavated material should be layered adjacent to the test pit in the sequence in which it is removed. In particular topsoil should be separated from the underlying layers. Removed soil should be placed a minimum of 3 feet from the final edge of the test pit.

6. If the total depth of the test pit is less than four feet, the test pit should have one vertical smooth wall for sample collection and one sloping or stepped wall for entrance into the test pit. A competent person will examine the test pit and determine if the test pit is safe prior to entering.

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7. Sample collection will take place in the test pit at intervals specified in the site-specific QAPP. Measure and mark the intervals to be sampled on the straight wall of the test pit.

8. The deepest marked sample interval should be scraped with a decontaminated stainless steel trowel or scoop, a Teflon scoop, or a disposable plastic scoop to expose a clean surface.

9. Scrape the cleaned interval with the sampling tool and place the soil in a stainless steel bowl or a clean decontaminated disposable foil pan. Care must be taken to avoid material from above falling into the sample tool or sample bowl.

10. Remove all coarse fragments greater than 0.5 inches from the bowl. Mix the remaining soil in the bowl with the sampling tool.

11. Transfer the soil sample directly into the appropriate sample container according to Community Soils Operable Unit (CS OU) SOP-SA-01 (*Soil Sample Packaging and Shipping*).

12. Record appropriate information about the sample collection on field data sheets and/or in the field logbook.

13. Decontaminate sampling tools according to procedures outlined in CS OU–SOP-DE-02 (*Equipment Decontamination*).

14. If sampling more than one interval in the test pit repeat the above procedure for each interval to be sampled. Always begin sampling with the deepest interval and collect samples working up to the interval closest to the surface.

15. Once sampling is complete the test pit should be filled in by the equipment operator, placing the soils in the test pit in the geologic sequence in which it was removed. Top soil should always be placed last and smoothed out to match the surrounding terrain as closely as possible.

16. The backhoe (or excavator) and other sampling equipment should undergo decontamination as outlined in CS OU-SOP-DE-02 (*Equipment Decontamination*).

DEEPER TEST PIT SAMPLING:

Test pits over 4 feet require a trench box for sampling or the sample material can be collected from removed soil piles placed by the equipment. If necessary samples can be collected from the equipment bucket once it has been placed on the ground and the equipment powered down.

1. Locate the site as directed in the appropriate QAPP .

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2. Prior to site entry have a utility locate performed. Walk through the site and determine any site specific hazards associated with the sampling area. Discuss these with sampling crew and note in the field logbook. Double check the utility locates information by identifying where natural gas pipes enter any structures on the property or if yard lights or street lights are present with no overhead lines. Determine if an underground sprinkling system is present. If sample locations have not been assigned in the SAP, note the marked and/or probable locations of underground utilities and try to avoid those areas when choosing sample locations. Note the locations of overhead lines and overhead hazards and avoid those areas if possible. If sample locations are identified in the SAP use the appropriate survey method to locate.

3. If sample locations are identified in the QAPP use the appropriate survey method to locate. Stake the locations for the test pits.

4. Most excavation companies have their own SOPs for excavations, these machine specific SOP's and safety requirements should be consulted prior to site entry and the sampling protocols adjusted accordingly. Prior to beginning excavation, on-site safety protocol for excavation and sampling will be reviewed by the excavator operator and sampling crew.

5. The decontaminated backhoe will excavate the test pit to the desired depth. The excavated material should be layered adjacent to the test pit in the sequence in which it is removed. In particular topsoil should be separated from the underlying layers. Removed soil should be placed a minimum of 3 feet from the final edge of the test pit.

6. Sample collection will take place from the test pit at intervals specified in the site-specific Sampling and Analysis Plan.

7. If using a trench box the sampling protocol will be similar to that described in the Shallow Test Pit Sampling.

8. To sample using the backhoe or excavator as a sampling tool, a member of the sampling team needs to observe the excavation process and indicate to the equipment operator which buckets of soil should be placed aside for subsequent sampling. The decision on what soils to sample will be based on the SAP and may call for sampling protocol based on depths, color or lithologic changes.

9. Once test pit excavation is complete and the equipment has powered down the samples can be collected from the placed piles of soil. A decontaminated stainless steel trowel or scoop, a Teflon scoop, or a disposable plastic scoop should be used to clean the area of the pile to be sampled if it appears to have soil other than the designated interval on the surface. Care must be taken to get soils from the appropriate interval. If a large amount of sample is needed, such as for a proctor analysis, a clean shovel can be used to place soil directly into a sample container.

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10. For smaller amounts place the soil in a stainless steel bowl or a clean decontaminated disposable foil pan. Soil from several different places in the pile can be collected for a more representative sample of the interval.

11. Remove all coarse fragments greater than 0.5 inches from the bowl. Mix the remaining material in the bowl with the sampling tool.

12. Transfer the soil sample directly into the appropriate sample container according to CS OU-SOP-SA-01 (*Soil Sample Packaging and Shipping)*.

13. Record appropriate information about the sample collection on field data sheets and/or in the field logbook.

14. Decontaminate sampling tools according to procedures outlined in CS OU–SOP-DE-02 (*Equipment Decontamination)*.

15. If sampling more than one soil pile from the test pit repeat the above procedure for each pile to be sampled.

16. Once sampling is complete the backhoe should fill in the test pit with the excavated material in the geologic sequence in which it was removed. The backfilled test pit should attempt to achieve a surface flush with surrounding terrain.

17. The backhoe and other sampling equipment should undergo decontamination as outlined in CS OU–SOP-DE-02 (*Equipment Decontamination)*.

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COMMUNITY SOILS OPERABLE UNIT

STANDARD OPERATING PROCEDURE
CSOU-SOP-D-01
ATTIC DUST SAMPLING WITH A MICRO-VACUUM

This SOP will discuss the procedures for attic dust sampling using the Gilian HFS 113A Micro-Vacuum (sampling pump). Micro-vacuum sampling is designed to collect dust using a collection nozzle attached to a filter holder (sampling cassette) that is connected to an air sampling pump. This SOP, in general, follows procedures outlined in ASTM D 7144-05A *Standard Practice for Collection of Surface Dust by Micro-vacuum Sampling for Subsequent Metals Determination.*

Prior to the sampling event:

1. Order the number of Micro-Vacuum sampling cassettes that will be required for the sample event. SKC, Omega Specialty Division, Micro-Vacuum Cassettes (SKC model number 225-9543) have been used successfully with the Gilian Micro-Vacuum. The sampling cassettes should be individually wrapped and sealed, and contain a 37 mm diameter MCE filter inside of a plastic case (cassette). The cassette should have 0.45 µm nozzle on one end and a means to connect tubing to the sampling pump on the other. Enough additional cassettes should be ordered so that duplicates and equipment blanks can be collected at the appropriate interval as described in the project specific SAP and/or QAPP and that there are replacements in case of a cassette malfunction or accidental contamination of a cassette.
2. The sample cassettes will be submitted to the analytical laboratory so that a pre-sample weight for each cassette can be determined. This pre-sample weight will be used to in calculating the mass of the collected dust for each sample. The weight of each sample cassette will be recorded by the laboratory and a unique laboratory identifier will be written on the original sample cassette storage bag so that the laboratory can readily access the information during analysis. The laboratory will place each sample cassette back in its original re-sealable plastic bag. Samplers will store the weighed sample cassettes in a plastic container to ensure that they are protected from moisture (moisture could alter the recorded pre-sample weight).
3. Charge the batteries on the sampling pumps. At least two pumps should be taken to each sample location in case there is a pump malfunction.
4. A flexible piece of silicone tubing should be attached to each sample pump. The silicon tubing to be used with the Gilian sampling pump has an inner diameter of 0.188 inches.
5. Contact residents and set up a time to sample. For attic sampling, the residents need to be present for at least the start of the sampling.
6. Assemble equipment as outlined on Attachment A.

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Calibration of the Gilian Sampling Pump:

Equipment required for calibration includes: the Sampling Pumps, the Gilbrator Calibration Meter© (2 pieces), a sampling cassette that has been designated for calibration, small screw driver and the soap bubble solution provided by the manufacturer. The calibration cassette does not need to be weighed and can be used for multiple calibrations. It should be marked as the calibration cassette and not be used for sampling. Replace the cassette in its re-sealable plastic bag after each calibration.

1. Prior to calibration, the sampling pumps should be fully charged. Calibration will be done **daily** prior to any sampling activities. Each pump that will be used for sampling during the day requires calibration. A post calibration check will be completed for each pump at the end of each day.
2. Insert the calibration cassette in the end of the flexible hose attached to the sampling pump. To attach, remove the blue cap from the "wagon wheel" end of the calibration cassette and push the tubing onto the connector.
3. Assemble and lock in place the 2 pieces of the Gilbrator Calibration Meter©. Add an appropriate amount of soap solution to the Gilbrator following the manufacturer's directions. If possible leave the calibration meter set up for the end of day calibration check.
4. Connect the calibration cassette to the Gilbrator Calibration Meter© using a piece of flexible tubing. To connect the calibration cassette remove the red tip from the nozzle of the cassette and slide the nozzle inside the flexible tubing.
5. Using a pin, pen or small screwdriver turn on the sampling pump to begin calibration. The on-off switch is located on the front of the pump, left side near the bottom and is recessed.
6. Push the black button on the Gilbrator Calibration Meter© 10 to 15 times to lubricate the meter. Bubbles should rise all the way to the top of the tube, if the bubbles burst before reaching the top, more soap solution should be added. Continue pressing the button until bubbles flow smoothly out of the solution and up the tube.
7. Each sampling pump should be set to pump at 2.5 ± 0.1 Liters a minute (Lmin). To measure the pump flow rate, push the reset button on the Gilbrator Calibration Meter©. This will delete all previous flow measurements. Once the red light on the front of the meter goes out, push and hold the black button on the calibration meter until the red light goes out. If the reading on the calibration meter is not 2.5 ± 0.1 L/min the flow rate for the sampling pump will need to be adjusted. Make adjustments to the flow rate using a small screwdriver and turning the flow adjustment screw on the pump slightly. The screw is located to the right of the on-off switch and is recessed. Turning the screw to the left will lower the flow rate, turning the screw to the right will raise the flow rate. Once the initial adjustment has been made, push and hold the black button on the calibration

ED_014362_00000173-00085

meter to determine the new flow rate. Continue adjusting the flow rate until it is very close to 2.5 L/min.

8. Once the flow rate on the pump has been set, begin taking readings with the Gilbrator Calibration Meter©. Press the reset button on the calibration meter to clear all previous flow readings. The red light will come on to show that it is deleting the information. Once the red light goes out, hold down the black button to release **ONE** bubble, the red light will again come on. When the red light comes on release the black button, this should release another bubble. Once the red light goes out again, press and hold down the black button until the red light goes on again. This provides 3 flow readings that the Gilbrator Calibration Meter© will average. If a fourth bubble is generated when the black button is released for the final time, delete the fourth reading by pushing the delete button on the calibration meter. Only 3 readings should be used for the average flow rate.
9. Push the button marked average on the Gilbrator Calibration Meter©. Record this reading as the "start" flow rate for each pump in the field logbook. This flow rate should be 2.5 ± 0.1 L/m. Each pump should be identified by their seriel number or other unique identifier. The "start" average flow rate for the pump used to collect a sample will also be recorded on the Attic Dust Sample Data Sheet. An example of the Attic Dust Data Sheet is included as Attachment B.

Post-Calibration or End of Day Calibration Check

1. Insert the connector on the calibration cassette into the flexible tubing attached to the sampling pump to be checked. To attach, remove the blue cap from the "wagon wheel" end of the calibration cassette and push the tubing onto the connector.
2. Assemble and lock in place the 2 pieces of the Gilbrator Calibration Meter©. Add an appropriate amount of soap solution following the manufacturer's directions.
3. Connect the calibration cassette to the Gilbrator Calibration Meter© with a piece of flexible tubing. To connect the calibration cassette, remove the red tip from the nozzle of the cassette and slide the nozzle into the flexible tubing.
4. Using a pin, pen or small screwdriver turn on the sampling pump.
5. Push the black button on the Gilbrator Calibration Meter© 10 to 15 times to lubricate the meter. This will release bubbles. Bubbles should go to the top of the tube, if the bubbles burst before reaching the top, add more soap solution. Continue pressing the button until bubbles flow smoothly out of the solution and up the tube.
6. Press the reset button on the calibration meter to clear all of the previous flow readings. A red light will come on to show that it is deleting the information. Once the red light turns off, hold down the black button to release **ONE** bubble, the red light will come on. Release the black button when the red light goes off, this should release another bubble. Once the red light goes out, press and hold down the black button until the red light goes on again. This should provide 3 flow readings that the Gilbrator Calibration Meter© will average. If a fourth bubble is generated when the black button is released for the final

ED_014362_00000173-00086

time, delete the fourth reading by pushing the delete button on the calibration meter. Only 3 readings should be used to calculate the average for the end of day flow rate check.

7. Record the average flow rate for each pump as the "end" flow rate in the field logbook and on the Attic Dust Sample Data Sheet. Make sure that the "end "reading for each pump is paired with the appropriate "start" reading.
8. Calculate the average of the starting and stopping flow rate to confirm that it is within 2.5 ± 0.1 L/min. If it falls outside of that range make a note in the logbook as a deviation to the sampling protocol.

Attic Dust Sampling

Attic dust sampling must be done in with a crew of no less than 2 people. One team member will enter the attic and sample (sampler), the non-entrant will monitor the sampler for any health or safety issues, and provide support by running the stopwatch, handing up appropriate equipment and making sure that the resident stays an appropriate distance away from sampling activities.

Unless otherwise specified in the SAP, attic dust sampling will be conducted only in homes where the attic can be routinely accessed (by stairway, or ladder). If the attic access is nailed or painted shut, requires moving a lot of items (i.e. in a closet under shelving), or is located outside the residence and greater than 6 feet above ground surface the sampling will need to be reevaluated. If it is determined that the sampling is necessary, additional safety measures will need to be put in place.

If vermiculite or asbestos is present in the attic, sampling will not be conducted.

In most cases, attics will **not** be entered. Instead, sampling will be conducted in the area surrounding the attic access. This is not ideal because the area is prone to disturbance, resulting in less dust then other areas of the attic. However, uncertainty about the structural integrity of the attic (e.g., rafters, boards etc.) presents a high degree of risk and could cause potential damage to the home.

1. The day before the sampling event, contact the resident to confirm the appointment. Plug in the sampling pumps to charge.
2. After arriving at the property, take a photo of the front of the residence, note north direction.
3. Knock on the door and introduce yourselves to the resident(s). Upon entering the house, don disposable booties as to not track any dirt or liquids into the house. Explain what the sampling will entail and ask about the attic access. Determine if it is safe to access the attic and what equipment will be required.
4. One team member should complete any required residential questionnaires as detailed in the SAP. Take a photograph of the attic access prior to attempting entry. The location of the attic access and photograph information should be recorded in the logbook.

ED_014362_00000173-00087

5. If a ladder is needed, 2 team members should carefully maneuver the ladder through the house to the attic access. A third person may be required to "spot" tight corners and low hanging doorways.
6. Lay a large sheet of plastic under the attic access. The plastic sheet should be big enough that there is a 2-3 foot buffer zone around the ladder or access stairs. Carefully set up the ladder on the plastic.
7. The sampler will don a Tyvek© suit, nitrile gloves and headlamp. The non-entrant will label the sampling canister with the appropriate sample identifier. The sample name, pump serial number or identifier, and sampler will be entered into the logbook and on the Attic Dust Sample Data Sheet. At this time, the sample pump should be turned on to warm up.
8. The sampler will pull down the attic stairs or climb the ladder (steadied by the non-entrant) and remove or open the access door. Be aware that objects may be resting on or adjacent to the access panel and may fall. If needed the non-entrant should place the access door in a location where it will not get soiled and out of the traffic area.
9. The sampler, without leaving the access point, will describe the attic features to the non-entrant who records the information in the logbook or on field data sheets. The sampler will remain on the stairs or ladder and will never fully enter the attic. Information of interest would include type of insulation, amount of insulation, the presence of ducts, and penetrations or openings from the roof to the attic.
10. Each sample of attic dust will consist of material collected from at least two different locations in the attic. Sampling locations adjacent to the access point should be identified by the sampler. Each sampling location needs to have an area of at least 100 cm^2. Wooden rafters, joists, planks or cardboard on the floor of the attic are ideal for sampling. Choose areas with heavy dust accumulation. If necessary, dust can be carefully collected from faced fiberglass batts. Once sample locations have been identified, pre-cut plexi-glass templates can be used to demarcate the area to be sampled. The templates can be placed directly on the sample surface and secured using tape. If templates will not work (sample area is to narrow or curved), a ruler or tape measure and masking tape can be used to mark an area of 100 cm^2. The non-entrant can hand up any equipment required.
11. The sampler should identify the location of the sample area (i.e west of access area, or north side of house). The non-entrant will write the sample number and location information on a 3" X 5" index card for each sample area. The cards and camera will be handed to the sampler and a photograph of each sample area should be taken. General attic conditions as well as any special features such as openings, insulation distribution, stored contents or the dust accumulation can also be photograph. Information on photographs should be recorded in the logbook by the non-entrant. Information for any SAP required questionnaires should be relayed by the sampler and recorded on the forms by the non-entrant.

ED_014362_00000173-00088

12. The non-entrant will don new nitrile gloves, turn the pump off and attach the labeled sample canister to the pump. This can be done by removing the blue cap on the "wagon-wheel" end of the sampling canister and inserting it into the flexible tubing attached to the pump. The red cap on the nozzle end of the sample canister should also be removed. Place both end caps back in the re-sealable plastic bag back for temporary storage. Hand the pump up carefully to the sampler.
13. The sampler should turn the pump on when he is in position at the first sample location. As specified in ASTM D-7144-05a, collect the sample by holding the nozzle immediately adjacent to the surface to be sampled. The nozzle, which is cut at a 45° angle, should lightly touch the sample surface, but not be pressed hard against the area being sampled. The sampler should indicate to the non-entrant when sampling begins. The non-entrant will then record the start time to the minute and second in the logbook and on the Attic Dust Sample Data Sheet. Sampling at each location will be conducted for **one minute.** The non-entrant will be responsible for timing, and after one minute has passed will say "STOP". The sampler will then lift the nozzle from the surface and turn the pump off. During the collection period the sampler will sweep the collection nozzle from one side of the sampling area to the other. The rate of movement should be no more than 1 sec/10 cm. The sample area should be "swept" in the same direction until the entire sampling area has been "vacuumed" with the collection nozzle. After 30 seconds, repeat in a different direction 90° from the initial sampling direction. Make sure to cover the entire sampling area. Record the stop time in the logbook and on the Attic Dust Sample Data Sheet.
14. Move to the second sample area and repeat Step 13. Sample collection time should always be 1 minute for each 100 cm^2. Once sampling has been completed in the second area, the sample cassette should be examined to see if there is visible dust on the filter. If there is only a small amount of dust in the sample cassette, more locations should be sampled, continue sampling areas as described in Step 13. Once sampling is completed, record the total surface area per sample, i.e. if two 100 cm^2 areas were sampled the total area would be 200 cm^2, in the logbook and on the Attic Dust Sample Data Sheet.
15. If the sample nozzle becomes clogged, the sampler should tell the non-entrant to stop timing, hold the nozzle in an upright configuration and tap against a hard surface. Once the pump appears to be drawing air into the filter, the sampler should say "start" and the timing and sampling should resume.
16. If the filter in the sample cassette becomes overloaded as indicated by a >10% drop in the measured flow rate, additional cassettes will be needed to complete collection of the sample. The sampler will notify the non-entrant when timing should be stopped. The full sample cassette will be removed from the pump and handed to the non-entrant. The stored end caps will be placed on the cassette and the cassette will be placed in the original re-sealable plastic bag. A new sample cassette, labeled with the same information as the first sample cassette, except for the addition of 1of 2, 2 of 2 etc., will

ED_014362_00000173-00089

be attached to the pump. The stop time for the first cassette and start time for the second cassette will be recorded in the logbook. Restart the stopwatch and continue timing until the sample location has been "vacuumed" for 1 minute.

17. Once sampling is complete, remove sampling cassette from the pump, replace both end caps and place in the original plastic re-sealable bag. Check that the sample number(s) are correct. Place all filters for 1 attic sample in a quart re-sealable plastic bag.
18. The non-entrant should review the collected information and make sure that there are no omissions. If it is determined that sampling is complete, the sampler can hand down equipment to the non-entrant and then exit the attic.
19. The sampler should stand on the plastic and carefully remove the Tyvek© coveralls and gloves, and immediately place them in a garbage bag. New gloves and booties should be donned and the sampler should climb the latter, steadied by the non-entrant, and restore the attic access cover to its original position.
20. The ladder should be lowered and carefully carried out by 2 people, if needed a 3rd person should be used as a spotter.
21. Carefully roll up the plastic that was under the ladder/access stairs and place in garbage bag. Use a handheld vacuum to touch up the area below the attic access.
22. Verify that field logbook entries and field data sheets are complete. Flaking paint in the sampled home or adjacent homes should be documented and photographed, if possible.
23. Thank resident.
24. Once outside, using wet-wipes or de-ionized water wetted paper towels, wipe off the pump, attached flexible tubing, ruler, templates, headlamp, handheld vacuum and the rails and feet of the ladder. Any other tools or equipment that may have come in contact with dust from the attic should also be wiped down.
25. The Tyvek© coveralls and plastic sheeting will be disposed of in accordance with SOP-DE-03 Investigation Derived Waste Handling.
26. Label the outer plastic re-sealable bag with the sample number and place in cooler for storage until the samples can be delivered to the laboratory for analysis. Ship in accordance with SOP-SA-01 Soil and Water Sample Packaging and Shipping.

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ATTACHMENT A

ATTIC DUST SAMPLING
SUPPLY AND EQUIPMENT CHECKLIST

Attic Dust Sampling
Supply and Equipment Checklist

- ☐ **Sampling Equipment**
  - ☐ Gilian hi-flow sampler with associated silicon tubing
  - ☐ Gilbrator calibration meter
  - ☐ Pre-weighed, assembled filter cassettes with nozzles
  - ☐ Re-sealable plastic bags (quart and gallon)
  - ☐ Plastic sampling templates
  - ☐ Roll of 6-mil poly drop cloth
  - ☐ Garbage bags (32-gallon)
  - ☐ Digital timer

- ☐ **Tools**
  - ☐ Mini screwdriver set
  - ☐ Screwdriver set
  - ☐ Tape measure
  - ☐ Small rulers with centimeter scale
  - ☐ Blue or tan ½-inch masking tape\
  - ☐ Duct tape
  - ☐ Small pry bar
  - ☐ Flashlights
  - ☐ Ladders with cleanable steps (6-, 8-, and 10-feet)
  - ☐ Compass
  - ☐ Small, hand-held vacuum
  - ☐ Wet-wipes for hands and equipment
  - ☐ Paper towels

- ☐ **PPE**
  - ☐ Steel-toed boots
  - ☐ Safety glasses
  - ☐ Disposable tyvek® booties
  - ☐ Disposable tyvek® coveralls with hoods (no booties)
  - ☐ Knee pads
  - ☐ Headlamp with extra batteries
  - ☐ Powderless, latex-free or nitrile sampling gloves
  - ☐ First aid kit
  - ☐ Eye wash bottle
  - ☐ Drinking water (gallon bottles)

- ☐ **Office Supplies**
  - ☐ Digital camera with date/time stamp
  - ☐ Plastic tote
  - ☐ Notepads, field log books, and supply forms
  - ☐ Pens and dry markers
  - ☐ 3x5 Blank file cards or small whiteboard
  - ☐ Scissors

ATTACHMENT B

ATTIC DUST SAMPLING
SAMPLE DATA SHEET

ATTIC DUST SAMPLE DATA SHEET
Revised 09/20/12

Address									
Sampler			Date			Time			
Sample ID				Duplicate Collected? Y / N					
Pump Serial Number									
Pump Calibration, (L/min)	Date		Start		Stop		Average		
Sample Duration, min									
Comments:									
Location(s) Sampled:									
Location: Surface: Area Sampled (cm²):	Location: Surface: Area Sampled (cm²):	Location: Surface: Area Sampled (cm²):	Location: Surface: Area Sampled (cm²):						
Lab Sample #:									

COMMUNITY SOILS OPERABLE UNIT

STANDARD OPERATING PROCEDURE
CSOU-SOP-SA-01
SOIL SAMPLE PACKAGING AND SHIPPING

1. In most cases, all sample containers collected from a specific sample location are placed in a large ziplock bag and shipped together. Samples will then be placed in a cooler which has been lined with a plastic bag. The samples will be surrounded with non-contaminating packaging materials to reduce movement and absorb any leakage. Ice will be double bagged and placed in the cooler. The plastic bag will then be sealed to contain the samples, packing material and ice.

2. The Field Team Leader or their designated representative will double check the chain-of-custody forms to assure those samples recorded on the chain-of-custody form are in the cooler. The Field Team Leader or the designated representative will then sign the chain-of-custody form to relinquish custody.

3. One copy of the signed chain-of-custody form will remain with the Field Team Leader. A photocopy may be made of the completed form if there are no carbon copies available. The paper work will then be placed in a sealed ziplock bag and taped to the inside of the cooler lid. If the shipping cooler contains more samples than can be analyzed in one analytical batch, the laboratory may request that the samples in the cooler be bagged for separate analytical batches. This may be necessary so that the appropriate Quality Control/Quality Assurance samples are included in each analytical batch. In this case separate chain-of-custody forms will be filled out for each batch and included in the appropriate bags. The chain-of-custody forms for each batch will be placed in a sealed ziplock bag and included at the top of the bag so that they are clearly visible to laboratory personnel when they open the bags.

4. The cooler will be labeled with the appropriate shipping labels (NOS, flammable liquids, flammable solids, this side up, fragile, etc.).

5. The cooler will then be closed and the appropriate shipping label (overnight shipping from Federal Express, UPS or the United States Postal Service or equivalent) will be affixed to the lid.

6. The Field Team Leader or the designated representative will sign COC seals and place the signed seals over the opening edge of the cooler.

7. Tape will then be placed over the custody seals and around the cooler.

8. The cooler(s) will then be transported to a secure storage, to the shipping agent, or directly to the laboratory.

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Note: Bagging of samples and lining of coolers will not be necessary if samplers transport samples directly to the laboratory.

ED_014362_00000173-00096

COMMUNITY SOILS OPERABLE UNIT

STANDARD OPERATING PROCEDURE
CSOU-SOP-SA-03
FIELD QUALITY CONTROL SAMPLES

Field quality control (QC) is a part of the Project Quality Assurance/Quality Control (QA/QC) program and is described in detail in the site-specific Quality Assurance Project Plan (QAPP). This standard operating procedure (SOP) describes the preparation and collection frequency of field duplicate samples.

At least one set of field QC samples will be prepared for each sampling event. QA/QC samples will be collected at a frequency of 1:20. If the number of field QC samples taken is not equal to an integer multiple of the interval, then the next higher multiple will be used. For example, if a frequency of 1:20 is indicated and 28 samples are taken, then two QC samples will be prepared.

All field QC samples shall be shipped with field samples to the contract laboratory as per Community Soils Operable Unit Standard Operating Procedure (*Soil Sample Packaging and Shipping*) (CSOU-SOP-SA-01).

One field duplicate will be taken 1:20 and as follows:

A field duplicate consists of two samples taken at the same location and time, but placed in different containers for separate analysis. Each duplicate shall be analyzed for identical chemical parameters.

1. Collect an adequate volume of sample to accommodate two sample containers.

2. Process the samples (as per SOPs) for each duplicate.

3. Label the two sample containers with appropriate sample numbers.

4. Record duplicate number, sample number, and sample location in the field log book.

ED_014362_00000173-00097

COMMUNITY SOILS OPERABLE UNIT

STANDARD OPERATING PROCEDURE
CSOU-SOP-SA-04
CHAIN OF CUSTODY FORMS FOR ENVIRONMENTAL SAMPLES

This standard operating procedure (SOP) establishes the requirements for documenting and maintaining environmental sample chain-of-custody from point of origin to receipt of sample at the analytical laboratory. This procedure shall apply to all types of air, soil, water, sediment, biological, and/or core samples collected in environmental investigations. It is applicable from the time of sample acquisition until custody of the sample is transferred to an analytical laboratory.

Chain-of-custody is an unbroken trail of accountability that ensures the physical security of samples, data and records. Custody refers to the physical responsibility for sample integrity, handling, and/or transportation. Custody responsibilities are effectively met if the samples are:

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

RESPONSIBILITIES

PROJECT MANAGER:

1. The Project Manager is responsible for overall management of environmental sampling activities, designating sampling responsibilities to qualified personnel, and reviewing any changes to the sampling plan.

FIELD TEAM LEADER:

1. The Project Manager may act as the Field Team Leader or may choose to appoint a Field Team Leader.

2. The Field Team Leader is responsible for general supervision of field sampling activities and ensuring proper storage/transportation of samples from the field to the analytical laboratory. Chain-of-Custody forms will be reviewed for accuracy and completeness to preserve sample integrity from collection to receipt by an analytical lab by the Field Team Leader. The review of chain-of-Custody forms may be delegated to qualified personnel. The Field Team Leader is responsible for sample custody until the sample has been properly relinquished as documented on the chain-of-custody form.

ED_014362_00000173-00098

FIELD SAMPLER

1. The Field Sampler is responsible for sample acquisition in compliance with technical procedures, initiating the Chain-of-Custody, and checking sample integrity and documentation prior to transfer.

2. Field samplers are also responsible for initial transfer of samples consisting of physical transfer of samples directly to the internal laboratory or transferred to a shipping carrier, (i.e., United Parcel Service or Federal Express) for delivery.

LABORATORY TECHNICIAN

1. The receiving Laboratory Technician is responsible for inspection of transferred samples to ensure proper labeling and satisfactory sample condition.

2. Unacceptable samples will be identified and segregated. The Laboratory Project Manager will be notified.

3. The Laboratory Technician will review the Chain-of-Custody for completeness and file as part of the project's permanent record.

EQUIPMENT AND MATERIALS

- Seals and Labels;
- Chain of Custody forms and chain of custody seals (provided by contracted laboratory); and
- Packing and shipping materials as necessary.

1. All samples shall be collected and handled in accordance with the appropriate Community Soils Operable Unit Standard Operating Procedure (SOP) or methods described in the project Quality Assurance Project Plan or work plan. Samples will be transported in insulated coolers with ice ('blue ice' is acceptable) as necessary to maintain temperature at 4° C+/- 2°C until receipt by the analytical laboratory.

2. The Field Team Leader or designated Field Sampler shall initiate the Chain-of-Custody form for the initial transfer of samples.

3. A Chain-of-Custody form will be completed and accompany every sample. The form includes the following information:

- Project code;
- Project name;
- Samplers signature;
- Sample identification;
- Date sampled;

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- Time sampled;
- Analysis requested;
- Remarks;
- Relinquishing signature, data, and time; and
- Receiving signature, date, and time.

4. The Field Sampler relinquishing custody and the responsible individual accepting custody shall sign, date, and note the time of transfer on the Chain-of-Custody form. (If the transporter is not an employee of sampling firm, the Field Sampler may identify the carrier and reference the bill of lading number in lieu of the transporter's signature.)

5. One copy of the Chain-of-Custody form shall be filed as a temporary record of sample transfer by the Field Sampler. The original form shall accompany the samples and shall be returned to the sampling firm as part of the contracted laboratory Quality Assurance/Quality Control (QA/QC) requirements. The original form will be filed as part of the project's permanent records.

6. The Project Manager (or designee) shall track the Chain-of-Custody to ensure timely receipt of samples by an analytical laboratory.

ED_014362_00000173-00100

COMMUNITY SOILS OPERABLE UNIT

STANDARD OPERATING PROCEDURE
CSOU-SOP-SA-05
PROJECT DOCUMENTATION

This Standard Operating Procedure (SOP) establishes the requirements for documenting and maintaining field logbooks and photographs. These procedures shall apply to all types of air, soil, water, sediment, biological, and/or core samples collected during Community Soils Operable Unit (CS OU) environmental investigations. These procedures apply from the time field work begins until site activities are completed.

RESPONSIBILITIES:

A designated field logbook will be used for each field project. If requested by the Project Manager a separate field logbook will be used for each field task within a larger project. Each logbook shall have a unique document control number. The logbooks will be bound and have consecutively numbered pages. The information recorded in these logbooks shall be written in ink. The author will initial and date entries at the end of each day. All corrections will consist of a single line-out deletion in ink, followed by the author's initial and the date. No bound field logbooks will be destroyed or thrown away even if they are illegible or contain inaccuracies that require a replacement document. These bound logbooks shall include the following entries:

1. A description of the field task.

2. Time and date fieldwork started.

3. Location and/or a description of the work areas, including sketches if needed, any maps or references needed to identify locations, and sketches of construction activities. If the location is an often visited field area changes in conditions from previous field events should be noted.

4. Names and company affiliations of field personnel.

5. Name, company affiliation or address, and phone number of any field contact or official visitors.

6. Meteorological conditions at the beginning of fieldwork and any ensuing changes in these conditions.

7. Details of the fieldwork performed and reference to field data sheets if used.

8. Deviation from the task-specific Sampling and Analysis Plan (SAP), Work Plan (WP) or SOP.

9. All field measurements made.

ED_014362_00000173-00101

10. Any field laboratory analytical results.

11. Personnel and equipment decontamination procedures, if appropriate.

For any field sampling work the following entries should be made:

1. Sample location and number.

2. Sample type and amount collected.

3. Date and time of sample collection.

4. Type of sample preservation.

5. Split samples taken by other parties. Note the type of sample, sample location, time/date name of person, person's company, and any other pertinent information.

6. Sampling method, particularly any deviations from the SOP.

7. Documentation or reference of preparation procedures for reagents or supplies that will become an integral part of the sample if available. This information may not be available for water or soil sampling bottles that come preserved from the laboratory or for preservative provided by the laboratory. Bottle blanks will need to be used to evaluate the provided reagents.

8. The laboratory where the samples will be sent.

Photographs will be taken of field activities using a digital camera. Photographs should include a scale in the picture when practical. Telephoto or wide-angle shots will not be used, since they cannot be used in enforcement meetings. The following items shall be recorded in the bound field logbook for each photograph taken:

1. The photographer's name, the date, the time of the photograph, and the general direction faced.

2. A brief description of the subject and the fieldwork portrayed in the picture.

3. Sequential number of the photograph.

An electronic copy and/or a hard copy of the photographs shall be placed in task files in the field office after each day of field activities. Any supporting documentation from the bound field logbooks or field data sheets shall be photo copied and placed in the task files to accompany the photographs once the field activates are completed.

ED_014362_00000173-00102

COMMUNITY SOILS OPERABLE UNIT

STANDARD OPERATING PROCEDURE
CSOU-SOP-DE-01
PERSONNEL DECONTAMINATION PROCEDURES

The purpose of this Standard Operating Procedure (SOP) is to provide instructions for decontamination of personnel leaving a contaminated area. This practice has been prepared for Community Soils Operable Unit (CS OU) remedial action work. All members of the workforce who conduct the work shall be trained and competent in the risk-assessed work.

DECONTAMINATION INSTRUCTIONS

PROTECTION:

1. Follow personal protective measures outlined in the Site Specific Health and Safety Plan (SSHASP).

2. In the event that personnel decontamination becomes necessary, the outer more heavily contaminated items should be soap (nonphosphate)/tap water washed. Rinse the items in tap water.

3. Next, the inner, less contaminated items should be soap (nonphosphate)/tap water washed. Rinse the items in tap water.

4. Store the items separately so they are used in contaminated areas only.

5. For contaminants other than those found typically at uncontrolled hazardous waste sites, see the Health and Safety Manager.

EMERGENCY DECONTAMINATION:

1. If the decontamination procedure is essential to the life-saving process, decontamination must be performed immediately.

2. If heat related illness develops, protective clothing should be removed as soon as possible. **WASH, RINSE AND/OR CUT OFF PROTECTIVE CLOTHING/EQUIPMENT.**

3. If medical treatment is required to save a life, decontamination should be delayed until the victim is stabilized. Wrap the victim to reduce contamination of others.

4. Alert medical personnel to the emergency and instruct them about potential contamination. Instruct medical personnel about specific decontamination procedures.

5. Dispose of contaminated clothing and equipment properly.

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HSSE CONSIDERATIONS

CHEMICAL HAZARDS:

HAZARDS	WHERE	HOW, WHEN, RESULT	CONTROLS
1. Contact with contaminants at hazardous waste sites.	Sites	Adverse health effects could result from ingesting inhaling and/or skin/eyes contact with contaminants.	Personnel will practice proper personal hygiene – wash hands prior to easting and when leaving the site. Work will be suspended during high wind conditions that may produce large amounts of visible dust. Employees will follow decontamination procedures as described above. Employees will wear nitrile/latex gloves when handling contaminated items.
2. Carbon Monoxide (CO).	Vehicle	Employees could be exposed to CO when traveling to and from job sites. Co from idling vehicles may cause irritated eyes, headache, nausea, weakness and dizziness.	Employees will minimize the time sitting in idling vehicles and will open window to increase ventilation.
3. Gasoline/diesel.	Vehicle	Exposure to gasoline/diesel could occur via inhalation and/or skin/eyes contact causing headache, dizziness, blurred vision and irritated skin.	Employees will fuel vehicles in a well-ventilated area and they will minimize splash hazards to prevent skin/eyes contact.

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ELECTRICAL HAZARDS:

HAZARDS	WHERE	HOW, WHEN, RESULT	CONTROLS
1. Lightning	Outdoor sites	Electrocution, injury to death, or equipment damage could be caused by lightning.	Employees will follow the 30/30 rule during lightning storms.

MOTION HAZARDS:

HAZARDS	WHERE	HOW, WHEN, RESULT	CONTROLS
1. Struck by and/or caught in between heavy equipment.	Construction sites.	Personnel could be injured if struck by and/or caught in between heavy equipment.	Employees will communicate with the contractor's contact person to let him/her know Pioneer personnel are on site. Personnel will avoid working near heavy equipment, when possible. High visibility clothing will be worn.

GRAVITY HAZARDS:

HAZARDS	WHERE	HOW, WHEN, RESULT	CONTROLS
1. Falls from slips and trips	Uneven terrain, slick surfaces and steep slopes at testing sites.	Workers could get injured if they fall causing bruises, scrapes, or broken bones.	Workers will wear work boots with good traction and ankle support. Employees will plan their path and walk cautiously.

THERMAL HAZARDS:

HAZARDS	WHERE	HOW, WHEN, RESULT	CONTROLS
1. Cold/heat stress.	Sites – weather.	Exposure to cold climates may result in cols burns, frostbites, and hypothermia. Exposure to high	Training on signs and symptoms of cold/heat stress. Personnel will wear appropriate clothing

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		temperatures may result in heat cramps, heat exhaustion, or heat stroke.	when working outdoors. Employees will remain hydrated and will have sufficient caloric intakes during the day.

RADIATION HAZARDS:

HAZARDS	WHERE	HOW, WHEN, RESULT	CONTROLS
1. Ultraviolet (UV) radiation.	Outdoors.	Employees could be exposed to UV radiation during summer months causing sun burns, skin damage and eye damage.	Employees will wear sunscreen, long-sleeve work shirts and long pants. Employees will also use safety glasses with tinted lenses.

BIOLOGICAL HAZARDS:

HAZARDS	WHERE	HOW, WHEN, RESULT	CONTROLS
1. Untrained worker.	Sites.	Adverse health effects or injury could result from lack of training.	Employees will take task training prior to performing decontamination procedures.
2. Hazardous plants and animals.	Testing sites.	Exposure to hazardous plants or animals may cause rashes, blisters, redness and swelling.	Training on the signs and symptoms of exposure to hazardous plants and animals. Avoid contact with hazardous plants and animals. First-aid kits will be available on site. Employees with allergies will notify their supervisor.

ADDITIONAL HSSE CONSIDERATIONS:

1. The required personnel protective equipment (PPE) during personnel decontamination is

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level D PPE (hard hat, safety glasses, high-visibility work shirt or vest, long pants, and steel toes boots).

2. The applicable Material Safety Data Sheets (MSDSs) are for heavy metals, gasoline/diesel, and carbon monoxide.

3. Additional training for personnel decontamination is OSHA 4-hour HAZWOPER/8-hour refresher.

TOOLS REQUIRED:

1. The tools required for personnel decontamination procedures are work vehicle, soap, tap water, tarps, decontamination tubs, brushes, and sprayers.

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COMMUNITY SOILS OPERABLE UNIT

STANDARD OPERATING PROCEDURE
CSOU-SOP-DE-02
EQUIPMENT DECONTAMINATION

All equipment leaving the contaminated area of a site must be decontaminated. Decontamination methods include removal of contaminants through physical, chemical or a combination of both methods. Decontamination procedures are to be performed in the same level of protection used in the contaminated area of a site. In some cases, decontamination personnel may be sufficiently protected by wearing one level lower protection. The information for site specific equipment decontamination and personnel protection levels as detailed in the sampling and analysis or work plan should be followed.

The following decontamination procedures are for typical uncontrolled hazardous waste sites, for a specific or unusual contaminant such as dioxins, see the site-specific Health and Safety Plan. Decontamination procedures should be used in conjunction with methods to prevent contamination of sampling and monitoring equipment. One time use equipment should be used if practical, and disposed of in accordance with the site-specific Health and Safety Plan

INORGANIC CONTAMINANTS - HEAVY METALS:

1. Remove gross contamination with a tap water rinse. If available, use pressurized or gravity flow tap water, if not a 5 gallon bucket of tap water and a stiff brush may be used.

2. Wash equipment in a solution of soap (no phosphate) and tap water with a stiff brush.

3. Triple rinse the equipment with tap water.

4. Triple rinse the equipment with de-ionized or distilled water.

5. If specified in the site Sampling or Work Plan, rinse the equipment with a mixture of 10:1 nitric acid in distilled water (10 parts water to 1 part nitric acid). In many cases, the tap water and de-ionized water rinses will be sufficient.

6. If a nitric rinse is used, rinse the equipment again with distilled water.

7. Place equipment on plastic sheeting or foil to air dry.

8. Wrap equipment in foil or plastic wrap to transport or store.

ORGANIC CONTAMINANTS:

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1. Remove gross contamination physically with a disposable paper towel or if available with a tap water rinse using pressurized or gravity flow. If water is not available on site the equipment can be rinsed using a five gallon bucket of tap water and a stiff brush

2. Wash equipment in a solution of soap (no phosphate) and tap water with a stiff brush.

3. Triple rinse the equipment in tap water.

4. Triple rinse the equipment with de-ionized water.

5. Rinse the equipment with methanol (if appropriate, see site Sampling Plan or Work Plan to determine appropriate chemical rinses). If testing for dioxins, a hexane triple rinse will be included as part of the decontamination.

EQUIPMENT USED FOR DECONTAMINATION:

1. Triple rinse equipment (brushes, buckets, tubs) used in the decontamination process with water, preferably pressurized.

2. Agitate the equipment used in the decontamination process in the soap/tap water solution. (The tub which holds the solution will only have the water rinse.)

3. Triple rinse equipment with tap water.

4. Place equipment in appropriate areas, so they are used only for decontamination purposes (label if necessary).

DISPOSAL OF DECONTAMINATION SOLUTIONS:

1. Proper disposal of the soap/tap water solution, the tap water rinse, and the de-ionized water rinse is to a proper waste water container.

2. Proper disposal of the solvent rinse is to a proper organic solvent waste container.

3. When contaminants have been identified, either in the solutions or elsewhere on the site, solutions should be disposed of appropriately as discussed in the site specific Health and Safety plan. If they are hazardous (characteristic, listed, etc.) dispose of them as such.

4. WHEN USING OTHER THAN THE ABOVE MENTIONED SOLUTIONS, BE SURE TO CHECK WITH THE HEALTH AND SAFETY OFFICER AND THE PROJECT MANAGER. SOME SOLVENTS MUST BE EVAPORATED.

EFFECTIVENESS OF DECONTAMINATION:

ED_014362_00000173-00109

1. Effectiveness of the decontamination procedures will be measured using field equipment rinsate blanks (see the site-specific Quality Assurance Project Plan).

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ATTACHMENT A-2

SOIL LABORATORY
STANDARD OPERATING PROCEDURES

Attachment A-2
Soil Laboratory SOP
Index

Laboratory	SOP Number	SOP Title	# Pages
Pace	S-MT-ME-041-Rev 00	Soil Sieve	9
Pace	S-MN-I-367-Rev 15	The Determination of Percent Moisture in Soil and Solid Samples	13
Pace	S-MN-I-460-Rev 17	Preparation of Solid Samples and Wipes for Analysis by ICP and ICP-MS	9
Pace	S-MN-I-313-Rev 25	Inductively Coupled Plasma Atomic Emission Spectroscopy	19
Pace	S-MN-I-492-Rev 23	Metals Analysis by ICP/MS	25

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STANDARD OPERATING PROCEDURE

SOIL SIEVE

Reference Methods: NA

Local SOP Number:	S-MT-ME-041-Rev.00
Effective Date:	Date of Final Signature
Supersedes:	New Issue

APPROVALS

Billings Laboratory Operations Manager	26 Mar 2015 Date
Laboratory General Manager	06 Apr 2015 Date
Laboratory Quality Manager	30 Mar 2015 Date

PERIODIC REVIEW

SIGNATURES BELOW INDICATE NO CHANGES HAVE BEEN MADE SINCE PREVIOUS APPROVAL.

Signature	Title	Date

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TABLE OF CONTENTS

SECTION PAGE

Error! Bookmark not defined.. **Purpose/Identification of Method** 3

2. Summary of Method 3

3. Scope and Application 3

4. Applicable Matrices 3

5. Limits of Detection and Quantitation 3

6. Interferences 3

7. Sample Collection, Preservation, Shipment and Storage 3

8. Definitions 3

9. Equipment and Supplies (Including Computer Hardware and Software) 3

10. Reagents and Standards 4

11. Calibration and Standardization 4

13. Quality Control 5

14. Data Analysis and Calculations 5

15. Data Assessment and Acceptance Criteria for Quality Control Measures 5

16. Corrective Actions for Out-of-Control Data 6

17. Contingencies for Handling Out-of-Control or Unacceptable Data 6

18. Method Performance 6

19. Method Modifications 6

20. Instrument/Equipment Maintenance 6

21. Troubleshooting 6

22. Safety 6

23. Waste Management 6

24. Pollution Prevention 6

25. References 6

26. Tables, Diagrams, Flowcharts, and Validation Data 7

27. Revisions 7

1. Purpose/Identification of Method

1.1. This standard operating procedure describes the process for drying and sieving soil samples to obtain a portion of soil for analysis

2. Summary of Method

2.1. The sample is split and a portion air dried. The air dried portion is then sieved through a selected sieve size. The portion that passes the sieve is then ready for analysis. Depending on client requirements, the original non-sieved portion and the portions larger than the selected sieve size may need to be archived.

3. Scope and Application

3.1. **Personnel:** This standard operating procedure is applicable to all analysts performing sieve analysis

3.2. **Parameters:** This standard operating procedure is applicable to soils or sediments.

4. Applicable Matrices

4.1. This SOP is applicable to soils and sediments.

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. The appropriate container for the analytical test requested should be used. For metals, samples are collected in 1 quart size plastic Ziploc bags. For organic parameters a 16 oz glass jar is used. Other sample containers sizes may be used with the goal of providing at least 2 cups of soil to sieve.

7.2. The appropriate preservation is determined by the analytical test requested. Mercury and organics samples are to be kept cooled, above freezing but below 6 C, from collection to placing the samples in the drying cabinets. Other metals may be stored at ambient temperature.

7.3. Samples are dried and sieved at room temperature. When thermal preservation is required the samples are then re-cooled after sieving to <6 C and shipped on ice to the analytical laboratory.

8. Definitions

8.1. Definitions are located in the Glossary section of the Quality Manual.

9. Equipment and Supplies (Including Computer Hardware and Software)

9.1. Equipment

Equipment	Vendor	Model / Version	Description / Comments
Ro-Tap sieve shaker	Various manufacture	--	--
Sieves	Gilson or equivalent	--	Stainless steel, #10, #60 or other sizes as needed
Sieve catch pans and lids	Gilson or equivalent	--	Stainless steel
Bakers racks	Restaurant Supply	--	To hold 18" x 26" trays
Drying Fans	Various manufacture	--	--
Mechanical Pulverizer	Retsch	RS100	--
Mortar and Pestle	--	--	Porcelain

9.2. Supplies

Supplies	Vendor	Model / Version	Description / Comments
Aluminum Trays	Restaurant Supply	--	~18" x 26" x 1"
Freezer Paper	Fisher Scientific or equivalent	50-200-5215	18" x 100'
Ziploc bags	--	--	Sandwich, quart, gallon sizes
Wooden Rolling Pin	Restaurant Supply	--	--
Rubber Mallet	--	--	--

10. Reagents and Standards

10.1. Not applicable to this SOP.

11. Calibration and Standardization

11.1. Not applicable to this SOP

12. Procedure

12.1. Place soils in the drying cabinet.

12.1.1. After samples are logged in they will be in a cooler or stored at ambient temperature depending on preservation requirements.

12.1.2. Pull a set of samples to be dried. Order the sample bags in numerical order. Make sure all the bags are present. Note: If standard reference material (SRM) samples were submitted by the client, those samples are not dried and sieved.

12.1.3. Line a tray with freezer paper wax side down. Fold the sides of freezer paper up about 2 inches on each side to form a "boat". Two folded freezer paper sheets should fit on one large tray.

12.1.4. Write the sample number on the freezer paper. Place the entire sample on the freezer paper. Multiple trays may be used for drying if a large sample volume was received.

NOTE: If some of the original sample is to be retained (archived) return about 1/3 of the sample to its original container after homogenization.

12.1.5. 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.

12.1.6. Record the sample numbers in the Soil Sieve Logbook (Attachment I, or equivalent replacement). Record the sample number, the date and the time 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.

12.2. Soil disaggregation procedure

12.2.1. After the samples are dried remove them from the drying cabinets. Record the date and time in the Soil Sieve Logbook. Trays may be stacked on top of each other but place freezer paper on top of the soil so that there is freezer paper between the soil and tray above it. The waxed side of the freezer paper should face away from the soil.

12.2.2. Place a tray on the counter. Carefully remove the freezer paper boat containing the dried sample onto another large tray. Pick any rocks, twigs or other foreign matter and set to the side of the freezer paper boat.

12.2.3. 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. Using a rolling pin, roll over the dried soil for 1-2 minutes. A rubber mallet may be used to disaggregate soil clumps. Take care that the sample remains on the freezer paper. Change gloves between samples.

12.3. Soil Sieving Procedure

12.3.1. Determine the sieve sized to be used to meet project specifications. Check with the project manager or lab manager. Generally soils are sieved using a #10 or #60 mesh sieve. Other sieve sizes may be used per project specifications. When sieve size is not specified by the client, default to #10.

12.3.2. Stack a set of sieves in the following order, bottom catch pan, #60 sieve, #10 sieve or catch pan and #10 sieve. Other sieve sizes may be used, stacking smallest on the bottom to largest on top. Pour the disaggregated soil onto #10 (or top) sieve. All contents are poured onto the sieve including the rocks and foreign matter that had been set to the side. Record the sample number on the side of the catch pan. An abbreviated number may be used such as 407-1. Use a sharpie marker to record.

12.3.3. Stack a second set of sieves, stacked catch pan, #60 sieve, #10 sieve. The stacked catch pan is a catch pan with a lipped underside so that it can be stacked on top of other sieves. Pour sample 2 onto the #10 sieve then stack this set of sieves on top of the other set of sieves. Record the sample number on the catch pan. Place a lid on the top #10 sieve.

12.3.4. Place these two sets of sieves on a RoTap. Tighten the RoTap adjustments so that the sieves fit tightly and securely in the RoTap. Set the timer for 10 minutes and begin the sieve shaking.

12.3.5. After10 minutes remove the sieves off the RoTap. Carefully separate the two sieve sets.

12.3.6. Label a glass jar or Ziploc bag with a printed sample label. Pour the contents in the catch pan into the labeled container. If a baggie is used, double bag to prevent spillage.

NOTE: Great care should be taken in matching the sample number written on the catch pans to the sample numbers on the labeled container.

12.3.7. 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 container, label with the lab number and mark "Coarse Fragments".

12.3.8. Record the sieve date, analyst, shaker ID and sieve size used on the Soil Sieve Logbook. Note yes or no if coarse fragments were retained.

12.4. 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 using the Retsch soil pulverizer. Instructions for the use of the Retsch pulverizer are attached (Attachment II)

12.5. Cleaning Sieves – the sieves must be washed and dried between each use.

12.5.1. 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.

12.5.2. Place the sieves and catch pans in an oven to dry. Alternatively allow to air dry overnight on the counter.

13. Quality Control

13.1. Not applicable to this 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. Not applicable to this SOP.

16. Corrective Actions for Out-of-Control Data

16.1. Not applicable to this SOP.

17. Contingencies for Handling Out-of-Control or Unacceptable Data

17.1. Not applicable to this SOP.

18. Method Performance

18.1. The analyst must read and understand this procedure with written documentation maintained in his/her training file which is located in the QA Office.

18.2. An initial demonstration of capability (IDC) must be performed per SOP S-All-Q-020, or equivalent replacement. A record of the IDC will be maintained in his/her file with written authorization from the Laboratory Manager and Quality Manager. Results are stored in the QA office.

19. Method Modifications

19.1. Not applicable to this SOP.

20. Instrument/Equipment Maintenance

20.1. All maintenance activities are listed daily in maintenance logs that are assigned to each separate instrument.

21. Troubleshooting

21.1. Not applicable to this SOP.

22. Safety

22.1. Chemicals or reagents are not used in this procedure.

22.2. Do not attempt to adjust the sieves in the "RoTap" while in use.

22.3. The "RoTap" sieve shaker should are placed in the drying room to reduce noise levels in the lab. Keep the drying room door closed during use to control noise levels.

23. Waste Management

23.1. Procedures for handling waste generated during this analysis are addressed in S-MT-S-001, Waste Handling and Management.

23.2. The Environmental Protection Agency (USEPA) requires that laboratory waste management practice be conducted consistent with all applicable rules and regulations. Excess reagents, samples and method process wastes are characterized and disposed of in an acceptable manner. For further information on waste management consult SOP S-MT-S-001, or equivalent replacement.

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. National Environmental Laboratory Accreditation Conference (NELAC), Chapter 5, "Quality Systems" - most current version.

25.3. The NELAC Institute (TNI); Volume 1, Module 2, "Quality Systems" - most current version

26. Tables, Diagrams, Flowcharts, and Validation Data

26.1. Attachment I – Soil Sieve Logbook, F-MT-ME-245 (or equivalent replacement)

26.2. Attachment II – RS100 Pulverizer Operating Instructions F-MT-I-192

27. Revisions

Document Number	Reason for Change	Date
S-MT-ME-041-Rev.00	First Issue	24Mar2015

ATTACHMENT I – Soil Sieve Logbook

Pace Analytical	Document Name: Soil Sieve Logbook	Document Revised: 25March13 Page 1 of 50
	Document No.: S-MT-ME-245 Rev.00	Issuing Authority: Pace Montana Quality Office

Sample	Drying		Sieving		Sieve Size	Coarse Frag Retained?	Pulverized?
	Date/ Time In	Date/ Time Out	Date	Shaker ID			
	Date In	Date Out	Date Sieved				
	_____	_____	_____				
	Time In	Time Out	Sieve Time				
	_____	_____	10 Minutes				
	Analyst		Analyst				
	_____		_____				

Shaker IDs: 1-11MT47 2-11MT48 3-11MT49 4-11MT50

Signed By/ Date: _____

ATTACHMENT II – RS100 Pulverizer Operating Instructions

Pace Analytical	Document Name: RS100 Pulverizer Operating Instructions	Document Revised: 15Mar2012 Page 1 of 1
	Document No.: F-MT-I-192-Rev.00	Issuing Authority: Pace Montana Quality Office

PACE MT RS100 PULVERIZER OPERATING INSTRUCTIONS

STATEMENT OF PURPOSE

To aid analysts with the operation of the RS100 Pulverizer.

TO OPEN PULVERIZER

Power on
Key unlocked
Push circle until green and red lights come on
Lift lid

TO OPERATE

Remove lid from grinding set
Leaving grinding tools in place, add sample
Brush excess sample off top of grinding tools
Replace lid
Place set on vibrating plate and clamp securely
Close lid, red light should turn off
Using + or - keys, set time in seconds (20-25 is usually sufficient)
Set speed (usually 1400)
Press blue S to start
Open lid when red light comes on
If washing between samples, you *must* dry the grinding set- otherwise it will rust

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STANDARD OPERATING PROCEDURE

THE DETERMINATION OF PERCENT MOISTURE IN SOIL AND SOLID SAMPLES

Reference Methods: ASTM D 2974-07

Local SOP Number:	S-MN-I-367-Rev.15
Effective Date:	Date of Final Signature
Supersedes:	S-MN-I-367-Rev.14

APPROVALS

Laboratory Assistant General Manager — Date: 7/31/2014

Laboratory Quality Manager — Date: 31 Jul 2014

PERIODIC REVIEW

SIGNATURES BELOW INDICATE NO CHANGES HAVE BEEN MADE SINCE PREVIOUS APPROVAL.

Signature	Title	Date

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TABLE OF CONTENTS

SECTION PAGE

1. Purpose/Identification of Method ... 3
2. Summary of Method ... 3
3. Scope and Application ... 3
4. Applicable Matrices ... 3
5. Limits of Detection and Quantitation ... 3
6. Interferences ... 3
7. Sample Collection, Preservation, Shipment and Storage ... 3
8. Definitions ... 3
9. Equipment and Supplies (Including Computer Hardware and Software) ... 4
10. Reagents and Standards ... 4
11. Calibration and Standardization ... 4
12. Procedure ... 4
13. Quality Control ... 5
14. Data Analysis and Calculations ... 9
15. Data Assessment and Acceptance Criteria for Quality Control Measures ... 9
16. Corrective Actions for Out-of-Control Data ... 9
17. Contingencies for Handling Out-of-Control or Unacceptable Data ... 9
18. Method Performance ... 9
19. Method Modifications ... 9
20. Instrument/Equipment Maintenance ... 10
21. Troubleshooting ... 10
22. Safety ... 10
23. Waste Management ... 10
24. Pollution Prevention ... 10
25. References ... 10
26. Tables, Diagrams, Flowcharts, and Validation Data ... 10
27. Revisions ... 10

1. PURPOSE/IDENTIFICATION OF METHOD

1.1. This standard operating procedure describes the gravimetric determination of the percent moisture by measuring the solids content of soils, peats, organic clays, silts, etc.

2. SUMMARY OF METHOD

2.1. A representative portion of a soil sample is dried in an oven and the solids content is determined by the weight loss. The percent moisture content is calculated from the solids content.

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. **Parameters**: This SOP is applicable to most moisture bearing solids and has the minimum detectable quantity of 0.1%.

4. APPLICABLE MATRICES

4.1. This SOP is applicable to most moisture bearing solids.

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 and Storage.

Sample type	Collection per sample	Preservation	Storage	Hold time
Soil/Solid	Collect samples in an unpreserved sample container. Store above freezing but below 6°C until analysis	n/a	Store above freezing but below 6°C until analysis	There is no specified holding time in ASTM 2974. The LIMS is set to 30 days from collection for the sake of an acode requirement, but data will not be qualified for holding time exceedances

8. DEFINITIONS

8.1. Definitions of terms found in this SOP are described in the Pace Analytical Services Quality Manual, Glossary Section.

8.2. Dry Weight – The weight of a sample based on percent solids after drying in an oven at a 105°C ±5°C.

8.3. Sample Delivery Group (SDG) – A unit within a single project that is used to identify a group of samples for delivery. An SDG is a group of 20 or fewer field samples within a project, received over a period of up to 14 calendar days. Data from all samples in an SDG are reported concurrently. A Sample Delivery Group is generally defined by one of the following, whichever occurs first:

8.3.1. All Samples within a project; or

8.3.2. Every set of 20 field samples within a project; or

8.3.3. All samples received within a 14-day calendar period.

8.3.4. Samples may be assigned to Sample Delivery Groups by matrix (i.e., all soil samples in one SDG, all water samples in another), at the discretion of the laboratory. Clients may establish different SDG classifications to meet project specific requirements.

9. EQUIPMENT AND SUPPLIES (INCLUDING COMPUTER HARDWARE AND SOFTWARE)

9.1. Table 9.1 - Equipment and Supplies

Supply	Description	Vendor/ Item # / Description
Weighing dish	Disposable aluminum foil	Fisher Scientific #08-732-101, or equivalent
Aluminum foil	Novelis Foil, or equivalent	Fisher Scientific 1217, or equivalent
Metal Spatula or Knife	Metal spatula, knife or spoon	N/A
Oven	Gravity Convection Oven	Baxter DS-64, or equivalent
Balance	Analytical with a minimum sensitivity of 0.01 g	SN H47315, or equivalent
Desiccator	With drierite in a metal tray	Fisher Scientific, or equivalent
Pace Workbench	Sample Preparation Logbook and Data Transmission Software	see master list for most current version
Horizon	Data Reporting Software	see master list for most current version

10. REAGENTS AND STANDARDS

10.1. Table 10.1 - Reagent and Standards

Reagent/Standard	Concentration/ Description	Requirements/ Vendor/ Item #
Drierite	Drierite will change from blue to pink when ineffective	Fisher Scientific. P/N 23005
Anhydrous calcium Sulfate	Color indicating	Fisher Scientific P/N 13005

11. CALIBRATION AND STANDARDIZATION

11.1. Initial Calibration

11.1.1. All balances and weights are calibrated by an outside agency on an annual basis.

11.2. Daily Calibration

11.2.1. Daily calibration of the balance using one high, one medium, and one low Class I Standard weight.

11.2.2. Each day calibrate the balance that will be used for the moisture analysis with **50.0 g**, **10.0 g** and **1.0 g** weights that are traceable to the National Institute of Standards. Record the appropriate information in a calibration logbook.

11.2.3. Record oven temperature daily in the appropriate logbook.

11.2.4. Note in comments section of logbook if the Drierite is to be replaced. This is determined by color. If pink, Drierite it no longer anhydrous and must be replaced with anhydrous blue-colored Drierite. See S-MN-O-557, Drierite Regeneration (or equivalent replacement), for procedure.

11.3. Calibrate thermometer in oven on an annual basis. Document calibration using Thermometer Calibration Benchsheet F-MN-L-281 (or equivalent replacement)..

11.4. Read the temperature of the oven on a daily basis. Document in the Oven Temperature logbook.

12. PROCEDURE

12.1. Building the Batch

12.1.1. The maximum batch size allowed is 20 samples. One duplicate should be performed for every 10 samples analyzed.

12.1.2. Double click on EPIC PRO icon and enter your username and password.

12.1.3. Left click on 'batching', left click on 'new batch'.

12.1.4. Type in 'MPRP" as the Queue and click ok.

12.1.5. Double click on the row for Dry Weight.

12.1.6. Click on build batch.

12.1.7. The first sample in the batch is automatically assigned a duplicate. Confirm that there is enough sample to run a duplicate (at least 12g). If the sample selected does not have enough volume available, highlight the DUP and click on the **X** box in the tool bar to remove it. Choose another sample and add a duplicate using the steps below. If there are more than 10 samples in the batch, select one of the samples and then click 'Add QC' and select 'Dup' and click 'Add QC'.

12.1.8. Delete the blank from the batch.

12.1.9. A sample highlighted in RED indicates that the sample is a RUSH.

12.1.10. Press save and print your batch worklist.

12.1.11. Obtain samples from the cooler and organize by batch onto trays.

12.1.11.1. Determine whether or not a specific container was collected for dry weight (normally a 20mL glass vial).

12.1.11.2. If not, a metals container should be utilized for dry weight if possible.

12.1.11.3. Moisture samples cannot be obtained from WIDRO, GRO or VOC sample container.

12.1.12. Dry weights are automatically assigned to samples having a solid matrix. Certain samples are not required to have dry weights. If these samples are logged in to the moisture queue, see a project manager to get the test removed.

12.1.12.1. TCLP

12.1.12.2. 8280 low res dioxin

12.1.12.3. VOC/GRO trip blanks

12.1.12.4. pH

12.1.12.5. Paint Filter

12.1.12.6. Flashpoint

12.2. Build the prep log template.

12.2.1. Launch the balance utility application (BalUtil) from the desktop before logging into the Workbench (the balance utility must be running in order for the balance to connect to the electronic prep log).

12.2.2. Log into the Workbench using your Epic Pro username and password (you must use the Firefox internet browser).

12.2.3. Single click on the Pace Electronic Log Icon (a new window will open).

12.2.4. Load a new 'ASTM D2974 | Percent Moisture / Percent Total Solids' template (F-MN-I-348, or equivalent replacement) in the workbench.

12.2.4.1. Click on tri-colored puzzle piece icon to the right of the "No Template Loaded" field.

12.2.4.2. Click drop down arrow for "search criteria".

12.2.4.3. In the "Qc Rule" field, type in *moisture* and hit search in order to locate the Dry Weight prep log.

12.2.4.4. Click on the Template that populated in the Search Results window.

In the far right pane, under Search by Batch, type in the HBN # for the dry weight batch you created in Epic Pro, click on the magnifying glass icon to display the samples in the HBN.

12.2.4.5. From the list of samples that populates, select all the samples you want to add to the prep log. Click and drag the samples into the sample area of the prep log.

12.2.4.6. Observe the sample position number that the prep log associates with each EPIC PRIO sample number. Use a black marker to write the EPIC Pro batch number on the first tray (empty tray).

12.2.4.7. Order the trays in the exact numerical order that is displayed on the prep log template. The tare masses MUST be obtained in this order.

12.3. Obtain Wet Weight

12.3.1. Click on the Balance icon to the left of the AutoPost button on the tool bar to connect to the balance.

12.3.2. Double click under "Dish Weight", in the bottom, middle pane, for the first sample.

12.3.2.1. Place a tin on the balance, wait for the balance to stabilize and press the red button next to the balance to send the weight to prep log. You should now see the tare mass displayed in the "Dish Weight" field for your sample. Tare all of the subsequent trays in this manner.

12.3.3. Enter the number '1', to correspond to your numbered tray, under the ID field that corresponds to the first sample in your batch. Click in the Dish weight field for the first sample to autopopulate the tray numbers for the remaining samples in the batch. Order the trays in the **exact** numerical order that is displayed on the prep log template. **The wet masses MUST be obtained in this order**.

12.3.4. Place the tray on the balance. The same tare mass that is recorded on the prep log template should be displayed on the balance. Confirm the lab ID with the one in the template.

12.3.5. Obtain a representative sample by stirring. Make a comment in the 'Sample Notes' field if a sample or its DUP is not homogenous. Do not remove any rocks that are smaller that pea size. Add 5.0-10.0g of sample to the tray.

12.3.6. Place tray on the balance and close both balance doors. Double click in the "Wet Weight w/Dish" field for the first sample. Press the red button that is located next to the balance. You should now see the wet mass that is displayed on the balance also displayed in the correct cell of the prep log template. Obtain the wet mass for all of the subsequent samples in this manner.

12.3.7. Click the Disk icon at the top of the prep log to save your work.

12.3.7.1. In the Batch Group pop up window save the prep log using the naming scheme: Batch# HBN# DW

12.4. Dry Samples

12.4.1. Place samples in the oven. Dry the sample overnight (minimum of 16 hours).

12.4.1.1. In the top Batch pane of the prep log template, double click in field under "Oven Temp In1" and enter the observed temperature of the oven in degrees Celsius. (Do not enter the corrected temperature; the prep log performs this calculation).

12.4.1.2. The correction factor of the thermometer ID associated with the oven will calculate and display the corrected temperature based on the observed temperature you recorded. The date, time that samples were placed in the oven, as well as the analyst initials, will populate once the observed temperature is entered.

12.4.2. Click the Disk icon at the top of the prep log to save your work.

12.4.3. If a sample was dried for less than 16 hours, it must be documented that constant weight was attained. To do this, record data for a minimum of two weigh/dry/desiccate weigh cycles with a minimum of 1 hour drying time in each cycle. Constant weight is defined as a loss in weight of <0.01 g between the start weight and final weight of the last cycle.

12.4.4. Samples should not be dried longer than 24hours. Remove the sample from the oven, record the date and time the samples were removed from the oven by entering the observed oven temperature in the top, Batch pane under "Oven Temp Out1". You should see the corrected temperature, date, time, and your initials populate in this field.

12.4.5. Please samples in a desiccator. In the top, Batch pane, double click the field under "Desic. In1" and enter the Instrument ID for the desiccator. You should see the date, time and your initials populate in this field. Allow samples to cool in the desiccator for at least 30 minutes.

12.4.6. Click the Disk icon at the top of the prep log to save your work.

12.5. Obtain Dry Weights

12.5.1. Open the Workbench and find your prep log template.

12.5.1.1. Click on the Magnifying Glass icon.

12.5.1.2. Click drop down arrow for "search criteria".

12.5.1.3. Search for the batch you plan to work with by Analyst Initials, HBN#, Batch#, or Batch Group Name.

12.5.1.4. Double click on the batch to reopen the prep log template.

12.5.2. In the top, Batch pane, double click the field under "Desic. Out1" and enter the desiccator ID to document the date/time/initials you removed the samples from the desiccator.

12.5.3. Click the Balance icon to the left of the AutoPost button to connect the balance to the prep log.

12.5.4. Order the trays in the **exact** numerical order that is displayed on the prep log template . **The dry masses MUST be obtained in this order**.

12.5.5. In the bottom, middle, Sample pane, double click in the field under Dry Weight 1 for the first sample.

12.5.6. Tare the balance, place sample tray on the balance. Close balance doors.

12.5.7. Press the red button that is located next to the balance. You should now see the dry mass that is displayed on the balance also displayed in the correct cell of the prep log template.

12.5.8. Obtain the dry mass for all of the remaining samples in this manner.

12.5.9. The TS Posted (%) and the Percent Moisture data will autopopulate based on the dry weight entered.

12.5.10. Calculate percent solids by the formula below. This calculation will be performed and reported in EPIC via the Workbench. This value will be used for calculating analytical concentration on a dry weight basis

$$\% \text{ Solids} = \frac{\text{Sample Dry Weight}}{\text{Sample Wet Weight}} \times 100$$

12.5.11. Add any batch notes or sample notes to the prep log as necessary. The Batch Notes field in the top pane can be used to document items that affect the whole batch. The Sample Notes field in the bottom, middle pane can be used to document items that are specific to that sample. Remember that all of these fields can be viewed by clients via a data package; take care to ensure spelling and grammar are correct and that no client/proprietary information is revealed.

12.5.12. Click the Disk Icon to save your work.

12.5.13. The RPD of the duplicate sample for each run must be evaluated using either the electronic Logbook or the Validation Report generated from the LIMS.

12.5.14. Validation Report

12.5.14.1. In LIMS under the Systems menu, select Submit Jobs,

12.5.14.2. Select Validation Report from the Jobs menu.

12.5.14.3. Enter the Queue (MPRP) and the HBN number.

12.5.14.4. Click OK and review the Validation Report on screen via PDF.

12.5.15. RPD calculation in the electronic Logbook

12.5.15.1. Once you dry weight data is entered, save the logbook template.

12.5.15.2. Click on the Menu button.

12.5.15.3. Select (DEBUG) Show QC from the menu

12.5.15.4. Review the RPR for the duplicate sample(s).

12.5.16. If the %RPD exceeds 30% for the duplicate sample, the batch must be re-prepped and re-analyzed.

12.6. Auto Posting to Epic

12.6.1. Review your data entries and verify all of the information in the prep log is correct for your batch, click on "AutoPost" to send your data to Epic Pro (once you autopost data you cannot make any changes to the prep log. See your supervisor in the event that any edits are needed after autoposting.).

12.6.2. Give the Batch Worklist you printed in step 12.1.10. to your supervisor for data validation according to SOP S-MN-L-132 (or equivalent replacement).

12.7. Review and validate data.

12.7.1. The data will be available in EPIC PRO in about 20 minutes.

12.7.2. Click on the **EPIC PRO** icon.

12.7.3. Login using your username and password.

12.7.4. Click Batching, Edit, By Batch, enter the Queue and Batch number and press F10.

12.7.5. Review the CC column for all samples in the batch, all entries should be "OK" and the A column should have a (*).

12.7.6. Click 'systems', then click 'Submit Jobs'

12.7.7. Select validation report. Enter the Queue and Batch number and press F10.

12.7.8. View the validation report on-screen.

12.7.9. Click on Batching, Data Review.

12.7.10. Verify Percent Moi column against the information on the validation report.

12.7.11. Double click the status box, click OK, save.

12.7.12. Log into the Work Bench (Electronic Prep Log).

12.7.13. Click on the Magnifying Glass icon and search for the batch you want to review.

12.7.14. After you have reviewed the data, click "Review". You should see the date, time and analyst initials appear in the Reviewed By field, documenting when you performed the review.

12.7.15. Click the Disk icon to save your work.

13. QUALITY CONTROL

13.1. QC Criteria Table 13.1

QC Sample	Components	Frequency	Acceptance Criteria	Corrective Action
Duplicate	Client provided sample	One sample must be prepared and analyzed in duplicate at a frequency of 1 in 10 samples or 1 per analytical batch, whichever is more frequent.	The RPD should be ≤ 30%.	If the dup fails the samples associated with that dup are put into re-run status and they are re-analyzed.

13.2. Assure Drierite is anhydrous. When color-indicating Drierite turns from blue to pink, it must be changed and is no longer anhydrous. Fill metal tray with fresh Drierite (non-indicating), and top with a sufficient amount of blue, color-indicating Drierite. See section 11.2.4 and procedure S-MN-O-557, Drierite Regeneration (or equivalent replacement) for more information.

14. DATA ANALYSIS AND CALCULATIONS

14.1. Calculate the RPD (relative percent difference) of the duplicates for each run as follows:

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

Where:

S = Sample result, (%w/w)
D = Duplicate result, (%w/w)

15. DATA ASSESSMENT AND ACCEPTANCE CRITERIA FOR QUALITY CONTROL MEASURES

15.1. Not applicable to this SOP.

16. CORRECTIVE ACTIONS FOR OUT-OF-CONTROL DATA

16.1. Not applicable to this SOP.

17. CONTINGENCIES FOR HANDLING OUT-OF-CONTROL OR UNACCEPTABLE DATA

17.1. Not applicable to this SOP.

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. **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 S-ALL-Q-020, Training Procedures, or equivalent replacement.

19. METHOD MODIFICATIONS

19.1. Not applicable to this SOP.

20. INSTRUMENT/EQUIPMENT MAINTENANCE

20.1. Not applicable to this SOP.

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. Material Safety Data Sheets (MSDSs) 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 S-MN-S-003, Waste Handling, 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. National Environmental Laboratory Accreditation Conference (NELAC), Chapter 5, "Quality Systems"- most current version.

25.3. The NELAC Institute (TNI); Volume 1, Module 2, "Quality Systems"- most current version.

25.4. EPA Contract Laboratory Program SOW for Inorganic Analysis Document ILM 05.4. December 1, 2006.

25.5. ASTM D 2974-07, Standard Test Methods for Moisture, Ash, and Organic Matter of Peat and Other Organic Soils.

26. TABLES, DIAGRAMS, FLOWCHARTS, AND VALIDATION DATA

26.1. Attachment I - Dry Weight Prep Log Instructions

27. REVISIONS

SOP Number	Revisions	Date
S-MN-I-367-Rev.14	Removed DoD reference from section 25 Removed Tongue depressors and add metal or knife in 9.1	05March 2014
S-MN-I-367-Rev.15	Updated SOP to include new procedure using the Pace Workbench, electronic prep log that replaced LimsLink. 9.1-Replaced Limslink with Pace Workbench in equipment list 11.3-Changed calibration documentation—F-MN-L-281 Benchsheet replaces the logbook 12.1.7 – changed to 12g 12.1.11.3 – changed to WIDRO 12.1.12.3 – 12.1.12.6 – added 12.3.3 – added procedure for dish weight entry 12.5.4 – removed "LimsLink worksheet" and added "prep log template" 12.5.10 – revised calculation 12.5.13-12.5.16 - added 12.2-Added a section to procedure explaining the process of building the prep log template; procedure pertaining to Limslink was deleted 12.3-modified 12.2 and created a separate section for the procedure for obtaining wet weight 12.4-created a sub-section for procedure in dealing with Dry Samples 12.5-modified procedure to apply to Obtaining Dry weights with Pace Workbench 12.6-Updated section to correctly explain Autoposting to Epic using Pace Workbench 12.7—updated Review and Data validation section to apply to Pace Workbench method 26.1—Added Attachment I (Dry weight instructions)	03Jul2014

Attachment I – Dry Weight Prep Log Instruction

Pace Analytical®	Document Name: Dry Weight Prep Log Instructions	Document Revised: 02Apr2014 Page 1 of 2
	Document No.: F-MN-C-141-rev.00	Issuing Authority: Pace Minnesota Quality Office

Logging into the Prep Log:

1. Launch the Balance Utility (BalUtil) from the desktop.
2. Open Pace WorkBench Desktop using FireFox.
3. Preplog username/password is the same as your Epic Pro login.
4. Single Click on *Pace Electronic Log* Icon (a new window will open).

Starting a new Prep Log for your batch:

1. Click on tri-colored puzzle piece icon to the right of the "No Template Loaded" field.
2. Click drop down arrow for "search criteria".
3. In the "Qc Rule" field, type in *moisture* and hit search in order to locate the Dry Weight prep log.
4. Click on the Template that populated in the Search Results window.
5. In the far right pane, under Search by Batch, type in the HBN # for the dry weight batch you created in Epic Pro, click on the magnifying glass icon to display the samples in the HBN.
6. From the list of samples that populates, select all the samples you want to add to the prep log.
   a. You can add the samples individually if you want to pull them into the log in a specific order by clicking on the sample and dragging it into the middle, bottom Sample pane of the prep log.
   b. You can add all the samples at once (samples will populate in the same order they appear in the Search pane) by clicking the first sample #, holding the shift key and clicking the last sample # and dragging the sample set into the middle, bottom Sample pane of the prep log.
7. Click on the Balance icon to the left of the AutoPost button on the tool bar to connect to the balance.
8. Double click under "Dish Weight", in the bottom, middle pane, for the first sample.
   a. Place a tin on the balance and press the red Balance Button to send the weight to prep log.
   b. Repeat for all tins used for all samples in batch.
9. Double click under "Wet Weight/w Dish" for the first sample.
   a. Place a tin on the balance, add sample, and press the red Balance Button to send the weight to prep log.
   b. Repeat for all samples in batch.
10. In the top Batch pane of the prep log, double click in field under "Oven Temp In1" and enter the observed temperature of the oven in degrees C
    a. Do not enter the corrected temperature; the prep log performs this calculation.
    b. Entering the temperature will automatically populate the date/time/initials of when samples were placed in the oven.
    c. The correction factor of the thermometer ID associated with the DW oven 10WET49 will calculate and display the corrected temperature based on the observed temperature you recorded.

    ** If you notice that the thermometer ID, oven ID or correction factor of the equipment in use does not match what is populated in the prep log you must notify QA immediately so that the prep log can be revised.*
11. Click the Disk icon at the top of the prep log to save your work.
    a. In the Batch Group pop up window save the prep log using the naming scheme: ***Batch# HBN# DW***
12. At this point you can either start another batch/prep log or log out and move onto a different task.

Reopening a new Prep Log for your batch:

1. Launch the Balance Utility (BalUtil).
2. Log into the Workbench
3. Click on the Magnifying Glass icon
4. Click drop down arrow for "search criteria".
5. Search for the batch you plan to work with by Analyst Initials, HBN#, Batch#, or Batch Group Name.
6. Double click on the batch to reopen the prep log.
7. In the top, Batch pane, double click the field under "Oven Temp Out1" and enter the observed oven temperature.
8. In the top, Batch pane, double click the field under "Desic. In1" and enter the Instrument ID for the desiccator "10WET50".

Attachment I – Dry Weight Prep Log Instruction (continued)

PaceAnalytical®	Document Name: Dry Weight Prep Log Instructions	Document Revised: 02Apr2014 Page 2 of 2
	Document No.: F-MN-C-141-rev.00	Issuing Authority: Pace Minnesota Quality Office

   a. Typing the Instrument ID will autopopulate the date/time/initials of when the samples were placed in the desiccator.
9. Save the prep log.
10. Repeat steps 1-5 above to log back in when taking samples out of the desiccator.
11. In the top, Batch pane, double click the field under "Desic. Out1" and enter 10WET50 to document the date/time/initials you removed the samples from the desiccator.
12. Click the Balance icon to the left of the AutoPost button to connect the balance to the prep log.
13. In the bottom, middle, Sample pane, double click in the field under Dry Weight 1 for the first sample.
    a. Place the sample on the balance and press the red Balance Button to send the weight to prep log.
    b. Repeat for all samples in batch.
    a. The TS Posted (%) and the Percent Moisture data will autopopulate based on the dry weight entered.
14. Add any batch notes or sample notes to the prep log as necessary. The Batch Notes field in the top pane can be used to document items that affect the whole batch. The Sample Notes field in the bottom, middle pane can be used to document items that are specific to that sample. Remember that all of these fields can be viewed by clients via a data package; take care to ensure spelling and grammar are correct and that no client/proprietary information is revealed.
15. Click the Disk icon to save your work.
16. Review your data entries and verify all of the information in the prep log is correct for your batch, click on "AutoPost" to send your data to Epic Pro.
    *Note: once you autopost data you cannot make any changes to the prep log. See your supervisor in the event that any edits are needed after autoposting.
17. At this point you can either work with another batch/prep log or log out and move onto a different task.

Dry Weight Prep Log Review and Validation:

1. Log into Epic Pro (LIMS)
    a. Click Batching, Edit, By Batch.
    b. Queue: MPRP, Batch: see prep log for this information.
    c. Review the CC column, all entries should be "OK" and the A column should have a (*).
    d. Click on Systems, F9, Validation Report to generate validation report as a PDF to view onscreen.
    e. Click on Batching, Data Review.
    f. Verify Percent Moi column against the information on the validation report.
    g. Double click the status box, click OK, save.
2. Log into the Work Bench (Electronic Prep Log)
    a. Click on the Magnifying Glass icon and search for the batch you want to review.
    b. After you have reviewed the data, click "Review". This will autopopulate the date/time/initials, documenting when you performed the review.

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STANDARD OPERATING PROCEDURE

PREPARATION OF SOLID SAMPLES AND WIPES FOR ANALYSIS BY ICP AND ICP-MS

Reference Methods: EPA 3050B

Local SOP Number:	S-MN-I-460-rev.17
Effective Date:	Date of Final Signature
Supersedes:	S-MN-I-460-rev.16

APPROVALS

Laboratory Assistant General Manager — Date: 5/1/2014

Laboratory Quality Manager — Date: 02May2014

PERIODIC REVIEW

SIGNATURES BELOW INDICATE NO CHANGES HAVE BEEN MADE SINCE PREVIOUS APPROVAL.

Signature	Title	Date

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TABLE OF CONTENTS

SECTION PAGE

1. Purpose/Identification of Method 3
2. Summary of Method 3
3. Scope and Application 3
4. Applicable Matrices 3
5. Limits of Detection and Quantitation 3
6. Interferences 3
7. Sample Collection, Preservation, Shipment and Storage 3
8. Definitions 3
9. Equipment and Supplies (Including Computer Hardware and Software) 3
10. Reagents and Standards 4
11. Calibration and Standardization 5
12. Procedure 5
13. Quality Control 7
14. Data Analysis and Calculations 7
15. Data Assessment and Acceptance Criteria for Quality Control Measures 7
16. Corrective Actions for Out-of-Control Data 7
17. Contingencies for Handling Out-of-Control or Unacceptable Data 7
18. Method Performance 7
19. Method Modifications 8
20. Instrument/Equipment Maintenance 8
21. Troubleshooting 8
22. Safety 8
23. Waste Management 8
24. Pollution Prevention 9
25. References 9
26. Tables, Diagrams, Flowcharts, and Validation Data 9
27. Revisions 9

1. Purpose/Identification of Method

1.1. The purpose of this SOP is to establish a procedure for the digestion of solid samples and wipes to be analyzed by ICP and ICP-MS as described in EPA Method 3050B.

2. Summary of Method

2.1. Inductively coupled plasma atomic emission spectroscopy (ICP-AES) and inductively coupled plasma mass spectrometry (ICP-MS) are utilized for the determination of metals in solution. The method is applicable to a large number of matrices.

2.2. The samples are digested in concentrated nitric acid, hydrochloric acid and hydrogen peroxide. After digestion, samples are filtered (unless ICPMS) and brought to volume.

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. **Parameters**: Not applicable to this SOP.

4. Applicable Matrices

4.1. This SOP is applicable to solid samples and wipes.

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	Plastic or glass containers. Pre-cleaned containers are purchased from a supplier.	N/A	Above freezing but below 6°C until digested if samples are to be tested for mercury too	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 0.01g	Fisher Scientific or equivalent
Resin beads	For solid matrix QC	Environmental Express or equivalent
Ghost Wipes	Meet ASTM E1792 criteria	Environmental Express

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 Spike - Stock solution standards for LCS and MS/MSD	The solution identifications are PA-STD-1B, PA-STD-2B and PA-STD-3B. See 10.1.1.	Purchased from Inorganic Ventures (or equivalent). Store at room temperature. Expires as specified by manufacturer.
ICP-MS Spike - Stock solution standards for LCS and MS/MSD	The solution identifications are XFSPA-656-250, XFSPA-221-250 and XFSPA-220-250. See 10.1.2.	Purchased from Spex CertiPrep (or equivalent). Store at room temperature. Expires as specified by manufacturer.

10.1.1. ICP Stock Standards Table

PA-STD-1B		PA-STD-2B		PA-STD-3B	
element	mg/L	element	mg/L	element	mg/L
As	200	Ag	100	Al	2000
Ba	200	B	200	Ca	2000
Be	200	Mo	200	Fe	2000
Cd	200	Sb	200	K	2000
Co	200	Sn	200	Mg	2000
Cr	200	Ti	200	Na	2000
Cu	200	Zr	200		
Li	200	Si	1000		
Mn	200				
Ni	200				
P	200				
Pb	200				
Se	200				
Sr	200				
Tl	200				
V	200				
Zn	200				

10.1.2. ICPMS Stock Standards Table

XFSPA-656-250		XFSPA-221-250		XFSPA-220-250	
Element	(µg/L)	Element	(µg/L)	Element	(µg/L)
Ca	250	Si	250	Ir	20
Fe	250	Sb	20	Pd	20
Mg	250	As	20	Pt	20
K	250	Sn	20		
Na	250				
Se	20				
Al	20				
Ba	20				
Be	20				
Bi	20				
B	20				
Cd	20				
Ce	20				
Cs	20				
Cr	20				
Co	20				
Cu	20				
La	20				
Li	20				
Mo	20				
Mn	20				
Pb	20				
Ni	20				
Ag	20				
Sr	20				
Tl	20				
V	20				
Zn	20				
U	20				

10.2. Table 10.2 - Working Standard Dilutions and Concentrations

Standard	Standard(s) Amount	Solvent	Solvent Volume	Final Total Volume	Final Concentration
Working LCS/MS Spike Solution. See 10.2.1	50 mL of each standard solution (PA-STD-1B, PA-STD-2B, PA-STD-3B)	DI water	50 mL	200 mL	Varies

10.2.1. Store at room temperature. Expires in 3 months.

11. Calibration and Standardization

11.1. Calibrate variable and fixed volume pipettes as specified in SOP S-MN-Q-264 – Support Equipment (or equivalent replacement). Calibration records are kept in the QA Office.

11.2. Calibrate the thermometer as specified in SOP S-MN-Q-264 – Support Equipment (or equivalent replacement). Calibration records are kept in the QA Office.

12. Procedure

12.1. Sample Preparation

12.1.1. Mix the sample thoroughly to achieve homogeneity. For each digestion procedure, weigh a 1-1.5g portion of sample (to the nearest 0.01g) and transfer to a 50 mL digestioncup. 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 supplicate (MSD) being sure to weigh them as close to the same weight as possible.

12.1.1.1. For wipes, place the entire wipe in plastic digestion cup.

12.1.1.1.1. Create a method blank and a laboratory control sample (LCS) by weighing out 1 gram of resin beads for each. For wipes, use blank wipes for the method blank and laboratory control sample(LCS).

12.1.1.1.2. Spike the LCS.

12.1.2. Add 10mL of DI water to each sample.

12.1.3. Add 7.5mL of concentrated HNO3, 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 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: record initial Hot Black temperature in the digestion log.

12.1.4. 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.

12.1.4.1. 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.

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

12.1.5. Add 5mL 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 ICPMS.

12.1.7. For ICP-AES, samples may be allowed to sit overnight while solid materials settle out or samples may be filtered. If filtered, use FilterMate plunge filters following manufacturers instructions. If samples are filtered all QC samples including the method blank and laboratory control sample (LCS) must also be filtered.

12.1.7.1. Note: The method modifications that have been utilized have been defined in the above process have been demonstrated effective in MDLs, DOCs, successful PTs, and ongoing precision and accuracy data samples.

12.2. Documentation

12.2.1. Standard Prep Logbook: 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 and LCS 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 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.

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 grams of resin beads. For wipes, use a new Ghost Wipe.	Prepared with each batch	See appropriate analysis SOP.	See appropriate analysis SOP.
Laboratory Control Sample (LCS)	For solids, weigh 1.0 gram of resin beads. For wipes, use a new Ghost Wipe. Spike with appropriate spiking solutions.	Prepared with each batch	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**: An MDL study must be conducted annually (per the method) per S-MN-Q-269 – Determination of Limit of Detection and Limit of Quantitation (or equivalent replacement) for each matrix per instrument.

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 S-ALL-Q-020 - Training Procedures (or equivalent replacement).

18.4. **Periodic performance evaluation (PE)** samples are analyzed to demonstrate continuing competence per SOP S-MN-Q-258 – Proficiency Testing Program (or equivalent replacement). Results are stored in the QA office.

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. Material Safety Data Sheets (MSDSs) 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 S-MN-S-003 - 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. National Environmental Laboratory Accreditation Conference (NELAC), Chapter 5, "Quality Systems"- most current version.

25.3. The NELAC Institute (TNI); Volume 1, Module 2, "Quality Systems"- most current version.

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

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-460-Rev.16	Implemented new corp-issued SOP format Removed "or Deena" from hot block in equipment list 12.1.1.2.1 – added 12.1.5 – added "do not add HCl…"	26Feb2013
S-MN-I-460-Rev.17	Title changed/1.1/4.1 – removed Filters 2.1 – removed matrices 9.1/10.1/13.1 - updated 12.1.1 – added 'Weigh out 3 aliquots…' 12.1.1.1 – removed filters 12.1.1.1.1 – adeed 12.1.1.1.2 – removed words after 'LCS' 12.1.4 – removed 5 mins; added note to and removed 12.1.5 12.1.5 – removed '(do not add…)' 12.1.6 – added 'invert several times…' 12.1.7 – modified 19.1 – 19.2 – modified; added 19.3 20.3 - added Removed DoD section from section 25	31Mar2014

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STANDARD OPERATING PROCEDURE

INDUCTIVELY COUPLED PLASMA ATOMIC EMISSION SPECTROSCOPY

Reference Methods: EPA 6010B, 6010C, and EPA 200.7

Local SOP Number:	S-MN-I-313-rev.25
Effective Date:	Date of Final Signature
Supersedes:	S-MN-I-313-rev.24

APPROVALS

Laboratory Assistant General Manager — Date: 5/1/2014

Laboratory Quality Manager — Date: 01 May 2014

PERIODIC REVIEW

SIGNATURES BELOW INDICATE NO CHANGES HAVE BEEN MADE SINCE PREVIOUS APPROVAL.

Signature	Title	Date

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TABLE OF CONTENTS

SECTION		PAGE
1.	Purpose/Identification of Method	3
2.	Summary of Method	3
3.	Scope and Application	3
4.	Applicable Matrices	3
5.	Limits of Detection and Quantitation	3
6.	Interferences	3
7.	Sample Collection, Preservation, Shipment and Storage	4
8.	Definitions	4
9.	Equipment and Supplies (Including Computer Hardware and Software)	4
10.	Reagents and Standards	5
11.	Calibration and Standardization	5
12.	Procedure	7
13.	Quality Control	9
14.	Data Analysis and Calculations	10
15.	Data Assessment and Acceptance Criteria for Quality Control Measures	10
16.	Corrective Actions for Out-of-Control Data	10
17.	Contingencies for Handling Out-of-Control or Unacceptable Data	11
18.	Method Performance	11
19.	Method Modifications	11
20.	Instrument/Equipment Maintenance	11
21.	Troubleshooting	11
22.	Safety	11
23.	Waste Management	11
24.	Pollution Prevention	12
25.	References	12
26.	Tables, Diagrams, Flowcharts, and Validation Data	12
27.	Revisions	12

1. Purpose/Identification of Method

1.1. The purpose of this SOP is to establish a procedure for the determination of metals by inductively coupled plasma atomic emissions spectroscopy (ICP-AES) as delineated in EPA Method 6010B, 6010C or 200.7.

2. Summary of Method

2.1. Prior to analysis, samples must be solubilized or digested using appropriate sample preparation methods.

2.2. This method describes the determination of elements by ICP-AES. The method measures element-emitted light by optical spectrometry. Samples are nebulized and the resulting aerosol is transported to the plasma torch. Element-specific atomic-line emission spectra are produced by a radio-frequency inductively coupled plasma. The spectra are dispersed by a grating spectrometer, and the intensities of the lines are monitored by a charge coupled device detector.

2.3. Background correction may be required. Background is measured adjacent to analyte lines on samples during analysis. The position selected for the background-intensity measurement, on either or both sides of the analytical line, will be determined by the complexity of the spectrum adjacent to the analyte line. The position used should be free of spectral interference and reflect the same change in background intensity as occurs at the analyte wavelength measured. Background correction is not required in cases of line broadening where a background correction measurement would actually degrade the analytical result. The possibility of additional interferences should also be recognized and appropriate corrections made as necessary.

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. **Parameters**: This SOP applies to the elements listed in Attachment I.

4. Applicable Matrices

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

5. Limits of Detection and Quantitation

5.1. The reporting limit (LOQ) for all analytes is listed in Attachment I. All current MDLs are listed in the LIMS and are available by request from the Quality Manager.

6. Interferences

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

6.1.1. Spectral overlap can be compensated by computer-correcting the raw data after monitoring and measuring the interfering element. Unresolved overlap requires selection of an alternate wavelength. Background contribution and stray light can usually be compensated for by a background correction adjacent to the analyte line. Interelement correction factors are used on the simultaneous ICP.

6.2. Physical Interferences are effects associated with the sample nebulization and transport processes. Changes in viscosity and surface tension can cause significant inaccuracies, especially in samples containing high dissolved solids or high acid concentrations. If physical interferences are present, they must be reduced by diluting the sample, by using a peristaltic pump, by using an internal

standard, or by using a high solids nebulizer. Another problem that can occur with high dissolved solids is salt buildup at the tip of the nebulizer, affecting aerosol flow rate and causing instrument drift.

6.3. Chemical interferences include molecular compound formation, ionization effects and solute vaporization effects. Normally, these effects are not significant with the ICP technique, but if observed, can be minimized by careful selection of operating conditions (incident power, observation position, and so forth), but buffering the sample, by matrix matching, and by standard addition procedures.

6.4. Memory interferences result when analytes in a previous sample contribute to the signals measured in the new sample. Memory effects can result from sample deposition on the uptake tubing to the nebulizer and from buildup of sample material in the plasma torch and spray chamber.

6.5. Users are advised that high salt concentrations can cause analyte signal suppressions and confuse interference tests.

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
Liquid	Glass containers. Collect dissolved metal samples and filter immediately through a 0.45-micron filter on-site by the sampler before adding preservative. If samples are filtered at the laboratory, use a polyethylene or glass container and preserve after filtration with HNO_3.	Preserve immediately with HNO_3 to bring the pH to <2 For samples received with a pH>2, additional nitric acid must be added upon receipt to dissolve the metals that may have adhered to the sample container. Sample receiving adds the additional acid, labels the samples with the amount of acid added, the lot number of the acid, date, time and initials of person that added the acid. The samples must not be analyzed for 24 hours from acid addition per the Method Update Rules.	Store total and dissolved metal samples at room temperature.	The maximum sample holding time for metals is 6 months from sample collection.
Solid	Glass or polyethylene container	N/A	Above freezing but below 6°C.	The maximum sample holding time for metals is 6 months from sample 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
Perkin Elmer Optima	4300 (or equivalent)	
Desktop computer and printer		
Perkin Elmer AS 93	Plus Autosampler	

Peristaltic pump and Fisherbrand pump tubing, or equivalent.		
Refrigerated Circulator		
Argon gas supply	high-purity grade, 99.99%	House Argon
Mechanical pipettes, and metals-free disposable pipet tips		
Glassware	Class A volumetric flasks, graduated cylinders and funnels (glass or metals-free class B plastic).	
Disposable digestion cups	50 mL	
Epic Pro	Data reporting software	See master list for current version
LimsLink	Data transmission software	See master list for current version
WinLab 32 ICP Continuous	Automated Analysis Control	See master list for current version

10. Reagents and Standards

10.1. Table 10.1 – Reagents and Standards

Reagent/Standard	Concentration/Description	Requirements/Vendor/Item #
De-ionized Water	ASTM Type II	House DI water
Concentrated Hydrochloric acid (HCl)	Trace Metals grade	Fisher
Concentrated Nitric Acid (HNO3)	Trace Metals grade	Fisher
Calibration Standard Stock Solutions	Custom blend	Spex Certiprep or equivalent
Initial Calibration Verification (ICV) Stock Standard solutions	Custom blend. Must be separate stock from the calibration standards.	CPI or equivalent
Profiling Solution	Manganese, at a concentration of 1 mg/L or 10 mg/L, is used to profile the system	
Internal Standards (optional)	Scandium (1 mg/L) or Yttrium (5 mg/L) may be used as an internal standard	

10.2. Table 10.2 - Working Standard Dilutions and Concentrations

10.2.1. See Attachment VII – ICP Standard Prep Log 8632

11. Calibration and Standardization

11.1. Table 11.1 – Calibration and Standardization

Calibration Metric	Parameter/Frequency	Criteria	Comments
Initial Calibration (ICAL)	Instruments must be calibrated at a minimum once every 24 hours or prior to use. The instrument standardization date and time must be included in the raw data. See Attachment VI for an example run sequence. A calibration curve must consist of a blank and at least one calibration standard	Linear regression: $r \geq 0.995$ for 6010B, 200.7 $r \geq 0.998$ for 6010C	If not met, remake standards and recalibrate and verify before sample analysis.
Second Source Verification Standard (ICV)	Immediately after the calibration standards have been analyzed, the accuracy of the initial calibration shall be verified and documented for every analyte by the analysis of an ICV Solution(s) at each	± 10% for method 6010B and 6010C or ± 5% for method 200.7 The RSD of the standards must be below 5% for 6010 and 3% for 200.7 W of the replicate readings.	Review the standard preparation. Remake the standard accordingly if that is the cause. Re-inject the ICV one more time, if it fails stop all analysis. Perform all necessary

	wavelength used for analysis. The Initial Calibration Verification (ICV) Solution(s) should be obtained from a different source than the calibration standards.		instrument maintenance and recalibrate the instrument. Only two injections are allowed back to back, then the system must be recalibrated.
Continuing Calibration Verification (CCV)	To ensure calibration accuracy during each analytical run, a CCV standard must be analyzed after every 10 samples and at the end of the run for each wavelength. The ICV solution can be utilized as the CCV.	For method 6010B, 6010C and 200.7, the CCV must be within ± 10% of the true value. The RSD of the standards must be below 5% for 6010 and 3% for 200.7 W of the replicate readings.	If the requirements for continuing calibration are not met, review for preparation error or instrument malfunction. A CCV may be repeated, but a second failure requires the system to be recalibrated prior to further analysis. If the samples bracketed are non-detect and the CCV is biased high, data may be reported as there is no impact from the high bias. If the samples associated are non-detect and the only detections are associated with the batch QC (LCS/MS) but the QC is within limits, the data can be reported. The QC should be flagged indicating that there was bias but that there was no impact to the associated samples. If the CCVs are biased low, reanalyze any samples impacted since the last passing CCV.
6010B/200.7 - Contract Required Detection Limit Sample (CRDL)	The CRDL must be analyzed at the beginning of each run for every analyte of interest. The CRDL is at or below the RL.	± 40% (or specified by the client)	. The system must be stopped. Perform any necessary maintenance and recalibrate accordingly.
6010C – Low Level Initial/Continuing Calibration Verification (LLICV/LLCCV)	The LLICV must be analyzed following the ICV at a concentration at or below the RL. Additionally, a LLCCV (may be the same solution as the LLICV) must be analyzed at a frequency of once at the end of each analytical batch, preferably every 10 samples following the CCV to minimize sample re-runs.	± 30%	. The system must be stopped. Perform any necessary maintenance and recalibrate accordingly.
Initial Calibration Blank (ICB)	An ICB must be analyzed immediately following ICV	All elements of interest must be evaluated to the method detection limit. Depending on the data	. If an analyte of interest is greater than the RL, the sample concentration must

	for each element of interest.	quality objective of the associated projects, a blank with detections less than the RL may be considered acceptable	be greater than 10 times the blank concentration or the element cannot be reported. If associated projects are evaluated to the method detection limits per data quality objects, the detections must be evaluated for data impact and the system evaluated for necessary corrective actions.
Continuing Calibration Blank (CCB)	A CCB must be analyzed, for each element of interest after every CCV within the analytical run and after the final CCV.	All elements of interest must be evaluated to the method detection limit. Depending on the data quality objective of the associated projects, a blank with detections less than the RL may be considered acceptable.	If an analyte of interest is greater than the RL, the sample concentration must be greater than 10 times the blank concentration or the element cannot be reported. If associated projects are evaluated to the method detection limits per data quality objects, the detections must be evaluated for data impact and the system evaluated for necessary corrective actions.
Interelement Correction Standard A (ICSA)	A solution containing high concentrations of Al, Ca Fe and Mg is analyzed at the beginning of each sample run sequence	Acceptance criteria for the spiked interferent elements are ± 20% and ± 2X the RL for target analytes.	If the ICSA fails criteria, stop analysis. Review the standard preparation, remake accordingly. Perform any necessary maintenance and recalibrate prior to sample analysis. Additional corrective actions may be required for client specific QAPP and Technical Specifications
Interelement Correction Standard AB (ICSAB)	A solution containing high concentrations of Al, Ca, Fe and Mg and low to mid-range concentrations of the target analytes is analyzed following the ICSA This is required by certain clients. It is not a method requirement and need be analyzed only for clients specifying this in the QAPP	The acceptance criteria are ± 20%.	If the ICSAB fails criteria, stop analysis. Review the standard preparation, remake accordingly. Perform any necessary maintenance and recalibrate prior to sample analysis. Additional corrective actions may be required for client specific QAPP and Technical Specifications

12. Procedure

12.1. Instrument Set up and Operation

12.1.1. Perform the daily maintenance if needed (change pump tubing, fill rinse solution container, drain waste container, etc.) and record in the daily maintenance log.

12.1.2. Start the ICP and let it warm up.

12.1.2.1. Check the gas supply.

12.1.2.2. Confirm the water circulator/chiller is on.

12.1.2.3. Turn on the computer.

12.1.2.4. Double click on the Optima (WinLab32) program icon. Open the workspace that has the method to run: it will open as well.

12.1.2.5. Click on the plasma icon.

12.1.2.6. Click on the startup/shutdown button.

12.1.2.7. The software will go through several steps until the argon is ignited. Check to be sure the gas flow and pump speed are correct. If not, make changes and click on Apply

12.1.2.8. Close the window.

12.1.3. Create an auto sampler file and pour samples into the corresponding location in the auto sampler tray.

12.1.3.1. Open sample information table by clicking on the button for Sample Info.

12.1.3.2. Click on New/File.

12.1.3.3. Enter in the batch ID, the name of the analyst and standard log numbers. Go down to the table and type in the samples to be analyzed.

12.1.3.4. Click on File Save As and type Date for the data file (which is usually the same as the batch ID).

12.1.3.5. Click on Setup and click on Open to choose which results data set to use. Choose or type in the data set, click OK.

12.1.3.6. Click on Analyze tab. Click on rebuilt list or reset sequence buttons to put the sample information into the analyze window.

12.1.3.7. Print the Sample Info page to get the run log.

12.1.4. Pour the standards and start the calibration of the instrument.

12.1.5. Monitor all initial QC checks. If initial QC fails, make instrument modifications and recalibrate. If checks pass criteria, continue with sample analysis.

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

12.1.7. In LIMS system enter any dilutions and any required footnotes. Print validation lists and complete checklist. Turn data in for validation.

12.1.8. ICP Routine Maintenance:

12.1.8.1. Change pump tubing if there is visible wear or flat spots.

12.1.8.2. Change filters as needed.

12.1.8.3. Clean optic window as needed.

12.1.8.4. Empty the waste container as needed.

12.1.8.5. Replace the torch as needed.

12.1.8.6. Clean injector as needed.

12.2. Sample Analysis

12.2.1. Load samples and batch QC into designated autosampler locations making sure the correct spot is used.

12.2.2. Start analytical procedure.

12.2.3. Monitor results and standard recoveries for problems.

12.2.4. At the end of the analytical run, perform the manual shut down.

12.2.4.1. If the analysis extends past working hours, set the instrument up for auto shutdown.

12.3. Daily File

12.3.1. Gather daily printouts.
12.3.2. Print out instrument raw data.
12.3.3. Include calibration summaries and runlogs.
12.3.4. Update and include necessary standard prep logs.
12.3.5. Label the daily file folder with date and instrument information.

13. Quality Control

13.1. Table 13.1 – Quality Control

QC Sample	Components	Frequency	Acceptance Criteria	Corrective Action
Method Blank (MB)	DI water for liquid samples Resin beads for solid samples	Prepared and analyzed with each group of samples digested. For 6010C - Carried through the appropriate steps of the analytical process. These steps may include, but are not limited to, prefiltering, digestion, dilution, filtering and analysis.	< absolute value of the reporting limit (RL) For 6010C - If the method blank does not contain target analytes at a level that interferes with project-specific DQOs, then the method blank would be considered acceptable.	If the concentration in the MB is greater than the RL, samples associated with that MB must be re-prepared, unless the samples are non-detect or greater than 10X the blank contamination. When reporting data with a hit in the MB, all samples affected will be footnoted with the appropriate flag to document contamination in the blank.
Laboratory Control Sample (LCS)	DI water for liquids and resin beads for solids, spiked with analytes of interest at same level as MS/MSD	Prepared and analyzed for every batch of 20 or less samples digested	80-120% for 6010B and 6010C 85-115% for 200.7	If the percent recovery for the LCS falls outside the control limits of 80-120% for 6010B and 6010C or 85-115% for 200.7, the analyses should be terminated, the problem corrected, and the samples associated with that LCS re-analyzed. If reanalysis of the samples fail, the samples affected by the failing LCS elements need to be re-digested and re-analyzed. ***EXCEPTION:*** if LCS fails high and samples are ND the data may be reported with appropriate qualification.
Matrix Spike (MS) / Matrix Spike Duplicate (MSD)	The spike is added to a well-mixed aliquot of a selected sample before the digestion (i.e., prior to the addition of other reagents).	One MS/MSD per batch. If >10 samples for 200.7, an additional MS is required. Clients may have requirements that create a higher frequency of MS/MSD samples.	75-125% for 6010B and 6010C 70-130% for 200.7 % RPD: 20% for 6010B/C; 20% for 200.7	If the percent recovery for the MS and MSD fall outside the control limits, the results are flagged that they are outside acceptance criteria along with the parent sample. If the RPD exceeds the acceptance criteria, the MSD sample and associated parent sample need to be flagged. For Minnesota Admin Contract clients – all MS/MSD failures require reanalysis of the MS/MSD and the original sample. If it is still out of control, investigate and document the

				cause in the associated narrative as well as qualifying appropriately.
Post Digestion Spike (PDS)	Spike is added to the native QC sample at the same concentration as the MS but at the instrument.	Required if reporting by 6010C and MS/MSD fail outside 75-125% and if the PDS also fails then a 5x dilution is made of the PDS.	75-125% for 6010B and 80-120% for 6010C	If PDS fails data is qualified
Internal Standard	The same concentration should be used for standards and samples throughout the entire analytical run.	Introduced automatically with every sample.	70-130% of its true concentration	If the recovery is outside the criteria, sample is reanalyzed at a 5X dilution.

13.2. If matrix interference is suspected, a serial dilution may be performed. One serial dilution test may be performed per batch of twenty samples per client contractual requirements. An analysis of a 1:5 dilution should agree within ± 10% of the original result. Additional dilutions may be required.

13.3. When reviewing total and dissolved metals, an RPD of ≤ 20% is considered to be within experimental error.

14. Data Analysis and Calculations

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

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

Where: SSR = Spike sample result, ug/L or mg/kg dry
SR = Sample result, ug/L or mg/kg dry
ST = Spike target, ug/L or mg/kg dry

14.2. The relative percent difference between the MS/MSD can be calculated as follows:

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

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

15. Data Assessment and Acceptance Criteria for Quality Control Measures

15.1. See tables in section 11 and 13.

16. Corrective Actions for Out-of-Control Data

16.1. See tables in section 11 and 13.

17. Contingencies for Handling Out-of-Control or Unacceptable Data

17.1. If not specifically listed in the tables in section 11 or 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**: An MDL study must be conducted annually (per the method) per S-MN-Q-269 – Determination of Limit of Detection and Limit of Quantitation (or equivalent replacement) for each matrix per instrument.

18.3. **Instrument Detection Limit (IDL) Study**: An IDL study must be conducted quarterly per S-MN-Q-269 – Determination of Limit of Detection and Limit of Quantitation (or equivalent replacement).

18.4. **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 S-ALL-Q-020 - Training Procedures (or equivalent replacement).

18.5. **Periodic performance evaluation (PE)** samples are analyzed to demonstrate continuing competence per SOP S-MN-Q-258 – Proficiency Testing Program (or equivalent replacement). Results are stored in the QA office.

19. Method Modifications

19.1. Not applicable for this SOP.

20. Instrument/Equipment Maintenance

20.1. All maintenance activities are listed daily in maintenance logs that are assigned to each separate instrument.

21. Troubleshooting

21.1. Not applicable for 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. Material Safety Data Sheets (MSDSs) 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 S-MN-S-003 - 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. National Environmental Laboratory Accreditation Conference (NELAC), Chapter 5, "Quality Systems"- most current version.
25.3. The NELAC Institute (TNI); Volume 1, Module 2, "Quality Systems"- most current version.
25.4. Test Methods for Evaluating Water and Solid Waste, SW-846 3rd Edition, Final Update III, Method 6010B.
25.5. Test Methods for Evaluating Water and Solid Waste, SW-846, Method 6010C Update IV, Feb. 2007.
25.6. Perkin Elmer Hardware Guide 1997.
25.7. Perkin Software Guide 2000.
25.8. Method 200.7 Revision 4.4, Determination of Metals and Trace Elements in Water and Wastes by Inductively Coupled Plasma-atomic Emission Spectrometry
25.9. Test Methods for Evaluating Solid Waste Physical/Chemical Methods, SW-846, Third Edition. Method 3050B

26. Tables, Diagrams, Flowcharts, and Validation Data

26.1. Attachment I - Target Analyte List and Reporting Limits (PRL)
26.2. Attachment II - Trace-ICP Working Calibration Standard
26.3. Attachment III - Trace-ICP Calibration Verification Standard
26.4. Attachment IV - ICSA
26.5. Attachment V - Trace CRDL #2
26.6. Attachment VI - Sample Run Sequence
26.7. Attachment VII – ICP Standards Prep Log 8632

27. Revisions

Document Number	Reason for Change	Date
S-MN-I-313-rev.24	Implemented new corp SOP format 18.3 – added 26.1-26.6 – changed "table" to "attachment" Added – attachment VII	01Aug2013
S-MN-I-313-rev.25	1.1/2.2 – added 'AES' to ICP 2.2 – edited Table 7.1 – updated sample type Table 9.1/10.1/11.1/13.1 – updated Added and changed method criteria (RPD) to 20% Added PDS criteria and frequency Removed DoD reference from section 25	30Apr2014

ATTACHMENT I – Target Analyte List and Reporting Limits (PRL)

Element	Wavelength [1](nm)	Water PRL (ug/L)	Soil PRL (mg/kg)
Aluminum	396	200	10
Antimony	217	20	1.0
Arsenic	193	20	1.0
Barium	233	10	0.50
Beryllium	313	5.0	0.25
Boron	249	150	7.5
Cadmium	228	3.0	0.15
Calcium	317	500	25
Chromium	267	10	0.50
Cobalt	228	10	0.50
Copper	327	10	0.50
Iron	238	50	2.5
Lead	220	10	1.0
Magnesium	279	500	25
Manganese	257	5.0	0.25
Molybdenum	202	15	0.75
Nickel	231	20	1.0
Potassium	766	2500	125
Selenium	196	20	0.75
Silver	328	10	0.50
Sodium	589	1000	50
Thallium	190	20	1.0
Tin	189	75	3.75
Titanium	334	25	1.25
Vanadium	290	15	0.75
Zinc	206	20	1.0
Hardness	N/A	3300	134

[1]The wavelengths listed are recommended because of their sensitivity and overall acceptance. Other wavelengths may be substituted when using a sequential instrument if they can provide the needed sensitivity and are treated with the same corrective techniques for spectral interference.

ATTACHMENT II – Trace-ICP Working Calibration Standard

Element	Stock Conc. (mg/L)	Aliquot (mL)	Final Volume (mL)	Cal STD Final Conc. (mg/L)
Ag	100	1.0	50	2
Al	2,000	0.5	50	20
As	200	1.0	50	4
Ba	200	1.0	50	4
Be	200	1.0	50	4
Ca	2000	0.5	50	20
Cd	200	1.0	50	4
Co	200	1.0	50	4
Cr	200	1.0	50	4
Cu	200	1.0	50	4
Fe	2000	0.5	50	20
K	2000	0.5	50	20
Mg	2000	0.5	50	20
Mn	200	1.0	50	4
Na	2000	0.5	50	20
Ni	200	1.0	50	4
Pb	200	1.0	50	4
Sb	200	1.0	50	4
Se	200	1.0	50	4
Tl	200	1.0	50	4
V	200	1.0	50	4
Zn	200	1.0	50	4
Mo	200	1.0	50	4
B	200	1.0	50	4
Sn	200	1.0	50	4
Ti	200	1.0	50	4

ATTACHMENT III – Trace-ICP Calibration Verification Standard

Element	Stock Conc. (mg/L)	Aliquot in (mL)	Final Volume (mL)	Final Conc. (mg/L)
Ag	50	1.0	50	1
Al	1000	0.5	50	10
As	100	1.0	50	2
Ba	100	1.0	50	2
Be	100	1.0	50	2
Ca	1000	0.5	50	10
Cd	100	1.0	50	2
Co	100	1.0	50	2
Cr	100	1.0	50	2
Cu	100	1.0	50	2
Fe	1000	0.5	50	10
K	1000	0.5	50	10
Mg	1000	0.5	50	10
Mn	100	1.0	50	2
Na	1000	0.5	50	10
Ni	100	1.0	50	2
Pb	100	1.0	50	2
Sb	100	1.0	50	2
Se	100	1.0	50	2
Tl	100	1.0	50	2
V	100	1.0	50	2
Zn	100	1.0	50	2
Mo	100	1.0	50	2
B	100	1.0	50	2
Sn	100	1.0	50	2
Ti	100	1.0	50	2

ATTACHMENT IV – ICSA

Element	Stock Conc. (mg/L)	Aliquot in (mL)	Final Volume (mL)	Final Conc. (mg/L)
Al	10000	2.5	1000	25
Ca	10000	50	1000	500
Fe	10000	7.5	1000	75
Mg	10000	15	1000	150

ATTACHMENT V – Trace CRDL2

Element	Stock Conc. (mg/L)	Aliquot in (mL)	Final Volume (mL)	Final Conc. (mg/L)
Ag	5	1.0	1000	0.005
Al	100	1.0	1000	0.100
As	5	1.0	1000	0.005
Ba	1.5	1.0	1000	0.0015
Be	5	1.0	1000	0.005
Ca	250	1.0	1000	0.250
Cd	0.5	1.0	1000	0.0005
Co	10	1.0	1000	0.010
Cr	10	1.0	1000	0.010
Cu	5	1.0	1000	0.005
Fe	50	1.0	1000	0.050
K	250	1.0	1000	0.250
Mg	250	1.0	1000	0.250
Mn	5	1.0	1000	0.005
Na	1000	1.0	1000	1.0
Ni	20	1.0	1000	0.020
Pb	3	1.0	1000	0.003
Sb	5	1.0	1000	0.005
Se	5	1.0	1000	0.005
Tl	10	1.0	1000	0.010
V	15	1.0	1000	0.015
Zn	20	1.0	1000	0.020
Mo	15	1.0	1000	0.015
B	75	1.0	1000	0.075
Sn	35	1.0	1000	0.035
Ti	10	1.0	1000	0.010

The CRDL standard is prepared by diluting the CRDL2 standard by a factor of 2.

ATTACHMENT VI – Sample Run Sequence

1. **Calibration Blank**
2. **Calibration Standard**
3. **ICV**
4. **LLICV (6010C)**
5. **ICB**
6. **CRDL (6010B and 200.7)**
8. **CRDL2 (6010B and 200.7)**
9. **ICSA**
10. **ICSAB**
11. **CCV**
12. **LLCCV (6010C)**
13. **CCB**
14. **SAMPLE 1**
15. **SAMPLE 2**
16. **SAMPLE 3**
17. **SAMPLE 4**
18. **SAMPLE 5**
19. **SAMPLE 6**
20. **SAMPLE 7**
21. **SAMPLE 8**
22. **SAMPLE 9**
23. **SAMPLE 10**
24. **CCV**
25. **CCB**
26. **CRDL (project specific)**
27. **CRDL2 (project specific)**
28. **ICSA (project specific)**
34. **ICSAB (project specific)**
35. **CCV**
36. **CCB**

ATTACHMENT VII - ICP Standards Prep Log 8632

ICP standard log 8632

ICP Standards Prep Log 8632

STD. ID	USED	Standard name	Elements	Initial		exp.date	stock ID	stock lot	stock conc (mg/L)	stock amt. (ml)	final vol. (ml)	Final conc. (ug/L)
N/A	y	Low Standard	N/A	N/A	N/A	N/A	N/A	—	—	10.0	40	5000/1000/500
8632- 2156	y	High Standard	see label	IP	7/24/2013	7/31/2013	8074-666	IV F2-MEB410061	2000	0.5	50	20000
8632- 2156	y	High Standard	see label	IP	7/24/2013	7/31/2013	8074-664	IV F2-MEB410060	200/100	1.0	50	4000/2000
8632- 2156	y	High Standard	see label	IP	7/24/2013	7/31/2013	8074-662	IV F2-MEB410059	200	1.0	50	4000
8632- 2156	y	High Standard	Au	IP	7/24/2013	7/31/2013	8074-357	Ricca 4112631	1000	0.1	50	2000
N/A	y	High Standard	S	N/A	N/A	N/A	8074-433	ULTRA P00037	1000	0.1	50	2000
8632- 2157	y	ICV/CCV	see label	IP	7/24/2013	7/31/2013	8074-530	SP 15-122CR	1000	0.5	50	10000
8632- 2157	y	ICV/CCV	see label	IP	7/24/2013	7/31/2013	8074-709	SP 19-029CR	100/50	1	50	2000/1000
8632- 2157	y	ICV/CCV	see label	IP	7/24/2013	7/31/2013	8074-668	SP 14-034CR	100	1	50	2000
8632- 2157	y	ICV/CCV	Au	IP	7/24/2013	7/31/2013	8074-471	FS 120437	1000	0.05	50	100
N/A	y	ICV/CCV	S	N/A	N/A	N/A	8074-469	SP 16-137S	10000	0.05	50	1000
8632- 2158	y	CRDL	see label	IP	7/24/2013	7/25/2013	8632-2120	—	various	15	30	various
8632- 2120	y	CRDL2	see label	IP	7/8/2013	7/29/2013	8074-737	see p. 652	various	2	1000	various
8632- 2047	y	ISCA	see label	IP	6/10/2013	12/10/2013	8074-772	CPI 13D009	5000/2000	20	200	500000/200000
8632- 2048	y	ISCAB	see label	IP	6/10/2013	10/15/2013	8074-772	CPI 13C018	5000/2000	20	200	500000/200000
8632- 2048	y	ISCAB	see label	IP	See Pg. 607	See Pg. 607	8632-1941	—	various	20	200	various
8632-2121	y	Tuning Solution	Mn	IP	See Pg. 666	See Pg. 666						
8632-2122	y	IS Std	Y	IP	See Pg. 666	See Pg. 666						

DI water: 2%HNO3 / 5% HCl (lot#): W 0133

Date prepared: 7/24/2013

Reviewer

Date

Page 678

I209 Rev.00 (24Feb2009)

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STANDARD OPERATING PROCEDURE

METALS ANALYSIS BY ICP/MS

Reference Methods: EPA Methods 6020/6020A/200.8

Local SOP Number:	S-MN-I-492-rev.23
Effective Date:	Date of Final Signature
Supersedes:	S-MN-I-492-Rev.22

APPROVALS

Laboratory Assistant General Manager — Date: 12/14/2014

Laboratory Quality Manager — Date: 15Dec2014

PERIODIC REVIEW

SIGNATURES BELOW INDICATE NO CHANGES HAVE BEEN MADE SINCE PREVIOUS APPROVAL.

Signature	Title	Date

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TABLE OF CONTENTS

SECTION PAGE

1. Purpose/Identification of Method 3
2. Summary of Method 3
3. Scope and Application 3
4. Applicable Matrices 4
5. Limits of Detection and Quantitation 4
6. Interferences 4
7. Sample Collection, Preservation, Shipment and Storage 6
8. Definitions 6
9. Equipment and Supplies (Including Computer Hardware and Software) 6
10. Reagents and Standards 7
11. Calibration and Standardization 8
12. Procedure 12
13. Quality Control 13
14. Data Analysis and Calculations 15
15. Data Assessment and Acceptance Criteria for Quality Control Measures 16
16. Corrective Actions for Out-of-Control Data 16
17. Contingencies for Handling Out-of-Control or Unacceptable Data 16
18. Method Performance 16
19. Method Modifications 16
20. Instrument/Equipment Maintenance 17
21. Troubleshooting 18
22. Safety 18
23. Waste Management 18
24. Pollution Prevention 18
25. References 18
26. Tables, Diagrams, Flowcharts, and Validation Data 19
27. Revisions 19

1. Purpose/Identification of Method

1.1. This Standard Operating Procedure provides a detailed description based on EPA Methods 200.8, 6020 and 6020A for analysis determining dissolved and total recoverable metals by Inductively Coupled Plasma – Mass Spectrometry (ICP-MS) in environmental samples.

2. Summary of Method

2.1. An aliquot of a well-mixed, homogeneous sample is accurately measured for sample processing. For total recoverable analysis of solids and liquids or dissolved liquid analysis requiring digestion, analytes are first solubilized by gently refluxing with nitric and hydrochloric acids. After cooling, the sample is brought to volume, mixed and allowed to settle overnight prior to analysis. For the determination of dissolved analytes in a filtered aqueous sample by 200.8, or for the "direct analysis" 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, and then mixed and allowed to set for the required time prior to analysis.

2.2. The method describes the determination of trace elements in aqueous solutions by ICP-MS. 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 separated on the basis of their mass-to-charge ratio by a quadrupole mass spectrometer. 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. Additional corrections are applied as necessary to correct for isobaric and poly atomic elemental interferences (Section 6).

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.

3.2. **Parameters**: This SOP applies to the determination of dissolved and total recoverable elements by Inductively Coupled Plasma-Mass Spectrometry (ICP-MS) in environmental samples.

3.3. This SOP is applicable to the determination of the following elements, at a minimum:

Analyte	Registry Number (CAS #)
Aluminum (Al)	7429-90-5
Antimony (Sb)	7440-36-0
Arsenic (As)	7440-38-2
Barium (Ba)	7440-39-3
Beryllium (Be)	7440-41-7
Bismuth (Bi)	7440-69-9
Boron (B)	7440-42-8
Cadmium (Cd)	7440-43-9
Calcium (Ca)	7740-70-2
Cesium (Cs)	7440-46-2
Cerium (Ce)	7440-45-1
Chromium (Cr)	7440-47-3
Cobalt (Co)	7440-48-4
Copper (Cu)	7440-50-8
Iron (Fe)	7439-86-6
Iridium (Ir)	7439-88-5
Lanthanum (La)	7439-91-1

Lead (Pb)	7439-92-1
Lithium (Li)	7439-93-2
Magnesium (Mg)	7439-95-4
Manganese (Mn)	7439-96-5
Mercury (Hg)	7439-97-6
Molybdenum (Mo)	7439-98-7
Neodymium (Nd)	7440-00-8
Nickel (Ni)	7440-02-0
Palladium (Pd)	7440-05-3
Platinum (Pt)	7440-06-4
Potassium (K)	7440-70-2
Rhenium (Re)	7440-15-5
Rhodium (Rh)	7440-16-6
Rubidium (Rb)	7440-17-7
Selenium (Se)	7782-49-2
Silicon (Si)	7740-21-3
Silver (Ag)	7440-22-4
Sodium (Na)	7740-23-5
Strontium (Sr)	7440-24-6
Thallium (Tl)	7440-28-0
Tin (Sn)	7440-31-5
Titanium (Ti)	7440-32-6
Vanadium (V)	7440-62-2
Zinc (Zn)	7440-66-6

4. Applicable Matrices

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

4.2. 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).

4.3. Where this method is approved for the determination of metal and metalloid contaminants in drinking water, samples are analyzed directly by pneumatic nebulization without acid digestion if the samples have been properly acid-preserved and have turbidity of < 1 NTU at the time of analysis, this total recoverable determination procedure is referred to as "direct analysis". Direct Analysis is only applicable to samples being analyzed by 200.8.

4.4. For the determination of total recoverable analytes in aqueous samples containing particulate and suspended solids a digestion step is required prior to analysis.

5. Limits of Detection and Quantitation

5.1. The reporting limit (LOQ) for all analytes ranges from 0.08 to 53.5 ug/L for waters and 0.08 to 50 mg/Kg for soils depending on the element for this method. All current method detection limits (MDLs) and LOQs are listed in the LIMS and are available by request from the Quality Manager.

6. Interferences

6.1. 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 instrument's 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.

6.1.1. All analytes listed in section 2.1 have at least one isotope free of isobaric elemental interference. Of the analytical isotopes recommended for use with this method (Table III), only molybdenum-98 (ruthenium) and selenium-82 (krypton) have isobaric elemental interferences. If alternative analytical isotopes having higher natural abundance are selected in order to achieve greater sensitivity, an isobaric interference may occur. All data obtained under such conditions must be corrected by measuring the signal from another isotope of the interfering element and subtracting the appropriate signal ratio from the isotope of interest. A record of this correction process should be included with the report of the data. Such corrections will only be as accurate as the accuracy of the isotope ratio used in the elemental equation for data calculations. Relevant isotope ratios should be established prior to the application of any corrections.

6.2. Abundance Sensitivity Interference – Abundance sensitivity interference refers to the degree of peak overlap that can occur between adjacent peaks. Interference can occur when the shoulder of a large peak significantly overlaps the peak of a neighboring minor peak, thereby contributing to its intensity. The potential for these interferences should be recognized and the spectrometer resolution adjusted to minimize them.

6.3. Isobaric Polyatomic Interference – 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. Examples include ArCl+ (mass 75) which interferes with As, ClO+ (mass 51) which interferes with V and must be corrected by measuring ClO+ at mass 53. These interferences are highly dependent on the matrix of the samples and day-to-day plasma conditions, so correction factors must be determined on the day of analysis. When possible, one should choose an interference-free isotope to measure.

6.4. Physical Interferences – Physical interferences result from the physical processes associated with the transport of sample to the plasma, sample behavior within the plasma, and transmission through the interface region between the plasma and the mass spectrometer. Viscosity and surface tension differences can affect results, as can deposits on the sampler and skimmer cones caused by large quantities of dissolved solids in the sample. The interferences can be compensated for by the use of internal standards that approximate the analytical behavior of the elements being determined. Additionally, it is recommended that dissolved solids in samples be kept below 0.2% (w/v).

6.5. Memory Interference – Memory interferences are related to sample transport and result when there is carryover from one sample to the next. Sample carryover can result from sample disposition on the sample and the skimmer cones and from incomplete rinsing of the sample solution from the plasma torch and the spray chamber between samples. These memory effects are dependent upon both the analyte being measured and sample matrix and can be minimized through the use of suitable rinse times.

6.5.1. The rinse times necessary for a particular analyte should be estimated prior to analysis. This may be achieved by aspirating a standard containing the analyte at a concentration ten times the LDR for the normal sample analysis period, followed by analysis of the rinse blank at designated intervals. The length of time required to reduce the analyte signal to less than ten times the method detection limit should be noted. The minimum rinse time between samples should be set to this time. Memory interferences may also be assessed within an analytical run by using three or more replicate integrations for data acquisition. If the integrated signal values drop consecutively, the analyst should check for the possibility of a memory effect. If the analyte concentration in the previous sample is high enough to suspect analyte carryover, the sample should be re-analyzed after a long rinse period.

6.6. Silver is only slightly soluble in the presence of chloride unless there is a sufficient chloride concentration to form the soluble chloride complex. Therefore, low recoveries of silver may occur in samples, fortified sample matrices and even fortified blanks if determined as a dissolved analyte or by "direct analysis" where the sample has not been processed using the total recoverable mixed acid digestion. For this reason samples are digested prior to the determination of silver. 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.

6.7. The total recoverable sample digestion procedure given in this method will solubilize and hold in solution only minimal concentrations of barium in the presence of free sulfate. For the analysis of barium in samples having varying and unknown concentrations of sulfate, analysis should be completed as soon as possible after sample preparation.

7. Sample Collection, Preservation, Shipment and Storage

7.1. Table 7.1 Collection, Preservation and Storage.

Sample type	Collection per sample	Preservation	Storage	Hold time
Aqueous	Plastic or glass, 250 mL minimum.	Acidified with nitric acid to pH<2; if received unpreserved at 1.5 mL nitric fore each liter received. Mix and confirm that the pH has been adjusted to <2 and allow the samples to sit for 24 hours prior to analysis.	<6°C if shared with mercury analysis; otherwise ambient if preserved properly with nitric acid	Must be prepped and analyzed within 180 days of collection for all other metals than mercury.
Solid	8 oz glass jar	None	<6°C, but above freezing	Must be prepped and analyzed within 180 days 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
ICPMS (Inductively Coupled Plasma Mass Spectrometer)	Cetac ASX-530 Autosampler, Niagra valve system and PC3 spray chamber chiller/valve system. Toshiba 1600 EP recirculating chiller or equivalent. Toshiba UPS system	Thermo Fisher Scientific XSeries 2 ICP-MS Agilent 7700 series ICPMS
Argon gas	High purity grade, 99.99%	Praxair or equivalent replacement
Collision Gas	High purity 7%H/93% He mix Ultra high purity He, Ultra high purity H_2	Praxair (Oxygen Services) or equivalent replacement
Autosampler tubes	15 mL metals free autosampler tubes	Mold Pro, MP100 or equivalent replacement
Rough pump oil	CMP-19	Fisher Scientific or equivalent replacement
Peristaltic pump tubing	Various sizes	Fisher Scientific or equivalent replacement
Analytical Balance	With the capability to measure to 0.1 mg	Sartorius BP 110S, A&D EK-610i, A&D FX1200, or Sartoriius LC612S-00MS or equivalent replacement
Mechanical pipettors	Capable of delivering volumes ranging from 10 to 5000 µL, and associated metal –free disposable pipet tips	Eppendorf, Fisher brand or equivalent replacement

Glassware	Class A volumetric flasks, graduated cylinders, funnels (glass and/or metal-free plastic	Fisher Scientific or equivalent replacement
Digestion cups	50 mL disposable digestion cups	Environmental Express SC475or equivalent replacement
Narrow mouth storage bottles	FEP (fluorinated ethylene propylene) with screw top closure, 135 mL to 1 L capacity	C&G Containers or equivalent replacement
Data Reporting Software		Horizon (also referenced Epic Pro)
Data Uploading Software	Pace internal software used to transfer data from the instrument to the LIMS	Limslink
Instrument software	PlasmaLab ICPMS software	Thermo Fisher

10. Reagents and Standards

10.1. Table 10.1 Reagents and Standards.

Reagent/Standard	Concentration/ Description	Requirements/ Vendor/ Item #
Hydrochloric acid (HCl)	Trace metals grade or better	Fisher Scientific, A-508-P212 or equivalent replacement
Nitric Acid (HNO_3)	Trace metals grade or better	Fisher Scientific, A-509-P212 or equivalent replacement
Deionized water (DI)	Reagent grade (DI) water – (18 MOhm resistivity)	Barnstead Epure System 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.	n/a
Calibration Stock Standard solutions	Custom blend of elements. See Attachment II for the standard preparation information	Spex Certiprep, XFSPA-221-250, XFSPA-656-250, XFSPA-220-250 or equivalent replacement
Initial Calibration Verification (ICV) Stock Standard solutions	Custom blend from of elements from a different source than the Calibration Stock Standard Solutions. See Attachment II for standard preparation information	Inorganic Ventures, XFSMN-26-250A, XFSMN-27-250A, XFSMN-28-250A; Peak Performance, Hg: 4400-1000331 or equivalent replacement
Internal Standard Stock Solution	Can be a purchased multi-element blend or single element standards to be mixed prior to use.	Peak Performance, In: S4400-1000241, Sc: S4400-100048, Y: S4400-1000671, Tb: S4400-1000571; Inorganic Ventures, Ge: CGGe1-1, Spex, Th: PL TH2-2y
Tuning Stock Solution	Purchased multi-element standard from a qualified vendor	Claritas PPT, CL-JUNG-1
Rinse Blank	2-5% (v/v) Nitric Acid solution for rinsing between runs. Prepared in 1G increments utilizing Nalgene bottles. This is measured by mixing 76 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.	n/a

10.2. Working Standard Dilutions and Concentrations

Standard	Standard(s) Used	Standard(s) Amount (mL)	Diluent	Solvent Volume (mL)	Final Total Volume (mL)	Final Concentration
Standard 1	Refer to Table 10.1, See Calibration Stock Standard Solutions	0.250	2% Nitric/1% HCl acid solution	9.75	10	5/62.5
Standard 2		0.500		9.5	10	10/125
Standard 3		1.000		9	10	20/250
Standard 4		0.1		9.9	10	200/2500
Standard 4		0.1		9.9	10	200/2500
Standard 4		0.1		9.9	10	200
Standard 5		0.05		9.95	10	500
Standard 5		0.05		9.95	10	250/500/2500
Standard 5		0.25		9.75	10	25000
CRDL	Inorganic Ventures, PACE-28	0.1		9.9	10	varied
CRDL2	Inorganic Ventures, PACE-28	0.2		9.8	10	varied
ICS-A	Inorganic Ventures 6020 ISC-OA	0.25		9.75	10	25000
ICS-AB	Refer to Table 10.1	0.25		9.65	10	100/1250/26250
ICV/CCV	Inorganic Ventures, PACE-5, PACE-4B	0.2		49.8	50	80/1000
ICV/CCV	Inorganic Ventures, PACE-5, PACE-4B	0.2		49.8	50	80/1000
Hg-Intermediate	Refer to 10.1 See Calibration Stock Standard Solutions	1.000		24.0	25	40
Standard 6		0.05		9.95	10	0.2
Standard 7		0.25		9.75	10	1.00
Standard 8		0.25		4.75	5	2.00
Standard 9		0.5		4.5	5	4.00
ICV/CCV/Hg*	Inorganic Venture, CGHG1-1	0.05		24.95	25	2

11. Calibration and Standardization

11.1. Table 11.1 Calibration and Standardization.

Calibration Metric	Parameter / Frequency	Criteria	Comments
Tune	Daily prior to any calibration	EPA performance report - print and file all reports, note failures and corrective actions taken. Adjust spectrometer resolution to produce a peak width of approximately 0.75 amu at 5% peak height. Adjust mass calibration if	The tune criteria must pass before instrument is calibrated and any samples are analyzed. Follow manufacturer guidelines for troubleshooting and maintenance for tune failures. Document all maintenance performed and return to compliance.

		it has shifted by more than 0.1 amu from unit mass. Note: The tuning criteria utilized is based on the 200.8 method. The mass criteria for 200.8 (0.75 amu at 5% peak height) is more stringent than the criteria for 6020A (0.9 amu at 10% peak height) therefore applicable for both methods.	
Calibration Curve Fit	Linear Regression	$r \geq 0.998$	Instruments are setup with the following calibration curve fit options; linear regression, linear regression through the blank, and weighted least squares. Analysis is conducted utilizing the calibration curve fit method recommended by the instrument manufacturer. If not met, remake standards and recalibrate and verify before sample analysis.
Second Source Verification Standard (ICV)	Immediately after each initial calibration	% Diff ± 10% of the true value %RSD between multiple integrations must be ≤ 5%	Review the standard preparation. Remake the standard accordingly if that is the cause. Re-inject the ICV one more time, if it fails stop all analysis. Perform all necessary instrument maintenance and recalibrate the instrument. Only two injections are allowed back to back, then the system must be recalibrated.
Initial Calibration Blank (ICB)	Immediately after the initial calibration verification	Evaluate the blank to the MDL depending on the data quality objective of the associated samples, a blank with detections less than the RL or have levels at least $1/10^{th}$ of that in the associated samples to be acceptable. Per client QAPP/Technical Specifications the blanks may require to be evaluated and clean to the MDL or ½ RL. WIDNR and West Virginia require samples to be reported to the MDL. The blanks must be clean to the data	If there is a detection, stop analysis and determine the source of the contamination. Perform any necessary maintenance and recalibrate the instrument accordingly.

		quality objectives.	
Contract Required Detection Limit Sample (CRDL) or Low Level Initial/Continuing Calibration Verification (LLICV/LLCCV)	6020/200.8 - The CRDL must be analyzed at the beginning of each run for every analyte of interest. 6020A - must be analyzed at the beginning of each run, and once at the end of each analytical batch. Per client QAPP/Technical Specifications a closing CRDL may be required every 8 hours or close of the sequence of client samples, whichever more frequent.	The CRDL/LLICV/LLCCV is at or below the RL. 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).	Evaluate standard preparation, re-prepare and analyze if suspected. Review the CRDL 2 standard for the failing elements. -If the CRDL2 passes, report only the samples that have detections greater than that concentration. -If the CRDL or CRDL2 are biased high, only samples that are non-detect below the reporting limit can be reported as there would be no impact from the high bias. -If the CRDL is biased low, no data can be reported for the target elements failing criteria including J-flagged data. The system must be stopped. Perform any necessary maintenance and recalibrate accordingly.
Interference Check Solutions (ICSA/AB)	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 C, Cl, Al, Ca, Fe, K, Mg, Mo, Na, P, S and Ti and mid-range concentrations of the remaining analyzed elements is analyzed at the beginning of each sample run sequence following the ICSA. 6020A requires the ICSA/AB be analyzed every 12 hours thereafter. Per client QAPP/Technical Specifications a closing sequence ICSA/AB may be required at a frequency of every 8 hours or end of sequence, whichever is more frequent.	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. Client QAPPs/Technical Specifications by provided alternate criteria for non-spiked elements and must be followed accordingly.	If the ICSA or AB fail criteria, stop analysis. Review the standard preparation, remake accordingly. Perform any necessary maintenance and recalibrate prior to sample analysis. Adjust the equations accordingly or recalibrate as needed to meet specified requirements. Note that monitoring the interference source does not necessarily require monitoring the interference itself, but that a molecular species may be monitored to indicate the presence of the interference Exception: If the minerals are high bias and the analytes of interest are not impacted, passing all criteria, the data may be reported.
Continuing Calibration Verification (CCV)	Prior to the analysis of any samples and after every 10 injections thereafter. Samples must	% Diff ± 10% of the true value %RSD between multiple	If the requirements for continuing calibration are not met, review for preparation error or instrument malfunction. A CCV may be

	be bracketed with a closing CCV standard.	integrations must be ≤ 5%	repeated, but a second failure requires the system to be recalibrated prior to further analysis. If the samples bracketed are non-detect and the CCV is biased high, data may be reported as there is no impact from the high bias. If the samples associated are non-detect and the only detections are associated with the batch QC (LCS/MS) but the QC is within limits, the data can be reported. The QC should be flagged indicating that there was bias but that there was no impact to the associated samples. If the CCVs are biased low, reanalyze any samples impacted since the last passing CCV.
Continuing Calibration Blank (CCB)	Following every CCV injection	Evaluate the blank to the MDL depending on the data quality objective of the associated samples, a blank with detections less than the RL or have levels at least 1/10th of that in the associated samples to be acceptable. Per client QAPP/Technical Specifications the blanks may require to be evaluated and clean to the MDL or ½ RL. WIDNR and West Virginia require samples to be reported to the MDL. The blanks must be clean to the data quality objectives.	If there is detection, stop analysis and determine the source of the contamination. Perform any necessary maintenance and recalibrate the instrument accordingly. Reanalyze any samples impacted since the last passing CCB.
Internal Standard Response	Monitor the signal intensity for the internal standard masses throughout the analytical run. This information is useful in detecting instrument drift, sensitivity shifts, and inherent internal standard (i.e., a natural constituent in a sample).	For method 6020, the absolute intensity of any one 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 absolute intensity of any one internal standard in the	If deviations greater than these are observed, flush the instrument with the rinse blank and re-analyze the calibration blank and examine the internal standard intensities with the following actions; If the intensities of the internal standards are acceptable, dilute a fresh aliquot of the sample and re-analyze. A 5X dilution or larger factor or multiple dilutions may be

		samples and remaining QC must not deviate more than 30-120% from its original intensity in the associated calibration blank. For method 6020A, the intensity of the internal standards must not fall below 70% for any standard or sample. An upper limit of 125% will be applied to the internal standard recoveries for analyses. For Method 200.8 the absolute intensity of any one internal standard in the samples and QC must not deviate more than 60-125% from its original intensity in the associated calibration blank.	required to achieve acceptable results. If the internal standards are still out-of-limits, terminate the analysis and determine the cause of the drift. Routine maintenance of the sampling interface or re-tuning the mass spectrometer may be required. The system must be calibrated and any samples not bordered by acceptable ICV/CCV samples re-analyzed.

12. Procedure

12.1. Perform all initial system set up procedures

12.1.1. Check waste jug. If more than half full, empty it into the acid waste stream.

12.1.2. Check the chiller temperature and water level. Add additional water as needed to return the instrument to specification. The system will not operate if the water is too low.

12.1.3. Check rough pump oil color. It should be amber or light yellow; if it is the color of coffee it should be changed.

12.1.4. Check pressure of collision gas cylinder, change if needed.

12.1.5. Open the torch chamber and visually inspect the cones if the instrument performance is not acceptable. If there are significant deposits, remove both cones and clean or replace them.

12.1.6. Check torch position and fittings. Adjust the position of the torch accordingly, reattach the fittings as necessary.

12.1.7. Check the peristaltic pump tubing. If it has flattened or plugged, replace it.

12.1.8. Turn plasma on by switching the instrument into the operating state. Place the probes in 2% nitric acid solution and allow the instrument to warm up for 20-30 minutes.

12.1.9. Check peristaltic pump flow by monitoring bubble movement in the pump tubing. Adjust tension as needed to achieve a smooth flow.

12.1.10. Perform daily tuning and optimization of the instrument in the following order:

12.1.10.1. Torchbox alignment, if torch chamber was opened.

12.1.10.2. EPA autotune wizard.

12.1.10.3. EPA performance report, print and file all reports. Note failures and corrective actions taken.

12.1.10.4. Detector cross calibration.

12.1.10.5. CCT-KED autotune wizard.

12.1.10.6. CCT-KED performance report, print and file all reports. Note failures and corrective actions taken.

12.1.10.7. CCT autotune wizard.

12.1.10.8. CCT performance report, print and file all reports. Note failures and corrective actions taken.

12.1.11. See Attachment I for an example of the Daily Operational Checklist Form F-MN-I-212, or equivalent replacement.

12.2. Analysis

12.2.1. General method development steps are outlined in the Training and Operations manuals from Thermo Scientific. Select masses carefully to avoid and/or minimize interferences. Make sure appropriate measures are in place to deal with interferences. Use an internal standard within 50 mass units of the analyte if possible.

12.2.2. Run analytical samples, appropriate batch and quality control samples.

12.2.3. Each sample preparation set will typically include a digestion blank, a laboratory control spike, matrix spike and sample duplicate, as defined in the Glossary Section of the PASI Quality Manual. More quality control samples may be necessary. In particular, it is suggested that the matrix spike be added in a form that tests the digestion efficiency.

12.2.4. CCV/CCB required every 10 samples using the same solution and control limits as the ICV and ICB standard and blank.

12.2.5. Review results of quality control samples for PASS/FAIL criteria.

12.2.6. Review the sample in-solution concentrations. Samples that have concentrations outside the linear range for each element will be diluted until they fall within range.

12.2.7. At the conclusion of the experiment, set the instrument to go into vacuum status. Selecting shut down will turn the vacuum off. Selecting or leaving the instrument at none will leave the plasma on.

12.3. Daily File (in hardcopy format; if electronic assembly is available the same images will be captured in pdf accordingly)

12.3.1. Gather all daily printouts.

12.3.2. Print out instrument raw data.

12.3.3. Include the calibration summaries and runlogs.

12.3.4. Update and include necessary standard preparation logbooks.

12.3.5. Label the daily folder with the date and instrument information.

13. Quality Control

13.1. Table 13.1 Quality Control and Corrective Action.

QC Sample	Components	Frequency	Acceptance Criteria	Corrective Action
Method Blank (MB)	Reagent water for water samples or Teflon chips/or nonmetal containing solid matrix for soil batches	One per 20 samples	Target analytes must be less than reporting limit. If results are reported to MDL, target analytes in MB should be non-detect. WIDNR and West Virginia require samples to be reported to the MDL. The blanks must be clean to the data quality objectives.	Re-analyze associated samples. ***Exceptions:*** If sample ND, report sample without qualification; If sample result >10x MB detects, report sample as not impacted by the blank contamination; If sample result <10x MB detects and sample cannot be reanalyzed, report sample with appropriate qualifier to indicate an estimated value. Client must be alerted and authorize this condition.

Laboratory Control Sample (LCS)	DI water spiked with all target compounds or a spiked Teflon chip/or nonmetal containing matrix for soils.	One per 20 samples	6020/6020A: 80-120% 200.8: 85-115%	The analyses should be terminated, the problem corrected, and the samples associated with that LCS re-analyzed. If reanalysis of the samples fail, the samples affected by the failing LCS elements need to be re-digested and re-analyzed. ***Exceptions:*** If LCS recovery is > QC limits and these compounds are non-detect in the associated samples, the sample data may be reported with appropriate data qualifiers.
Matrix Spike (MS)	Client sample spiked with all target compounds	One per 20 samples for 6020 and 6020A One per 10 samples for 200.8	6020/6020A : 75-125% 200.8: 70-130%	If LCS and MBs are acceptable, the MS/MSD chromatogram should be reviewed and it may be reported with appropriate footnote indicating matrix interferences. Perform a PDS on any elements that failed to meet criteria. For Minnesota Admin Contract clients – all MS/MSD failures require analysis of the MS/MSD and the original sample. If it is still out of control, investigate and document the cause in the associated narrative as well as qualifying appropriately.
MSD / Duplicate	MS Duplicate *OR (alternative)* Sample Dup	One for every 20 samples for 6020 and 6020A.	%Diff ≤ 20%	Report results with an appropriate footnote.
Serial Dilution	A 5-fold dilution of a digested sample. See section 13.1.1.	One per batch of 20 samples or less	fivefold dilution must agree within ± 10% of the original determination if analyte concentration is >50x MDL	If criteria is not met, original sample and dilution shall be reanalyzed.
Post Digestion Spike (PDS)	An aliquot of the parent sample used for the MS, prepared at the same dilution as the parent sample. Spiked with 20 uL of 20/250 mg/L stock concentration.	One per batch if there is a MS failure.	80-120%	If the element fails to meet the recovery criteria, then the dilution test on the PDS must be performed. If the dilution test fails, it is determined to be matrix interference,
Serial Dilution of Post Digestion Spike	The above PDS is diluted 1:5 and treated as a serial dilution	Required by certain client QAPPs. This is not a standard practice	fivefold dilution must agree within ± 10% of the original determination	If this fails data is qualified

13.1.1. To prepare a 5-fold dilution: take a 1 mL aliquot from the sample and add to 4 mL of 2% HNO3 / 1% HCl DI water diluents. 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.

14. Data Analysis and Calculations

14.1. Percent Recovery Equation:

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

Where:
SSR = Spike sample result, µg/L or mg/kg dry
SR = Sample result, µg/L or mg/kg dry
ST = Spike target, µg/L or mg/kg dry

14.2 Relative Percent Difference Calculation

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

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

14.3 Concentration Calculation for Soils

$$(W) \times (1 - (M/100)) = C$$

$$\frac{(K) \times (D) \times (F)}{(C \times 1000)} = X$$

Where:
W = original sample weight (g)
M = percent moisture
K = on column analyte concentration (ug/L)
D = digested volume (mL)
F = dilution factor
C = moisture corrected weight (g)

14.4 Linear Regression Equation

$$y = mx + b$$

Where:
y = instrument response
m = slope of calibration function
x = analyte concentration (ug/L)
b = blank concentration (ug/L)

14.5 The Weighted Least Squares Regression of each point (w_i) is calculated as follows:

$$w_i = S_i^{-2} / (\sum_i S_i^{-2} / n)$$

Where:
w_i = weight of each point
S_i = standard deviation of each point
n = number of point

15. Data Assessment and Acceptance Criteria for Quality Control Measures

15.1. See tables in section 11 & 13.

16. Corrective Actions for Out-Of-Control Data

16.1. See tables in section 11 % 13.

17. Contingencies for Handling Out-Of-Control or Unacceptable Data

17.1. If not specifically listed in the tables in section 11 & 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**: An MDL study must be conducted annually (per the method) per S-MN-Q-269, Method Detection Limit Studies for each matrix per instrument. For 200.8, a new MDL must be performed with each new analyst begins work as well.

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 S-ALL-Q-020, Training Procedures.

18.4. **Periodic performance evaluation (PE)** samples are analyzed to demonstrate continuing competence per SOP S-MN-Q-258, or equivalent replacement. Results are stored in the QA office.

18.5. An Instrument Detection Limit (IDL) Study is required quarterly. This consists of three runs on three consecutive days from the analysis of a reagent blank with seven consecutive measurements per day. The data will be maintained in the metals department.

19. Method Modifications

19.1. The tuning criteria utilized is based on the 200.8 method. The mass criteria for 200.8 (0.75 amu at 5% peak height) is more stringent than the criteria for 6020A (0.9 amu at 10% peak height) therefore applicable for both methods

19.2. Sample digestates are allowed to settle overnight. If undissolved material does not settle out the sample digestate is filtered prior to analysis on the instrument. The laboratory does not centrifuge samples as suggested in the method.

19.3. Aqueous samples with less than 1%-undissolved solids are brought up to a final volume of 100 mL (no preparation step is performed at the instrument).

19.4. LRB results are not subtracted from LFB results for calculation of LFB recoveries. The level of contamination in the LRB should be minimal when compared to the LFB spiking levels.

19.5. Instruments utilize dual detector scanning, analog and pulse counting. The detector cross calibration is used to convert the data acquired using analog detector mode into the equivalent pulse counting

data. During detector cross calibration, a polynomial fit line is applied by comparing masses found on the y-axis against x-axis. External calibration is completed each day by utilizing 100/1250 ug/L solution. Internal instrument calibrations are also obtained by the instrument during pauses in operation, utilizing obtained integrated counts per second across the mass range and creating a separate polynomial fit line.

19.6. Calibration

19.6.1. Linear Regression

19.6.1.1. Utilizing a standard linear regression format (See Equation 14.4). All points of the calibration curve including the blank are plotted as regular fitted points.

19.6.2. Weighted Least Squares Regression

19.6.2.1. Utilizing Weighted Least Squares Regression in Section 14.5. When data with more than one repetition is used to create a calibration curve, weighted regression can be selected. The count error for data collected in this manner is represented as the standard deviation of the counts. Usually the count error for higher concentrations is larger than that for lower concentrations. When weighted regression is selected, lower concentrations are given more weight because it is more desirable that the curve pass through points having lower error than points having higher error.

19.7. Analyte List from Section 1.2

19.7.1. The following elements are not listed in the method 6020A recommended analyte list; bismuth, boron, lithium, molybdenum, palladium, platinum, silica, silicon, strontium, tin, titanium, and uranium-238. 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.

19.7.2. 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

20. Instrument/Equipment Maintenance

20.1. Please refer to the instrument manual for maintenance procedures performed by the lab.

20.2. Routine Maintenance (Document all routine maintenance in the Routine Maintenance Logbook at the time of performing the maintenance)

20.2.1. Weekly Maintenance.

20.2.1.1. Replace peristaltic pump tubing for sample, internal standard and waste.

20.2.1.2. Clean spray chamber and nebulizer.

20.2.1.3. Clean and/or replace torch (optional or as needed).

20.2.1.4. Check and clean air filters id necessary.

20.2.1.5. Check multiplier voltages.

20.2.2. Monthly Maintenance

20.2.2.1. Check rotary pump oil.

20.2.2.2. Check oil mist filters.

20.2.2.3. Replace sample uptake tubing.

20.2.2.4. Check chiller water level in reservoir

20.2.2.5. Bi-Annual Maintenance

20.2.2.6. Examine lens system and clean, if necessary.

20.2.2.7. Examine penning gauge and clean, if necessary.

20.2.2.8. Change rotary pump oil (perform more frequently based on sample type and load).

20.2.2.9. Annual Maintenance

20.2.2.10. Replace worn o-rings.

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. Material Safety Data Sheets (MSDSs) 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 S-MN-S-003, Waste Handling, 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. National Environmental Laboratory Accreditation Conference (NELAC), Chapter 5, "Quality Systems"- most current version.

25.3. The NELAC Institute (TNI); Volume 1, Module 2, "Quality Systems"- most current version.

25.4. 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.

25.5. Fisons –VG Genesis Users Manual.

25.6. Region 9 Laboratory Standard Operating Procedure 130, Glassware Cleaning Procedures.

25.7. Region 9 Laboratory Standard Operating Procedure 462, Analysis of Total Suspended Solids By EPA Method 160.2.

25.8. U.S. Environmental Protection Agency. SW846 Method 6020, Inductively Coupled Plasma – Mass Spectrometry, Revision 0, 9/94.

25.9. U.S. Environmental Protection Agency. SW846 Method 6020A, Inductively Coupled Plasma – Mass Spectrometry, Revision 1, 92/2007.

25.10. Test Methods for Evaluating Solid Waste Physical/Chemical Methods, SW-846, Third Edition. Method 3020A.

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

26. Tables, Diagrams, Flowcharts, and Validation Data

26.1. Attachment I – ICPMS Daily Operational Checklist
26.2. Attachment II – IS Reference ICPMS.
26.3. Attachment III - Method 6020/6020A/200.8 Analyte List and PRL
26.4. Table I – Internal Standards and Limitation of Use.
26.5. Table II – Recommended Elemental Equations for Data Calculations.
26.6. Table III – Recommended Analytical Isotopes and Additional Masses.

27. Revisions

Document Number	Reason for Change	Date
S-MN-I-492-rev.21	2.1 – removed HCl for soil only reference 9.1 – removed ESI SC4 DX autosampler, replaced with Niagra valve system; vendor updated 9.1 – collision gas row – added ultra high purity… 10.1 – 10.2 – updated 11.1 – added RSD criteria to ICV/CCV 13.1 – Added Serial Dilution of PDS 19.5 – removed Added PDS criteria in section 13.1 table Attachment I-II updated to current revision Attachment III – standard prep log example removed Attachment IV (now III) – updated Removed DoD reference from section 25	30Apr2014
S-MN-I-492-rev.22	Table 10.2 – Changed Pace-9 to Pace-28 for CRDL/CRDL2; Changed standard amount, solvent volume and final concentration for ICV/CCV 18.2 – added MDL frequency with a new analyst per 200.8 19.7 – added section 1.2 analyte list deviation for 6020A and 200.8 Table 11.1 – added C and P in ICSA/ICSB Attachment I – corrected title	28Oct2014
S-MN-I-492-rev.23	11.1 and 13.1, added state regulation requirements for blanks for WIDNR and West Virginia	20Nov2014

Attachment I – ICPMS Daily Operational Checklist

Pace Analytical	Document Name: ICPMS Daily Checklist	Document Revised: 24Feb2014 Page 1 of 25
	Document No.: F-MN-I-212-rev.08	Issuing Authority: Pace Minnesota Quality Office

ICPMS Daily Operational Checklist

Date	Analyst	Sample Tube Lot #	Waste Jug	Chiller Temp (° C)	Default Corrections Equations changed?	Vacuum Pressure (torr)	Peristaltic Tubing Changed?	Manual shutdown?	Comments & Maintenance
			☐ ok		☐ yes ☐ no if yes, please provide analyte and multiplier value:_____		☐ IS ☐ Sample ☐ Waste ☐ None	☐ yes ☐ no	
			☐ ok		☐ yes ☐ no if yes, please provide analyte and multiplier value:_____		☐ IS ☐ Sample ☐ Waste ☐ None	☐ yes ☐ no	
			☐ ok		☐ yes ☐ no if yes, please provide analyte and multiplier value:_____		☐ IS ☐ Sample ☐ Waste ☐ None	☐ yes ☐ no	
			☐ ok		☐ yes ☐ no if yes, please provide analyte and multiplier value:_____		☐ IS ☐ Sample ☐ Waste ☐ None	☐ yes ☐ no	
			☐ ok		☐ yes ☐ no if yes, please provide analyte and multiplier value:_____		☐ IS ☐ Sample ☐ Waste ☐ None	☐ yes ☐ no	
			☐ ok		☐ yes ☐ no if yes, please provide analyte and multiplier value:_____		☐ IS ☐ Sample ☐ Waste ☐ None	☐ yes ☐ no	
			☐ ok		☐ yes ☐ no if yes, please provide analyte and multiplier value:_____		☐ IS ☐ Sample ☐ Waste ☐ None	☐ yes ☐ no	
			☐ ok		☐ yes ☐ no if yes, please provide analyte and multiplier value:_____		☐ IS ☐ Sample ☐ Waste ☐ None	☐ yes ☐ no	
			☐ ok		☐ yes ☐ no if yes, please provide analyte and multiplier value:_____		☐ IS ☐ Sample ☐ Waste ☐ None	☐ yes ☐ no	
			☐ ok		☐ yes ☐ no if yes, please provide analyte and multiplier value:_____		☐ IS ☐ Sample ☐ Waste ☐ None	☐ yes ☐ no	
			☐ ok		☐ yes ☐ no if yes, please provide analyte and multiplier value:_____		☐ IS ☐ Sample ☐ Waste ☐ None	☐ yes ☐ no	
			☐ ok		☐ yes ☐ no if yes, please provide analyte and multiplier value:_____		☐ IS ☐ Sample ☐ Waste ☐ None	☐ yes ☐ no	
			☐ ok		☐ yes ☐ no if yes, please provide analyte and multiplier value:_____		☐ IS ☐ Sample ☐ Waste ☐ None	☐ yes ☐ no	
			☐ ok		☐ yes ☐ no if yes, please provide analyte and multiplier value:_____		☐ IS ☐ Sample ☐ Waste ☐ None	☐ yes ☐ no	

Reviewed by:_____Date:_____

Attachment II - IS Reference ICPMS

Pace Analytical™	Document Name: IS Reference ICPMS	Document Revised: 27Jan2014 Page 1 of 1
	Document Number: F-MN-I-260-rev.01	Issuing Authority: Pace Minnesota Quality Office

Instrument: Thermo XII

IS	45 Sc-CCT	45 Sc	72 Ge	115 In	159 Tb	232 Th
	7 Li	23 Na	59 Co	88 Sr	205 Tl	238 U
	9 Be	25 Mg	60 Ni	95 Mo	208 Pb	
	10 B	27 Al	63 Cu	107 Ag	209 Bi	
		28 Si	66 Zn	111 Cd		
		39 K	75 As	118 Sn		
		43 Ca	78 Se	121 Sb		
		47 Ti		137 Ba		
		51 V				
		52 Cr				
		54 Fe				
		55 Mn				

Instrument: Agilent 7700

IS	45 Sc -H_2	45 Sc	72 Ge-H_2	72 Ge	115 In	159 Tb	232 Th
	7 Li	23 Na	78 Se	59 Co	95 Mo	205 Tl	238 U
	9 Be	24 Mg		60Ni	107 Ag	208 Pb	
	10 B	27 Al		63 Cu	111 Cd	209 Bi	
	28 Si	39 K		66 Zn	118 Sn		
		43 Ca		75 As	121 Sb		
		47 Ti		88 Sr	138 Ba		
		51 V					
		52 Cr					
		55 Mn					
		56 Fe					

Attachment III: – Method 6020/6020A/200.8 Analyte List and PRL

Analyte	CAS#	Water PRL (ug/L)	Soil PRL (mg/kg)
Aluminum	7429-90-5	10.0	10.0
Antimony	7440-36-0	0.5	0.5
Arsenic	7440-38-2	0.5	0.5
Barium	7440-39-3	0.3	0.3
Beryllium	7440-41-7	0.2	0.2
Bismuth	7440-69-9	0.5	0.5
Boron	7440-42-8	5.0	5.0
Cadmium	7440-43-9	0.08	0.08
Calcium	7740-70-2	40.0	40.0
Chromium	7440-47-3	0.5	0.5
Cobalt	7440-48-4	0.5	0.5
Copper	7440-50-8	1.0	1.0
Iron	7439-86-6	50.0	50.0
Lead	7439-92-1	0.1	0.1
Lithium	7439-93-2	0.5	0.5
Magnesium	7439-95-4	10.0	10.0
Manganese	7439-96-5	0.5	0.5
Molybdenum	7439-98-7	0.5	0.5
Nickel	7440-02-0	0.5	0.5
Palladium	7440-05-3	0.5	-
Platinum	7440-06-4	0.5	-
Potassium	7440-70-2	50.0	50.0
Selenium	7782-49-2	0.5	0.5
Silica	7631-86-9	53.5	53.5
Silicon	7740-21-3	50.0	50.0
Silver	7440-22-4	0.5	0.5
Sodium	7740-23-5	50.0	50.0
Strontium	7440-24-6	0.5	0.5
Thallium	7440-28-0	0.1	0.1
Tin	7440-31-5	0.5	0.5
Titanium	7440-32-6	1.0	1.0
Vanadium	7440-62-2	1.0	1.0
Zinc	7440-66-6	5.0	5.0
Uranium-238	7440-61-1	0.5	0.5

NOTE: Reporting Limits are current at the time of issuing this SOP. For the most current reporting limits, refer to LIMS system.

Table I - Internal Standards and Limitations of Use

Internal Standard	**Mass**	**Possible Limitation**
Lithium	6	a
Scandium	45	polyatomic ion interference
Yttrium	89	a, b
Rhodium	103	
Indium	115	isobaric interference by Sn
Terbium	159	
Holmium	165	
Lutetium	175	
Bismuth	209	

a- May be present in environmental samples
b- In some instruments Yttrium may form measurable amounts of YO^+ (105 amu) and YOH^+ (106 amu). If this is the case, care should be taken in the use of the cadmium elemental correction equation.

Internal standards recommended for use with this method are underlined.

Table II - Recommended Elemental Equations for Data Calculations

Element	Elemental Equation	Note
Al	(1.000) (^{27}C)	
Sb	(1.000)(^{123}C)	
As	1.000(^{75C})-3.1278(^{77}C)+0.815(^{82}C)	1
Ba	1.000(^{137}C)	
Be	1.000(^{9}C)	
Cd	1.000(^{111}C) -1.073 (^{108}C)+0.76398(^{106}C)	2
Cr	1.000(^{52}C)	3
Co	1.000(^{59}C)	
Cu	1.000(^{63}C)	
Pb	1.000(^{206}C)+1.000 (^{207}C)+1.000(^{208}C)	4
Mn	1.000(^{55}C)	
Mo	1.000(^{98}C) -0.146 (^{99}C)	5
Ni	1.000(^{60}C)	
Se	1.000(^{82}C)	6
Ag	1.000(^{107}C)	
Tl	1.000(^{205}C)	
Th	1.000(^{232}C)	
U	1.000(^{238}C)	
V	1.000(^{51}C) -3.1081 (^{53}C)+0.353351(^{52}C)	7
Zn	1.000(^{66}C)	
Bi	1.000(^{209}C)	
Sc	1.000(^{45}C)	
Tb	1.000(^{159}C)	
Y	1.000(^{89}C)	
Hg	1.000(^{202}C)	

C Calibration blank subtracted counts at specified mass.
1 Correction equation for As taken from EPA method 6020. Isobaric correction for ArCl.
2 Correction for MoO, Sn.
3 The background for ClOH will normally be small and can be estimated from the reagent blank.
4 Allowance for isobaric variablility of lead isotopes.
5 Isobaric elemental correction for Ru.
6 Some Ar supplies contain Kr as an impurity. Se is corrected for Kr by background subtraction.
7 Correction for chloride interference with adjustment for Cr. ClO 51/53 ratio may be determined from the reagent blank. Isobaric mass 52 must be from Cr only not ArC^{+}.

Table III - Recommended Analytical Isotopes

Element of Interest	Isotope
Aluminium	27
Antimony	121,123
Arsenic	75
Barium	135, 137
Beryllium	9
Cadmium	106, 108, 111, 114
Chromium	52, 53
Cobalt	59
Copper	65
Lead	206,207,208
Manganese	55
Mercury	202
Molybdenum	95, 97, 98
Nickel	62
Selenium	77, 82
Silver	107, 109
Thallium	203, 205
Vanadium	51
Zinc	66, 67, 68
Ruthenium	99
Palladium	105
Tin	118, 120

NOTE: Isotopes recommended for analytical determination are underlined.

ATTACHMENT A-3

ATTIC DUST LABORATORY
STANDARD OPERATING PROCEDURES

Attachment A-3
Attic Dust Laboratory SOP
Index

Laboratory	SOP Number	SOP Title	# Pages
Pace	ICP6010 rev. 01	Analysis of Paint, Dust Wipe, Air Filter, and Soil Samples for Lead and other Metals by ICAP/OES.	23
Pace	S-MN-I-604-rev.00	Preparation of Attic Dust and Filter Cartridge Samples for Analysis by ICP-MS	8
Pace	PBDUSTWIPEDIG rev. 02	Dust Wipe Digestion	12

DESCRIPTION:

Analysis of Paint, Dust Wipe, Air Filter, and Soil samples for Lead and other metals by ICAP/OES.

SUMMARY:

This SOP describes the technique for the determination of lead and other metals in solution by inductively coupled argon plasma (ICAP/OES) optical emission spectroscopy. This SOP may be used for the analysis of paints, soils/solids and dust wipes by SW-846 6010C; and air filters by NIOSH 7303.

SCOPE:

This SOP is applicable for analysis of paint chips, soils/solids, air filters, and dust wipes regulated under the American Industrial Hygiene Association (AIHA-LAP, LLC) Environmental Lead Laboratory Accreditation Program (ELLAP).

DOCUMENT CONTINUITY:

This document replaces Pace Analytical Services, Inc. SOP ICP6010, Revision 0.

SIGNATURES:

Quality Manager Craig Foxhoven **Date** 12-16-14
Craig Foxhoven

Technical Lead _____ **Date** 12/12/14
Cynthia Schultz

General Manager _____ **Date** 12-16-14
Derrick Friedrich

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1.0 PERSONNEL QUALIFICATIONS:

1.1 Personnel performing this procedure must be qualified per the requirements outlined in Chapter 1 of the IH Laboratory Quality Assurance Manual (QAM).

1.2 AIHA-LAP, LLC requires analysts performing this procedure to have analyzed and passed four independent test runs of each matrix (paint, dust wipes, air filters, and soil). Each test run must have five known samples. The samples are analyzed, and at least 75% of the samples for each matrix must pass the requirements.

2.0 SAFETY:

2.1 Personnel performing this procedure must follow general laboratory safety practices as defined in Chapter 1 of the QAM.

2.2 Safety practices include: wearing safety glasses, lab coat and gloves when handling samples and chemicals and using proper ventilation for all tasks. Review Safety Data Sheets for chemicals you are not familiar with.

3.0 DEFINITIONS:

3.1 Refer to standardized definitions as described in Chapter 4 of the QAM.

4.0 FORMS & RECORDS:

4.1 Data is electronically transferred from ICP data files to a LIMS (Promium's Element).

5.0 EQUIPMENT & SUPPLIES:

5.1 Perkin Elmer Optima 5300 DV ICP-OES (AES)

5.2 Perkin Elmer S10 Autosampler , or equivalent

5.3 Recirculating Chiller

5.4 Desktop Computer system with WinLab32

5.5 Liquid Argon, UN 1951 grade

5.6 Eppendorf pipettors (or equivalent) and disposable tips.

5.7 Various class A volumetric glassware

5.8 Filter paper – Whatman #1, or equivalent (Environmental Express Filter Mates can also be used).

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6.0 REAGENTS & STANDARDS:

6.1 Ultra Pure De-ionized water: UPDI

6.2 Nitric Acid (HNO_3), Trace metal grade or better.

6.3 Hydrochloric Acid (HCl), Trace metal grade or better.

6.4 Commercially prepared ICAP single element stock solutions at various concentrations for spiking, IEC and IFA/B standards

6.5 Cesium nitrate dry reagent ($CsNO_3$).

6.6 Commercially prepared multi-element standard mixes, from 2 different approved vendors or at least from 2 different lot numbers from the same approved vendor for calibration and second source standards.

	Source1			Source 2		
Cat#	XFSMN-26-250A	XFSMN-27-250A	XFSMN-28-250A	PA-STD-1B	PA-STD-2B	PA-STD-3B
	Pace STD-1A	Pace STD-2A	Pace Std-3A	Pace STD-1B	Pace STD-2B	Pace STD-3B
Elements	mg/L	mg/L	mg/L	mg/L	mg/L	mg/L
Ag		50			100	
Al			1000			2000
As	100			200		
B		100			200	
Ba	100			200		
Be	100			200		
Ca			1000			2000
Cd	100			200		
Co	100			200		
Cr	100			200		
Cu	100			200		
Fe			1000			2000
Li	100			200		
Mg			1000			2000
Mn	100			200		
Mo		100			200	
Na			1000			2000
Ni	100			200		
P	100			200		
Pb	100			200		
Sb		100			200	

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	Pace STD-1A	Pace STD-2A	Pace Std-3A	Pace STD-1B	Pace STD-2B	Pace STD-3B
Elements	mg/L	mg/L	mg/L	mg/L	mg/L	mg/L
Se	100			200		
Si		500			1000	
Sn		100			200	
Sr	100			200		
Ti		100			200	
Tl	100			200		
V	100			200		
Zn	100			200		
Zr		100			200	

6.7 Interference Check Standard A (IFA)—Prepare a solution containing 500 mg/L of Ca, Al, Mg; 200 mg/L of Fe; and 100 mg/L K and Na. Prepare this with the same proportion of acids as the calibration standards. Use the 10,000 mg/L single element stock solutions for Al, Ca, K, Mg, Na, and Fe.

6.8 Interference Check Standard AB (IFB) - Prepare a solution containing 500 mg/L Al, Ca, and Mg; 200 mg/L Fe; 100 mg/L Na and K; spike the remaining elements at 0.5-1.0 ppm using the mixed stock standards. Use the 10,000 mg/L single element stock solutions for Al, Ca, K, Mg, Na, and Fe.

6.9 Low-level Calibration Verification standards (LCV) – Prepare a solution containing the requested elements with a concentration at or below the in-solution reporting limit.

6.10 Lead LDR Solution – Prepare a solution significantly high enough (i.e. 50 mg/L) to encompass the range of concentrations of the samples being analyzed.

6.11 Lead Solution, 10 ppm, for spiking the Reporting Limit matrix sample (SRM – MRL spike). Prepare a 1 to 100 dilution of 1000 mg/L lead stock standard in 4% HNO3/5% HCl.

6.12 Manganese profiling solution, 1 mg/L for axial view and 10 mg/L for radial view. Prepare a 1 to 100 and a 1 to 1000 dilution of the 1000 mg/L Manganese stock standard in 4% HNO3/5% HCl.

6.13 Internal Standard solution—Prepare a solution containing 2 mg/L of Yttrium and 2500 mg/L of cesium nitrate ($CsNO_3$) in a 5% HNO_3 solution. Add 1.25 grams $CsNO_3$ (Reagent 6.5), 1 mL of 1000 mg/L Yttrium, and 5 mL HNO_3 to a container and dilute to 500 mL.

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6.14 Certified Reference Materials containing lead, such as these obtained from Inorganic Ventures: "Trace Metals – Sandy Loam 7" (part no. CRM023-050) and "Powdered Paint" (part no. CRM017-020). Reference materials from other vendors may also be used as long as they are NIST traceable.

7.0 INTERFERENCES & PROCEDURAL LIMITATIONS:

7.1 Spectral interferences can be categorized as overlap of a spectral line from another element; unresolved overlap molecular ban spectra; background contribution for continuous or recombination phenomena and background contribution from stray light from the line emission of high concentration elements. Spectral interferences are minimized through background correction and inter-element correction factors. These corrections are done through the instrument software. For further detail of spectral interferences, refer to the method reference.

7.2 Physical interferences are generally considered to be effects associated with the sample introduction process. Physical interferences can be minimized by filtering, diluting the samples, and the use of internal standards. For further detail of physical interferences, refer to the method references.

7.3 Chemical interferences are characterized by: molecular compound formation, ionization effects and solute vaporization effects. Chemical interferences can be minimized by diluting the samples. For further detail of chemical interferences, refer to the method references.

8.0 SAMPLE ACCEPTANCE & HOLDING TIMES:

8.1 Soil or solid samples, paints, air filter cassettes, and dust wipes can be stored at ambient temperature.

8.2 Sample holding time is 180 days from collection.

9.0 CALIBRATION & STANDARDIZATION

9.1 The instrument is calibrated prior to each sequence using a calibration blank and a minimum two-point calibration curve. The curve must have a correlation coefficient of 0.995 or better to pass.

Note: AIHA-LAP, LLC ELLAP policies require a minimum of a two-point plus blank calibration curve for ICP-AES analyses.

9.2 An Initial Calibration Verification (ICV) standard, prepared from a stock solution obtained from a different source than the calibration standards, must be analyzed to verify the prepared calibration standards. For SW 846-6010C and NIOSH 7303 requirements, analyze this standard daily after calibration. The results must be ± 10% of the true value. If it is not, try a new preparation of the ICV. If this does not

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correct the problem, recalibrate the instrument and reanalyze the ICV. If there is still a problem, prepare new calibration standards and recalibrate the instrument. This solution is also used as the Continuing Calibration Verification (CCV) and is analyzed prior to running samples, after every 10 samples and at the end of the sequence.

9.3 After analysis of the ICV, analyze an Initial Calibration Blank (ICB) check. The results should be less than 10% of the regulatory levels of lead in paint, dust, air, or soil. If it fails, repeat the analysis of the blank. If it's still outside the limits, prepare another calibration blank (if contamination is suspected) or reanalyze the previous set of samples that had results over the method reporting limit, using a new calibration curve. This solution is also used as the Continuing Calibration Blank (CCB) and is analyzed following each CCV.

9.4 Analyze the LCV(s) following the ICB. The recovery must be ±30% of the spiked amount. If the recovery fails, analyze another LCV sample at a higher concentration, but still at or below the in-solution method reporting limits for the different matrices. This solution is run prior to analyzing samples and at the close of the sequence.

9.5 To verify that the interfering element correction (IEC) and background correction factors in the instrument software are correcting for spectral interferences properly, analyze the interference check standards at the beginning and end of each run or twice every eight hours. For the IFA, the absolute value of the analyte results should not exceed the reporting limit of the analyte. The analyte results for IFB should be ±20% of the true value. If analytes are out of control in either of these standards, correction factors may need to be changed so that they properly correct for spectral interferences. Following each set of IFA/IFB standards, analyze an Instrument Blank (IBK).

9.6 The linearity for Lead under the ELLAP program cannot exceed the high calibration point or the proven LDR in the sequence of interest. After the calibration, prior to analyzing samples, run the standard for lead from 6.10. The acceptance criteria for this is +/- 10% of the true value. Samples can be reported to within 10% of the true concentration of this standard.

9.7 The linear dynamic range (LDR) for an analyte wavelength may be determined by calibrating the instrument and then analyzing a series of standards with concentrations above the highest calibration standard. An upper limit of the LDR may be set at a concentration where analysis of a standard at that concentration does not exceed ±10% of the true value of that standard. The LDRs should be checked semi-annually.

10.0 PROCEDURE:

10.1 Check the argon level and line pressure at the manifold.

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10.2 Turn on the recirculating chiller.

10.3 Log into the computer and open WinLab32 software, make sure you are not using the off-line version. If there has been no purge gas flowing to the spectrophotometer, the system will require a purge delay.

10.4 Fill the rinse container with 4% HNO3/5% HCl rinse solution. Make sure the drain from the autosampler empties into the waste container.

10.5 Fit the pump tubing into the peristaltic pump. Make sure the spray chamber waste flows to the waste container and the sample and Internal Standard flow into the spray chamber. Do not clamp the tubing at this time.

10.6 Click on the Plasma button to open the Plasma Control window. Check the settings and ignite the plasma by turning it ON. After the ignition sequence and after the plasma has lit, the pump will turn on. Check to make sure the tubing is in the channels correctly then apply the clamps. Check the flow and adjust as needed.

10.7 While the instrument is warming up, create a sequence in Element (LIMS). Make sure that ICV, ICB, LCVs, IFA, IFB, IBL, CCVs, and CCBs are included and at the correct frequency in the sequence (i.e., CCV and CCB after every ten samples). Also include the LCVs, IFA, IFB and IBK, CCV, CCB at the very end.

10.8 To build a new sequence in WinLab32, open a new Sample Information File (SIF). Enter the correct standard and sample IDs and autosampler locations. The Cal Blank and Standard IDs do not need to be entered since they are built into the method and will be added in the Automated Analysis window. Save as a new SIF using the YYMMDD format.

10.9 Open the Automated Analysis window. In the Set Up tab, enter the correct method ID (typically the most recent). Open the correct SIF to be analyzed and create a new Data Set to save the data in (Use the same ID as the SIF). Open the analyze tab and check the sequence.

10.10 Open the Spectra window and Results window. Under the Tools menu, open the Spectrophotometer Control. Place the sample and IS line in the 1 ppm Mn solution and align the axial view. Next, put both lines in the 10 ppm Mn solution and align the radial view. Flush the lines with rinse upon completion. Close the Spectrophotometer Control.

10.11 Place the sample probe back into the autosampler arm and place the IS line into the IS container. Make sure there is enough IS to complete the sequence before starting.

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10.12 Fill the autosampler racks with standards and samples according to the sequence, beginning with the calibration standards. Paint, soil, and dust wipe samples should be filtered prior to analysis to avoid plugging the nebulizer.

10.13 Highlight the Automated Analysis window again. Make sure the sample probe is in rinse and the IS probe is in the IS container. Click on the Analyze All button to run all defined standards and samples on the list.

10.14 If a sample's concentration of a requested analyte is greater than the highest calibration standard, greater than 90% of the proven LDR, or there's a matrix interference, determine the dilution needed and dilute with blank solution (4% HNO_3/ 5%HCl). Add the diluted sample to the WinLab32 SIF and Element sequence as a re-shot (RE).

10.15 The IS percent recovery requirements are 70-130%. For any sample where the IS recovery is outside of this range, dilute the sample and re-analyze. This process must be repeated until the IS recovery is within acceptable limits.

10.16 After completion of the sequence, flush the lines with DI for several minutes. Open the Plasma Control window and switch off the plasma. Unclamp and loosen the pump tubing from the peristaltic pump and cap opened standards.

10.17 Check the results of the analysis of the interference check standards (IFA and IFB) at the beginning and end of the run. Refer to Step 9.5 for acceptance limits. If any analytes are out of control and IEC factors in the instrument software need to be changed, refer to Appendix A.

11.0 CALCULATIONS:

11.1 WinLab32 reports CRM and soil samples in mg/L. Element will perform the calculation to convert to mg/kg. The calculation for CRM and soil samples into mg/kg, is as follows:

$$\frac{C \times V \times D}{W} = \text{mg/kg of lead}$$

Where C = Concentration, in mg/L, from the standard curve
V = Final volume, in mLs
D = Dilution factor (if any)
W = Weight of sample or Certified Reference Material, in grams

11.2 WinLab32 reports paint samples in mg/L. Element will perform the calculation to convert to % weight for paint samples. The calculation is as follows:

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$$\frac{C \times V \times D}{W} \times 0.0001 = \%\ Weight\ of\ Lead$$

Where C = Concentration, in mg/L, from the standard curve
V = Final volume, in mLs
D = Dilution factor (if any)
W = Weight of sample in grams
0.0001 = Factor to convert units to % weight

11.3 WinLab32 reports dust wipe samples in mg/L. Element will perform the calculation to convert to µg of lead for blank wipe samples or samples where the area of the surface sampled is not known. The calculation is as follows:

$$C \times V \times D = \mu g\ of\ lead$$

Where C = Concentration, in mg/L, from the standard curve
V = Final volume, in mLs
D = Dilution factor (if any)

11.4 If the area of the surface that was sampled for a dust wipe is known, Element will perform the calculation to convert to µg/ft². The calculation is as follows:

$$\frac{C \times V \times D}{A} = \mu g/ft^2\ of\ lead$$

Where C = Concentration, in mg/L, from the standard curve
V = Final volume, in mLs
D = Dilution factor (if any)
A = Area, in square feet (144 in.² = 1 ft²)

11.5 For composited dust wipe samples, the sub-sampled wipes are analyzed separately and the results for each sub-sample are added together to get one final result for the sample.

11.6 WinLab32 reports air filter samples in mg/L. Element will perform the calculation to convert to µg of lead for blank filter samples or samples where the volume of air sampled is not known. The calculation is as follows:

$$C \times V \times D = \mu g\ of\ lead$$

Where C = Concentration, in mg/L, from the standard curve
V = Final volume, in mLs
D = Dilution factor (if any)

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11.7 For air filter samples where the volume of air that was sampled is known, Element will perform the calculation to convert to µg/m³. The calculation is as follows:

$$\frac{C \times V \times D}{A} = \mu g/m^3 \text{ of lead}$$

Where C = Concentration, in mg/L, from the standard curve
V = Final volume, in mLs
D = Dilution factor (if any)
A = Volume of air sampled, in m³ (1000 liters = 1 m³)

11.8 Accuracy:

$$\% \text{ Spike Recovery} = \frac{SSR - USR}{SA} \times 100$$

SSR = Spiked Sample Result
USR = Unspiked Sample Result
SA = Spike Added

11.9 Precision: Relative Percent Difference (RPD)

$$RPD = \frac{(\text{Sample A} - \text{Sample B})}{(A+B)/2} \times 100$$

Where A = sample result or matrix spike result
B = sample duplicate result or matrix spike duplicate result

12.0 DATA REDUCTION & RECORDS:

12.1 The data packet represents a single sequence and will include: a copy of the Element preparation bench sheets, a copy of the Element analysis sequence, a print-out of the raw data from WinLab32, a copy of the Element Data Review Report, and the QC Checklist sheet.

12.2 Reagents and standards used for each analysis must be kept current with traceable documentation in Element.

12.3 Data is submitted to a technical peer for review/verification. This verification is documented in Element.

13.0 REPORTING:

13.1 Refer to Appendix B.

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14.0 QUALITY CONTROL:

14.1 Analyze a Reporting Limit matrix sample (SRM – MRL spike) for each matrix that is run each day. The SRM sample is prepared by spiking the appropriate amount of standard to a blank matrix sample (paint, wipe, filter, or sand) to obtain an analyte concentration at or below the method reporting limit in the particular matrix. The SRM – MRL sample is prepared as a QC sample in the prep batch and digested at the same time as the batch and other QC samples. The result should be ± 20% of the true value. Additional SRM samples with alternate concentrations may also be prepared. The method reporting limit of the samples would then be adjusted to the higher concentration.

14.2 Digest and analyze a reagent blank (BLK1) and a matrix blank (BLK2) with each batch of up to 20 samples. The reagent blank shall consist of only the reagents used in the digestion. The matrix blank shall consist of blank matrix (paint, sand, filter, or wipe) with the reagents. Both blanks will be digested the same as the samples. These blanks shall be a check for any contamination from the reagents used, the digestion containers, the matrix, or the digestion process. Blanks shall not be used to correct sample results. The blank results must be less than 10% of the regulatory level of lead in soil, paint, or dust wipes. If not, re-analyze the blanks, if the results are still out of control, re-digest the samples if possible, or footnote the results, make note on the QC checklist and report the blank concentration. If the sample concentrations are non-detect or the in-solution sample concentration is greater than 10X the blank concentration, make note of this on the QC checklist and no further action is required.

14.3 Digest and analyze a blank spike/blank spike duplicate (BS/BSD) with each batch of up to 20 samples. For paints and dust wipes, weigh out and record an amount of Certified Reference Material (CRM) containing lead (Reagent 6.14) for each BS/BSD set. For the dust wipe BS/BSD, add the CRM to a blank wipe. For soil samples, analyze a BS/BSD set and also a Certified Reference Material (SRM) sample. The soil BS/BSD consist of the acid reagents spiked with a known amount of analyte. For air filter samples, analyze BS/BSD of a known amount of analyte spiked on blank filters. Recovery must be ±20% of the true value, or within established control limits, and the RPD between the BS/BSD concentrations must be <20 RPD, or within established control limits. If the results are out of control, re-analyze the BS/BSD, if the results are still out of control, re-digest the samples if possible, or footnote the results and note this on the QC checklist.

14.4 For paint and soil samples, digest and analyze a matrix spike/ matrix spike duplicate (MS/MSD) with each batch of up to 20 samples. Choose a sample to spike and weigh out three similar weight portions of it. Spike a known amount of stock solution into two of the portions and label as "MS" and "MSD". Digest them the same as the other samples. Default acceptance limits are ± 25% for the spike recovery and <20 RPD between the duplicate spike sample concentrations. If an MS or MSD recovery

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or the RPD is out of control footnote the unspiked source sample. If an analyte's concentration in the unspiked source sample is greater than four times the level spiked, the MS/MSD recoveries for that analyte are not reported.

14.5 If out-of-control data must be reported, footnote the data in Element and document the information on the QC Checklist sheet in the data packet.

14.6 Once every three months, analyze dust wipe samples taken in the laboratory to check for possible lead contamination in areas where samples are prepared and analyzed. If the concentration in any of the samples exceeds the detection limit in $\mu g/ft^2$, the area should be thoroughly cleaned and re-sampled.

15.0 METHOD PERFORMANCE:

15.1 This procedure has been validated as fit for its intended use. See Appendix C for example method performance results.

15.2 Refer to Appendix D for factors that may contribute to measurement of uncertainty in this procedure.

16.0 DETECTION LIMITS:

16.1 Approximate nominal Method Detection Limits (MDL) and Reporting Limits (RL) for Lead are shown below.

	MDL	RL	
Paints	0.00087	0.005	%weight
Dust wipes	0.45	5.0	micrograms
Soils	1.6	10.0	mg/kg
Air filters	0.047	1.0	micrograms

17.0 REFERENCES:

17.1 PRIMARY REFERENCES:

17.1.1 Test Methods for Evaluating Solid Waste, SW 846, Revision 3, February 2007, Method 6010C.

17.1.2 NIOSH Manual of Analytical Methods, 4th Edition, 3/15/2003, Method 7303.

17.1.3 AIHA-LAP, LLC Policy Module 2C, Effective May 13, 2014

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18.0 WASTE MANAGEMENT & POLLUTION PREVENTION:

18.1 Please refer to the general policies and procedures outlined in Chapter 7 of the QAM regarding waste management and pollution prevention.

18.2 Digested samples are very acidic. They must be neutralized before going into the sewer system.

19.0 REVISIONS:

19.1 Major changes to this document are summarized in the table below.

Document ID	Changes made	Effective Date
ICP6010, Rev. 1	Addition of Revisions section 19.0	12/11/14
	Varian ICP details replaced with Perkin Elmer ICP details throughout the SOP	12/11/14
	The preparation of standards has been revised to reflect different stock sources	12/11/14
	Added the Lead high calibration (HCV/LDR) standard analysis with acceptance criteria	12/11/14

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Appendix A
Correcting IEC Factors

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Procedure to Correct Interfering Element Correction (IEC) Factors

1. From the analysis of the IFA determine how much correction is needed. For example, in IFA, Pb should be 0 mg/L and in IFB, it should be 1.0 mg/L. If analysis of IFA gave a Pb result of –0.25 mg/L, it is being over corrected and needs to be adjusted accordingly.

2. Determine which interfering element (Al, Ca, Fe, or Mg) is interfering with the analyte and note the concentration of that interfering element. (Single-element solutions of Al and Fe may be run to help determine the interference effects of Al and Fe individually). Some of the analytes that occasionally need IEC factors changed are shown below:

Analyte	Interfering element(s)
As	Al, Fe
B	Fe
Be	Fe
Pb	Fe, Al
Sb	Fe
Se	Al, Fe
Sr	Fe

3. To determine how much to change the IEC factor, take the result determined in Step 1 (make sure to include the positive or negative sign), and divide it by the concentration of the interfering element. For example: if analyte Pb was –0.25 mg/L and interfering element Al was 505 mg/L, then –0.25 / 505 = –0.0005.

4. Open the IEC model associated with the method being used. Go to the factors tab and adjust the IEC factor. Save the IEC model and click to update the method and then save the method with the updated IEC table.

5. Open the Data Reprocess window and highlight the data set of interest. Highlight the data through the first IFA/IFB and reprocess. Check the results for the IFA/IFB to endure the proper correction is being applied and readjust as needed. When the right corrections have been achieved, save the reprocessed data into a new data set, set the data to print and reprocess the data.

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Appendix B
Electronic Data Transfer Instructions

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Procedure for Electronic Data Transfer into Element

1. Check the Element sequence to make sure it includes all samples and QC that were run. Change the "*Sequence matrix*" to the matrix of the data to be transferred. The sequence matrix may need to be changed and re-saved depending on the number of matrices in the sequence being transferred.

2. Open the Data Manager window in the WinLab32 software in the Utilities option under the File drop-down menu.

3. Highlight the Data Set to be transferred and click the Export button. Select Export Design – Check to use an existing template and then click on the browse button to select the design to use. The current export design is Element Test. At this point, you can click on Finish and then click Export Data Set. If different options are needed, click on Next.

4. Select Samples to Export – be default, all samples in the data set are highlighted to export. This can be edited to export only the samples needed. Click on Next.

5. Select Analytes to Export – by default, all analytes ran under the method are highlighted. This can be edited to select only the needed elements. Click on Next

6. Export Options – This is where the export file gets named. The default is to name it the same as the Data Set, this can be changed if needed. The remaining settings need to be left as is for the file to export into Data Tool properly. Click on finish at this point if it wasn't done at step 4.Click on Export Data Set and close out the window.

7. Go back into Element and click on the Laboratory drop-down menu. Select the Data Entry/Review option and highlight the sequence being exported. Make sure all elements being exported are highlighted and click on Create in the Data Entry tab. Click on Export and save the transfer file using the ICP file. Select Yes to save over the existing file.

8. Click on Data Tool. On the right side of the Data Tool window check to make sure the file type is PE WinLab.prn. If you do not see the exported data sets on the bottom of the left side, check to make sure that the path to the exported files is correct. Double click on the export file corresponding to the sequence being transferred. The File Name and Sample/Info for the data will show up on the right side for all of the samples that were exported. Click on the Auto Select button and all IDs that match the LIMs sequence will move to the bottom to be merged. Check to make sure that all needed samples and standards have been selected, then click done. Samples can be manually included or removed by highlighting and clicking on the Include or Remove option.

9. Check to make sure the needed cross table is selected, the file IDs are right then click on Merge Files. This will bring you to the Data Transfer screen. Make sure the desired wavelength to report for each analyte is designated in the "*ELMAnalyte*" column in the analyte cross table if needed, there should be only one wavelength used for each analyte. Close the Analyte Cross Table window ("X") and click "*Save*". Highlight the "*ICP.xls*" file and click "*Save*". Answer "*Yes*" to "*Do you want to replace it?*".

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10. The Data Entry/Review Table will open up. Click "*Save*", then "*OK*", and then "*Query*". Click on "*Edit*" and scroll across to review data, making sure dilution factors (Diln), detection limits (IMDL/IMRL), control limits (LCL/UCL), and analyst's initials are correct. (While in the edit mode, corrections can be made in columns where the column heading is in blue). When everything is correct, click "*Save*" and then "*OK*".

11. Lock the data and update the status to "*Analyzed*". Print out a copy of the Data Review Report and QC Checklist to be included in the data packet. To transfer data of other matrices, change the "*Sequence matrix*" in the Element sequence to the desired matrix, and repeat steps 7 through 10.

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Appendix C
Method Performance

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Results of Environmental Lead Proficiency Analytical Testing (ELPAT) program, Round 088:

Contaminant	Units	#	Reported Result	Reference Value	Lower Limit	Upper Limit	Z-Score	Rating
Paint Chips	%	1	0.0426	0.433	.0324	0.542	-0.2	A
	%	2	0.6405	.6108	.4952	.7263	0.8	A
	%	3	3.1957	3.0732	2.5809	3.5656	0.7	A
	%	4	2.0849	2.0228	1.6069	2.4386	0.4	A
Soil	mg/kg	1	69.9	71.1	52.3	89.9	-0.2	A
	mg/kg	2	323.2	337.5	281.5	393.4	-0.8	A
	mg/kg	3	175.9	176.2	147.4	205.0	0.0	A
	mg/kg	4	240.2	248.4	207.8	289.1	-0.06	A
Dust Wipe	ug	1	309.9	301.0	232.5	371.6	0.3	A
	ug	2	148.8	152.8	115.2	190.4	-0.3	A
	ug	3	223.4	202.2	151.9	252.5	1.3	A
	ug	4	89.7	84.5	62.1	107.0	0.7	A

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Appendix D
Measurement of Uncertainty Table

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Measurement of Uncertainty Table

Measuring Instruments:	Representative and Applicable QC Data	Comments to Clarify Contributor Effects
Transportation/Storage/Handling:		
shipping time, container and temperature	NA	Usually no impact if recommended shipping conditions and holding times in referenced methods are maintained
lab storage time, conditions and temperature	NA	Usually no impact if recommended storage conditions and holding times in reference method are maintained. Unpreserved water samples and soils are stored at refrigeration temperature. Preserved water samples and filters are stored at ambient temperature.
contamination in lab storage areas	NA	Usually no impact if recommended storege conditions and holding times in reference methods are maintained.
Laboratory Subsampling:		
sample nonhomogeneity	MS/MSD	Sample composition, such as small stones in soils and paint samples may include non-paint material. Attempt to obtain subsemples that are representative of the entire sample.
blending techniques	MS/MSD	Attempt to stir soils and to crush and blend paint samples.
sample size	MS/MSD	Large enough to allow adequate subsampling. Normal subsampling sizes are about 0.5 grams for soils, 0.2 – 0.25 grams for paints. For wipes and filters, the entire sample is used.
Sample Preparation:		
Digestion tubes	BS/BSD	Accuracy of volume markings.
dispensing device	BS/BSD	Pipettors, and other types of dispensers that are not Class A.
balance	MS/MSD	Balance error is often insignificant compared to other MU sources.
temperature	BS/BSD	Hot Block or microwave oven temperature variations.
sample extraction	BS/BSD	Applies to BS/BSD if it goes through the entire sample preparation process.
extractant background	BS/BSD, MB	Presence of analyte or interferant in acids, solvents, etc.
Lab Environmental Conditions:		
temperature variance	NA	Usually no impact.
humidity variance	NA	Usually no impact.
Analysts:		
different analysts	BS/BSD	BS/BSD results reflect variability due to different analysts, as applicable, on different days
analyst training level and experience	BS/BSD	BS/BSD results reflect variability due to different analysts, as applicable, on different days
data interpretation by analyst	BS/BSD	BS/BSD results reflect variability due to different analysts, as applicable, on different days
Measuring Instruments:		
instrument stability	BS/BSD	Baseline drift, repeatability of averaged readings, lab environmental stability, etc.
carryover effects	BS/BSD, MB	Impact of high samples on following sample readings; can be monitored by proper use of CCBs.
day to day calibration differences	BS/BSD	Variation in instrument response and calibration process
interferences	MS/MSD	Due to matrix, inter-element effects.
Calibration Standards/Reference Materials:		
preparation variances	BS/BSD	Due to analysts, dispensing devices used, etc
calibration stock material uncertainty	CERTIFICATE	Obtain from certificate or estimate. Can be ignored if less than 1/3 of the largest contributor.
LCS stock material uncertainty	CERTIFICATE	Only has impact when LCS data are used to correct customer sample results. Obtain from certificate or estimate. Can be ignored if less than 1/3 of the largest contributor. Note that use of an LCS with a large uncertainty can result in over estimation of overall analytical uncertainty.
Data Manipulation:		
sampling media/blank correction	NA	Blank correction is not done.
instrument blank correction	NA	Blank correction is not done.
accuracy of calculations	BS/BSD, MS/MSD	Most calculations are done by LIMS.
area or air volume sampled	NA	Determined by hand calculation. The correct area or volume must be entered into LIMS.

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Where:

BS/BSD = Laboratory Control samples, matrix matched and typically taken through the entire analytical process with each sample batch
MS/MSD = Matrix Spike/Matrix Spike Duplicate samples, resulting from subsampling and taken through the entire analytical process with each sample batch
MB = Method or Matrix Blank, typically taken through the entire analytical process with each sample batch
NA = Not Applicable
CERTIFICATE = certificate of analysis

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STANDARD OPERATING PROCEDURE

PREPARATION OF ATTIC DUST AND FILTER CARTRIDGE SAMPLES FOR ANALYSIS BY ICP-MS

Reference Methods: EPA 3050B

Local SOP Number:	S-MN-I-604-rev.00
Effective Date:	Date of Final Signature
Supersedes:	N/A

APPROVALS

Laboratory General Manager — Date: 03Nov2015

Laboratory Quality Manager — Date: 03Nov2015

PERIODIC REVIEW

SIGNATURES BELOW INDICATE NO CHANGES HAVE BEEN MADE SINCE PREVIOUS APPROVAL.

Signature	Title	Date

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TABLE OF CONTENTS

SECTION PAGE

1. Purpose/Identification of Method 3
2. Summary of Method 3
3. Scope and Application 3
4. Applicable Matrices 3
5. Limits of Detection and Quantitation 3
6. Interferences 3
7. Sample Collection, Preservation, Shipment and Storage 3
8. Definitions 3
9. Equipment and Supplies (Including Computer Hardware and Software) 3
10. Reagents and Standards 4
11. Calibration and Standardization 5
12. Procedure 5
13. Quality Control 6
14. Data Analysis and Calculations 6
15. Data Assessment and Acceptance Criteria for Quality Control Measures 6
16. Corrective Actions for Out-of-Control Data 7
17. Contingencies for Handling Out-of-Control or Unacceptable Data 7
18. Method Performance 7
19. Method Modifications 7
20. Instrument/Equipment Maintenance 7
21. Troubleshooting 7
22. Safety 7
23. Waste Management 8
24. Pollution Prevention 8
25. References 8
26. Tables, Diagrams, Flowcharts, and Validation Data 8
27. Revisions 8

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 50mL.

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. **Parameters**: 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 0.01g	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 XFSPA-656-250, XFSPA-221-250 and XFSPA-220-250. See 10.1.2.	Purchased from Spex CertiPrep (or equivalent). Store at room temperature. Expires as specified by manufacturer.

ICPMS Stock Standards Table

XFSPA-656-250		XFSPA-221-250	
Element	(µg/L)	Element	(µg/L)
Ca	250	Si	250
Fe	250	Sb	20
Mg	250	As	20
K	250	Sn	20
Na	250		
Se	20		
Al	20		
Ba	20		
Be	20		
Bi	20		
B	20		
Cd	20		
Ce	20		
Cs	20		
Cr	20		
Co	20		
Cu	20		
La	20		
Li	20		
Mo	20		
Mn	20		
Pb	20		
Ni	20		
Ag	20		
Sr	20		
Tl	20		
V	20		
Zn	20		
U	20		

10.2. Table 10.2 - Working Standard Dilutions and Concentrations

Standard	Standard(s) Amount	Solvent	Solvent Volume	Final Total Volume	Final Concentration
Working LCS/MS Spike Solution. See 10.2.1	50 mL of each standard solution (PA-STD-1B, PA-STD-2B, PA-STD-3B)	DI water	50 mL	200 mL	Varies

10.2.1. Store at room temperature. Expires in 3 months.

11. Calibration and Standardization

11.1. Calibrate variable and fixed volume pipettes as specified in SOP S-MN-Q-264 – Support Equipment (or equivalent replacement). Calibration records are kept in the QA Office.

11.2. Calibrate the thermometer as specified in SOP S-MN-Q-264 – 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 grams. 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.

12.1.1.1.1. Create a method blank, laboratory control sample (LCS), and laboratory control sample duplicate (LCSD) by weighing out 1 gram of resin beads for each. Spike the LCS and LCSD. If sample volume permits, preparation of a matrix spike (MS) and matrix spike duplicate (MSD) will be employed.

12.1.2. Add 10mL of DI water to each sample.

12.1.3. Add 7.5mL of concentrated HNO3, 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 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: record initial Hot Black temperature in the digestion log.

12.1.4. 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.

12.1.4.1. 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.

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

12.1.5. Add 5mL 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 ICPMS.

12.1.6.1. Note: The method modifications that have been utilized have been defined 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: 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 and LCS 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 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.

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 grams of resin beads.	Prepared with each batch	See appropriate analysis SOP.	See appropriate analysis SOP.
Laboratory Control Sample (LCS)	For solids, weigh 1.0 gram of resin beads. Spike with appropriate spiking solutions.	Prepared with each batch	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**: An MDL study must be conducted annually (per the method) per S-MN-Q-269 – Determination of Limit of Detection and Limit of Quantitation (or equivalent replacement) for each matrix per instrument.

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 S-ALL-Q-020 - 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. Material Safety Data Sheets (MSDSs) 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 S-MN-S-003 - 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. National Environmental Laboratory Accreditation Conference (NELAC), Chapter 5, "Quality Systems"- most current version.

25.3. The NELAC Institute (TNI); Volume 1, Module 2, "Quality Systems"- most current version.

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

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.00	Original Document	26Oct2015

DESCRIPTION:

Dust Wipe Digestion

SUMMARY:

This SOP describes the procedure for the digestion of Environmental Express brand "Ghost Wipes" for the determination of Lead and other metals that may be requested. This SOP also includes additional steps that may need to be used for digesting wipes that are not "Ghost Wipes" and do not readily dissolve in the acids. The Ghost Wipes are heated in nitric and hydrochloric acids to dissolve the wipe and all lead compounds. Additional steps, if necessary, would involve the addition of hydrogen peroxide. The digested samples are then analyzed by inductively coupled argon plasma spectroscopy (ICAP). This procedure follows a method provided by Environmental Express.

SCOPE:

This SOP is applicable for dust samples collected on Environmental Express "Ghost Wipes" and other types of wipes. The SOP follows the quality requirements of the AIHA Environmental Lead program.

DOCUMENT CONTINUITY:

This document replaces Braun Intertec SOP PBDUSTWIPEDIG, Revision #1.

SIGNATURES:

Quality Manager Craig Foxhoven **Date** 10-23-14
Craig Foxhoven

Technical Lead _____ **Date** 10/22/14
Cynthia Schultz

General Manager _____ **Date** 10-23-14
Derrick Friedrich

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1.0 PERSONNEL QUALIFICATIONS:

1.1 Personnel performing this procedure must be qualified per the requirements outlined in chapter 1 of the Pace Analytical Services, Inc. Industrial Hygiene Laboratory Quality Assurance Manual (QAM).

1.2 AIHA requires analysts performing this procedure to have prepared and passed four independent test runs of the procedure. Each test run must have five known samples. The samples are analyzed and at least 75% of the samples must have recoveries of 90 – 110% of the assigned value.

2.0 SAFETY:

2.1 Personnel performing this procedure must follow general laboratory safety practices as defined in chapter 1 of the QAM.

2.2 Safety practices include wearing safety glasses, lab coat, and gloves when handling chemicals; and performing the digestion in a fume hood. Always add acid to water, not water to acid. Review Safety Data Sheets (SDSs) for chemicals you are not familiar with.

3.0 DEFINITIONS:

3.1 Refer to standardized Pace Analytical Services, Inc. definitions as described in chapter 4 of the QAM.

3.2 LIMS: Laboratory Information Management System. The laboratory is currently using Promium Element 6.

4.0 FORMS & RECORDS:

4.1 Element (LIMS) generated benchsheet and reporting limit sample record. (See Appendix A for examples.)

5.0 EQUIPMENT & SUPPLIES:

5.1 Environmental Express "Hot Block" digestion system.

5.2 Unused "Ghost Wipes" from Environmental Express (part no. SC4210 or SC4250).

5.3 Analytical balance, capable of weighing to 0.0001 g.

5.4 Plastic 50 mL graduated digestion tubes with screw caps (Environmental Express part no. SC475 or equivalent).

5.5 Nitrile gloves

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5.6 Eppendorf pipettor and tips.

5.7 Graduated cylinder (10, 25, or 50 mL).

5.8 Plastic ribbed 44 mm watch glass covers (Environmental Express part no. SC505 or equivalent).

6.0 REAGENTS & STANDARDS:

6.1 Nitric Acid (HNO_3) concentrated, reagent grade or better.

6.2 Hydrochloric Acid (HCl) concentrated, reagent grade or better.

6.3 Hydrogen peroxide (H_2O_2) 30%, reagent grade.

6.4 Ultra pure deionized water: UPDI.

6.5 Lead stock standard, NIST traceable, 1000 µg/mL, ICP grade.

6.6 Lead Spike Solution, 10 ppm, for spiking the reporting limit sample. Prepare a 1 to 100 dilution of the lead stock standard (6.6).

6.7 Certified Reference Material containing lead, obtained from Inorganic Ventures (catalog no. CRM023-050). Reference material from other vendors may also be used as long as it is NIST traceable.

7.0 INTERFERENCES & PROCEDURAL LIMITATIONS:

7.1 Interferences are discussed in the analytical SOP.

8.0 SAMPLE ACCEPTANCE & HOLDING TIMES:

8.1 The wipe material used for dust wipe samples must meet the specifications given in ASTM E1792, "Standard Specification for Wipe Sampling Materials for Lead in Surface Dust", or meet specifications issued by EPA. Environmental Express "Ghost Wipes" meet these specifications. For any wipe samples where it cannot be verified that the wipe material meets these specifications, the data must be qualified with a footnote stating this.

8.2 Dust wipe samples are not preserved and may be stored at room temperature in plastic or glass containers.

9.0 CALIBRATION & STANDARDIZATION:

9.1 Calibration and standardization is discussed in the analytical SOP.

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10.0 PROCEDURE:

10.1 Create the batch of samples in Element (LIMS).

10.1.1 Obtain the Chain of Custody, or other paperwork received with the samples, to determine if the sample areas have been recorded. If the sample areas are available, when creating the batch choose "Surface" as the Batch Matrix in Element. If there are no sample areas, choose "Wipe" as the Batch Matrix. This is to enable Element to designate the correct final result units.

10.1.2 The batch should have no more than 20 samples. It should include two Method Blanks (one for the reagent blank and one for the wipe matrix blank), a Blank Spike (BS), and a Blank Spike Duplicate (BSD). If there are samples with composited wipes, see 10.1.3, otherwise go to 10.1.4.

10.1.3 For samples with composited wipes, the wipes in these samples must be separated into sub-samples with a single wipe in each sub-sample. Note on the benchsheet when a sample consists of sub-samples. Also, when a batch contains sub-samples, the total number of wipes to be digested cannot exceed the 20 sample limit for the batch.

10.1.4 For samples where the area of surface that was sampled is known, enter these areas (in square feet) into the "Initial" column of the benchsheet. Print the benchsheet(s) and label the digestion tubes. In the Laboratory Standards section of Element create a standard ID for the reporting limit sample. Print the standard ID report for this sample and attach to the benchsheet.

10.2 If the wipes were received in containers other than the 50 mL plastic digestion tubes, remove the wipes from the containers (while wearing nitrile gloves) and place them into labeled 50 mL plastic digestion tubes. (If a wipe was received in a plastic digestion tube, it may be digested in the same tube). The Method Blanks, Blank Spike (BS), Blank Spike Duplicate (BSD), and the reporting limit samples are also started at this time. Refer to section 14.0 for instructions on making these quality control samples.

10.3 Add 10-15 mLs of UPDI water to all of the tubes.

10.4 To each tube add 2.0 mLs of concentrated HNO_3 and 2.5 mLs of concentrated HCl. Cover each with a plastic 44 mm watch glass cover.

10.5 Place the samples in the Hot Block at 90- 95°C and heat for 45 minutes. Ghost Wipes normally will completely dissolve into solution during this time. (If a sample does not dissolve into solution, it is probably not a Ghost Wipe and will require additional steps).

10.6 For samples that are Ghost Wipes, remove them from the Hot Block for cooling and proceed to Step 10.11. For wipe samples that are not Ghost Wipes, additional heating is

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necessary to help break down the wipe material. For these samples proceed to Steps 10.7 – 10.10.

10.7 *Additional steps for non-Ghost Wipe samples:* Add an additional 5 mLs of UPDI water to help keep the samples submerged in liquid. Continue heating for another 30 - 45 minutes and then remove them from the Hot Block.

10.8 Allow the samples to cool for a few minutes and then add 1.0 mL of 30% hydrogen peroxide.

10.9 Place samples back in the Hot Block and heat until effervescence subsides (approximately 20 minutes). Remove from Hot Block.

10.10 Repeat Steps 10.8 and 10.9, adding 1.0 to 2.0 mLs of 30% hydrogen peroxide each time, up to a total of no more than 5.0 mLs of the hydrogen peroxide.

10.11 When finished heating, allow the samples to cool to room temperature. Add UPDI water to the digestion tube to bring to a final volume of 50 mLs. Cap the tube and shake to mix. Store them at room temperature for analysis.

10.12 Prior to analysis, filter the samples with Whatman #41 filter paper or equivalent.

11.0 CALCULATIONS:

11.1 Calculations are included in the analytical SOP.

11.2 For composited wipe samples, the sub-samples are analyzed separately and the results for each sub-sample are added together to get one final result for the sample.

12.0 DATA REDUCTION & RECORDS:

12.1 The batch of samples is created in Element (LIMS). Enter the weights of the Certified Reference Material samples (for BS/BSD) into LIMS. Also, if the area of surface that was sampled for the wipes is known, enter these areas (in square feet) into the "Initial" area of the LIMS benchsheet. Print and include the benchsheet with the data packet. Log in the reporting limit sample in the Standards section of LIMS, print the report, and include it with the benchsheet.

12.2 Data is submitted to a technical peer for review/verification. This review/verification is documented.

13.0 REPORTING:

13.1 Reporting of data is included in the analytical SOP.

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14.0 QUALITY CONTROL:

14.1 Digest and analyze a reagent blank and a matrix blank with each batch of up to 20 samples. The reagent blank shall consist of only the acids used in the digestion. The matrix blank shall consist of an unused wipe and the acids used in the digestion. Digest both blanks the same as the samples. These blanks shall be a check for contamination from the acids, the wipes, or the digestion process. Blanks shall not be used to correct sample results. If analysis of the blank shows contamination, any samples that have results above the reporting limit and less than ten times the level of contamination shall be footnoted.

14.2 Digest and analyze duplicate Certified Reference Material samples (BS/BSD) with each batch of up to 20 samples. Weigh into a digestion tube and record on the LIMS benchsheet an amount (0.15-0.26 grams) of Certified Reference Material (Reagent 6.7). Add an unused wipe to them and digest the same as the samples. Calculate the mg/kg of lead. Calculate the percent of the lead recovered and the RPD between the duplicates. If a recovery is outside of the control limits, the BS/BSD's shall be redigested and then reanalyzed with the samples.

14.3 Digest and analyze a reporting limit sample. Spike an appropriate amount of lead spike solution (Reagent 6.6) to a blank wipe, so that the final concentration of the sample is at or below the reporting limit for lead in dust wipes. Another reporting limit sample spiked at a higher lead concentration can also be digested, in case the first one fails. Digest these samples the same as the other samples. The percent recovery must be 80-120%. If it's out of control, analyze the other reporting limit sample with the higher concentration of lead and raise the reporting limit of the samples to the higher concentration. Log the reporting limit sample into LIMS as a standard with a unique traceability number.

14.4 After the analysis is complete, for any quality control data that is out of control, document the information on the QC Checklist form. If out-of-control data is to be reported, footnote the affected samples.

15.0 METHOD PERFORMANCE:

15.1 This procedure has been validated as fit for its intended use. Results of a demonstration of capability study are shown below:

LIMS Standard ID	Reported Results µg/wipe	Reference Value µg/wipe	Acceptable range lower, µg	upper, µg	% rec
B1G0030-BS1	201	213	170.4	255.6	94.2
B1G0030-BS2	186	213	170.4	255.6	87.2
B1G0030-BS3	209	213	170.4	255.6	98.0

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B1G0030-BS4	203	213	170.4	255.6	95.4

15.2 Refer to Appendix B for factors that may contribute to measurement of uncertainty in this procedure.

16.0 DETECTION LIMITS:

16.1 Method detection limit (MDL) and Reporting limit (RL) are included in the analytical SOP.

17.0 REFERENCES:

17.1 PRIMARY REFERENCE:

17.1.1 Environmental Express HotBlock Operation Guide, "Sample Preparation for Lead Analysis with the Ghost Wipe", page 33.

17.2 SECONDARY REFERENCE:

17.2.1 Test Methods for Evaluating Solid Waste, SW-846, 3rd Edition, December 1996, Method 3050B.

18.0 WASTE MANAGEMENT & POLLUTION PREVENTION:

18.1 Please refer to the general policies and procedures outlined in chapter 7 of the QAM regarding waste management and pollution prevention.

18.2 Digested samples are very acidic. They must be neutralized before going into the sewer system.

19.0 REVISIONS:

19.1 Major changes to this document are summarized in the table below.

Document ID	Changes made	Effective Date
PBDUSTWIPEDIG, Rev. 2	Changed from Braun Intertec format to Pace Analytical Services, Inc. format	10/22/14
	Added section 19.0 on major revisions.	10/22/14

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Appendix A
Example of a LIMS Benchsheet and Reporting Limit Sample Record

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PREPARATION BENCH SHEET

B4H0185

Pace Analytical Services, Inc. (IH Laboratory)

Matrix: Wipe **Prepared using: ENVMETALS - EPA 3050B** **Printed: 10/22/2014 1:12:20PM**

Lab Number	Cont.	Prepared	Initial (Wipe)	Final (mL)	Spike ID	uL Spike	Source ID	Due Date	Comments
1404038-09	A	08/14/14 09:09	1	50				08/14/14 00:00	
METALS DIGESTION F1		*6010C LEAD WIPE*							
1404038-10	A	08/14/14 09:09	1	50				08/14/14 00:00	
METALS DIGESTION F1		*6010C LEAD WIPE*							
1404038-11	A	08/14/14 09:09	1	50				08/14/14 00:00	
METALS DIGESTION F1		*6010C LEAD WIPE*							
1404038-12	A	08/14/14 09:09	1	50				08/14/14 00:00	
METALS DIGESTION F1		*6010C LEAD WIPE*							
1404038-13	A	08/14/14 09:09	1	50				08/14/14 00:00	
METALS DIGESTION F1		*6010C LEAD WIPE*							
B4H0185-BLK1		08/14/14 09:09	1	50					
B4H0185-BLK2		08/14/14 09:09	1	50					Matrix Blank
B4H0185-BS1		08/14/14 09:09	1	50	3L12019	265.6			
B4H0185-BSD1		08/14/14 09:09	1	50	3L12019	250.2			

Reagent	Description
3L12019	ICP Soil CRM (CRM023-050)
4E03004	Hydrochloric Acid (Trace Metals Grade)
4F19004	Nitric Acid (Trace metal grade)

ED_014362_00000173-00229

Analytical Standard Record

Pace Analytical Services, Inc. (IH Laboratory)

4H14006

Description:	Wipe CRL	Expires:	12/07/14
Standard Type:	Calibration Standard	Prepared:	08/14/14
Solvent:	4% HNO3/5% HCl	Prepared By:	Cynthia Schultz
Final Volume (mls):	50	Department:	ENVMETALS
Vials:	1	Last Edit:	08/14/14 15:32 by CJS

AIHA Wipe, with batch

Analyte	CAS Number	Concentration	Units
Nitric Acid	7697-37-2	0.0401	ug/mL
Lead	7439-92-1	0.1	ug/mL
Hydrochloric Acid	7647-01-0	0.05	ug/mL

Parent Standards used in this standard:

Standard	Description	Prepared	Prepared By	Expires	Last Edit	(mls)
4E03004	Hydrochloric Acid (Trace Metals G	05/03/14	** Vendor **	08/01/16	05/03/14 14:07 by DRM	2.5
4F10023	ICP Lead CRL spike sol. 10 mg/L	06/10/14	David R. Mullenbac	12/07/14	06/10/14 10:18 by DRM	0.5
4F19004	Nitric Acid (Trace metal grade)	06/19/14	** Vendor **	04/01/16	07/03/14 15:09 by DRM	2

Reviewed By Date

Appendix B
Measurement of Uncertainty Table

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Measurement of Uncertainty Table

Measuring Instruments:	Representative and Applicable QC Data	Comments to Clarify Contributor Effects
Transportation/Storage/Handling:		
shipping time, container and temperature	NA	Usually no impact if recommended shipping conditions and holding times in referenced methods are maintained
lab storage time, conditions and temperature	NA	Usually no impact if recommended storage conditions and holding times in reference method are maintained. Wipe samples are not preserved and may be stored at ambient temperature in plastic or glass containers.
contamination in lab storage areas	NA	Usually no impact if recommended storage conditions and holding times in reference methods are maintained.
Laboratory Subsampling:		
sample nonhomogeneity	NA	There is no impact since the entire wipe sample is digested.
blending techniques	NA	There is no impact since the entire wipe sample is digested
sample size	NA	There is no impact since the entire wipe sample is digested
Sample Preparation:		
Digestion tubes	BS/BSD	Accuracy of volume markings.
dispensing device	BS/BSD	Pipettors, and other types of dispensers that are not Class A.
balance	BS/BSD	Balance error is often insignificant compared to other MU sources.
temperature	BS/BSD	Hot Block temperature variations.
sample extraction	BS/BSD	Applies to BS/BSD if it goes through the entire sample preparation process.
extractant background	BS/BSD, MB	Presence of analyte or interferent in acids, solvents, etc.
Lab Environmental Conditions:		
temperature variance	NA	Usually no impact.
humidity variance	NA	Usually no impact.
Analysts:		
different analysts	BS/BSD	BS/BSD results reflect variability due to different analysts, as applicable, on different days
analyst training level and experience	BS/BSD	BS/BSD results reflect variability due to different analysts, as applicable, on different days
data interpretation by analyst	BS/BSD	BS/BSD results reflect variability due to different analysts, as applicable, on different days
Measuring Instruments:		
instrument stability	BS/BSD	Baseline drift, repeatability of averaged readings, lab environmental stability, etc.
carryover effects	BS/BSD, MB	Impact of high samples on following sample readings; can also be monitored by proper use of CCBs.
day to day calibration differences	BS/BSD	Variation in instrument response and calibration process
interferences	BS/BSD	Due to matrix, inter-element effects.
Calibration Standards/Reference Materials:		
preparation variances	BS/BSD	Due to analysts, balances, dispensing devices used, etc
calibration stock material uncertainty	CERTIFICATE	Obtain from certificate or estimate, can be ignored if less than 1/3 of the largest contributor.
LCS reference material uncertainty	CERTIFICATE	Only has impact when LCS data are used to correct customer sample results. Obtain from certificate or estimate. Can be ignored if less than 1/3 of the largest contributor. Note that use of an LCS with a large uncertainty can result in over estimation of overall analytical uncertainty.
Data Manipulation:		
sampling media/blank correction	NA	Blank correction is not done.
instrument blank correction	NA	Blank correction is not done.
accuracy of calculations	BS/BSD	Calculations are done by LIMS.
area or air volume sampled	NA	The area sampled for each wipe sample (if given) must be correctly entered into LIMS.

Where:

BS/BSD = Laboratory Control sample, matrix matched and typically taken through the entire analytical process with each sample batch
MB = Method or Matrix Blank
NA = Not Applicable
CERTIFICATE = certificate of analysis

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ATTACHMENT B

TECHNICAL MEMORANDA

Attachment B-1:	**Technical Memorandum #2 – Modifying the Sample Depth Intervals Used to Make Remedial Decisions for Arsenic and Lead in Yard Soils**
Attachment B-2:	**Technical Memorandum #1 – Basis for Excluding Living Space Dust and Addressing Only Accessible Attic Dust through Analysis of the Existing Data**
Attachment B-3:	**Agency Approval Letter for Technical Memoranda #1 and #2**

ATTACHMENT B-1

TECHNICAL MEMORANDUM #2
MODIFYING THE SAMPLE DEPTH INTERVALS USED TO MAKE REMEDIAL DECISIONS FOR ARSENIC AND LEAD IN YARD SOILS

ANACONDA COMMUNITY SOILS REMEDIAL ACTION WORK PLAN TECHNICAL MEMORANDUM #2 – MODIFYING THE SAMPLE DEPTH INTERVALS USED TO MAKE REMEDIAL DECISIONS FOR ARSENIC AND LEAD IN YARD SOILS

Background and Purpose

In 2002 the *Final Residential Soils Remedial Action Work Plan/Final Design Report (RAWP/FDR)* and the Sampling and Analysis Plan attached to the RAWP/FDR specified surface samples (0-2 inch depth) to be collected along with 2-6 and 6-12 inch depth samples (if the 0-2 weighted average exceeded the action level) with Remedial Action (RA) performed to the depth of the exceedance or to 18 inches if the 6-12 inch interval exceeded the action level. These sampling depths were developed to meet the arsenic cleanup requirements specified in the 1996 Community Soils Operable Unit (CSOU) Record of Decision (ROD). In 2013 EPA released an amendment to the 1996 CSOU ROD which did not specify any changes to these depth intervals for arsenic sampling/remediation, but specified that the requirements of the *Superfund Lead-Contaminated Residential Sites Handbook* (EPA 2003) should be followed for lead. The 2013 amendment provided a discontinuity in the methods for sampling arsenic (As) and lead (Pb) in soils. On March 27, 2014 Atlantic Richfield (AR) submitted an outline of the proposed CSOU Remedial Action Work Plan (RAWP) and the CSOU Residential Soils and Interior Dust Sampling and Analysis Plan (SAP) for implementing the work required under the ROD amendment to EPA and DEQ (the Agencies). Two issues concerning differences in proposed sampling depth intervals between the 2014 RAWP outline and the previous documents were identified in a meeting held on April 23, 2014 between AR and the Agencies and in subsequent Agency comments to the outline. Specifically:

1. Combining the 0-1 and 1-6 inch depth intervals for lead into a 0-6 inch sample, as allowed for by the lead handbook if supported by data analysis. Similarly, AR suggested combining the 0-2 and the 2-6 inch depth intervals for arsenic as proposed in the RAWP and SAP outline: "Two separate composite samples will be developed for each yard component. The first composite sample will consist of subsamples from the 0 to 6-inch depth interval. A second composite sample will consist of subsamples from the 6 to 12-inch depth interval." Area weighted averages (AWA) would be calculated from these depth intervals.
2. Limiting the maximum depth of sampling and remediation for all yard components to 12 inches, except for vegetable gardens, which would be sampled and remediated to 24 inches as proposed in the RAWP and SAP outline: "RA intervals will be the 0 to 6-inch and 6 to 12-inch depth intervals with a maximum depth of 12-inches." This suggestion is consistent with the *Superfund Lead-Contaminated Residential Sites Handbook* (the lead handbook), but represents a change for arsenic from what was required under the 2002 RAWP/FDR.

This memo is intended to address these issues by providing data and information in support of the protectiveness /effectiveness of ARs proposed approach and to provide the Basis of Design for deviations from the 2013 ROD Amendment, as those deviations may require documentation in an Explanation of Significant Differences.

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Issue 1: Combine 0-1 and 1-6 sampling depth intervals for lead and combine 0-2 and 2-6 sampling depth intervals for arsenic

The lead handbook suggest that the 0-1 and 1-6 inch depth intervals can be combined if *"...after collecting a statistically valid number of both 0–1" and 1–6" samples, the project manager may want to compare both sample horizons (e.g., paired-sample t-test; Wilcoxon Rank Sum test) (Gilbert, 1987; Snedecor and Cochran, 1989) to determine if the 0–1" depth can be eliminated (i.e., sample from 0–6"), to further decrease sampling costs."* A paired-samples t-test was conducted to compare lead in the 0-2 inch depth interval to lead in the 2-6 inch depth interval to see if there was a significant difference between the two depth intervals[1]. The paired-samples t-test indicated that there were significant differences between lead in the 0-2 inch depth interval (mean = 589.40) and lead in the 2-6 inch depth interval (mean=334.98), $t_{(2036)}=11.61$, $p=1.62\times10^{-30}$. These results suggest that there is a significant difference between the 0-2 inch depth interval and the 2-6 inch depth interval.

As an alternative or supplement to the lead handbook paired sample t-test, EPA expressed in the April 23, 2014 meeting that combing the 0-1 and 1-6 inch depth intervals for lead could be appropriate if analysis of the current data show that a majority of the yard components that would receive RA based on a surface sample would still receive RA if a single 0-6 inch depth interval was used. To evaluate this alternative, historic sample data that have been collected from the start of the original CSOU RAWP implementation were analyzed. The following mixing calculation was applied using the 0-2 and 2-6 depth interval data to estimate a 0-6 inch depth-weighted interval concentration for comparing the number of yard components that exceed the lead action level in each depth interval to the number that exceed based on a combined depth interval.

$$C = \frac{(A*2)+(B*4)}{6}$$

where:
A = 0-2 inch depth concentration
B = 2-6 inch depth concentration
C = calculated 0-6 inch depth concentration

The analysis of the lead data revealed that the majority (87%) of the yard components that had a 0-2 inch depth interval sample exceeding the 400 mg/kg action level for lead would still exceed the action level if a 0-6 inch depth interval was used. Similarly if a yard component had a lead exceedance in the 2-6 in depth interval but not in the 0-2 inch interval, the majority (85%) would still exceed the action level if a 0-6 inch depth interval was used.

Anaconda NPL CSOU Database Lead 0-6 inch Data summary	
1,019	Total number of sample locations with both a 0-2 and a 2-6 inch depth interval
0-6 inch Lead screening with an exceedance in the 0-2 depth interval	

[1] The dataset does not include sample results for 0-1 vs 1-6 inch depth intervals, but within that dataset, 1,019 yard component samples have lead data for both the 0-2 inch and 2-6 inch depth intervals. The 0-2 inch and 2-6 inch depth intervals can reasonably be used as a surrogate for the 0-1 and 1-6 inch sampling depth intervals recommended in the lead handbook.

ED_014362_00000173-00236

516	# of Yard Components with a Pb exceedance in the 0-2 inch depth interval with data in 2-6 inch depth interval
447	# of Yard Components with a Pb exceedance based on the estimated 0-6 inch depth interval concentration
87%[2]	Percentage of Yard components with a Pb exceedance in the 0-2 inch depth interval that are also exceeded based on the estimated 0-6 inch depth interval concentration
0-6 inch Lead screening with an exceedance in the 2-6 inch depth interval	
20	# of Yard Components with a Pb exceedance in the 2-6 inch depth interval but not the 0-2 inch depth interval
17	# of Yard Components with a Pb exceedance based on the estimated 0-6 inch depth interval concentration
85%[2]	Percentage of Yard components with a Pb exceedance in the 2-6 inch depth interval but not the 0-2 inch depth interval that are also exceeded based on the estimated 0-6 inch depth interval concentration

Although this analysis of a weighted 0-6 inch depth interval showed that a majority of the yard components would still receive RA, given that the paired sample t-test does not support combining the two depth intervals AR has decided not to pursue combining neither the 0-1 and 1-6 depth interval for lead nor the 0-2 and 2-6 inch depth interval for arsenic for sampling. However, for consistency with historic and currently proposed arsenic sampling AR is proposing the use of 0-2 and 2-6 inch depth intervals for sampling both arsenic and lead.

Issue 2: Change the maximum sampling/remediation depth for arsenic from 18 inches to 12 inches

When performing RA under the original 2002 CSOU design, residential soil removal was extended to a maximum depth of 18". This 18" Remedial Action Objective (RAO) was based on the maximum rooting depth of native dry land vegetation used as a capping component in Waste Management Areas (WMAs) within other OUs at the Anaconda Smelter NPL Site. The 18" depth RAO had been established at the site for WMAs and was subsequently transferred to address residential soils within the CSOU. However, this RAO is not directly associated with human health risk reduction. Therefore, under the 2014 design proposed in the CSOU RAWP and SAP outline, soil removal will extend to a maximum depth of 12". The 12" maximum depth of removal is appropriate as it attains the human health risk reduction objectives for metals as specified in the lead handbook. Also consistent with the lead handbook, soil removal within vegetable gardens will extend to a maximum depth of 24".

Conclusions

Consistent with the original CSOU design, remedial action decisions applicable to removal of soils with arsenic concentrations exceeding the 250 ppm action level will be based on an area weighted average basis. As stated above the analysis of existing data showed that a majority of the yard components get an RA based on a 0-2 or 2-6 inch exceedance would still receive an RA based on a 0-6 inch depth interval. However, given that the paired sample t-test does not support combining the two depth intervals AR has decided not to pursue combining the 0-1 and 1-6

[2] The 85% and the 87% do not consider any overlap.

depth interval for lead or the 0-2 and 2-6 inch depth interval for arsenic. However, for consistency with historic and currently proposed arsenic sampling AR is proposing the use of 0-2 and 2-6 inch depth intervals for sampling both arsenic and lead.

Furthermore, under the design proposed in the updated CSOU RAWP outline, soil removal will extend to a maximum depth of 12 inches and 24 inches for vegetable gardens. The 12 inch maximum depth of removal and 24 inch maximum depth for vegetable gardens is appropriate, as it attains the human health risk reduction objectives for metals as specified in the *Superfund Lead-Contaminated Residential Sites Handbook*. In addition, any area that is not a vegetable garden during the implementation of the CSOU RAWP will still be addressed under the Development Permit System, the Institutional Controls Management Plan and/or the Soil Swap Program.

References

Atlantic Richfield, 2002. *Final Residential Soils Remedial Action Work Plan/Final Design Report (RAWP/FDR)* Atlantic Richfield Company, July 19, 2002.

EPA, 1996. *Record of Decision, Community Soils Operable Unit, Anaconda Smelter NPL Site, Anaconda MT*, United States Environmental Protection Agency and Montana Department of Environmental Quality, September 25, 1996.

EPA, 2003. *Superfund Lead-Contaminated Residential Sites Handbook*, United States Environmental Protection Agency Lead Sites Workgroup. August, 1996.

EPA, 2013. *Record of Decision Amendment, Community Soils Operable Unit, Anaconda Smelter National Priorities List Site, Anaconda, MT*. United States Environmental Protection Agency and Montana Department of Environmental Quality, September, 2013.

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ATTACHMENT B-2

TECHNICAL MEMORANDUM #1
BASIS FOR EXCLUDING LIVING SPACE DUST AND ADDRESSING ONLY ACCESSIBLE ATTIC DUST THROUGH ANALYSIS OF EXISTING DATA

ANACONDA COMMUNITY SOILS REMEDIAL ACTION WORK PLAN TECHNICAL MEMORANDUM #1 - BASIS FOR EXCLUDING LIVING SPACE DUST AND ADDRESSING ONLY ACCESSIBLE ATTIC DUST THROUGH ANALYSIS OF THE EXISTING DATA

Background and Purpose

In 1996 EPA released a Record of Decision (ROD) for the Community Soils Operable Unit (CSOU) which specified arsenic action levels for residential properties of 250 mg/kg without assigning an action level to lead or requiring interior dust sampling. In 2013 EPA released an amendment to the 1996 CSOU ROD which expanded on the original ROD to include a lead action level of 400 mg/kg as well as requiring sampling of accessible interior dust along with yard soil when residential sampling is conducted under the program. On March 27, 2014 Atlantic Richfield (AR) submitted an outline of the proposed CSOU Remedial Action Work Plan (RAWP) for implementing the work required under the ROD amendment to EPA and DEQ (the Agencies) in which AR suggested "Interior/attic dust to be sampled only when requested by landowner; and only when an exposure pathway from an attic to living space is present." A subsequent meeting between AR and the Agencies was held on April 23, 2014 to discuss the CSOU RAWP outline. At the meeting and in the subsequent *Comments to the Residential Soils and Interior Dust Remedial Action Work Plan/Final Design Report Annotated Outline Community Soils Operable Unit Anaconda Smelter NPL Site* the Agencies identified there may be appropriate alternatives to address interior dust (living space dust) including developing a basis for excluding interior dust (living space dust) and addressing only accessible attic dust through analysis of the existing data base. This memorandum is to provide data and information in support of the protectiveness/effectiveness of ARs proposed approach as identified in the Agencies' alternative above and to provide the Basis of Design for deviations from the 2013 ROD Amendment, as those deviations may require documentation in an Explanation of Significant Differences.

Conceptual Site Model

This section includes a review of the Conceptual Site Model (CSM) for the original sources of lead and arsenic in living space dust and the mechanisms for transport between living space dust, attic dust and soils to assist in the interpretation of the analysis results. For approximately 100 years the Old Works and Washoe Smelters' emissions of arsenic and lead into the air may have resulted in deposition of particulates containing arsenic and lead onto soils and other surfaces, including inside living spaces and attics within the Anaconda area. While other sources of lead and arsenic emissions to the air over the same timeframe may also have contributed to soil and dust concentrations, the dominant air emission source was likely the smelters. Once deposited onto soil or interior surfaces, further movement of the arsenic and lead in soil and dust would have depended upon a variety of chemical/physical fate and transport factors (e.g., sorption, resuspension and redeposition, etc.), as well as activity-based factors (e.g., track-in, cleaning, tilling garden soil, etc.).

Because living spaces are typically cleaned periodically by residents, recent accessible living space dust concentrations would not be expected to be representative of long-term historical deposition from the air. In contrast, attics are typically not cleaned and tend to be seldom disturbed. Thus, little movement of settled arsenic and lead in attics that originated from historical smelter emissions would be expected in undisturbed attics. Only when the attic dust is

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disturbed and has a transmission pathway would it migrate into the living space. EPA's *Guidance for the Sampling and Analysis of Lead in Indoor Residential Dust for use in the Integrated Exposure Uptake Biokinetic (IEUBK) Model* describes this more thoroughly: "Contamination of attic dust occurs when ambient air containing pollutants enters the attic and airborne particulate matter settle on attic surfaces such as the floor, insulation, roof, and rafters. In a typical attic, warm air is vented near the peak of the roof. As the warm air is vented, cooler outside air is drawn into the attic through vents and other openings located lower in the attic (e.g., soffit/eave vents). Reduced air flow promotes increased particulate deposition rate and less re-entrainment. Over time, contaminated particles build up in the attic and may remain in the attic for the life of the home if left undisturbed."

Attics in older homes would be expected to have higher concentrations of both lead and arsenic given they were exposed to sources of dust dominated by the smelter emissions for a longer period of time and when smelter emissions were more concentrated (i.e., prior to installation and/or upgrade of pollution controls). Attics in homes built after the smelter was shut down in 1980 would be expected to have lower concentrations of lead and arsenic.

Due to the high prevalence of older homes within Anaconda, deteriorating lead paint on the exteriors and interiors of homes represents another likely source of lead in surface soils and living space dust. Unlike initial deposition of smelter emission sources of lead and arsenic, which ceased to influence lead and arsenic in soils once the smelters were shut down, lead paint on homes built before 1978 can continue to influence lead in soils if the paint is allowed to deteriorate. Given that attic interiors are typically not painted, attics are not expected to have been or continue to be influenced by deteriorating lead paint regardless of when the home was built.

Regardless of the original source(s) of lead and arsenic, transfer of soil particles to interior living spaces can occur as blown or tracked-in dust from soils in the yard. Tracking in particles from attics to interior living spaces is also possible, but it is expected to be a much less significant contributor to living space dust concentrations in homes with infrequently accessed attics. In contrast, renovations or repairs that affect attic spaces represent a temporary yet significant potential contributor of particulate materials from attics to living spaces.

Given the conceptual model described above, we would expect attic dust lead and arsenic concentrations to most closely represent historical smelter emissions in homes built before the smelters ceased operations, particularly the oldest homes that were present throughout most of the 100 year smelting history. In homes built more recently, attic dust concentrations would reflect non-smelter air emission sources. Lead and arsenic in soils may also reflect inputs of historical smelter emissions, though we would not expect the relationship (i.e. ratios) between lead and arsenic concentrations in soils to be as consistent as in attic dust due to the chemical-specific effects of soil on their fate and transport and the additional variable contribution of non-smelter sources (e.g., deteriorating lead paint) to soil concentrations across Anaconda properties. Similarly, lead and arsenic in living space dust are not likely to be very representative of historical smelter deposition from air given typical household activities and the presence of non-smelter sources (e.g., deteriorating lead paint). Living spaces are likely to be influenced by transfer of yard soil particles via blown in or tracked in soil, regardless of the original source of the particles in soil. However, to this point, it is important to note that the procedure for

calculating soil cleanup levels for arsenic and lead accounts for incidental ingestion of indoor dust that is derived from soil. Therefore, actions taken for soil are expected to address living space dust derived from soil.

Summary of Living Space and Attic Dust Data

In 2006 and 2007 the exterior, living space, and attics (if accessible) of 52 residential homes within the Anaconda area were sampled and reported in the *Draft Final Community Soils Interior and Attic Dust Characterization Study Data Summary Report (DSR)* (Atlantic Richfield 2008). The primary objective of the characterization study was to determine the distribution and concentration of arsenic and lead in living space and attic dust. In addition yard soils were collected by Atlantic Richfield and EPA for 34 of the 52 residential homes sampled in 2006 and 2007 for dust.

Of the 52 residential homes sampled all 52 had living space dust sampled (see Table 1). The median arsenic concentration for lead and arsenic was 186.5 and 53.1 respectively for the 52 residential homes samples.

Of the 52 residential homes sampled 46 had accessible attics sampled (see Table 1). Consistent with the CSM described above, attic dust samples tend to have greater median concentrations of both lead and arsenic when compared to the living space dust samples. Additionally, none of the attic dust exceedances were in houses constructed after the smelter shut down in 1980.

Table 1 - Living Space Arsenic Dust Summary

	Living Space		Attic	
	Arsenic	Lead	Arsenic	Lead
Total Number of Homes Sampled	52		46	
Minimum Concentration (mg/kg)	11.1	41.8	1.05	2.61
Maximum Concentration (mg/kg)	322	10,700	1,560	2,620
Median Concentration (mg/kg)	53.1	186.5	336.5	497
# Greater than 250 mg/kg As, 400 mg/kg Pb	1	12	26	27
% Greater than 250 mg/kg As, 400 mg/kg Pb	1.90%	23.10%	56.50%	58.70%

Relationship between Arsenic and Lead Concentration in Dust and Soil

The relationship between arsenic and lead concentrations in dust and soil was further evaluated by comparing the geometric mean of lead to arsenic concentration ratios in soil, and attic and living space dust. The values in Table 2 were first calculated by determining the ratio of lead to arsenic for each media lead/arsenic sample. Second the geometric mean was calculated from the lead/arsenic ratios from each media. This approach provides a measure of the central tendency value and is less likely to be influenced by outliers than a mean would be within the dataset. The results of this analysis are summarized in Table 2.

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Table 2 - Geometric mean of Lead to Arsenic concentration Ratios

Attic Pb/As	1.75
Yard Pb/As	2.10
Living Space Pb/As	4.01

As shown in Table 2 the concentration of lead is 1.75 times the concentration of arsenic in attic dust. As noted in our description of the CSM, we expect undisturbed attic dust to be most stable with regard to the relationship between arsenic and lead concentrations in smelter related dust, particularly in older homes that were subject to the longest period of smelter impacts. The ratio of the concentrations of lead to arsenic in soils was similar to attics though slightly higher at 2.10 suggesting a slight influence on the lead concentration from an additional sources or weathering of As. However, in living space dust the concentration of lead was a little more the 4 times (4.01) the concentration of arsenic. If soil was the primary source of indoor dust we would not expect the ratio of lead to arsenic in soil and living space dust to be significantly different. Given the lead to arsenic ratio in soil is not similar to the lead to arsenic ratio in living space dust, it is reasonable to conclude that another source is contributing to lead in living space dust. Consistent with the CSM described previously, deteriorating lead paint on interior surfaces is a likely additional source of lead in living space dust and could explain why the concentration of lead is so much higher than arsenic in living space dust.

Conclusions

Based on the presented analysis and the CSM, the following conclusions can be made from the data:

- Existing data show that living space dust is unlikely to exceed arsenic action levels. Given that living space dust is transient, in the unlikely event that living space dust exceeded the arsenic action level due to transfer from yard soils or accessible attics, it would be addressed by remediation of the soil or attic source. Therefore, separate sampling of living space dust should not be required.
- The much higher ratio of lead to arsenic concentrations in living space dust, relative to soil or attics, suggests that a secondary source (most likely deteriorating interior lead paint) is a primary contributor to the living space dust lead concentrations. Development of soil cleanup standards for Pb and As already includes estimation of risk due to exposure of interior dust derived from soil. Thus, collection of living space dust will not inform Superfund cleanup requirements.
- The high rate of arsenic and lead exceedances in attics, which are unlikely to be influenced by deteriorating lead paint, justifies retaining attic dust sampling as an option if there is a pathway for exposure. However, the lack of exceedances in attic dust in homes built after 1980 and lack of exposure to attics without an existing transmission pathway provides the basis for limiting attic sampling to accessible attics in homes built prior to 1980 with a transmission pathway.

References

Atlantic Richfield, 2008. *Draft Final Community Soils Operable Unit Interior and Attic Dust Characterization Study Data Summary Report (DSR).* January 4, 2008.

EPA, 1996. *Record of Decision, Community Soils Operable Unit, Anaconda Smelter NPL Site, Anaconda MT*, United States Environmental Protection Agency and Montana Department of Environmental Quality, September 25, 1996.

EPA, 2013. *Record of Decision Amendment, Community Soils Operable Unit, Anaconda Smelter National Priorities List Site, Anaconda, MT.* United States Environmental Protection Agency and Montana Department of Environmental Quality, September, 2013.

EPA, 2008. *Guidance for the Sampling and Analysis of Lead in Indoor Residential Dust for use in the Integrated Exposure Uptake Biokinetic (IEUBK) Model.* Prepared by the Lead Committee of the Technical Review Workgroup for Metals and Asbestos Office of Superfund Remediation and Technology Innovation United States Environmental Protection Agency, December 2008. Available online at: http://www.epa.gov/superfund/lead/products/dust_sampling_guidance_final.pdf

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Attachment 1
Data Table

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Address	Year Built	Arsenic in Yard Soil AWA (mg/kg)	Arsenic in Living Space Dust (mg/kg)	Arsenic in Attic Dust (mg/kg)	Lead in Yard Soil AWA (mg/kg)	Lead in Living Space Dust (mg/kg)	Lead in Attic Dust (mg/kg)	Noted Pathway
214 Locust	1915	331.20	42.00	294.00	1,096.92	649.0	462.0	
1002 W. 4th	1928	179.60	21.60	4.98	517.46	151.0	141.0	
620 W. 3rd	1912	214.60	48.80	360.00	538.71	247.0	955.0	X
811 W. 3rd	1923		29.40	407.00		927.0	511.0	
608 Walnut	1925	157.10	26.40	605.00	616.77	89.5	2580.0	
611 W. 6th	1917	116.50	53.60	462.00		189.0	459.0	
1806 W. Park	1954	89.70	16.20	5.36	183.06	85.1	13.7	
1603 Copper Road	1958	75.80	24.30	27.10	99.06	70.5	101.0	
1610 Tammany	1951	64.50	30.80	236.00	210.89	144.0	306.0	
422 Hickory	1880	142.20	52.30	368.00	886.49	4850.0	2620.0	
1707 Ogden	1953		101.00	6.90		70.3	19.5	
604 W. Park	1950	129.32	69.10	1460.00	543.92	408.0	1320.0	
620 W. 4th	1912	187.20	37.90	878.00	799.58	136.0	1560.0	
803 W. 7th	1955	256.30	38.80	875.00	151.69	70.4	575.0	
2008 W. Park	1955		35.10	45.90		103.0	115.0	
805 W. 3rd	1922	168.50	59.80	130.00	451.71	351.0	196.0	
217 Maple	1905		91.80			790.0		
813 E. Park	1905	300.90	73.40	617.00	1,126.96	304.0	776.0	
107 N. Main	1910	164.10	62.50	722.00	530.02	355.0	903.0	
801 E. 3rd	1906	334.40	30.90	928.00	350.39	172.0	804.0	
821 E. 5th	1881	185.10	61.30	313.00	553.38	322.0	480.0	
612 E. 4th	1908	120.80	35.60	806.00	341.99	184.0	1020.0	X
521 E. Commercial	1910	199.50	91.90	796.00		10700.0	1630.0	
712 Chestnut	1922	202.90	88.30	1420.00	587.99	446.0	2180.0	X
603 E Front	1916	199.90	81.20	1450.00	569.78	639.0	1140.0	X
600 Alder	1917	224.10	41.40	1460.00	772.17	672.0	1600.0	
708 Alder	1900	112.90	77.20	1520.00		450.0	1210.0	
600 E. Commercial	1955	228.30	46.10	297.00	572.78	244.0	483.0	
303 Ash	1920	283.00	65.50	1140.00		598.0	935.0	
606 E. Commercial	1912	217.30	46.80	471.00	662.34	297.0	526.0	
900 E. 6th St	1923	361.10	81.30	584.00	324.75	232.0	1870.0	
806 E. 5th	1916	78.70	52.60	1560.00		328.0	1700.0	X
616 Rickards	1976	99.90	28.00	204.00	183.24	41.8	190.0	

Address	Year Built	Arsenic in Yard Soil AWA (mg/kg)	Arsenic in Living Space Dust (mg/kg)	Arsenic in Attic Dust (mg/kg)	Lead in Yard Soil AWA (mg/kg)	Lead in Living Space Dust (mg/kg)	Lead in Attic Dust (mg/kg)	Noted Pathway
410 Rickards	1957	56.90	91.90	648.00	132.50	116.0	601.0	
814 Rickards	1954		55.30	56.90		122.0	315.0	X
1110 Smith St.	1969	75.60	42.60	5.09		71.7	6.7	
103 Stewart	1930	69.80	82.70	16.90	330.22	344.0	29.1	
600 Rickards	1920	162.50	49.70	5.17	234.37	110.0	7.8	
808 Stewart	1930	68.40	37.40	155.00	87.91	112.0	149.0	
408 S. Hauser	1910	99.00	161.00	183.00		275.0	903.0	
515 Stewart	1920	199.90	117.00	84.40	195.50	197.0	64.2	X
305 S. Hauser	1976	53.00	11.10	14.90		43.7	22.7	
4507 Crackerville	1978	131.50	31.80	6.94	454.41	755.0	22.9	
1218 Hwy 1 West	1930	58.20	61.50	1070.00		129.0	822.0	
46 Mountain View	1955		56.20	76.30		102.0	106.0	
100 Cutthroat Trl	1999	680.90	167.00		351.43	148.0		
100 Outlaw Trail	1994	801.40	322.00		403.22	203.0		
100 N. Juniper	2001		36.00	2.27		96.4	24.3	X
1101 Sunlight Basin	2003		35.70	1.05		65.7	2.6	X
6285 Aspen Hills Road	2007	496.90	69.60		341.85	103.0		
6300 Aspen Hills	2005	128.80	89.80		168.04	64.1		
1157 Galen Rd.	2001		113.00			99.8		

ATTACHMENT B-3

AGENCY APPROVAL LETTER
FOR TECHNICAL MEMORANDA #1 AND #2

UNITED STATES ENVIRONMENTAL PROTECTION AGENCY
REGION 8, MONTANA OFFICE
FEDERAL BUILDING, 10 W. 15th STREET, SUITE 3200
HELENA, MONTANA 59626

Ref: 8MO

September 8, 2014

Mr. Luke Pokorny
Atlantic Richfield Company
317 Anaconda Road
Butte, Montana 59701

Dear Luke:

EPA, in consultation with DEQ, approves, with comment, Atlantic Richfield's *Community Soils Operable Unit (CSOU) Residential Soils and Attic Dust Remedial Action Work Plan/Final Design Report and SAP Annotated Outlines, dated August 15, 2014.* No revisions of the outlines are necessary. Please proceed with the development of the draft Remedial Action Work Plan/Final Design Report (RAWP/FDR).

Please note that some of the proposed work plan elements differ from the 2013 CSOU ROD Amendment. These changes will need to be described in an EPA Explanation of Significant Differences (ESD). In order to coordinate the development of the RAWP/FDR and ESD, I suggest that we set up a meeting at your earliest convenience. If you have any questions, please give me a call.

Sincerely,

Charles Coleman
Anaconda Project Manager

Enclosure

cc: Andy Lensink, ENF-L
Joel Chavez, DEQ
Katherine-Haque-Hausrath, DEQ
Ken Brockman, BOR
Gunnar Emilsson, CDM
Connie Daniels, ADL

Printed on Recycled Paper

AR: Roy Thun, AR
Shannon Dunlap, AR
Jill Kelley, AR
John Davis, PRR
Kevin Bethke, TREC
Duane Logan, PTS

Comments to the
Residential Soils and Interior Dust Remedial Action Work Plan/Final Design Report Annotated Outline
Community Soils Operable Unit
Anaconda Smelter NPL Site

General Comments

The Atlantic Richfield Company (AR) submitted an outline of the Community Soils OU Residential Soils and Interior Dust Remedial Action Work Plan/Final Design Report (RAWP/FDR) to EPA and DEQ (the Agencies) on March 27, 2014. Upon initial review, the Agencies provided a list of discussion items which were discussed with AR on April 23. Following this meeting, the Agencies submitted comments to the outlines On May 8, 2014.

In response to these comments, on August 15, 2014, AR submitted the following documents:

1. Comment Responses to the Draft CS OU Residential Soils and Attic Dust Remedial Action Work Plan/Final Design Report Annotated Outline;

2. Comment Responses to the Draft CS OU Residential Soils and Attic Dust Sampling and Analysis Plan Annotated Outline;

3. Draft Final CS OU Residential Soils and Attic Dust Remedial Action Work Plan/Final Design Report Annotated Outline;

4. Draft Final CS OU Residential Soils and Attic Dust Sampling and Analysis Plan Annotated Outline;

5. Anaconda Community Soils Remedial Action Work Plan Technical Memorandum #1 – Basis for Excluding Living Space Dust and Addressing Only Accessible Attic Dust through Analysis of the Existing Data; and

6. Anaconda Community Soils Remedial Action Work Plan Technical Memorandum #2 – Modifying the Sample Depth Intervals used to make Remedial Decisions for Arsenic and Lead in Yard Soils.

The technical memoranda were submitted in response to the Agencies' comment that the basis for proposed design criteria presented in the RAWP/FDR outline should be presented for review as either part of the design or as stand-alone technical memoranda. As these technical memoranda support the proposed design presented in the outline, they will be reviewed first.

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Attachment 5 - Anaconda Community Soils Remedial Action Work Plan Technical Memorandum #1 – Basis for Excluding Living Space Dust and Addressing Only Accessible Attic Dust through Analysis of the Existing Data

This technical memorandum summarizes the existing interior and attic dust sampling and analytical results completed to date, and provides a justification for excluding interior dust sampling from the RA while retaining accessible attic dust sampling in homes built before 1980 (when the smelter ceased operations).

The conceptual site model (CSM) for dust presented in the technical memorandum is well written and is consistent with the 1996 Community Soils OU ROD CSM, which was used to develop the Selected Remedy (e.g., cleaning up the soil will clean up the interior dust). The statistical analysis presented in the technical memorandum supports the CSM assumption that the primary source of elevated lead in interior dust in older homes is likely lead paint.

Analysis of the provided data set indicates that only one of the 52 homes sampled for interior dust exceeded the 250 mg/kg action level for arsenic. That home is located in the Aspen Hills subdivision, and had soils area-weighted arsenic concentration of 801 mg/kg (the highest arsenic concentration for soils for the 52 residences). This affirms the CSM that the principal source of arsenic to interior dust is the soils.

The October 2008 Residential Soils Data Interpretation and Analysis Report (CDM Smith 2008) showed that the correlation between arsenic and lead concentrations in soil was statistically significant. This technical memorandum shows that there is no correlation between arsenic and lead for interior dust in the available data set, with the reasonable postulated thesis that arsenic and lead from the smelter would have been cleaned up, so that the likely source of the elevated lead in interior dust (12 out of the 52 homes sampled) is from deteriorating lead paint in older homes. The technical memorandum provides ratios of the geometric means arsenic and lead concentrations of soil, interior dust, and attic dust, and notes that while the soil and attic dust ratios are similar, the interior dust is skewed towards lead. Review of the 12 residences where lead in interior dust exceeds 400 mg/kg show that they are older homes with one exception. That exception, a home built in 1978, is located in Crackerville, an area previously identified as an elevated lead area due to the presence of fluvial tailings deposited from past Silver Bow Creek flooding. That residence also had lead exceeding 400 mg/kg in the soils.

In summary, the analysis and conclusions presented in Technical Memorandum #1 are supported by the data, and it can be approved without comment.

Attachment 6 - Anaconda Community Soils Remedial Action Work Plan Technical Memorandum #2 – Modifying the Sample Depth Intervals used to make Remedial Decisions for Arsenic and Lead in Yard Soils

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This technical memorandum reviews the proposed sampling depth interval modifications that were proposed in the draft March 27, 2014 outline. The proposed modification was use 0 to 6 inch and 6 to 12 inch sampling depth increments, in lieu of the 0 to 2 inch, 2 to 6 inch, and 6 to 12 inch increments used historically in Anaconda and the 0 to 1 inch, 1 to 6 inch, and 6 to 12 inch increments used in EPA's Lead Handbook. Following Lead Handbook guidance, AR used the existing screening level data for Pb and completed a paired sample t-test, the results of which suggested that "that there is a significant difference between the 0-2 inch depth interval and the 2-6 inch depth interval".

Based on this difference, AR has elected not to pursue the 0 to 6 inch and 6 to 12 inch sampling increment proposal, but to sample both arsenic and lead following the existing 0 to 2 inch, 2 to 6 inch, and 6 to 12 inch sampling increments. The Agencies concur, as keeping with the previous sampling intervals will allow these data to be more readily compared to the historical data set for Five Year Review purposes.

The second objective of Technical Memorandum #2 is to justify limiting the remedial action to 18 inches. The technical memorandum notes that the 18 inch removal criteria was carried over in the 1996 Community Soils OU Record of Decision from criteria original developed for minimum plant growth media thickness placed over waste material. As the 2003 Pb Handbook identified a 12 inch cover as protective of human health, changing the arsenic cleanup requirement to be consistent with the Pb Handbook is appropriate. Note that vegetable gardens will still receive 24 inches of cleanup where warranted.

This will require the Agencies to evaluate this proposal as part of an Explanation of Significant Differences (ESD) to the 1996 ROD in the future. As currently drafted, the DPS requires cleanup of all contaminated soil exceeding the cleanup levels. In order to ensure that the remedy remains protective, the DPS must continue to address cleanup of soil that exceeds the residential cleanup levels from 0-2 feet bgs.

Attachments 1 and 3 - Draft Final CS OU Residential Soils and Attic Dust Remedial Action Work Plan/Final Design Report Annotated Outline and Comment Responses

Overall, the comment responses are acceptable. The Agencies have a few points of clarification that should be addressed in the RAWP/FDR submittal (no response to comments is necessary).

Section 1.5 Community Soils OU Remedial Action Objectives, last bullet states that attic dust will be sampled if accessed more than once a week or the property owner has remodeling plans and **"and a construction permit has been granted by ADLC."** It is the Agencies' understanding that ADLC does not issue construction permits for all renovation projects. Therefore, the Development Permit System (DPS) likely must address the issuance of construction permits for all projects that could spread contaminated dust from attics to the interior. This needs to be addressed in the DPS revisions currently under development.

Consistent with previous Community Soils OU RA, all visible wastes should continue to be removed, regardless of depth.

Section 3.0, Basis of Design, third bullet states: "This 18 inch Remedial Action Objective (RAO) was based on the maximum rooting depth of native dry land vegetation used as a capping component in Waste Management Areas (WMAs) within other OUs at the Anaconda Smelter NPL Site." This statement is inaccurate. Section 9.1 of CSOU ROD states: "The maximum 18-inch depth is based upon possible activities that might be conducted in a yard (i.e., garden, play area, or other excavation)."

Section 3.0, Basis of Design, sixth bullet states: "Soil removal within residential yards will extend laterally until encountering an existing barrier (e.g., concrete sidewalk or foundation, tree roots, **property boundary,** asphalt pavement, etc.)." It is the Agencies' understanding that the area that is remediated will be tracked in the GIS database, which will provide notice to ADLC and property owners that contaminated soil and/or waste around tree roots, under pavement, etc., was/will not be remediated, and such contamination will be addressed by the DPS if disturbed in the future. However, please delete "property boundary" from this list, because contaminated soil should be cleaned up from adjacent properties unless Atlantic Richfield (AR) is unable to obtain access.

Sections 3.0, 5.2, 6.2: These sections state: "Insulation will not be replaced in attics unless/until all electrical components that may be present in the attic comply with applicable residential electrical codes." The document implies that insulation may be removed and not replaced. Given Montana's climate, it is not acceptable to remove attic insulation without replacing it. The work plan should clarify that no insulation will be removed unless AR will replace it.

Section 5.6.1: "A maximum of eight (8) soil subsamples will be collected from any individual residential yard, school, park, etc., component regardless of the surface area of that component." Please note that larger parcels, including schools, may require multiple components to ensure protectiveness.

Section 8.0, Remedial Action Schedule. In this section, AR proposes five years for property owners to sign up for the "test by request" program, although the yard cleanups might continue for longer than five years. After five years, the test by request program would be closed, but the DPS would apply if soil is disturbed on those properties. In order to ensure that property owners have adequate time to sign up for the test by request program, the Agencies would like the deadline to sign up for the test by request program to extend at least a year past the time that the majority of the yard cleanups are completed. This would provide for a "last call," so that property owners who see remediation occurring elsewhere are able to sign up for the test by request program.

Attachments 2 and 4 - Draft Final CS OU Residential Soils and Attic Dust Sampling and Analysis Plan Annotated Outline and Response to Comments

In response to the Agencies' comment to Section 3 that "EPA has established a method to determine if the cleanup was effective. Refer to: Lead Dust Sampling Technician Field Guide (EPA Office of Pollution Prevention and Toxics, EPAW- 04-022, May 2009)", AR responded with "comment noted." The Agencies would like to clarify that the confirmation sampling methodology and a clearance level of 40 $\mu g/ft^2$ for floors, that is presented in the guidance document, will be provided in the QAPP.

End of comments.

ATTACHMENT C

SAMPLING FIELD DATA SHEETS

Attachment C-1: **Residential Yard Sampling Field Data Sheet**
Attachment C-2: **Unpaved Roads & Alleys Sampling Field Data Sheet**
Attachment C-3: **Attic Dust In-Home Sampling Data Sheet**

ATTACHMENT C-1

RESIDENTIAL YARD SAMPLING FIELD DATA SHEET

Anaconda Smelter NPL Site – Community Soils Operable Unit
Residential Yard Sampling Field Data Sheet

Sample Identifier – _____ Date-_____ Time - _____

Owner_____

Address - _____ Phone Number - _____

Samplers - _____ Weather - _____

Owner Present - _____

Describe Hazards/Obstacles in the Yard and Control Measures Identified:

COMPONENTS SAMPLED

Component	Feet^2	No. of Samples	No. of Holes	Sample in Drip Zone	Comments

PHOTOGRAPHS

House and Trim Color:
Photographers Name:

Number	Time	Description
		Front of house looking (N, S, E, W)

Location	Sample ID	Component	Depth	QA/QC
A – Anaconda OP – Opportunity R – Regional Area	0001 – first sample location 0002 – second sample location	FY-front yard, BY-back yard, ED-earthen drive, FG-flower garden, , VG-vegetable garden, , PA-play area, BA-bare area, SA-source area, BV-boulevard	0002 – 0" to 2" 0206 – 2" to 6" 0612 – 6" to 12" Also for VG and FG only: 1218 – 12" to 18" 1824 – 18" to 24"	D-Duplicate B-Blank M-2586 M2-2711a

Examples: A-0001-BA-0206-B; A-0005-FG-0612

Anaconda Smelter NPL Site – Community Soils Operable Unit
Residential Yard Sampling Field Data Sheet

GENERAL INFORMATION:

Type of Roof: _____ Condition: _____
Type of Siding: _____ Condition: _____
Exterior Lead Paint? Y / N Peeling Paint? Y / N
Type of Trim (Windows, doors, eaves) _____
Condition of trim: _____
Outbuildings/Type? _____ Condition: _____
Porch/Other: _____ Condition: _____
Gutters? _____ Type(s):_____ Condition:_____
Sprinkler System Present? _____ Location: _____
Fence Present?_____ Type(s):_____ Condition:_____

SAMPLES COLLECTED

Metals to Analyze for: **As, Pb**

Sample ID Sample Identifier :	Time	No. of Holes	QA/QC	Paint chips /Amount	Comments

COMMENTS:

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Anaconda Smelter NPL Site – Community Soils Operable Unit
Residential Yard Sampling Field Data Sheet

Sample Identifier – _____ Date-_____ Time - _____

CONTINUATION: PHOTOGRAPHS

House and Trim Color:
Photographers Name:

Number	Time	Description

ED_014362_00000173-00260

Anaconda Smelter NPL Site – Community Soils Operable Unit
Residential Yard Sampling Field Data Sheet

CONTINUATION: SAMPLES COLLECTED
Metals to Analyze for: **As, Pb**

Sample ID Sample Identifier :	Time	No. of Holes	QA/QC	Paint chips /Amount	Comments

ATTACHMENT C-2

UNPAVED ROADS & ALLEYS SAMPLING FIELD DATA SHEET

Anaconda Smelter NPL Site – Community Soils Operable Unit
Unpaved Roads and Alley Sampling Field Data Sheet

Sample Identifier – _____ Date-_____ Time - _____

Owner_____ Phone Number - _____

Samplers - _____ Operator - _____ Weather - _____

Describe Hazards/Obstacles in the Yard and Control Measures Identified:

PHOTOGRAPHS

Photographers Name:

Number	Time	Description

Anaconda Smelter NPL Site – Community Soils Operable Unit
Unpaved Roads and Alley Sampling Field Data Sheet

Site Code	Sequential Letter Code	Sequential Number Code	Letter Code	QA/QC
A – Anaconda **OP – Opportunity** **R – Regional** **Alley**	**A –** First unpaved road or alley segment from south to north. **B –** Second unpaved road or alley segment south to north.	**01** – First Unpaved road or alley segment of block from west to east. **02** – Second unpaved road or alley segment of block from south to north.	**A** – Alley	**D**-Duplicate **B**-Blank **M**-2586 **M2**-2711a

Examples: A-B-02-A, A-B-02-A-M

SAMPLES COLLECTED

Metals to Analyze for: **As, Pb**

Sample ID Sample Identifier :	Time	No. of Holes	QA/QC	Paint chips /Amount	Comments

COMMENTS:

ATTACHMENT C-3

ATTIC DUST IN-HOME SAMPLING DATA SHEET

Anaconda Smelter NPL Site – Community Soils Operable Unit
Attic Dust In-Home Sampling Data Sheet

Circle most correct answer(s)	
Date:	**Address:**
Team member:	**Area:**

Features of Attic				
Insulation	PACM vermiculite	fibrous glass batt, faced	fibrous glass batt, unfaced	Blown in, type:

Does the current attic electrical wiring meet code?	Yes		No	

Photos Checklist	Unique attic features	
	Sampling sites ( )	
	General attic configuration	
	Dust accumulation	
	Other:	

Sampling Information			
Sampler		Time	
Sample ID		Duplicate Collected? Y / N	
Pump Serial Number			

Pump Calibration, (L/min)	Date		Start		Stop		Average	

Sample Duration, (min)	
Comments:	

Location(s) Sampled:			
Location: Surface: Area Samples (cm^2):	Location: Surface: Area Samples (cm^2):	Location: Surface: Area Samples (cm^2):	Location: Surface: Area Samples (cm^2):

Lab Sample #:	
Comments:	

Instructions: Circle description(s) ( ) = Fill in number in category Type: specify in

ATTACHMENT D

LEAD DUST SAMPLING TECHNICIAN FIELD GUIDE

What Is the Field Guide?

This guide will help determine that a recently-renovated area has been cleaned sufficiently. The Lead Dust Sampling Technician Field Guide should be used by lead dust sampling technicians. The guide provides protocols for conducting post-renovation clearance under EPA's Renovation, Repair, and Painting Rule (RRP) covering housing and child-occupied facilities built before 1978, and clearance examinations under HUD's Lead Safe Housing Rule (LSHR) in federally-assisted housing built before 1978. This guide also provides federal standards for maximum allowable contamination levels of residual lead dust.

How To Use This Guide

Take this guide with you on site when you perform clearance, including visual inspections. It serves as a quick reminder of:

- When and where to take lead dust clearance samples;
- The step-by-step instructions for taking a dust wipe sample; and
- EPA/HUD clearance standards for lead dust.

When To Perform Lead Dust Clearance Tests

Renovation activities that disturb lead-based paint can create lead dust so proper cleanup after these jobs is critical. The purpose of lead dust clearance is to determine if the area is safe for re-occupancy.

Lead dust clearance is performed:

- After renovation, repair, painting, and cleaning activities are finished in property built before 1978 and where children are assumed to spend time.
- After hazard reduction or maintenance activities in most federally-assisted properties built before 1978 that are covered by HUD's LSHR.

Lead dust sampling technicians should NEVER perform post-abatement clearance. (Abatement—as opposed to renovation, repair and painting—is a term used for the complete removal of lead.) When performing clearance, the lead dust sampling technician is required to bring a copy of his or her certificate of initial training to the worksite.

Where To Collect Samples for Lead Dust Clearance Tests

If there is more than one room, hallway, or stairwell within the work area, take:

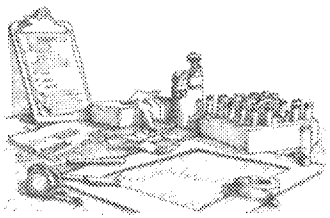
- One windowsill sample and one floor sample within each room, hall way, or stairwell (no more than four rooms, hallways, or stairwells need be sampled).
- If the windows were not closed and covered with plastic during the renovation, also take one window trough sample in each room, hall way, or stairwell (no more than four need be sampled).
- One floor sample adjacent to the work area, but not in an area that has been cleaned.

For federally-assisted housing, take these samples if the work area is contained, otherwise, clear the whole unit.

If the work area is a single room, hallway, or stairwell, or a smaller area, take:

- One windowsill sample and one floor sample.
- If the windows were not closed and covered with plastic during the renovation, also take one window trough sample.
- One floor sample adjacent to the work area, but not in an area that has been cleaned.

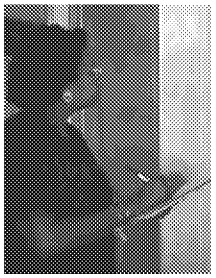
Equipment List



- Disposable lead dust wipes (individually wrapped)
- Disposable gloves
- Disposable shoe covers
- Sample tubes with caps
- Re-usable templates
- Masking or painter's tape
- Ruler
- Sample collection forms
- Chain-of-custody forms
- Markers, trash bags, labels, pens, re-sealable storage bags
- Calculator
- Sanitary wipes

Check with your laboratory for their sampling requirements

Visual Inspections



Lead dust clearance testing for both EPA's RRP Rule and HUD's LSHR requires a visual inspection as a first step in the clearance process:

- Under both HUD and EPA rules, the visual inspection is designed to determine if the area is free of visible dust and debris before lead dust clearance testing can begin.

In addition, under HUD's rule the visual inspection determines whether the unit/work area (interior and exterior) is clear of visible conditions that can result in exposure to lead-based paint hazards:

- Deteriorated paint
- Chips or debris
- Visible dust

Lead Dust Wipe Sampling

Single or composite samples can be taken; however, single-surface sampling is recommended to get results for specific surfaces. Use durable, re-usable 12" x 12" sampling templates, a disposable template, or use tape to lay out the sampling area.

Step One: Put on disposable shoe covers and lay out the sample area

- Clean template with a new wipe.
- Tape template to surface.
- If no template, outline with tape.
- Using tape to lay out the sample area, make sure that on floors the tape is laid in a square. On sills and troughs, the tape should be laid perpendicular to the sill.
- DO NOT touch the area inside the template.

Note: Use disposable shoe covers when walking between buildings and remove shoe covers before entering your vehicle to help minimize the spreading of settled lead dust from one location to another.

Step Two: Prepare the sample tubes

- Use clean tubes.
- Label tube with ID number.
- Record ID number on sample collection form and chain-of-custody form.
- Partially unscrew tube cap.
- Place tube near sample area.

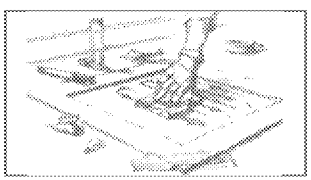
Step Three: Put on clean gloves

- Use disposable gloves.
- Use new gloves for each sample.
- DO NOT touch anything except the wipe after putting on the gloves.

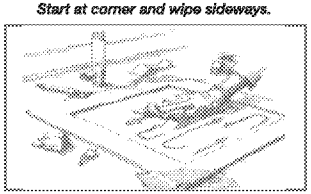
Step Four: Wipe sample area and place wipe in sample tube

- Do not touch other objects.
- Press the wipe down firmly at an upper corner of the sample area.
- Make as many "S"-like motions as needed to wipe the entire sample area, moving from side to side. Do not cross the outer border of the tape or template.
- Fold the wipe in half, keeping the dirty side in, and repeat the wiping procedure in the original direction in a forward and back motion.
- Fold the wipe again and repeat the wiping procedure, concetrating on collecting dust from the edges and corners of the sample area.

Floors



Start at corner and wipe sideways.



Now wipe in a forward and back motion.

Step Four: **(Continued)**

- Fold the wipe again with the sample side folded in, and place the folded wipe into the sample tube.
- Cap the container. Discard the gloves into a trash bag.
- Label the centrifuge tube and record the dimensions of the sampling area.

Step Five: **Measure the sample area**

- Measure width and length (unless template was used).
  - Length of sill or trough between edges of tape
  - Width of sill or trough, measure at tape
- Measure to 1/8 inch.
- Do not remove tape until after measurements are taken.

Step Six: **Record sample area dimensions on forms**

- Calculate the sample area and record on sample collection form and laboratory chain-of-custody form.

Step Seven: **Clean up**

- Clean template with a clean wipe; place in a plastic bag for storage.
- Remove materials from site:
  - Gloves, tape from floors and windows, and used shoe covers
  - Put items in trash bag, NOT in client's trash containers
- Clean face and hands with warm, soapy water.
  - Use sanitary wipes if you do not have access to warm, soapy water
- Send the samples to a laboratory recognized by the National Lead Laboratory Accreditation Program (NLLAP) as being proficient in lead in dust analysis. For information on locating EPA-accredited labs, visit http://www.epa.gov/lead/pubs/nllaplist.pdf.

Evaluate the Results

- Compare the laboratory results to the EPA clearance standards for maximum allowable residual lead dust provided below:
  - Floors: 40 micrograms per square foot ($\mu g/ft^2$)
  - Interior windowsills: 250 $\mu g/ft^2$
  - Window troughs: 400 $\mu g/ft^2$

These standards are for single-surface samples. The clearance standards for composite samples will be different depending on how many sub-samples are collected. Before collecting composite samples, check with your laboratory. Note that HUD discourages composite sampling when clearing federally-assisted housing.

Write the Report

- Use the standard report format.
- Sign the report.

Useful Resources

National Lead Information Center
1-800-424-LEAD (1-800-424-5323)
http://www.epa.gov/lead/pubs/nlic.htm

For a wide range of lead information—from outreach brochures to technical reports—on lead-based paint in the home.

National Lead Laboratory Accreditation Program
http://www.epa.gov/lead/pubs/nllaplist.pdf

For information on locating EPA-accredited labs.

Office of Pollution Prevention and Toxics
U.S. Environmental Protection Agency
1200 Pennsylvania Avenue, NW (7404T)
Washington, DC 20460
202-566-0500
http://www.epa.gov/lead

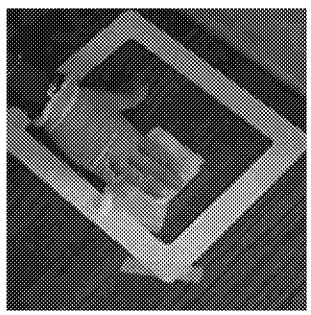
For information on EPA lead-based paint regulations.

Office of Healthy Homes and Lead Hazard Control
U.S. Department of Housing and Urban Development (HUD)
451 Seventh Street, SW
Washington, DC 20410
202-755-1785
http://www.hud.gov/offices/lead

For information on the HUD lead-based paint regulations and technical assistance in complying with the HUD regulations for HUD-funded work.

United States Environmental Protection Agency
Office of Pollution Prevention and Toxics
EPA-W-04-022
May 2009

Lead Dust Sampling Technician Field Guide



ATTACHMENT E

CORRECTIVE ACTION REPORT

Corrective Action Report/ Corrective Action Plan

Project ID	Project Name	Document ID

Preparer's Signature/Submit Date	Submitted to:

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

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Corrective Action Report/ Corrective Action Plan

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

ATTACHMENT F

LEVEL A/B SCREENING CHECKLIST

I. General Information

Site:
Project:
Client:
Sample Matrix:

II. Screening Results

Data are:

1) Unusable ☐
2) Level A ☐
3) Level B ☐

I. Level A Screening

Criteria – The following must be fully documented:	Yes/No	Comments
1. Sampling date	Yes	
2. Sampling team or leader	Yes	
3. Physical description of sampling location	Yes	
4. Sample depth (soils)	N/A	
5. Sample collection technique	Yes	
6. Field preparation technique	N/A	
7. Sample preservation technique	Yes	
8. Sample shipping records and laboratory analysis dates	Yes	
9. Companion sampling efforts	Yes	
10. Visual classification of samples	Yes	

II. Level B Screening

Criteria – The following must be fully documented:	Yes/No	Comments
1. Field/laboratory instrumentation, standardization and methods/procedures	Yes	
2. Proper sample containers and container preparation	Yes	
3. Collection of field replicates (1/20 minimum)	Yes	
4. Proper and decontaminated sampling equipment	N/A	
5. Identity of sample taker	Yes	
6. Field custody documentation	Yes	
7. Shipping custody documentation	Yes	
8. Traceable sample designation number	Yes	
9. Field notebooks, custody records in secure repository	Yes	

10. Properly prepared and complete field forms	Yes	
11. Physical data/observations date and time	Yes	
12. Physical data/observations recorder, team leader	Yes	
13. Physical data/observation location	Yes	

ED_014362_00000173-00275

ATTACHMENT G

QAPP CROSSWALK

EPA REGION 8 QA DOCUMENT REVIEW CROSSWALK

QAPP/FSP/SAP for: *(check appropriate box)*		**Entity** *(grantee, contract, EPA AO, EPA Program, Other)*	**Regulatory Authority and/or Funding Mechanism**	___**40 CFR 31 for Grants** ___**48 CFR Part 46 for Contracts** ___ **Interagency Agreement** ___ **EPA Administrative Order** ___ **EPA Program Funding** ___ **EPA Program Regulation** ___ **EPA CIO 2105**
	GRANTEE	PRP		
	CONTRACTOR			
	EPA			
X	**Other**			
Document Title *[Note: Title will be repeated in Header]*		Draft Final Residential Soils/Dust Quality Assurance Project Plan		
QAPP/FSP/SAP Preparer		Pioneer Technical Services, Inc.		
Period of Performance *(of QAPP/FSP/SAP)*		Fall 2015 through completion	**Date Submitted for Review**	01/12/16
EPA Project Officer **EPA Project Manager**		Charlie Coleman	**PO Phone #** **PM Phone #**	(406) 457-5038
QA Program Reviewer or Approving Official		Terry Moore	**Date of Review**	01/12/16

Documents to Review:

1. QAPP written by Grantee or EPA must also include for review:
   Work Plan(WP) / Statement of Work (SOW) / Program Plan (PP) / Research Proposal (RP)

2. QAPP written by Contractor must also include for review:
   a) Copy of signed QARF for Task Order
   b) Copy of Task Order SOW
   c) Made available hard or electronic copy of approved QMP
   d) If QMP not approved, provide Contract SOW

3. For a Field Sampling Plan (FSP) or Sampling & Analyses Plan (SAP), the Project QAPP must also be provided.
   OR
   The FSP or SAP must be clearly identified as a stand-alone QA document and must contain all QAPP required elements (Project Management, Data Generation/Acquisition, Assessment and Oversight, and Data Validation and Usability).

Documents Submitted for QAPP Review:

1. QA Document(s) submitted for review:

QA Document	Document Date	Document Stand-alone	Document with QAPP
QAPP		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 _____

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

1. Comment #1
2. Comment #2
3. Comment #3
4. **The PRP 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)".**

Element	Acceptable *Yes/No/NA*	Page/ Section	Comments
A. Project Management			
A1. Title and Approval Sheet			
a. Contains project title		Title Page	
b. Date and revision number line (for when needed)		Following TOC	
c. Indicates organization's name		Title Page	
d. Date and signature line for organization's project manager		Approval Page	Page i of vii
e. Date and signature line for organization's QA manager		Approval Page	Page i of vii
f. Other date and signatures lines, as needed		Approval Page	Page i of vii
A2. Table of Contents			
a. Lists QA Project Plan information sections		TOC	Page iii through vii
b. Document control information indicated		Following TOC	Page v of vii
A3. Distribution List			
Includes all individuals who are to receive a copy of the QA Project Plan and identifies their organization		Distribution List	Page ii of vii
A4. Project/Task Organization			
a. Identifies key individuals involved in all major aspects of the project, including contractors		Section 2.1 & Figure 1	
b. Discusses their responsibilities		Section 2.1	
c. Project QA Manager position indicates independence from unit generating data		Section 2.1	
d. Identifies individual responsible for maintaining the official, approved QA Project Plan		Section 2.1	
e. Organizational chart shows lines of authority and reporting responsibilities		Figure 1	
A5. Problem Definition/Background			
a. States decision(s) to be made, actions to be taken, or outcomes expected from the information to be obtained		Section 2.2	
b. Clearly explains the reason (site background or historical context) for initiating this project		Section 2.2	

c. Identifies regulatory information, applicable criteria, action limits, etc. necessary to the project		Section 2.4.2	
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		Section 2.3	
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		Section 2.3 & Table 1	
c. Details geographical locations to be studied, including maps where possible		Section 2.2 & Figure 2	
d. Discusses resource and time constraints, if applicable		N/A	Long term project.
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		Section 2.4	
b. Discusses precision		Sections 3.3.1 & 3.3.2 Attachments A-2 & A-3	
c. Addresses bias		Section 3.3.2 Attachments A-2 & A-3	
d. Discusses representativeness		Section 2.4.2	
e. Identifies the need for completeness		Section 2.4.2	
f. Describes the need for comparability		Section 2.4.2	

g. Discusses desired method sensitivity		Attachments A-2 & A-3	
A8. Special Training/Certifications			
a. Identifies any project personnel specialized training or certifications		Section 2.5	
b. Discusses how this training will be provided		Section 2.5	
c. Indicates personnel responsible for assuring training/certifications are satisfied		Section 2.5	
d. identifies where this information is documented		Section 2.5	
A9. Documentation and Records			
a. Identifies report format and summarizes all data report package information		Section 2.6.6	
b. Lists all other project documents, records, and electronic files that will be produced		Section 2.6	
c. Identifies where project information should be kept and for how long		Section 2.6.7	Long term project. Project information maintained indefinitely in GIS database.
d. Discusses back up plans for records stored electronically		Section 3.6	
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		Section 2.5	
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		Section 3.0	
b. Details the type and total number of sample types/matrix or test runs/trials expected and needed		Section 3.1	
c. Indicates where samples should be taken, how sites will be identified/located		Section 3.1	
d. Discusses what to do if sampling sites become inaccessible		Section 3.1.1.3	
e. Identifies project activity schedules such as each sampling event, times samples should be sent to the laboratory, etc.		Section 4.2 & Table 1	

f. Specifies what information is critical and what is for informational purposes only		Sections 2.6.2 & 2.6.7 Attachment F	
g. Identifies sources of variability and how this variability should be reconciled with project information		Sections 2.4.7, 3.3.2 & 4.1	
B2. Sampling Methods			
a. Identifies all sampling SOPs by number, date, and regulatory citation, indicating sampling options or modifications to be taken		Attachment A-1	
b. Indicates how each sample/matrix type should be collected		Attachment A-1	
c. If in situ monitoring, indicates how instruments should be deployed and operated to avoid contamination and ensure maintenance of proper data		N/A	
d. If continuous monitoring, indicates averaging time and how instruments should store and maintain raw data, or data averages		N/A	
e. Indicates how samples are to be homogenized, composited, split, or filtered, if needed		Attachment A-1	
f. Indicates what sample containers and sample volumes should be used		Attachment A-1	
g. Identifies whether samples should be preserved and indicates methods that should be followed		Attachment A-1	
h. Indicates whether sampling equipment and samplers should be cleaned and/or decontaminated, identifying how this should be done and by-products disposed of		Attachment A-1	
i. Identifies any equipment and support facilities needed		Attachment A-1	
j. Addresses actions to be taken when problems occur, identifying individual(s) responsible for corrective action and how this should be documented		Section 4.0 & Attachment A-1	
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		Section 5.2.2	
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)		Section 3.1 Attachments A-1, A-2 & A-3	
c. Indicates how sample or information handling and custody information should be documented, such as in field notebooks and forms, identifying individual responsible		Section 2.6 Attachment A-1	
d. Discusses system for identifying samples, for example, numbering system, sample tags and labels, and attaches forms to the plan		Sections 3.1.1.5.1, 3.1.1.5.2, 3.1.2.3 & 3.1.2.5	
e. Identifies chain-of-custody procedures and includes form to track custody		Section 2.6.4 Attachment A-1	
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		Section 3.2 Attachments A-2 & A-3	
b. Identifies equipment or instrumentation needed		Section 3.2 Attachments A-2 & A-3	
c. Specifies any specific method performance criteria		Attachments A-2 & A-3	
d. Identifies procedures to follow when failures occur, identifying individual responsible for corrective action and appropriate documentation		Section 4.1 Attachments A-2 & A-3	
e. Identifies sample disposal procedures		Attachments A-2 & A-3	
f. Specifies laboratory turnaround times needed		Not Specified	As needed to complete annual DSR and possible short turnaround time on occasion to meet RA schedule.

g. Provides method validation information and SOPs for nonstandard methods		Attachments A-2 & A-3	
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		Section 3.3 Attachments A-2 & A-3	
b. Details what should be done when control limits are exceeded, and how effectiveness of control actions will be determined and documented		Section 4.1 Attachments A-2 & A-3	
c. Identifies procedures and formulas for calculating applicable QC statistics, for example, for precision, bias, outliers and missing data		Attachments A-2 & A-3	
B6. Instrument/Equipment Testing, Inspection, and Maintenance			
a. Identifies field and laboratory equipment needing periodic maintenance, and the schedule for this		Attachments A-1, A-2 & A-3	
b. Identifies testing criteria		Attachments A-1, A-2 & A-3	
c. Notes availability and location of spare parts		Attachments A-1, A-2 & A-3	
d. Indicates procedures in place for inspecting equipment before usage		Attachments A-1, A-2 & A-3	
e. Identifies individual(s) responsible for testing, inspection and maintenance		Attachments A-1, A-2 & A-3	
f. Indicates how deficiencies found should be resolved, re-inspections performed, and effectiveness of corrective action determined and documented		Section 4.1 Attachments A-1, A-2 & A-3	
B7. Instrument/Equipment Calibration and Frequency			
a. Identifies equipment, tools, and instruments that should be calibrated and the frequency for this calibration		Attachments A-1, A-2 & A-3	

b. Describes how calibrations should be performed and documented, indicating test criteria and standards or certified equipment		Attachments A-2 & A-3	
c. Identifies how deficiencies should be resolved and documented		Section 4.1 Attachments A-1, A-2 & A-3	
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		Section 3.5 Attachments A-1, A-2 & A-3	
b. Identifies the individual(s) responsible for this		Section 4.1 Attachments A-1, A-2 & A-3	
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		Section 2.4.2	
b. Describes the intended use of this information and the rationale for their selection, i.e., its relevance to project		Sections 2.4.2, 2.4.6 & 3.1	
c. Indicates the acceptance criteria for these data sources and/or models		Sections 2.4.2, 2.4.6 & 3.1	
d. Identifies key resources/support facilities needed		Attachments A-1, A-2 & A-3	
e. Describes how limits to validity and operating conditions should be determined, for example, internal checks of the program and Beta testing		Sections 3.3 & 5.0 Attachments A-2 & A-3	
B10. Data Management			
a. Describes data management scheme from field to final use and storage		Sections 3.0, 4.0 & 5.0	

b. Discusses standard record-keeping and tracking practices, and the document control system or cites other written documentation such as SOPs		Sections 2.6, 3.0 & 4.0	
c. Identifies data handling equipment/procedures that should be used to process, compile, analyze, and transmit data reliably and accurately		Sections 2.6.5, 3.6	
d. Identifies individual(s) responsible for this		Section 5.1.1	
e. Describes the process for data archival and retrieval		Section 3.6	
f. Describes procedures to demonstrate acceptability of hardware and software configurations		Section 3.6	
g. Attaches checklists and forms that should be used		Section 3.6	
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		Section 4.0	
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		Section 4.0	
c. Describes how and to whom assessment information should be reported		Section 4.0	
d. Identifies how corrective actions should be addressed and by whom, and how they should be verified and documented		Section 4.0	
C2. Reports to Management			
a. Identifies what project QA status reports are needed and how frequently		Section 4.3	
b. Identifies who should write these reports and who should receive this information		Section 4.3	
D. Data Validation and Usability			
D1. Data Review, Verification, and Validation			
Describes criteria that should be used for accepting, rejecting, or qualifying project data		Section 5.0	
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		Section 5.1	
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.		Section 5.1	
c. Identifies issue resolution process, and method and individual responsible for conveying these results to data users		Section 5.1	
d. Attaches checklists, forms, and calculations		Section 5.0 Attachment F	
D3. Reconciliation with User Requirements			
a. Describes procedures to evaluate the uncertainty of the validated data		Section 5.0	
b. Describes how limitations on data use should be reported to the data users		Section 5.0	