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

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Atlantic Richfield Company

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November 5, 2021

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Helena, Montana 59620-0901

RE: Draft Final RMAP School Sampling Field Sampling Plan (FSP) Submittal #8 (Covering Additional Investigation at Butte High School/Annex)

Dear Agency Representatives:

I am writing to you on behalf of Atlantic Richfield Company to submit the *Draft Final Residential Metals Abatement Program (RMAP) School Soil Sampling Field Sampling Plan Submittal #8* which addresses additional soil sampling at Butte High School/Annex under the RMAP program. The report, tables, and figures may be downloaded at the following link:

https://pioneertechnicalservices.sharepoint.com/:f:/s/submitted/EnQS8xMofq1Milr47LpMqw4B_gB7Yep1MP7jHHaUiJGyCA

If you have any questions or comments, please call me at (406) 723-1822.

Sincerely,

Mike McAnulty

Mike Mc Anulty
Liability Manager
Remediation Management Services Company
An affiliate of **Atlantic Richfield Company**

cc: Patricia Gallery / Atlantic Richfield - email
Chris Greco / Atlantic Richfield – email
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Loren Burmeister / Atlantic Richfield – email
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Becky Summerville / MR - email
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Leo Berry / BNSF - email
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Connie Logan/ Pioneer – email
Ian Magruder/ CTEC- email
CTEC of Butte – email
Scott Juskiewicz / Montana Tech – email

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

Draft Final

2021 Residential Metals Abatement Program (RMAP) School Soil Sampling Field Sampling Plan (FSP)
Submittal #8
[Covering Additional Investigation at Butte High School/Annex]

Butte-Silver Bow County

and

Atlantic Richfield Company

November 5, 2021

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

Draft Final

2021 Residential Metals Abatement Program (RMAP) School Soil Sampling Field Sampling Plan (FSP)
Submittal #8
[Covering Additional Investigation at Butte High School/Annex]

Prepared for:

Butte-Silver Bow County
Superfund Division
155 W. Granite
Butte, Montana 59701

and

Atlantic Richfield Company
317 Anaconda Road
Butte, Montana 59701

Prepared by:

Pioneer Technical Services, Inc.
1101 S. Montana Street
Butte, Montana 59701

November 5, 2021

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DOCUMENT MODIFICATION SUMMARY

Modification	Author	Version	Description	Date
0	Jesse Schwarzrock	Draft Final	Issued for Agency Review	11/05/21

1.0 INTRODUCTION

This Field Sampling Plan (FSP) was developed to outline a portion of the 2021 Residential Metals Abatement Program (RMAP) school soil sampling plan for Butte, Montana, area schools. Soil sampling procedures, data quality objectives (DQOs), standard operating procedures (SOPs), sampling analytical methods, sampling equipment, quality control (QC) samples, and data validation and assessment will be in accordance with the *Final Residential Metals Abatement Program (RMAP) Quality Assurance Project Plan* (QAPP) *(Non-Residential Parcels)* (Butte-Silver Bow County and Atlantic Richfield Company, 2021a) provided in Attachment A.

2.0 SCHOOL SOIL SAMPLING SCOPE

The scope of work covered by this FSP includes the following school(s):

- Butte High School/Annex.

Butte High School was sampled per *2021 Residential Metals Abatement Program (RMAP) School Soil Sampling Field Sampling Plan (FSP) Submittal #4* (Butte-Silver Bow County and Atlantic Richfield Company, 2021b) between August 2, 2021 and August 10, 2021. As a result of this sampling event, remedial action construction was determined to be needed within polygons GA3, GA5, and GA7 (see Individual Site Work Plan in Attachment B). Excavation of soil within GA3 in October 2021 revealed visually suspect material along the eastern boundary of GA3 extending north into GA2. At Agency direction, a small amount of soil was removed from the southeast corner of GA2 (see Figure 2). This additional excavation revealed more visually suspect material in both the northern and western vertical faces of the excavation footprint. At this point, Agency representatives requested additional sampling in this portion of GA2 to quantify any potential additional removals.

The attached figure set depicts the additional investigation area to be sampled as well as the August 2021 sampling polygons and subsample locations. Table 1 lists the school properties (along with Resident ID’s, geocodes, ownership information, and age of school), and Table 2 shows the anticipated sampling quantities for the school(s) covered by this FSP.

3.0 SCHOOL SOIL SAMPLING SCHEDULE

Sampling schedules will be finalized through ongoing conversations with appropriate school representatives. Ideally, this additional sampling investigation will be conducted during November 2021. The appropriate utility locating service (i.e., One Call Utility Locate Services) will be contacted and informed of sampling activities 48 hours prior to commencing soil sampling activities.

4.0 FIELD SAMPLING PLAN

4.1 Soil Sampling Procedures

Soil sampling procedures will be completed as stated in Section 3.2.2 of the QAPP (Attachment A).

4.1.1 Soil Sampling Density, Location and Compositing

Soil sampling density, location and compositing decisions will be made according to the information provided in Section 3.2.1 of the QAPP (Attachment A).

4.1.2 Soil Sampling Depths

Sampling depths will be selected as stated in Section 3.2.2 of the QAPP (Attachment A).

4.1.3 Equipment Decontamination

This sampling effort will primarily use disposable sampling equipment (disposable scoops, etc.) Re-usable equipment such as shovels will be decontaminated between sampling sites as described in Section 3.2.4 of the QAPP (Attachment A).

4.2 Sampling Polygon Delineation

Below is documentation detailing the reasoning behind the delineation of sampling polygons. This information was collected from school representatives as well as school site visits. Field sampling crews will have the flexibility to make minor modifications to these polygons in the field as needed in consultation with the Agencies’ field representative.

4.2.1 Butte High School/Annex

- Land Use Category #1 (Playgrounds) – this land use category is not applicable to the additional investigation work at Butte High School.

- Land Use Category #2 (high access areas/barren sports areas) – this land use category is not applicable to the additional investigation work at Butte High School.

- Land Use Category #3 (grass areas/turf covered sports fields) – as outlined in Section 2.0, visually suspect material is located within the southeast corner of sampling polygon GA2. The additional investigation will focus on this area. The additional investigation area depicted in Figure 2 is bounded to the south by the GA2 polygon boundary and to the east by the property boundary. The northern and western boundaries are determined by existing mature tree canopies. School officials have expressed on multiple occasions how few trees they have on campus and how important they are to the school district. As such, it is appropriate to stop the additional sampling boundary before getting into areas with dense tree roots.

- Land Use Category #4 (low access areas/low maintenance areas) – this land use category is not applicable to the additional investigation work at Butte High School.

- Land Use Category #5 (garden areas) – this land use category is not applicable to the additional investigation work at Butte High School.

4.3 Deviations

This section addresses any deviations to the Agencies-approved QAPP (Attachment A) pertaining to 2021 BPSOU RMAP school soil sampling in or near Butte. Deviations include the following:

- No known deviations at this time.
- Any future deviations will be discussed with the Agencies' field representative, documented in the field, and addressed through forthcoming Data Summary Reports.

5.0 LABORATORY METHODS

5.1 Soil Metals Analyses Methods

Soil metals analyses will be conducted as stated in Section 3.5 of the QAPP (Attachment A).

6.0 QUALITY CONTROL

6.1 Field Quality Control Samples

Field QC will be conducted as stated in Sections 3.6 and 3.6.1 of the QAPP (Attachment A).

7.0 REFERENCES

Butte-Silver Bow County and Atlantic Richfield Company, 2021a. Silver Bow Creek/Butte Area NPL Site Butte Priority Soils Operable Unit. Final Residential Metals Abatement Program (RMAP) Quality Assurance Project Plan (QAPP). July 2021.

Butte-Silver Bow County and Atlantic Richfield Company, 2021b. Final Residential Metals Abatement Program (RMAP) School Soil Sampling Field Sampling Plan (FSP) Submittal #4. July 2021.

FIGURES

LEGEND

- 🟥 LAND USE CATEGORY #1 (PLAYGROUND AREAS)
- ▢ LAND USE CATEGORY #2 (HIGHLY ACCESSIBLE AREAS/BARREN SPORTS FIELDS)
- 🟩 LAND USE CATEGORY #3 (MAINTAINED GRASS AREAS/GRASS SPORTS FIELDS)
- 🟦 LAND USE CATEGORY #4 (LOW ACCESS AREAS/LOW MAINTENANCE AREAS/OPEN SPACE)
- 🟨 LAND USE CATEGORY #5 (GARDEN AREAS)
- ▨ OPPORTUNISTIC SAMPLES
- ▢ NON-SAMPLING AREAS
- ☐ SAMPLING POLYGON BOUNDARY
- ☐ PROPERTY BOUNDARY
- ● BPSOU RMAP SCHOOL SUB-SAMPLE LOCATION

N

DISPLAYED AS:
PROJECTION/ZONE: MSP
DATUM: NAD 83
UNITS: FEET
SOURCE: PIONEER

0 75 150 300
Feet

FIGURE 1

BUTTE HIGH SCHOOL ADDITIONAL INVESTIGATION FIELD SAMPLING PLAN

PIONEER TECHNICAL SERVICES, INC.

DATE: 11/3/2021

Path: Z:\Shared\Active Projects\ARCO\BPSOU\RMAP\GIS\School RAWP Figures\ButteHigh\Additional Investigation\RMAP_AI_ButteHighSchool.mxd

LEGEND

- ☒ NO ACTION AREA PER AUGUST 2021 SAMPLING EVENT
- ▨ REMEDIATED IN OCTOBER 2021
- ▭ PROPOSED ADDITIONAL INVESTIGATION AREA
- ☐ SAMPLING POLYGON BOUNDARY
- ● AUGUST 2021 BPSOU RMAP SCHOOL SUB-SAMPLE LOCATION
- ▲ PROPOSED APPROXIMATE SAMPLING LOCATIONS

DISPLAYED AS:
PROJECTION/ZONE: MSP
DATUM: NAD 83
UNITS: FEET
SOURCE: PIONEER

FIGURE 2

PIONEER
TECHNICAL SERVICES, INC.

BUTTE HIGH SCHOOL
ADDITIONAL INVESTIGATION
FIELD SAMPLING PLAN

DATE: 11/4/2021

Path: Z:\Shared\Active Projects\ARCO\BPSOU\RMAP\GIS\School RAWP Figures\ButteHigh\Additional Investigation\RMAP_AI_ButteHighSchool2.mxd

TABLES

TABLE 1: 2021 RMAP SCHOOL SOIL SAMPLING PROPERTY LIST
(FSP Submittal #8)

Count	Res-ID	Geocode	Name	Owner	Construction Date
1	S-0009	01119713454100000	Butte High School/Annex	School District #1	1937/1968

TABLE 2: 2021 RMAP SCHOOL
SOIL SAMPLING QUANTITY SUMMARY
(FSP Submittal #8)

Figure #	Res-ID	School/Park	Owner	Total Area (SF)	Polygon ID	Polygon Areas (SF)	# of Subsample Locations	# of Composite Samples	0-2"	2-6"	6-12"	12-18"	18-24"	Notes
1	S-0009	Butte High School/Annex	School District #1	842	GA2, Subarea 1	842	5	3	As/Pb/Hg	As/Pb/Hg	As/Pb/Hg	-	-	-
			Totals (SF):	**842**	**-**	**842**	**5**	**3**						
			Totals (AC):	**0.02**	**-**	**0.02**								

ATTACHMENT A

FINAL RMAP QAPP

(NON-RESIDENTIAL PARCELS)

Atlantic Richfield Company

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July 9, 2021

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Jonathan Morgan, Esq.
DEQ, Legal Counsel
P.O. Box 200901
Helena, Montana 59620-0901

RE: Final RMAP QAPP (Non-Residential RMAP Parcels)

Dear Agency Representatives:

I am writing to you on behalf of Atlantic Richfield Company to submit the Final Residential Metals Abatement Program (RMAP) Quality Assurance Project Plan (QAPP) (Non-Residential RMAP Parcels) which addresses schools, parks, and non-residential daycares under the RMAP program for your records. This submittal addresses EPA's comments from the July 8, 2021 Conditional Approval Letter. The report and appendices may be downloaded at the following link:

https://pioneertechnicalservices.sharepoint.com/:f:/s/submitted/EgiAkGHgzaRGpiq-jEk5aHEB9Qj5Q6IHKwYcbqvuXpUYmg

If you have any questions or comments, please call me at (406) 723-1822.

Sincerely,

Mike McAnulty

Mike Mc Anulty
Liability Manager
Remediation Management Services Company
An affiliate of **Atlantic Richfield Company**

cc: Patricia Gallery / Atlantic Richfield - email
Chris Greco / Atlantic Richfield – email
Mike Mc Anulty / Atlantic Richfield - email
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Ray Vinkey / NRDP - email
Harley Harris / NRDP - email
Katherine Hausrath / NRDP - email
Meranda Flugge / NRDP - email
Ted Duaime / MBMG - email
Gary Icopini / MBMG - email
Becky Summerville / MR - email
Kristen Stevens / UP - email
Robert Bylsma / UP - email
John Gilmour / Kelley Drye - email
Leo Berry / BNSF - email
Robert Lowry / BNSF - email
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BPSOU SharePoint - upload

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Ref: 8MO

July 8, 2021

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

Re: Approval letter for the Butte Priority Soils Operable Unit (BPSOU) Draft Final Residential Metals Abatement Program (RMAP), Quality Assurance Project Plan (QAPP), Non-Residential Parcels (dated July 7, 2021)

Dear Mike:

The U. S. Environmental Protection Agency (EPA), in consultation with the Montana Department of Environmental Quality (DEQ), is approving the *Draft Final Residential Metals Abatement Program (RMAP), Quality Assurance Project Plan, Non-Residential Parcels (dated July 7, 2021),* with the following comments:

- Section 2.7.1, DQO Step 3: Please revise the text to state that XRF data can be validated, or simply delete the following sentence - “Also, data validation cannot be completed on XRF data.”

  Atlantic Richfield Company Response (7/9/21) – Comment addressed by removing requested sentence.

- Section 2.7.1, DQO Step 3: The term “wet chemistry” is normally associated with parameters such as fluoride, chloride, etc. Please change “wet chemistry” to “inorganic analyses” to reference the arsenic, lead and mercury analyses to be conducted.

  Atlantic Richfield Company Response (7/9/21) – Comment addressed.

- Section 2.7.1, DQO Step 3: Please provide more information and detail regarding that statement that “expedited laboratory analysis and data validation options should be investigated.” For example, it is understood that data validation will be performed in 7 days after the data package is received. Please specify here and in subsequent QAPP sections (i.e., Section 5.3) the desired laboratory analysis and data validation turnaround times.

Atlantic Richfield Company Response (7/9/21) – Comment addressed.

- Section 2.9.2: As discussed on the July 8, 2021 conference call, please expand the discussion describing the process for agency approval of major deviations from the SOP or QAPP.

  Atlantic Richfield Company Response (7/9/21) – Comment addressed.

- Section 5.0: The text states that "Internal field and laboratory audits will be performed by AtlanticRichfield or their approved representative, BSB, their contractor(s), or a contracted laboratory as necessary." Please specify the conditions for which an audit will be deemed necessary. Please provide additional details on the internal and laboratory audits that will be completed as part of the non-residential exterior sampling effort. For example, please specify how many audits will beperformed and when they will be conducted.

  Atlantic Richfield Company Response (7/9/21) – Comment addressed.

- Table 3: The criteria limits specified in Table 3 for LCS and MS samples are more stringent than the limits set by the EPA National Functional Guidelines, which indicate an acceptable range of 70-130% for LCS and 75-125% for mercury MS. Please clarify if the more stringent limits are a project-specific requirement. Note that use of more stringent requirements has the potential to result in more qualified data, which may influence the specification of screening versus enforcement quality data.

  Atlantic Richfield Company Response (7/9/21) – Comment addressed. Table 3 has been updated.

- Please update the date on the document prior to distribution.

  Atlantic Richfield Company Response (7/9/21) – This Final document has been dated 7/9/21.

- If the content or the technical approach provided in the plan has changed or requires modification, please submit the revised plan to EPA and DEQ for review and approval.

  Atlantic Richfield Company Response (7/9/21) – No changes to content or technical approach beyond Agency requested updates in this memo.

- Please address comments and distribute the Final QAPP with the attached signature/approval page and the EPA approved crosswalk.

  Atlantic Richfield Company Response (7/9/21) – Comment addressed.

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

Sincerely,

NIKIA
GREENE

Digitally signed by NIKIA
GREENE
Date: 2021.07.08
20:11:53 -06'00'

Nikia Greene

Remedial Project Manager

Attachments:
EPA crosswalk
EPA and MDEQ Signature Page

cc: (email only)
Butte File
Jenny Chambers; DEQ
Matt Dorrington, DEQ
Daryl Reed; DEQ
Jon Morgan; DEQ counsel
Carolina Balliew; DEQ
Harley Harris; NRDP
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Loren Burmeister; AR
Josh Bryson; AR
Mike Mcanulty; AR
Dave Griffis; AR
Jean Martin; Counsel AR
Mave Gasaway; attorney for AR
Adam Cohen; Counsel for AR
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Jean Belille; EPA
Ian Magruder; CTEC (Tech Advisor)
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Ref: 8MO

July 8, 2021

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

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- Section 2.7.1, DQO Step 3: Please provide more information and detail regarding that statement that “expedited laboratory analysis and data validation options should be investigated.” For example, it is understood that data validation will be performed in 7 days after the data package is received. Please specify here and in subsequent QAPP sections (i.e., Section 5.3) the desired laboratory analysis and data validation turnaround times.
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- Section 5.0: The text states that “Internal field and laboratory audits will be performed by Atlantic Richfield or their approved representative, BSB, their contractor(s), or a contracted laboratory as necessary.” Please specify the conditions for which an audit will be deemed necessary. Please provide additional details on the internal and laboratory audits that will be completed as part of the

non-residential exterior sampling effort. For example, please specify how many audits will be performed and when they will be conducted.

- Table 3: The criteria limits specified in Table 3 for LCS and MS samples are more stringent than the limits set by the EPA National Functional Guidelines, which indicate an acceptable range of 70-130% for LCS and 75-125% for mercury MS. Please clarify if the more stringent limits are a project-specific requirement. Note that use of more stringent requirements has the potential to result in more qualified data, which may influence the specification of screening versus enforcement quality data.
- Please update the date on the document prior to distribution.
- If the content or the technical approach provided in the plan has changed or requires modification, please submit the revised plan to EPA and DEQ for review and approval.
- Please address comments and distribute the Final QAPP with the attached signature/approval page and the EPA approved crosswalk.

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

Sincerely,
NIKIA GREENE
Digitally signed by NIKIA GREENE
Date: 2021.07.08 20:11:53 -06'00'
Nikia Greene
Remedial Project Manager

Attachments:
EPA crosswalk
EPA and MDEQ Signature Page

cc: (email only)
Butte File
Jenny Chambers; DEQ
Matt Dorrington, DEQ
Daryl Reed; DEQ
Jon Morgan; DEQ counsel
Carolina Balliew; DEQ
Harley Harris; NRDP
Katherine Hausrath; NRDP
Jim Ford; NRDP
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Anne Walsh; UP
Robert Bylsma; UP counsel
Leo Berry; BNSF and UP counsel
Mark Engdahl; BNSF
Brooke Kuhl; BNSF counsel
Jeremie Maehr; Kennedy Jenks for BNSF and UP
Annika Silverman; Kennedy Jenks for BNSF and UP
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Becky Summerville; counsel for Inland Properties Inc.
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Mike Mcanulty; AR
Dave Griffis; AR
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Scott Bradshaw; TREC
Mike Borduin; Pioneer for AR
Karen Helfrich; Pioneer for AR
Andy Dare; Pioneer for AR
Scott Sampson; Pioneer for AR
Brad Archibald; Pioneer for AR
Andy Dare; Pioneer for AR
Tina Donovan; Woodardcurran for AR
Don Booth; AR consultant
Ted Duaime; MBMG
Gary Icopini; MBMG
David Shanight, CDM Smith
Curt Coover, CDM Smith
Chapin Storrar; CDM Smith
Erin Agee, EPA
Joe Vranka; EPA
Chris Wardell; EPA
Dana Barnicoat; EPA
Charlie Partridge; EPA
Jean Belille; EPA
Ian Magruder; CTEC (Tech Advisor)
Janice Hogan; CTEC
Kristi Carroll; Montana Tech Library

UNITED STATES ENVIRONMENTAL PROTECTION AGENCY
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Ref: 8MO

June 22, 2021

Mr. Eric Hassler
Superfund Program Data Administrator
Butte-Silver Bow Planning Department
155 West Granite Street
Butte, MT 59701

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

Re: Comments for: BPSOU Draft Final Residential Metals Abatement Program (RMAP) Quality Assurance Project Plan (QAPP) – Non-Residential Parcels (aka schools and parks), dated June 7, 2021

Dear Eric and Mike:

The U. S. Environmental Protection Agency (EPA), in consultation with the Montana Department of Environmental Quality (DEQ), is providing comments on the *Draft Final Residential Metals Abatement Program (RMAP) Quality Assurance Project Plan (QAPP) – Non-Residential Parcels (dated June 7, 2021)* that was prepared by Pioneer Technical Services, Inc., on behalf of the Butte-Silver Bow County (BSB) and Atlantic Richfield Company.

Comments have been stratified into the follow sections – General Comments, Specific Comments, Comments on Figures, Tables, and Attachments, and Minor/Editorial Changes. Those comments that have the potential to change the study design or approach are indicated in **bold** text and may require additional discussion. Please incorporate these comments and submit the final version of the report for review.

General Comments:

1. The Residential RMAP QAPP was used as the basis for this document; however, there are several aspects of schools that differ from homes both with regard to terminology and area use (e.g., living space, attic dust, yard soil). While most references to “yard” or “attics” have been removed, remaining residential-specific language and references to indoor media, such as dust and paint, should be removed as well.

   Atlantic Richfield Company Response (6/30/21) – Comment addressed.

2. Please clarify if there have been any previous sampling efforts conducted at the non-residential properties anticipated for evaluation as part of this QAPP. If so, the QAPP should specify what

those results show (or indicate that this information would be included in the property-specific field sampling plan [FSP]).

Atlantic Richfield Company Response (6/30/21) – Comment addressed in Section 3.2.3.

3. Mentions of lead-based paint (LBP) should be clear that it is specific to the exterior of the schools (i.e. chance of contaminating or re-contaminating soils. Please remove reference to lead solder for Non-Residential sections. Any interior work will require a revision or a new QAPP.

   Atlantic Richfield Company Response (6/30/21) – Comment addressed. All lead based paint references have been removed (per Agency comment #1) with the exception of Section 2.7.1 Step 3 (which references the likelihood of lead based paint chips influencing lead soil concentrations). All solder references have been removed.

4. Please ensure the QAPP is consistent in that all three metals (arsenic, lead, and mercury) are required to be analyzed in all samples.

   Atlantic Richfield Company Response (6/30/21) – Comment addressed.

5. Please ensure that supplemental text provided by Lester Dupes in the June 10, 2021 email regarding the preparation and analysis of samples for elemental mercury are incorporated into the final version of the QAPP.

   Atlantic Richfield Company Response (6/30/21) – Comment addressed.

6. **In the past, RMAP investigations have sieved soils to 250 micrometers (µm). However, more recent EPA guidance (EPA OLEM Directive 9200.1-128) requires sieving to 150 µm. For the purposes of the Non-Residential RMAP QAPP, given the schedule constraints, EPA agrees with targeting a particle size of 250 µm for the 2021 investigation. The DQOs and study design of this QAPP can reflect this agreement. However, EPA requires the performance of a demonstration pilot study to assess potential differences in enrichment between the 250-µm and 150-µm size fractions before use of the 250-µm fraction will be approved for broader use in other RMAP investigations.**

   Atlantic Richfield Company Response (6/30/21) – Section 3.2.5 updated to address comment.

   A statement has been added to the QAPP regarding the temporary agreement to use the less than 250 µm fraction, explaining the OLEM directive, the possibility of enrichment, and the plan for a demonstration study. We will work with EPA to develop a mutually acceptable plan for a pilot study to assess potential enrichment between the <150 micron and <250 micron soil fractions. OLEM Directive 9200.1-128 9 (USEPA 2016) cites 20 articles reporting higher lead concentrations in finer fractions of soil, but only one of those articles (Juhasz et al. 2011) included data for sieve sizes close to those of interest. Juhasz evaluated 16 soil samples from a range of mining/smelting sites, shooting ranges, incinerators, a gas works and historical fill areas for particle size fractions of <50, <100, <250, and <2000 microns. When we considered the enrichment in six samples with lead concentrations between 250 and 2,000 mg/kg, a range that is most relevant to most sites, the enrichment for the <100 vs. <250 micron size fractions ranged from 1.04 to 1.10 with a mean of 1.08. Based on the data reported by Juhasz et al., enrichment of lead concentrations in soil samples in the concentration range of interest (i.e., 250 ppm to 2,000 ppm) is predicted to be less than 10%, suggesting that the use of the finer sieve size may not be a significant factor in increasing lead concentrations.

USEPA. 2016. Recommendations for sieving soil and dust samples at lead sites for assessment of incidental ingestion. OLEM Directive 9200.1-128. U.S. Environmental Protection Agency, Assessment and Remediation Division, Office of Superfund Remediation and Technology Innovation. July.

Juhasz AL, Weber J, Smith E. 2011. Impact of Soil Particle Size and Bioaccessibility on Children and Adult Lead Exposure in Peri-Urban Contaminated Soils. Journal of Hazardous Materials, 186: 1870-1879.

Specific Comments:

1. Distribution List, page ii – Please add contact information for the Field Team Leader and the analytical laboratory(ies).

   Atlantic Richfield Company Response (6/30/21) – Comment addressed.

2. Section 1.0, page 1 – In the introduction section where the overall RMAP is being described, please also mention the use of medical monitoring to evaluate the effectiveness of the program.

   Atlantic Richfield Company Response (6/30/21) – Comment addressed.

3. Section 2.5, Problem Definition and Background – Mercury should be identified to be elemental mercury in this section and throughout the report where appropriate.

   Atlantic Richfield Company Response (6/30/21) – Comment addressed.

4. Section 2.6, page 5 – This section should focus on the non-residential aspects of the RMAP and references to residential-specific considerations should be removed.

   Atlantic Richfield Company Response (6/30/21) – Comment addressed.

5. Section 2.6.1, page 6 – This section should specify the desire (with certain caveats about unknowns and schedule changes) to complete outdoor sampling and remediation (if needed) prior to the school fall session beginning.

   Atlantic Richfield Company Response (6/30/21) – Comment addressed.

6. Section 2.7.1, DQO Step 3 – There are additional information inputs that should be listed as part of this step. For example, information will be needed on the land use of the different areas within the

parks and schools should be provided. In addition, information will also be needed on the sampling locations and the area that each sample represents (i.e., the field teams will need to document the sample coordinates and generate field sketches or map polygons to document each sampling unit).

Atlantic Richfield Company Response (6/30/21) – Comment addressed.

7. Section 2.7.1, DQO Step 3 – Please explain why residential action levels have been selected for use (i.e., there are no school-specific action levels and residential were selected to be conservative).

Atlantic Richfield Company Response (6/30/21) – Comment addressed.

8. Section 2.7.1, DQO Step 3 –
    a. Given that one of the concerns for the schools is being able to assess and, if necessary, remediate areas this summer, it is unclear why use of x-ray fluorescence (XRF) analysis for lead and arsenic is not discussed as a potentially viable analytical method. This section should discuss the potential use of XRF and why this analytical method was not selected for use (considering this has been the preferred RMAP analytical method up to this point). This section should also discuss how EPA has indicated that XRF is not a viable analytical method for analysis of mercury.

Atlantic Richfield Company Response (6/30/21) – Comment addressed.

    b. Please discuss the required laboratory turn-around times that will be necessary to achieve the assessment/remediation goals for non-residential properties.

Atlantic Richfield Company Response (6/30/21) – Comment addressed.

    c. **This section should discuss any sample collection/preparation requirements (e.g., sieving, drying, storage temperature). Specifically, the fact that past RMAP investigations have sieved to 250 µm and that more recent EPA guidance (EPA OLEM Directive 9200.1-128) requires sieving to 150 µm should be discussed here and EPA's temporary agreement with use of 250 µm pending the outcome of a particle size enrichment demonstration study.**

Atlantic Richfield Company Response (6/30/21) – A statement has been added to the QAPP regarding the temporary agreement to use the less than 250 µm fraction, explaining the OLEM directive, the possibility of enrichment, and the plan for a demonstration study. We will work with EPA to develop a mutually acceptable plan for a pilot study to assess potential enrichment between the <150 micron and <250 micron soil fractions. OLEM Directive 9200.1-128 9 (USEPA 2016) cites 20 articles reporting higher lead concentrations in finer fractions of soil, but only one of those articles (Juhasz et al. 2011) included data for sieve sizes close to those of interest. Juhasz evaluated 16 soil samples from a range of mining/smelting sites, shooting ranges, incinerators, a gas works and historical fill areas for particle size fractions of <50, <100, <250, and <2000 microns. When we considered the enrichment in six samples with lead concentrations between 250 and 2,000 mg/kg, a range that is most relevant to most sites, the enrichment for the <100 vs. <250 micron size fractions ranged from 1.04 to 1.10 with a mean of 1.08. Based on the data reported by Juhasz et al., enrichment of lead concentrations in soil samples in the concentration range of interest (i.e., 250 ppm to 2,000 ppm) is predicted to be less than 10%, suggesting that the use of the finer sieve size may not be a significant factor in increasing lead concentrations.

USEPA. 2016. Recommendations for sieving soil and dust samples at lead sites for assessment of incidental ingestion. OLEM Directive 9200.1-128. U.S. Environmental Protection Agency, Assessment and Remediation Division, Office of Superfund Remediation and Technology Innovation. July.

Juhasz AL, Weber J, Smith E. 2011. Impact of Soil Particle Size and Bioaccessibility on Children and Adult Lead Exposure in Peri-Urban Contaminated Soils. Journal of Hazardous Materials, 186: 1870-1879.

d. This section should specify if there are any constraints as to the types of sampling designs that will be considered for this program (e.g., incremental, composite, discrete). If project objectives can be accomplished by multiple sampling designs, please discuss why one design might be preferred over another (e.g., use of incremental or composite collection methods would result in lower analytical costs relative to discrete methods).

Atlantic Richfield Company Response (6/30/21) – Comment addressed.

9. Section 2.7.1, DQO Step 4 – The DQOs should specify why each of these different depth intervals are being targeted. The text should discuss if the need for different depth intervals is related to differences in exposure potential and/or if this is being done to help refine removal extents.

Atlantic Richfield Company Response (6/30/21) – Comment addressed.

10. Section 2.7.1, DQO Step 4 – The inclusion of a vegetable garden land use seems more specific to a residential scenario. Please confirm if this category is relevant to non-residential (e.g., schools, parks) and, if not, please modify the land use category appropriately.

Atlantic Richfield Company Response (6/30/21) – While not present at every location, this category is relevant to non-residential (e.g., schools, parks) RMAP parcels. While school outreach meetings are still underway, vegetable gardens have been documented at the Silver Bow Montessori School in Butte.

11. Section 2.7.1, DQO Step 4 – Please revise this section to include a more expansive discussion on sampling density requirements for each land-use category.

Atlantic Richfield Company Response (6/30/21) – Comment addressed.

12. Section 2.7.1, DQO Step 4 –

    a. This section should discuss any temporal requirements on the sampling, not the remediation (i.e., specify if there are any temporal constraints on the sampling investigation). Specifically, this section should discuss the need to assess all schools prior to school starting in the fall. Additionally, a discussion of the challenges of unknowns and schedule changes could be useful here.

    Atlantic Richfield Company Response (6/30/21) – Comment addressed.

    b. No temporal variability in soil concentrations is expected, so the sampling effort should be primarily dictated by when it is easiest to conduct sampling, meaning when no snow is present and when school facilities are not in use (i.e., summer).

    Atlantic Richfield Company Response (6/30/21) – Comment addressed.

13. Section 2.7.1, DQO Step 4 – The sampling design has not yet been established at this stage in the DQO process (the study design is established in Step 7). Thus, stating that the decision unit (DU) is equal to the extent of a single composite soil sample does not have inherent meaning. If the goal is to make remedial decisions on a sample-by-sample basis, this section should discuss how the DU will be set equal to the sampling unit (SU) and the SU extent should be specified as the maximum area for decision-making by land use type (i.e., for playgrounds the DU/SU size is 6,250square feet).

    Atlantic Richfield Company Response (6/30/21) – Comment addressed.

14. Section 2.7.1, DQO Step 6 – At this stage in the DQO process, the sampling design has not yet been selected (the study design is established in Step 7). The tolerable limits should not be dictated by the selected study design, rather the selected study design should be developed based on the performance criteria. Tolerable limits for decision errors should be specified whenever the problem question is a decision question. Please modify this section to specify the desired limits for making a Type I or Type II decision error.

    Atlantic Richfield Company Response (6/30/21) – Comment addressed.

15. Section 2.7.1, DQO Step 7 – Please explain in the DQOs why you have chosen to collect a single composite vs. other potentially viable sampling designs, such as a single ISM, multiple composites, or multiple discrete samples, and explain how the selected design will achieve the objectives stated in DQO Steps 1 through 6.

    Atlantic Richfield Company Response (6/30/21) – Comment addressed.

16. Section 2.7.2 –

    a. Precision: Please update this section to specify the frequency requirements and the collection of field and laboratory quality control (QC) samples that will be used to determine precision. Also, laboratory precision goals should be specified in the QAPP, and only those laboratories that can demonstrate they can meet these goals should be considered for use in performing analyses for this QAPP.

Atlantic Richfield Company Response (6/30/21) – Sections 2.7.2 and 3.5.2 have been updated. Table 3 (QC Sample Acceptance Criteria) has also been added.

b. Accuracy and Bias: Please update this section to include information about blank requirements. In addition, please specify the acceptance criteria for samples (e.g., laboratory control samples and matrix spikes) that will be used to assess accuracy and bias. Please also indicate how information on percent recovery will be used to assess bias (e.g., recovery less than 100% would suggest a possible negative bias).

Atlantic Richfield Company Response (6/30/21) – Sections 2.7.2 and 3.5.2 have been updated. Table 3 (QC Sample Acceptance Criteria) has also been added.

c. Completeness: Please establish a target goal for completeness. There should be two completeness goals, one for the number of samples collected compared to what was supposed to be collected, and one for the number of usable results compared to the total number of results expected.

Atlantic Richfield Company Response (6/30/21) – Sections 2.7.2 and 3.5.2 have been updated. Table 3 (QC Sample Acceptance Criteria) has also been added.

d. Sensitivity: Please modify this section to evaluate if the selected analytical methods for use in this QAPP will be sufficient to achieve the target method sensitivity. Please also specify how non-detect results will be reported (i.e., will they be reported relative to the method detection limit [MDL]or the method reporting limit [MDL]?).

Atlantic Richfield Company Response (6/30/21) – Sections 2.7.2 and 3.5.2 have been updated. Table 3 (QC Sample Acceptance Criteria) has also been added.

17. Section 2.9, page 13 – Update the existing BPSOU Data Management Plan (Atlantic Richfield Company, 2017) reference to BPSOU Data Management Plan (Atlantic Richfield Company, 2020). The 2020 version of the document is currently under review but should ultimately be the guiding version going forward.

Atlantic Richfield Company Response (6/30/21) – Comment addressed.

18. Section 2.9.2, page 14 – Please discuss the process for agency approval for major deviations from the SOP or QAPP.

Atlantic Richfield Company Response (6/30/21) – Comment addressed.

19. Section 2.9.2, page 14 – The bulleted list describing the field documentation includes "all field measurements made". Please elaborate on the types of field measurements that will be made.

Atlantic Richfield Company Response (6/30/21) – Comment addressed.

20. Section 2.9.2, Field Documentation, Last Paragraph - There is discussion of submitting sample information and results to the landowners. It is recommended that the sample results be validated before being given to the landowner. Please modify this section accordingly. Note: Depending upon schedule changes, there could be times where unvalidated results would be shared and protections would be discussed and implemented with the landowner.

    Atlantic Richfield Company Response (6/30/21) – Comment addressed.

21. **Section 2.9.6, page 17 – This section indicates that sampling data will be forwarded to the agencies for review and approval in the form of an annual data summary report (DSR). The agencies should be provided access to all sampling data well before the completion of an annual DSR. The agencies should also be allowed to review any results letters prior to submittal to the landowners. It is recommended that validation reports be provided to EPA for review on a monthly basis and/or by property (e.g., school, park). A formal DSR and/or write up is not required for this pre-review and EPA is only looking for a "real time" review and will provide formal comment one the formal DSR is submitted.**

    Atlantic Richfield Company Response (6/30/21) – Comment addressed.

22. Section 2.9.6, page 17 – The DSR should also include copies of all analytical reports, electronic data deliverables (EDDs), validation reports, and define when the annual reports will be submitted. Please specify when the DSR report would be prepared and estimated formal submittal (e.g., within three months of validation completion and approximately one month for formal submittal).

    Atlantic Richfield Company Response (6/30/21) – Comment addressed.

23. Section 2.9.6, Project Data Reports – There is discussion of sample results being provided to the individual landowners. It is recommended that the sample results be validated before being given to the landowner. Please modify this section accordingly. Note: Depending upon schedule, changes there could be times where unvalidated results would be shared and protections would be discussed and implemented with the landowner.

    Atlantic Richfield Company Response (6/30/21) – Comment addressed.

24. Section 2.9.7, Quality Records, Last paragraph – This section states that project data will be maintained indefinitely in the BPSOU Residential Soils and Attic Dust Global Information System (GIS) database. Please confirm this is the correct database for maintaining the non-residential sample results. Also, it may be appropriate to discuss that the database has not been completely developed and AR/BSB will be working with the Agencies to finalize the database.

    Atlantic Richfield Company Response (6/30/21) – The BPSOU Residential Soils and Attic Dust Global Information System(GIS) database is the correct reference. This QAPP addresses RMAP sampling work (albeit for RMAP non-residential properties) and therefore the resultant data will be housed with the RMAP residential data so that all RMAP data resides in one location.

Text was added to reflect the current status of the database as requested.

25. Section 3.1, page 18 – Please specify if a note will be placed on the property title and/or how it will be tracked in the event that access is refused (as is the case for residential properties).

Atlantic Richfield Company Response (6/30/21) – Text has been updated to reflect that property owners declining access or are non-responsive to contact attempts will be flagged in the project database (consistent with current RMAP program). Deed attachments need the owner's permission.

26. Section 3.2.1, page 20 – While it may be true that removals would not be performed under tree canopies, it is not clear why this should preclude sampling in these areas. Contamination status beneath the tree canopy is still worth understanding and the sampling design should be modified accordingly. Please note that sampling should also take place underneath the canopy. It is understood that remediation will not always be possible under a canopy; this sampling is done mainly to track any waste left in place.

Atlantic Richfield Company Response (6/30/21) – Comment addressed. Sentence in question has been removed.

27. Section 3.2.1.1 to 3.2.1.4 – Please modify the sample collection discussions in each subsection to specify that each subsample should have approximately similar mass to each other so that each location is equally represented in the total sample mass. Please also describe how the gallon bag will be subsampled to ensure representativeness of the aliquot submitted for analysis.

Atlantic Richfield Company Response (6/30/21) – Comment addressed.

28. Section 3.2.1.5, page 22 – Please define the sample density requirement for this land use category in this section (i.e., it appears to be 25 sq. feet.).

Atlantic Richfield Company Response (6/30/21) – Section 3.2.1.5 and Table 1 have been updated to address comment.

29. Section 3.2.4, page 23 – The equipment decontamination standard operating procedure (SOP) mentions the collection of equipment rinsate blanks, yet there is no mention of these field QC samples in this QAPP. Please update the QAPP to discuss the collection, analysis, and interpretation of equipment rinsate blanks.

Atlantic Richfield Company Response (6/30/21) – The reference to equipment rinsate blanks in the Equipment Decontamination SOP was inadvertent and has been removed. Given the currently proposed re-usable equipment decontamination procedures along with the currently proposed residential action levels, Atlantic Richfield Company does not believe equipment rinsate blanks are applicable for this project. If we were looking for very low-level metal concentrations, equipment rinsate blanks may be more appropriate.

30. Section 3.2.5, page 23 – As noted previously, EPA agrees with the proposed language provided by Lester Dupes regarding mercury sampling and analysis. However, some of the new mercury text presented in Section 3.2.1 should be moved into Section 3.2.5, since it is more relevant to sample preparation.

    Atlantic Richfield Company Response (6/30/21) – Comment addressed.

31. **Section 3.2.5, page 23 – Please discuss the basis for the size fraction of 250 µm, mention that EPA guidance (OLEM Directive 9200.1-128) specifies 150 µm for soil samples analyzed for lead, and note that the appropriate size fraction for other investigations will be determined based on a site-specific particle size enrichment study. As noted in earlier comments, sieve size requirements should be set forth in the DQOs (Step 3).**

    Atlantic Richfield Company Response (6/30/21) – Section 3.2.5 updated to address comment.

    A statement has been added to the QAPP regarding the temporary agreement to use the less than 250 µm fraction, explaining the OLEM directive, the possibility of enrichment, and the plan for a demonstration study. We will work with EPA to develop a mutually acceptable plan for a pilot study to assess potential enrichment between the <150 micron and <250 micron soil fractions. OLEM Directive 9200.1-128 9 (USEPA 2016) cites 20 articles reporting higher lead concentrations in finer fractions of soil, but only one of those articles (Juhasz et al. 2011) included data for sieve sizes close to those of interest. Juhasz evaluated 16 soil samples from a range of mining/smelting sites, shooting ranges, incinerators, a gas works and historical fill areas for particle size fractions of <50, <100, <250, and <2000 microns. When we considered the enrichment in six samples with lead concentrations between 250 and 2,000 mg/kg, a range that is most relevant to most sites, the enrichment for the <100 vs. <250 micron size fractions ranged from 1.04 to 1.10 with a mean of 1.08. Based on the data reported by Juhasz et al., enrichment of lead concentrations in soil samples in the concentration range of interest (i.e., 250 ppm to 2,000 ppm) is predicted to be less than 10%, suggesting that the use of the finer sieve size may not be a significant factor in increasing lead concentrations.

USEPA. 2016. Recommendations for sieving soil and dust samples at lead sites for assessment of incidental ingestion. OLEM Directive 9200.1-128. U.S. Environmental Protection Agency, Assessment and Remediation Division, Office of Superfund Remediation and Technology Innovation. July.

Juhasz AL, Weber J, Smith E. 2011. Impact of Soil Particle Size and Bioaccessibility on Children and Adult Lead Exposure in Peri-Urban Contaminated Soils. Journal of Hazardous Materials, 186: 1870-1879.

32. Section 3.2.6, page 23 – Please update this section to incorporate the necessary changes to include the mercury-specific collection containers (i.e., glass jars).

    Atlantic Richfield Company Response (6/30/21) – Comment addressed.

33. Section 3.3, page 24 – Please update this section to describe the shipping requirements, such as which samples should be kept on ice and if any samples will be hand delivered. Also, please include the laboratory, address, and point of contact in the QAPP that should be identified as the shipment recipient.

    Atlantic Richfield Company Response (6/30/21) – Comment addressed.

34. Section 3.5.2 – Please update this section to specify laboratory control limits for each type of laboratory QC analysis.

    Atlantic Richfield Company Response (6/30/21) – Section 3.5.2 has been updated. Additionally, Table 3 has been added to the document.

35. Section 3.5.2, Laboratory Quality Control Samples, Laboratory Blanks – Please modify this section to include initial calibration blanks and continuing calibration blanks as they will be reviewed during validation.

    Atlantic Richfield Company Response (6/30/21) – Section 3.5.2 has been updated. Additionally, Table 3 has been added to the document.

36. Section 3.5.2, Laboratory Quality Control Samples – Please modify this section to discuss ICP interference check samples, internal standards, and tunes if there is the potential they will be reviewed if ICP-MS analyses is utilized.

    Atlantic Richfield Company Response (6/30/21) – Section 3.5.2 has been updated. Additionally, Table 3 has been added to the document.

37. Section 3.5.2, Laboratory Quality Control Samples, Matrix Spikes – Please confirm that qualifiers will not be applied if the sample concentration is greater than 4 times the spike concentration. If so, this should be explicitly stated in the text.

    Atlantic Richfield Company Response (6/30/21) – Section 3.5.2 has been updated. Additionally, Table 3 has been added to the document.

38. Section 3.6, page 28 – Please verify the statement that all sampling equipment is single use. If sampling equipment such as metal trowels and augers will be used, this statement should be removed.

    Atlantic Richfield Company Response (6/30/21) – All sampling equipment is anticipated to be single use. No edit necessary.

39. Section 3.6.1, page 29 – The section text is inconsistent with the SOP. The SOP indicates a fieldduplicate is collected at the same location, but the section text refers to the creation of a

split. Please clarify if the parent sample is being split and, if so, how sample splitting will be accomplished. If the duplicate sample is actually a second aliquot collected at the same location,please specify the target proximity to the parent sample (e.g., 6 inches).

Atlantic Richfield Company Response (6/30/21) – Field duplicates will be "splits" as indicated by the original QAPP text. The SOP has been revised to reflect this.

40. Section 3.7, page 29 – Please update this section to discuss the disposal of any excess soil mass that is not included in the aliquot submitted to the laboratory.

    Atlantic Richfield Company Response (6/30/21) – Comment addressed.

41. Section 3.10 Data Management Procedure, page 30 – Please update the existing BPSOU Data Management Plan (Atlantic Richfield Company, 2017) reference to BPSOU Data Management Plan (Atlantic Richfield Company, 2020). The 2020 version of the document is currently under review but should ultimately be the guiding version going forward.

    Atlantic Richfield Company Response (6/30/21) – Comment addressed.

42. Section 3.10 Data Management Procedure, page 30 – As part of the discussion of how field and laboratory data will be compiled int the project database, please include an overarching statement that data management activities for the RMAP program will be further defined in the BPSOU Data Management Plan (Atlantic Richfield Company, 2020).

    Atlantic Richfield Company Response (6/30/21) – Comment addressed.

43. Section 3.10, page 30 – Please discuss if there is a required laboratory EDD format. If no format has been developed yet, it is recommended a standard template be developed that allows for easy upload into the project database.

    Atlantic Richfield Company Response (6/30/21) – Comment addressed.

44. **Section 4.0, page 31 – Insert the following sentence at the beginning of this paragraph: "Should sample results indicate that removal of soils at a school, park, or non-residential daycare is warranted, a removal work plan shall be submitted by BSB and Atlantic Richfield for approval by the Agencies. All materials used…"**

    Atlantic Richfield Company Response (6/30/21) – Comment addressed.

45. Section 5.0, page 32 – Please provide details on whether any internal or external audits are planned for completion as part of the non-residential exterior sampling effort.

    Atlantic Richfield Company Response (6/30/21) – Comment addressed.

46. Section 5.3, page 33 –

    a. The laboratory TATs should be dictated by the need to support remedial decisions this summer, not the annual DSR. This section needs to establish the required laboratory TATs.

    Atlantic Richfield Company Response (6/30/21) – Comment addressed.

    b. The DSR also needs to include the results of the validation and data usability assessments.

    Atlantic Richfield Company Response (6/30/21) – Comment addressed.

47. Section 5.3, Reports to Management – Please modify this section to specify that individual data validation reports will be provided to the agencies on a monthly basis (or per school) when the validation is complete (no formal DSR or write up is required for these interim submittals). See earlier comment on Section 2.9.6.

    Atlantic Richfield Company Response (6/30/21) – Comment addressed.

48. Section 6.0, Data Review and Usability – There are new 2020 EPA National Functional Guidelines. When QAPP updates are developed next year, the new guidelines should be used. Please use these guidelines now at your discretion.

    Atlantic Richfield Company Response (6/30/21) – Comment addressed in Section 6.0 and 6.2.

49. Section 6.1.3, Laboratory Data Verification – Please confirm if qualifiers are required, that they will be added to the laboratory EDDs and then uploaded into the database.

    Atlantic Richfield Company Response (6/30/21) – Comment addressed.

50. Section 6.2, Verification and Validation Methods – Please confirm if the non-detect values will be reported to the MDL or MRL. The "UJ" qualifier indicates the "analyte was not detected above the sample reporting limit." Please be consistent throughout the text.

    Atlantic Richfield Company Response (6/30/21) – Comment addressed.

51. Section 6.3, Reconciliation and User Requirements, Enforcement/Screening Designation Table – It is appropriate to note that sample results qualified as estimated "J" by the laboratory because, if the result is between the MDL and MRL, values are considered enforcement data if no other qualifiers were required during validation.

    Atlantic Richfield Company Response (6/30/21) – Comment addressed.

52. Section 6.2.3, page 39 – Please develop a periodic stage 4 validation by a random selection of 10% of laboratory jobs on an annual basis.

Atlantic Richfield Company Response (6/30/21) – Comment addressed.

53. Section 6.3- page 39, Step 3 – There are no statistical tests that are planned in the interpretation of the non-residential soils results; please modify the components of the data quality assessment to be consistent with the planned data use.

Atlantic Richfield Company Response (6/30/21) – There are no statistical tests that are planned in the interpretation of the non-residential soils results; laboratory results will be compared directly to action limits defined in the DQOs (Section 2.7.1). Text in Section 6.3 has been updated.

Comments on Figures, Tables, and Attachments:

1. Please add a new figure that indicates the locations of all the school and parks listed in Tables 3 and 4.

Atlantic Richfield Company Response (6/30/21) – Figure 4 showing the Butte area schools and former schools has been added to the figure set. Additional investigation is required into the park geocodes and property boundaries before an accurate park figure set can be developed. Given the schools are the current top priority, this park investigation will be conducted at a future time and the resultant figures provided through forthcoming QAPP revisions.

2. Figure 2: Please specify the Field Team Leader and laboratory point of contact in the organizational chart.

Atlantic Richfield Company Response (6/30/21) – Comment addressed.

3. Figure 3: There are multiple areas identified with the same code (e.g., PA1) on this map example. Please clarify if these should be different PA locations with a unique identifier.

Atlantic Richfield Company Response (6/30/21) – Comment addressed.

4. Table 1: Because are no differences between the sampling requirements for each chemical, this table appears redundant and could be simplified by listing each land use type once and specifying the metal-specific requirements stacked within each cell (or present the metal-specific action label and method as a separate Panel B to the table).

Atlantic Richfield Company Response (6/30/21) – Comment addressed.

5. Table 2: Please add two columns to this table to specify acceptance criteria and corrective actions if these criteria are not met.

Atlantic Richfield Company Response (6/30/21) – The requested information has been provided in a new Table 3.

6. Tables 3 and 4: Please add a new column to both tables to specify if any of these properties have been sampled in the past.

   Atlantic Richfield Company Response (6/30/21) – Per EPA General Comment #2, *"Please clarify if there have been any previous sampling efforts conducted at the non-residential properties anticipated for evaluation as part of this QAPP. If so, the QAPP should specify what those results show (or indicate that this information would be included in the property-specific field sampling plan [FSP])."*

   As noted in response to EPA General Comment #2: Section 3.2.3 has been amended to state, "Butte-Silver Bow County will review the Program database to identify properties that were previously sampled but have incomplete data sets. This information will be provided to the Agencies in the form of Field Sampling Plan (FSP) submittals."

7. Table 3: Please confirm the geocode for the Butte High School Annex.

   Atlantic Richfield Company Response (6/30/21) – This Table has been renumbered from 3 to 4. Butte High School and the Butte High School Annex share one common geocode. Table 4 has been updated to reflect this.

8. Attachment C1: Please determine if the test pit SOP is necessary to include for the non-residential sampling. If not, it should be deleted from this QAPP.

   Atlantic Richfield Company Response (6/30/21) – Test Pit SOP removed per Agency request.

9. Attachment C1: The field SOPs do not have a lot of detail that is not already in the QAPP. Please update the soil collection SOP text to clearly specify the sampling equipment that will be used (e.g., augers, trowels, cores) and how samples will be collected and composited.

   Atlantic Richfield Company Response (6/30/21) – Comment addressed.

10. Attachment C1, SOP-1A, page 1: Please confirm that some land use areas (e.g., sports fields) may be comprised of multiple composites.

    Atlantic Richfield Company Response (6/30/21) – Comment addressed.

11. Attachment C-1, SOP-DE-01: The Personnel Decontamination Procedures SOP would be better suited for inclusion in the SSHASP. Please consider moving this SOP into the SSHASP.

    Atlantic Richfield Company Response (6/30/21) – Personnel Decontamination SOP removed per Agency request.

12. Attachment C2:
    a. The sample processing SOP (Section 11.4 and 11.5) specifies that samples will be pulverized to obtain the desired mesh sizes. Samples should not be pulverized prior to sieving, rather only those particles (in their natural state) that pass through the desired mesh size should be analyzed. Please modify the sample processing SOP accordingly.

    Atlantic Richfield Company Response (6/30/21) – The SOP in question was for the Pace Sheridan, WY facility, which was originally planned to be part of the lab analytical network. However, the Sheridan facility has been replaced with the Pace Green Bay, WI facility. The original Sheridan SOP has been replaced with the Green Bay SOP (which has no language regarding pulverizing of samples).

    b. The soil preparation SOP should be revised to be consistent with the target particle size. Currently, this SOP describes sieving to a 60 mesh (250 µm), but 100 mesh would be needed to achieve 150 µm.

    Atlantic Richfield Company Response (6/30/21) – The 2021 investigation will utilize a 250 micrometer fine fraction (See Agency Comment #6). Therefore, this SOP should be appropriate for 2021 work. SOPs will be updated through future QAPP revisions, as appropriate.

    c. Please update the laboratory soil preparation methods to incorporate any mercury-specific requirements.

    Atlantic Richfield Company Response (6/30/21) – The Mercury analytical SOP (ENV-SOP-MIN4-0054) includes all soil preparation details.

13. Attachment E2: Please consider adding the first paragraph from the ‘no action’ letter (regarding the UAO) to the beginning of the ‘remediation action’ letter for context.

    Atlantic Richfield Company Response (6/30/21) – Comment addressed.

Minor/Editorial Changes:

1. Please perform an editorial review of this document to ensure that all acronyms are defined only once at first use and the acronym is used thereafter.

    Atlantic Richfield Company Response (6/30/21) – Comment addressed.

2. Document titles included in the text should be italicized.

    Atlantic Richfield Company Response (6/30/21) – Comment addressed.

3. Section 1.0, page 1, and Section 2.6, Objective 2 – Presumably, the interior school assessments may need to be completed before the next annual review (i.e., June 2022); therefore, please strike “annual” in the context of QAPP revisions.

Atlantic Richfield Company Response (6/30/21) – Comment addressed.

4. Section 2.5, page 4 – The term “monitoring” implies ongoing evaluation. Because what is being conducted at the schools is a one-time remedial evaluation, the term “assessment” may be more appropriate here.

   Atlantic Richfield Company Response (6/30/21) – Comment addressed.

5. Section 2.7.1, DQO Step 2 – Consider rephrasing the Primary question to: *Are soil concentrations of arsenic, lead and/or mercury at non-residential properties present at levels that may pose a risk to human health (e.g., above the action levels)?*

   Atlantic Richfield Company Response (6/30/21) – Comment addressed.

6. Section 2.9.1, page 13 – Please clarify here that, for non-residential properties like schools and parks, property owners may include the city or other entities and agencies.

   Atlantic Richfield Company Response (6/30/21) – Comment addressed.

7. Section 2.9.2, page 14 – The text states that field data “may be” converted to electronic storage. Please specify the conditions for when this conversion would be deemed necessary.

   Atlantic Richfield Company Response (6/30/21) – Sentence removed.

8. Section 3.2.1, page 20 –

   a. Please add a cross-reference to Table 1 in this section to refer to the sample density requirements for each land use category.

   Atlantic Richfield Company Response (6/30/21) – Comment addressed.

   b. The second paragraph, when referring to components between neighboring/adjacent structures, is unclear. Please modify this discussion to clarify the intent of this sentence.

   Atlantic Richfield Company Response (6/30/21) – Comment addressed.

9. Section 3.2.1.4, page 21 – One-half acre is equal to 21,780 sq. ft.; please correct this typographical error.

   Atlantic Richfield Company Response (6/30/21) – Comment addressed.

10. Section 3.2.1.5, page 22 – Table 1 states there is a *maximum* of 2 subsamples, however the text is stating a *minimum* of 2 subsamples. Please modify the table to be consistent with the text.

Atlantic Richfield Company Response (6/30/21) – Section 3.2.1.5 and Table 1 have been updated to address comment.

11. Section 3.2.4, Soil Sample Equipment Decontamination – The text states, "re-usable equipment *may be* decontaminated." Re-usable equipment *must be* decontaminated between each sample location.

    Atlantic Richfield Company Response (6/30/21) – Comment addressed.

12. Section 3.5.2, page 27 – The in-text table is redundant with Table 2 and should be deleted.

    Atlantic Richfield Company Response (6/30/21) – Comment addressed.

13. Section 3.6 – Please define what is meant by a "sampling event" (i.e., one per sampling day, one per school, etc.?)

    Atlantic Richfield Company Response (6/30/21) – Comment addressed.

14. Section 3.8.1, page 29 – Please clarify if equipment be inspected before the first use each day or just the first use on the program.

    Atlantic Richfield Company Response (6/30/21) – Comment addressed.

15. Section 3.10, page 30 – In the bulleted list of records, please include property owner letters, other reports, and other correspondence.

    Atlantic Richfield Company Response (6/30/21) – Comment addressed.

16. Section 6.1.2.1, Field Data Verification – The Level A criteria bullet for "Field *preservation* technique" should be changed to "Field *preparation* technique". Also, please add "Sample preservation technique" to the bullet list. (These criteria lists are correct in the actual Level A/B Assessment Checklists.)

    Atlantic Richfield Company Response (6/30/21) – Comment addressed.

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

Sincerely,

NIKIA GREENE

Digitally signed by NIKIA GREENE
Date: 2021.06.22 16:36:51 -06'00'

Nikia Greene

Remedial Project Manager

Attachment: EPA Crosswalk

cc: (email only)
Butte File
Jenny Chambers; DEQ
Matt Dorrington, DEQ
Daryl Reed; DEQ
Jon Morgan; DEQ counsel
Carolina Balliew; DEQ
Harley Harris; NRDP
Katherine Hausrath; NRDP
Jim Ford; NRDP
Ray Vinkey; NRDP
John Gallagher; BSBC
Mollie Maffei; BSBC
Eric Hassler; BSBC
Brandon Warner; BSBC
Chad Anderson; BSBC
Karen Sullivan; BSBC
Julia Crain; BSBC
Abby Peltomaa; BSBC
Jeremy Grotbo; BSBC
Anne Walsh; UP
Robert Bylsma; UP counsel
Leo Berry; BNSF and UP counsel
Mark Engdahl; BNSF
Brooke Kuhl; BNSF counsel
Jeremie Maehr; Kennedy Jenks for BNSF and UP
Annika Silverman; Kennedy Jenks for BNSF and UP
Bob Andreoli; Patroit/RARUS
Becky Summerville; counsel for Inland Properties Inc.
Robert Lowry, BNSF counsel
Loren Burmeister; AR
Josh Bryson; AR
Mike Mcanulty; AR
Dave Griffis; AR
Jean Martin; Counsel AR
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Scott Bradshaw; TREC
Mike Borduin; Pioneer for AR
Karen Helfrich; Pioneer for AR
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Tina Donovan; Woodardcurran for AR
Don Booth; AR consultant
Ted Duaime; MBMG
Gary Icopini; MBMG
David Shanight, CDM Smith
Curt Coover, CDM Smith
Chapin Storrar; CDM Smith
Erin Agee, EPA
Joe Vranka; EPA
Chris Wardell; EPA
Dana Barnicoat; EPA
Charlie Partridge; EPA
Jean Belille; EPA
Ian Magruder; CTEC (Tech Advisor)
Janice Hogan; CTEC
Kristi Carroll; Montana Tech Library

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Ref: 8MO

June 22, 2021

Mr. Eric Hassler
Superfund Program Data Administrator
Butte-Silver Bow Planning Department
155 West Granite Street
Butte, MT 59701

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

Re: Comments for: BPSOU Draft Final Residential Metals Abatement Program (RMAP) Quality Assurance Project Plan (QAPP) – Non-Residential Parcels (aka schools and parks), dated June 7, 2021

Dear Eric and Mike:

The U. S. Environmental Protection Agency (EPA), in consultation with the Montana Department of Environmental Quality (DEQ), is providing comments on the *Draft Final Residential Metals Abatement Program (RMAP) Quality Assurance Project Plan (QAPP) – Non-Residential Parcels (dated June 7, 2021)* that was prepared by Pioneer Technical Services, Inc., on behalf of the Butte-Silver Bow County (BSB) and Atlantic Richfield Company.

Comments have been stratified into the follow sections – General Comments, Specific Comments, Comments on Figures, Tables, and Attachments, and Minor/Editorial Changes. Those comments that have the potential to change the study design or approach are indicated in **bold** text and may require additional discussion. Please incorporate these comments and submit the final version of the report for review.

General Comments:

1. The Residential RMAP QAPP was used as the basis for this document; however, there are several aspects of schools that differ from homes both with regard to terminology and area use (e.g., living space, attic dust, yard soil). While most references to "yard" or "attics" have been removed, remaining residential-specific language and references to indoor media, such as dust and paint, should be removed as well.
2. Please clarify if there have been any previous sampling efforts conducted at the non-residential properties anticipated for evaluation as part of this QAPP. If so, the QAPP should specify what

those results show (or indicate that this information would be included in the property-specific field sampling plan [FSP]).

3. Mentions of lead-based paint (LBP) should be clear that it is specific to the exterior of the schools (i.e. chance of contaminating or re-contaminating soils. Please remove reference to lead solder for Non-Residential sections. Any interior work will require a revision or a new QAPP.
4. Please ensure the QAPP is consistent in that all three metals (arsenic, lead, and mercury) are required to be analyzed in all samples.
5. Please ensure that supplemental text provided by Lester Dupes in the June 10, 2021 email regarding the preparation and analysis of samples for elemental mercury are incorporated into the final version of the QAPP.
6. **In the past, RMAP investigations have sieved soils to 250 micrometers (µm). However, more recent EPA guidance (EPA OLEM Directive 9200.1-128) requires sieving to 150 µm. For the purposes of the Non-Residential RMAP QAPP, given the schedule constraints, EPA agrees with targeting a particle size of 250 µm for the 2021 investigation. The DQOs and study design of this QAPP can reflect this agreement. However, EPA requires the performance of a demonstration pilot study to assess potential differences in enrichment between the 250-µm and 150-µm size fractions before use of the 250-µm fraction will be approved for broader use in other RMAP investigations.**

Specific Comments:

1. Distribution List, page ii – Please add contact information for the Field Team Leader and the analytical laboratory(ies).
2. Section 1.0, page 1 – In the introduction section where the overall RMAP is being described, please also mention the use of medical monitoring to evaluate the effectiveness of the program.
3. Section 2.5, Problem Definition and Background – Mercury should be identified to be elemental mercury in this section and throughout the report where appropriate.
4. Section 2.6, page 5 – This section should focus on the non-residential aspects of the RMAP and references to residential-specific considerations should be removed.
5. Section 2.6.1, page 6 – This section should specify the desire (with certain caveats about unknowns and schedule changes) to complete outdoor sampling and remediation (if needed) prior to the school fall session beginning.
6. Section 2.7.1, DQO Step 3 – There are additional information inputs that should be listed as part of this step. For example, information will be needed on the land use of the different areas within the parks and schools should be provided. In addition, information will also be needed on the sampling locations and the area that each sample represents (i.e., the field teams will need to document the sample coordinates and generate field sketches or map polygons to document each sampling unit).
7. Section 2.7.1, DQO Step 3 – Please explain why residential action levels have been selected for use (i.e., there are no school-specific action levels and residential were selected to be conservative).
8. Section 2.7.1, DQO Step 3 –

   a. Given that one of the concerns for the schools is being able to assess and, if necessary, remediate areas this summer, it is unclear why use of x-ray fluorescence (XRF) analysis for lead and arsenic is not discussed as a potentially viable analytical method. This section should discuss the potential use of XRF and why this analytical method was not selected for use (considering this has been the preferred RMAP analytical method up to this point). This section should also discuss how EPA has indicated that XRF is not a viable analytical method for analysis of mercury.
   b. Please discuss the required laboratory turn-around times that will be necessary to achieve the assessment/remediation goals for non-residential properties.
   c. **This section should discuss any sample collection/preparation requirements (e.g., sieving, drying, storage temperature). Specifically, the fact that past RMAP investigations have sieved to 250 µm and that more recent EPA guidance (EPA OLEM Directive 9200.1-128) requires sieving to 150 µm should be discussed here and EPA's temporary agreement with use of 250 µm pending the outcome of a particle size enrichment demonstration study.**
   d. This section should specify if there are any constraints as to the types of sampling designs that will be considered for this program (e.g., incremental, composite, discrete). If project objectives can be accomplished by multiple sampling designs, please discuss why one design might be preferred over another (e.g., use of incremental or composite collection methods would result in lower analytical costs relative to discrete methods).

9. Section 2.7.1, DQO Step 4 – The DQOs should specify why each of these different depth intervals are being targeted. The text should discuss if the need for different depth intervals is related to differences in exposure potential and/or if this is being done to help refine removal extents.
10. Section 2.7.1, DQO Step 4 – The inclusion of a vegetable garden land use seems more specific to a residential scenario. Please confirm if this category is relevant to non-residential (e.g., schools, parks) and, if not, please modify the land use category appropriately.
11. Section 2.7.1, DQO Step 4 – Please revise this section to include a more expansive discussion on sampling density requirements for each land-use category.
12. Section 2.7.1, DQO Step 4 –
    a. This section should discuss any temporal requirements on the sampling, not the remediation (i.e., specify if there are any temporal constraints on the sampling investigation). Specifically, this section should discuss the need to assess all schools prior to school starting in the fall. Additionally, a discussion of the challenges of unknowns and schedule changes could be useful here.
    b. No temporal variability in soil concentrations is expected, so the sampling effort should be primarily dictated by when it is easiest to conduct sampling, meaning when no snow is present and when school facilities are not in use (i.e., summer).
13. Section 2.7.1, DQO Step 4 – The sampling design has not yet been established at this stage in the DQO process (the study design is established in Step 7). Thus, stating that the decision unit (DU) is equal to the extent of a single composite soil sample does not have inherent meaning. If the goal

is to make remedial decisions on a sample-by-sample basis, this section should discuss how the DU will be set equal to the sampling unit (SU) and the SU extent should be specified as the maximum area for decision-making by land use type (i.e., for playgrounds the DU/SU size is 6,250 square feet).

14. Section 2.7.1, DQO Step 6 – At this stage in the DQO process, the sampling design has not yet been selected (the study design is established in Step 7). The tolerable limits should not be dictated by the selected study design, rather the selected study design should be developed based on the performance criteria. Tolerable limits for decision errors should be specified whenever the problem question is a decision question. Please modify this section to specify the desired limits for making a Type I or Type II decision error.
15. Section 2.7.1, DQO Step 7 – Please explain in the DQOs why you have chosen to collect a single composite vs. other potentially viable sampling designs, such as a single ISM, multiple composites, or multiple discrete samples, and explain how the selected design will achieve the objectives stated in DQO Steps 1 through 6.
16. Section 2.7.2 –
    a. Precision: Please update this section to specify the frequency requirements and the collection of field and laboratory quality control (QC) samples that will be used to determine precision. Also, laboratory precision goals should be specified in the QAPP, and only those laboratories that can demonstrate they can meet these goals should be considered for use in performing analyses for this QAPP.
    b. Accuracy and Bias: Please update this section to include information about blank requirements. In addition, please specify the acceptance criteria for samples (e.g., laboratory control samples and matrix spikes) that will be used to assess accuracy and bias. Please also indicate how information on percent recovery will be used to assess bias (e.g., recovery less than 100% would suggest a possible negative bias).
    c. Completeness: Please establish a target goal for completeness. There should be two completeness goals, one for the number of samples collected compared to what was supposed to be collected, and one for the number of usable results compared to the total number of results expected.
    d. Sensitivity: Please modify this section to evaluate if the selected analytical methods for use in this QAPP will be sufficient to achieve the target method sensitivity. Please also specify how non-detect results will be reported (i.e., will they be reported relative to the method detection limit [MDL]or the method reporting limit [MDL]?).
17. Section 2.9, page 13 – Update the existing BPSOU Data Management Plan (Atlantic Richfield Company, 2017) reference to BPSOU Data Management Plan (Atlantic Richfield Company, 2020). The 2020 version of the document is currently under review but should ultimately be the guiding version going forward.
18. Section 2.9.2, page 14 – Please discuss the process for agency approval for major deviations from the SOP or QAPP.

19. Section 2.9.2, page 14 – The bulleted list describing the field documentation includes “all field measurements made”. Please elaborate on the types of field measurements that will be made.

20. Section 2.9.2, Field Documentation, Last Paragraph - There is discussion of submitting sample information and results to the landowners. It is recommended that the sample results be validated before being given to the landowner. Please modify this section accordingly. Note: Depending upon schedule changes, there could be times where unvalidated results would be shared and protections would be discussed and implemented with the landowner.

21. Section 2.9.6, page 17 – This section indicates that sampling data will be forwarded to the agencies for review and approval in the form of an annual data summary report (DSR). The agencies should be provided access to all sampling data well before the completion of an annual DSR. The agencies should also be allowed to review any results letters prior to submittal to the landowners. It is recommended that validation reports be provided to EPA for review on a monthly basis and/or by property (e.g., school, park). A formal DSR and/or write up is not required for this pre-review and EPA is only looking for a “real time” review and will provide formal comment one the formal DSR is submitted.

22. Section 2.9.6, page 17 – The DSR should also include copies of all analytical reports, electronic data deliverables (EDDs), validation reports, and define when the annual reports will be submitted. Please specify when the DSR report would be prepared and estimated formal submittal (e.g., within three months of validation completion and approximately one month for formal submittal).

23. Section 2.9.6, Project Data Reports – There is discussion of sample results being provided to the individual landowners. It is recommended that the sample results be validated before being given to the landowner. Please modify this section accordingly. Note: Depending upon schedule, changes there could be times where unvalidated results would be shared and protections would be discussed and implemented with the landowner.

24. Section 2.9.7, Quality Records, Last paragraph – This section states that project data will be maintained indefinitely in the BPSOU Residential Soils and Attic Dust Global Information System (GIS) database. Please confirm this is the correct database for maintaining the non-residential sample results. Also, it may be appropriate to discuss that the database has not been completely developed and AR/BSB will be working with the Agencies to finalize the database.

25. Section 3.1, page 18 – Please specify if a note will be placed on the property title and/or how it will be tracked in the event that access is refused (as is the case for residential properties).

26. Section 3.2.1, page 20 – While it may be true that removals would not be performed under tree canopies, it is not clear why this should preclude sampling in these areas. Contamination status beneath the tree canopy is still worth understanding and the sampling design should be modified accordingly. Please note that sampling should also take place underneath the canopy. It is understood that remediation will not always be possible under a canopy; this sampling is done mainly to track any waste left in place.

27. Section 3.2.1.1 to 3.2.1.4 – Please modify the sample collection discussions in each subsection to specify that each subsample should have approximately similar mass to each other so that each location is equally represented in the total sample mass. Please also describe how the gallon bag will be subsampled to ensure representativeness of the aliquot submitted for analysis.

28. Section 3.2.1.5, page 22 – Please define the sample density requirement for this land use category in this section (i.e., it appears to be 25 sq. feet.).
29. Section 3.2.4, page 23 – The equipment decontamination standard operating procedure (SOP) mentions the collection of equipment rinsate blanks, yet there is no mention of these field QC samples in this QAPP. Please update the QAPP to discuss the collection, analysis, and interpretation of equipment rinsate blanks.
30. Section 3.2.5, page 23 – As noted previously, EPA agrees with the proposed language provided by Lester Dupes regarding mercury sampling and analysis. However, some of the new mercury text presented in Section 3.2.1 should be moved into Section 3.2.5, since it is more relevant to sample preparation.
31. **Section 3.2.5, page 23 – Please discuss the basis for the size fraction of 250 µm, mention that EPA guidance (OLEM Directive 9200.1-128) specifies 150 µm for soil samples analyzed for lead, and note that the appropriate size fraction for other investigations will be determined based on a site-specific particle size enrichment study. As noted in earlier comments, sieve size requirements should be set forth in the DQOs (Step 3).**
32. Section 3.2.6, page 23 – Please update this section to incorporate the necessary changes to include the mercury-specific collection containers (i.e., glass jars).
33. Section 3.3, page 24 – Please update this section to describe the shipping requirements, such as which samples should be kept on ice and if any samples will be hand delivered. Also, please include the laboratory, address, and point of contact in the QAPP that should be identified as the shipment recipient.
34. Section 3.5.2 – Please update this section to specify laboratory control limits for each type of laboratory QC analysis.
35. Section 3.5.2, Laboratory Quality Control Samples, Laboratory Blanks – Please modify this section to include initial calibration blanks and continuing calibration blanks as they will be reviewed during validation.
36. Section 3.5.2, Laboratory Quality Control Samples – Please modify this section to discuss ICP interference check samples, internal standards, and tunes if there is the potential they will be reviewed if ICP-MS analyses is utilized.
37. Section 3.5.2, Laboratory Quality Control Samples, Matrix Spikes – Please confirm that qualifiers will not be applied if the sample concentration is greater than 4 times the spike concentration. If so, this should be explicitly stated in the text.
38. Section 3.6, page 28 – Please verify the statement that all sampling equipment is single use. If sampling equipment such as metal trowels and augers will be used, this statement should be removed.
39. Section 3.6.1, page 29 – The section text is inconsistent with the SOP. The SOP indicates a field duplicate is collected at the same location, but the section text refers to the creation of a split. Please clarify if the parent sample is being split and, if so, how sample splitting will be accomplished. If the duplicate sample is actually a second aliquot collected at the same location, please specify the target proximity to the parent sample (e.g., 6 inches).

40. Section 3.7, page 29 – Please update this section to discuss the disposal of any excess soil mass that is not included in the aliquot submitted to the laboratory.

41. Section 3.10 Data Management Procedure, page 30 – Please update the existing BPSOU Data Management Plan (Atlantic Richfield Company, 2017) reference to BPSOU Data Management Plan (Atlantic Richfield Company, 2020). The 2020 version of the document is currently under review but should ultimately be the guiding version going forward.

42. Section 3.10 Data Management Procedure, page 30 – As part of the discussion of how field and laboratory data will be compiled int the project database, please include an overarching statement that data management activities for the RMAP program will be further defined in the BPSOU Data Management Plan (Atlantic Richfield Company, 2020).

43. Section 3.10, page 30 – Please discuss if there is a required laboratory EDD format. If no format has been developed yet, it is recommended a standard template be developed that allows for easy upload into the project database.

44. **Section 4.0, page 31 – Insert the following sentence at the beginning of this paragraph: "Should sample results indicate that removal of soils at a school, park, or non-residential daycare is warranted, a removal work plan shall be submitted by BSB and Atlantic Richfield for approval by the Agencies. All materials used…"**

45. Section 5.0, page 32 – Please provide details on whether any internal or external audits are planned for completion as part of the non-residential exterior sampling effort.

46. Section 5.3, page 33 –
    a. The laboratory TATs should be dictated by the need to support remedial decisions this summer, not the annual DSR. This section needs to establish the required laboratory TATs.
    b. The DSR also needs to include the results of the validation and data usability assessments.

47. Section 5.3, Reports to Management – Please modify this section to specify that individual data validation reports will be provided to the agencies on a monthly basis (or per school) when the validation is complete (no formal DSR or write up is required for these interim submittals). See earlier comment on Section 2.9.6.

48. Section 6.0, Data Review and Usability – There are new 2020 EPA National Functional Guidelines. When QAPP updates are developed next year, the new guidelines should be used. Please use these guidelines now at your discretion.

49. Section 6.1.3, Laboratory Data Verification – Please confirm if qualifiers are required, that they will be added to the laboratory EDDs and then uploaded into the database.

50. Section 6.2, Verification and Validation Methods – Please confirm if the non-detect values will be reported to the MDL or MRL. The "UJ" qualifier indicates the "analyte was not detected above the sample reporting limit." Please be consistent throughout the text.

51. Section 6.3, Reconciliation and User Requirements, Enforcement/Screening Designation Table – It is appropriate to note that sample results qualified as estimated "J" by the laboratory because, if

the result is between the MDL and MRL, values are considered enforcement data if no other qualifiers were required during validation.

52. **Section 6.2.3, page 39 – Please develop a periodic stage 4 validation by a random selection of 10% of laboratory jobs on an annual basis.**
53. Section 6.3- page 39, Step 3 – There are no statistical tests that are planned in the interpretation of the non-residential soils results; please modify the components of the data quality assessment to be consistent with the planned data use.

Comments on Figures, Tables, and Attachments:

1. Please add a new figure that indicates the locations of all the school and parks listed in Tables 3 and 4.
2. Figure 2: Please specify the Field Team Leader and laboratory point of contact in the organizational chart.
3. Figure 3: There are multiple areas identified with the same code (e.g., PA1) on this map example. Please clarify if these should be different PA locations with a unique identifier.
4. Table 1: Because are no differences between the sampling requirements for each chemical, this table appears redundant and could be simplified by listing each land use type once and specifying the metal-specific requirements stacked within each cell (or present the metal-specific action label and method as a separate Panel B to the table).
5. Table 2: Please add two columns to this table to specify acceptance criteria and corrective actions if these criteria are not met.
6. Tables 3 and 4: Please add a new column to both tables to specify if any of these properties have been sampled in the past.
7. Table 3: Please confirm the geocode for the Butte High School Annex.
8. Attachment C1: Please determine if the test pit SOP is necessary to include for the non-residential sampling. If not, it should be deleted from this QAPP.
9. Attachment C1: The field SOPs do not have a lot of detail that is not already in the QAPP. Please update the soil collection SOP text to clearly specify the sampling equipment that will be used (e.g., augers, trowels, cores) and how samples will be collected and composited.
10. Attachment C1, SOP-1A, page 1: Please confirm that some land use areas (e.g., sports fields) may be comprised of multiple composites.
11. Attachment C-1, SOP-DE-01: The Personnel Decontamination Procedures SOP would be better suited for inclusion in the SSHASP. Please consider moving this SOP into the SSHASP.
12. Attachment C2:
    a. The sample processing SOP (Section 11.4 and 11.5) specifies that samples will be pulverized to obtain the desired mesh sizes. Samples should not be pulverized prior to sieving, rather only those particles (in their natural state) that pass through the desired mesh size should be analyzed. Please modify the sample processing SOP accordingly.

   b. The soil preparation SOP should be revised to be consistent with the target particle size. Currently, this SOP describes sieving to a 60 mesh (250 µm), but 100 mesh would be needed to achieve 150 µm.
   c. Please update the laboratory soil preparation methods to incorporate any mercury-specific requirements.

13. Attachment E2: Please consider adding the first paragraph from the ‘no action’ letter (regarding the UAO) to the beginning of the ‘remediation action’ letter for context.

Minor/Editorial Changes:

1. Please perform an editorial review of this document to ensure that all acronyms are defined only once at first use and the acronym is used thereafter.
2. Document titles included in the text should be italicized.
3. Section 1.0, page 1, and Section 2.6, Objective 2 – Presumably, the interior school assessments may need to be completed before the next annual review (i.e., June 2022); therefore, please strike “annual” in the context of QAPP revisions.
4. Section 2.5, page 4 – The term “monitoring” implies ongoing evaluation. Because what is being conducted at the schools is a one-time remedial evaluation, the term “assessment” may be more appropriate here.
5. Section 2.7.1, DQO Step 2 – Consider rephrasing the Primary question to: *Are soil concentrations of arsenic, lead and/or mercury at non-residential properties present at levels that may pose a risk to human health (e.g., above the action levels)?*
6. Section 2.9.1, page 13 – Please clarify here that, for non-residential properties like schools and parks, property owners may include the city or other entities and agencies.
7. Section 2.9.2, page 14 – The text states that field data “may be” converted to electronic storage. Please specify the conditions for when this conversion would be deemed necessary.
8. Section 3.2.1, page 20 –
   a. Please add a cross-reference to Table 1 in this section to refer to the sample density requirements for each land use category.
   b. The second paragraph, when referring to components between neighboring/adjacent structures, is unclear. Please modify this discussion to clarify the intent of this sentence.
9. Section 3.2.1.4, page 21 – One-half acre is equal to 21,780 sq. ft.; please correct this typographical error.
10. Section 3.2.1.5, page 22 – Table 1 states there is a *maximum* of 2 subsamples, however the text is stating a *minimum* of 2 subsamples. Please modify the table to be consistent with the text.
11. Section 3.2.4, Soil Sample Equipment Decontamination – The text states, “re-usable equipment *may be* decontaminated.” Re-usable equipment *must be* decontaminated between each sample location.
12. Section 3.5.2, page 27 – The in-text table is redundant with Table 2 and should be deleted.

13. Section 3.6 – Please define what is meant by a "sampling event" (i.e., one per sampling day, one per school, etc.?)
14. Section 3.8.1, page 29 – Please clarify if equipment be inspected before the first use each day or just the first use on the program.
15. Section 3.10, page 30 – In the bulleted list of records, please include property owner letters, other reports, and other correspondence.
16. Section 6.1.2.1, Field Data Verification – The Level A criteria bullet for "Field *preservation* technique" should be changed to "Field *preparation* technique". Also, please add "Sample preservation technique" to the bullet list. (These criteria lists are correct in the actual Level A/B Assessment Checklists.)

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

Sincerely,

NIKIA GREENE

Digitally signed by NIKIA GREENE
Date: 2021.06.22 16:36:51 -06'00'

Nikia Greene
Remedial Project Manager

Attachment: EPA Crosswalk

cc: (email only)
Butte File
Jenny Chambers; DEQ
Matt Dorrington, DEQ
Daryl Reed; DEQ
Jon Morgan; DEQ counsel
Carolina Balliew; DEQ
Harley Harris; NRDP
Katherine Hausrath; NRDP
Jim Ford; NRDP
Ray Vinkey; NRDP
John Gallagher; BSBC
Mollie Maffei; BSBC
Eric Hassler; BSBC
Brandon Warner; BSBC
Chad Anderson; BSBC
Karen Sullivan; BSBC
Julia Crain; BSBC
Abby Peltomaa; BSBC
Jeremy Grotbo; BSBC
Anne Walsh; UP

Robert Bylsma; UP counsel
Leo Berry; BNSF and UP counsel
Mark Engdahl; BNSF
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Chris Wardell; EPA
Dana Barnicoat; EPA
Charlie Partridge; EPA
Jean Belille; EPA
Ian Magruder; CTEC (Tech Advisor)
Janice Hogan; CTEC
Kristi Carroll; Montana Tech Library

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

Final

Residential Metals Abatement Program (RMAP) Quality Assurance Project Plan (QAPP) (Non-Residential Parcels)

Butte-Silver Bow County

and

Atlantic Richfield Company

July 9, 2021

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

Final

Residential Metals Abatement Program (RMAP) Quality Assurance Project Plan (QAPP) (Non-Residential Parcels)

Prepared for:

Butte-Silver Bow County
Superfund Division
155 W. Granite
Butte, Montana 59701

and

Atlantic Richfield Company
317 Anaconda Road
Butte, Montana 59701

Prepared by:

Pioneer Technical Services, Inc.
1101 S. Montana Street
Butte, Montana 59701

July 9, 2021

APPROVAL PAGE

Quality Assurance Project Plan for
BPSOU Residential Metals Abatement Program
(Non-Residential Parcels)
Butte Area NPL Site

NIKIA GREENE Digitally signed by NIKIA GREENE
Date: 2021.07.08 20:17:57 -06'00'

Approved: ______________________ Date: __________
Nikia Greene, Project Manager, EPA, Region 8
Quality Assurance Approval Official

Approved: Daryl Reed Date: 7/8/2021
Daryl Reed, Project Officer, Montana DEQ

Approved: ______________________ Date: 7/6/2021
Eric Hassler, Director
Department of Reclamation and Environmental Services
Butte-Silver Bow County

Approved: Julia M Crain Date: 7 July 2021
Julia Crain, Assistant Director
Department of Reclamation and Environmental Services
Butte-Silver Bow County

Approved: ______________________ Date: 7/6/21
Chad Anderson, Manager
Human Health/RMAP Division
Department of Reclamation and Environmental Services
Butte-Silver Bow County

Approved: ______________________ Date: 7/6/21
Abigail Peltomaa, Manager
Data Management Division/Quality Assurance Manager
Department of Reclamation and Environmental Services
Butte-Silver Bow County

Approved: Mike McAnulty Date: 7/1/21
Mike Mc Anulty, Liability Manager
Atlantic Richfield Company

Plan is effective on date of approval.

Revision 0

DISTRIBUTION LIST
Quality Assurance Project Plan for BPSOU Residential Metals Abatement Program (Non-Residential Parcels) Butte Area NPL Site

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A complete list of personnel to receive this document is provided on the associated cover letter distribution list. Atlantic Richfield Company will distribute the original Agency approved document. Subsequent annual revisions will be distributed by the Butte-Silver Bow County Department of Reclamation and Environmental Services Quality Assurance (QA) Manager.

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DOCUMENT MODIFICATION SUMMARY

Modification	Author	Version	Description	Date
0	Jesse Schwarzrock	Draft Final	Issued for Agency Approval	06/07/2021
1	Jesse Schwarzrock	Final	Issued for Agency Approval	07/02/21
2	Jesse Schwarzrock	Final	Issue Final for Agency Records	07/09/21

LIST OF ACRONYMS

Acronym	Definition	Acronym	Definition
Agencies	U.S. Environmental Protection Agency and Montana Department of Environmental Quality	**LBP**	Lead-based paint
ASA	American Society of Agronomy	**LCS**	Laboratory Control Sample
Atlantic Richfield		**LMS**	Laboratory Matrix Spike
bgs	Below Ground Surface	**MDL**	Method Detection Limit
BHRS	Butte Hill Revegetation Specifications	**mg/kg**	milligram per kilogram
BPSOU	BPSOU Butte Priority Soils Operable Unit	**ml**	milliliters
BSB	Butte-Silver Bow	**MS**	Matrix Spike
CAR	Corrective Action Report	**MSD**	Matrix Spike Duplicate
CERCLA	Comprehensive Environmental Response, Compensation, and Liability Act,	**NPL**	National Priorities List
CFRSSI	Clark Fork River Superfund Site Investigations	**pdf**	Portable document format
CLP	Contract Laboratory Program	**QA**	Quality Assurance
COC	Contaminant of Concern	**QAPP**	Quality Assurance Project Plan
DEQ	Montana Department of Environmental Quality	**QC**	Quality Control
DMP	Data Management Plan	**QMP**	Quality Management Plan
DQA	Data Quality Assessment	**RL**	Reporting Limit
DQO	Data Quality Objectives	**RMAP**	Residential Metals Abatement Program
DSR	Data Summary Report	**ROD**	Record of Decision
EBL	Elevated Blood Lead	**RPD**	relative percent difference
EDD	Electronic Data Deliverable	**SAP**	Sampling Analysis Plan
EPA	U.S. Environmental Protection Agency	**SOP**	Standard Operating Procedure
ESD	Explanation of Significant Differences	**SOW**	Statement of Work
ft^2	square feet	**SQL**	Structured Query Language
GIS	Geographical Information System	**SRM**	Standard Reference Material
HAZWOPER	Hazardous Waste Operations and Emergency Response	**SSHASP**	Site-Specific Health and Safety Plan
HEPA	High Efficiency Particulate Air	**SSSA**	Soil Science Society of America, Inc.
HUD	U.S. Housing and Urban Development	**UAO**	Unilateral Administrative Order
IC	Institutional controls	**USDA**	U.S. Department of Agriculture
ICIAP	Institutional Controls Implementation and Assurance Plan	**XRF**	X-ray fluorescence
ICP-AES	Inducted Coupled Plasma Atomic Emission Spectroscopy	**°C**	degrees Celsius

ICP-MS	Inductively-Coupled Plasma Mass Spectrometry	**$\mu g/m^3$**	Micrograms per cubic meter
ISWP	Individual Site Work Plan	**µm**	micron

1.0 INTRODUCTION

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

The contamination may originate from both mining-related (waste rock, tailings, aerial emissions) and non-mining-related sources. The potential sources of arsenic, lead, and/or mercury exposure addressed in the Program include lead, arsenic, and total mercury present in soil. The Program uses remediation and abatement of contaminated properties, and community awareness and education to ensure its effectiveness.

The Program requires systematic sampling of residential soil within the BPSOU. For areas outside of BPSOU, but within the 2020 RMAP Area boundary shown on Figure 1, a test-by-request campaign will be implemented in place of a systematic sampling approach to identify sampling efforts and potentially necessary remedial work. The Program also requires systematic sampling of playground and play areas (e.g., schools and parks) within the 2020 RMAP Area (see Figure 1). This QAPP addresses soil sampling of non-residential parcels (schools, parks, non-residential daycares) that fall under the RMAP umbrella. Interior assessments and sampling of these non-residential structures will be addressed through forthcoming QAPP revisions. Additionally, a separate QAPP will be prepared to support the assessment of residential RMAP parcels/properties.

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

1.1 Purpose

The BPSOU Quality Management Plan (QMP) (Atlantic Richfield Company, 2016) provides guidance to ensure quality environmental data collected for the BPSOU meet requirements mandated by the U.S. Environmental Protection Agency (EPA). The purpose of this Quality Assurance Project Plan (QAPP) is to provide guidance for future RMAP sampling and analyses of non-residential properties (e.g., schools, parks, and non-residential daycares) and to describe the QA/quality control (QA/QC) policies and procedures to be used during these efforts. The current Program boundary (depicted as the 2020 RMAP Area Boundary) is shown on Figure 1. This QAPP functions as the Program sampling and analysis plan (SAP) for all future non-residential sampling activities. A separate QAPP is being developed to address residential

RMAP parcels (including residential daycares and commercial properties containing living space).

This QAPP has been composed of standard recognized elements referenced in the *EPA Requirements for Quality Assurance Project Plans, EPA QA/R-5* (EPA, 2001); the *Guidance on Systematic Planning Using the Data Quality Objectives Process, EPA QA/G4* (EPA, 2006a); and the *EPA Region 8 Quality Assurance Document Review Crosswalk checklist* (EPA, 2016) provided in Attachment A. This QAPP includes the following four key elements:

- Program management and organization (Section 2.0).
- Measurement and data acquisition (Section 3.0).
- Reclamation material (Section 4.0).
- Assessment and oversight (Section5.0).
- Data review and usability (Section 6.0).

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

2.0 PROGRAM MANAGEMENT AND ORGANIZATION

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

2.1 Agency Oversight

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

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

2.2 Atlantic Richfield Company

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

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

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

Butte-Silver Bow is responsible for notifying qualifying property owners of potential exposure within the property, obtaining property owner access to conduct sampling and abatement (as needed), maintaining all Program data, and coordinating abatement activities. Key individuals comprising the BSB Department of Reclamation and Environmental Services are shown on Figure 2. The **Program project team** responsibilities are described below.

Director – Eric Hassler
The Director will oversee all activities throughout the department and is responsible for maintaining the official approved QAPP and for ensuring that the work is performed in accordance with the requirements contained herein. The Director is also responsible for consulting with the Assistant Director regarding any project deficiencies and resolutions.

Assistant Director – Julia Crain
The Assistant Director will perform various coordinating responsibilities across operable units while assisting with data related activities.

Manager, Human Health/RMAP Division - Chad Anderson
The Human Health/RMAP Division Manager will coordinate all RMAP activities and oversee division crews and staff. Furthermore, the Manager is responsible for verifying effective implementation of QAPP requirements and procedures and scheduling sampling work to be completed. This includes reviewing field and laboratory data and evaluating data quality. The Manager will also complete a site walk-through, prepare a site-specific work plan for approval of all reclamation projects prior to implementing, and provide project oversight.

The Manager will also be responsible for the oversight of field team laborers during abatement activities to complete the duties listed below:

- Scheduling sampling work to be completed.
- Managing requests for property access, tracking the status of access requests, and maintaining copies of completed agreements received from property owners (refer to Section 2.9.1 and 3.1).
- Ensuring completed agreements are photocopied, scanned, and the electronic version stored on a hard drive.
- Ensuring a copy of the individual access agreement is included in the project record files.
- Ensuring that all team members have reviewed the QAPP and the QAPP procedures are properly followed during field activities.
- Conducting daily safety meetings, assisting in field activities, and documenting activities in the field logbook or appropriate field collection device.
- Coordinating field activities and managing equipment.
- Solving problems and making decisions in the field.
- Managing technical aspects of the project.

- Maintaining an on-the-ground overview of the project tasks by observing site activities.
- Ensuring compliance with technical project requirements and the Site-Specific Health and Safety Plan (SSHASP).
- Identifying issues during field activities and reporting all issues to the RMAP Coordinator.

Data Management Division/Quality Assurance Manager – Abigail Peltomaa
The Data Management Division Manager assumes the role of Program QA Manager and is responsible for the data management and QA/QC of all field data, reviewing and maintaining laboratory data packages, compiling an annual Data Summary Report (DSR), maintaining quality records (as per described in Section 2.9.7), and reporting final remediated property requirements to the Agencies.

2.4 Analytical Laboratory

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

2.5 Problem Definition and Background

Contamination of properties described herein may originate from both mining-related (waste rock, tailings, aerial emissions) and non-mining-related sources. The potential sources of arsenic, lead, and/or mercury exposure addressed in the Program include arsenic, lead, and total mercury in soil.

Assessment is needed to determine remediation or abatement requirements if non-residential parcel soil (schools, parks, or non-residential daycares) exceeds solid media action levels.

This QAPP was developed in response to the Agencies *2006 BPSOU Record of Decision* (BPSOU ROD) (EPA, 2006b) and *Explanation of Significant Differences* (ESD) to the *2006 Butte Priority Soils Operable Unit Record of Decision* (EPA, 2011a). The ESD modified the soil sampling depth from 0 to 2 inches to the depth intervals discussed in Section 3.2; changed the soil removal from a minimum depth of 18 inches to the minimum depth of 12 inches or to the soil bedrock interface if less than 12 inches; and extended the project schedule to accommodate expansion of the Program.

This QAPP was also developed in response to the Agencies *2020 Unilateral Administrative Order Amendment* (UAO Amendment) *for "Partial Remedial Design/Remedial Action Implementation and Certain Operation and Maintenance at the Butte Priority Soils Operable Unit/Butte Site"* (EPA Docket No. CERCLA-08-2011-0011) (EPA, 2020a). The UAO Amendment expanded the RMAP boundary (see Figure 1) and also expanded to include schools, parks, and daycare facilities.

RMAP program representatives will provide results of monitoring and sampling data to the Agencies and notify property owners of necessary abatement (as needed).

2.6 Project Description and Schedule

The Program is designed to mitigate exposure to sources of arsenic, lead, and mercury contamination to residents of the BPSOU and Expanded Area .

In 2020, the Program was expanded to perform sampling within the 2020 RMAP Area boundary provided in Figure 1. Specific exclusion areas are also identified in Figure 1. Sampling outside of the BPSOU but within the expanded boundary will be performed on a test-by-request basis.

Components of the Program include environmental sampling and remediation, long-term tracking and data management, and education and outreach. Medical monitoring is conducted as a sister program to the Program. The long-term tracking and data management ensures properties will be sampled, evaluated, and remediated, if necessary. The long-term tracking and data management will be continued for the life of the Program. The BPSOU *Final Data Management Plan (DMP)* (Atlantic Richfield Company, TBD) describes the data management.

The Program includes systematic sampling for additional specific areas within the 2020 RMAP Area such as parks and play areas, schools, and non-residential daycares. Program eligibility is described in the *Revised Final Multi-Pathway Residential Metals Abatement Program (RMAP) Plan* (BSB and Atlantic Richfield Company, 2020).

The objectives of this QAPP are as follows:

1. Provide consistent means and methods of non-residential parcel (schools, parks, and non-residential daycares) soil sampling and analyses associated with the Program sampling activities and ensure compliance with performance standards. Interior assessment/sampling of these parcels will be addressed under forthcoming QAPP revisions.
2. Describe the requirements for sample collection and analyses.
3. Provide data to identify and mitigate potentially harmful exposure to sources of arsenic, lead, and mercury.

2.6.1 Project Schedule

Environmental assessment of schools, non-residential daycare facilities, playgrounds and play areas soils and vegetated areas will begin in 2021 with the goal of completing as much sampling and subsequent remediation work as possible prior to the start of the 2021-2022 academic calendar year. A systematic schedule to complete environmental assessments of structures and properties presently used as schools, playgrounds and play areas will be proposed annually. The annually proposed schedule will account for the results of previously completed environmental assessments, provision of access, and the availability of Program resources to implement and oversee subsequent environmental assessments and remediation, if required. Environmental assessment of playgrounds and play areas within designated parks will be coordinated with the entity responsible for their management (e.g., BSB Parks and Recreation).

2.7 Quality Objectives and Criteria

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

2.7.1 Data Quality Objectives

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

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

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

Step 1: State the Problem - *The purpose of this step is to describe the problem to be studied so that the focus of the investigation will not be ambiguous.*

> **Describing the problem.** Properties in Butte and within the Expanded 2020 RMAP Area (see Figure 1) had the potential to be contaminated by historical mining activities and related contaminants. The proximity of properties to mining wastes and operations may have resulted in contamination of non-residential properties such as schools, parks, and non-residential daycare facilities.

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

Establishing the planning team. Project personnel, roles, and responsibilities are detailed in Sections 2.1 through 2.3 of this document.

Describing the conceptual model of the potential hazard. Historical surface and underground mining activities resulted in the presence of contaminants in soil around Butte due to waste dumping and deposition of aerial emissions from smelters/mills. Other, non-mining sources have also resulted in contamination in some areas. People may contact contaminated soil at non-residential properties through pathways such as dermal contact and incidental ingestion; for example, children playing at a park may have skin contact with exposed soil, some of which could be ingested through hand to mouth transfer. When people contact contaminated soil, they may be exposed to contaminants, which could pose a health risk if concentrations are above health-protective concentrations, such as action levels. In order to investigate this problem, data quantifying contaminant concentrations will need to be collected, compared to the appropriate project action levels, and used for remedial decision making.

Identifying available resources, constraints, and deadlines. Atlantic Richfield Company (Section 2.2) and Butte-Silver Bow (Section 2.3) will provide necessary project resources (financial and staffing) to properly implement the program. Project schedule details are provided in Section 2.6 and 2.6.1.

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

Key elements/questions. The Program requires that all area schools, parks, and non-residential daycare facilities within the BPSOU be sampled and assessed. The goal is to use best efforts to obtain access to all applicable properties within the expanded 2020 RMAP Area (see Figure 1) that have not previously been sampled in accordance with current methodology to complete outdoor assessments. Exterior soil sampling will be addressed by this version of the QAPP. Interior assessments/sampling will be addressed at a later date under a future QAPP revision.

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

Are soil concentrations of arsenic, lead and/or mercury at non-residential properties present at levels that may pose a risk to human health (e.g., above the action levels)?
Determining alternative actions. Possible alternative actions are as follows:

- Take no action – If all analyte concentrations are below the appropriate project action level.
- Complete Remedial Action – If an analyte concentration is above the appropriate project action level. Remedial action would consist of soil removal and disposal at an

Agency approved repository followed by backfill with Agency approved borrow material.

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

- Determine whether Remedial Action (soil removal) is required.

Step 3: Identify the Information Inputs - *The purpose of this step is to identify the informational variables that will be required to resolve the decision statements and determine which variables require environmental measurements.*

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

Information regarding the land use of the different areas within the parks and schools should inform the sampling design for each area. Five primary land uses have been identified for non-residential RMAP properties. These land use categories help inform the approach for sampling each property, and include:

Land Use Category #1: playground areas.

Land Use Category #2: highly accessible areas/barren sports fields.

Land Use Category #3: maintained grass areas/grass sports fields.

Land Use Category #4: low access areas/low maintenance areas/open space.

Land Use Category #5: flower/vegetable gardens.

Land use information should be used to make decisions about the appropriate sample count/density and depth intervals to be sampled for each area, and to identify action levels that are protective of the specified land uses.

Sample coordinates and depth intervals should also be documented so that sample results are linked to specific locations and depths, to inform remediation decisions. If chips from building exterior lead based paint (LBP) are identified in a sampled area, this should also be documented as it is likely to influence lead concentrations in soil.

Identifying the number of variables to be collected. Arsenic, lead, and mercury concentrations should be determined for each sample collected.

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

Based on a review of the basis of the soil action levels, the residential soil action levels should be employed in evaluating the soil sampling results for the schools. The application of the residential action levels is conservative for a school scenario; however, use of more conservative action levels is appropriate, especially considering the school setting and community sensitivity to childhood exposures. The use of the residential action level in making cleanup decisions is consistent with what has been done historically for Butte parks. Additionally, residential soil action levels are also being used for the Anaconda Smelter site when making cleanup decisions for schools.

The BPSOU residential action levels (Arsenic – 250 mg/kg, Lead – 1,200 mg/kg, Mercury – 147 mg/kg) will be utilized for all work completed under this QAPP (see Table 1).

Identifying appropriate sampling and analysis methods. Multiple sampling strategies (discrete, incremental, composite, etc.) should be considered for potential use on this project. Given the large areas contemplated for this project, exclusive discrete sampling may not be the most appropriate option given its common deficiencies including poor spatial coverage, inadequate sample density, or data that cannot be used to statistically represent the entire area of interest with a reasonable level of confidence. While incremental sampling is a type of composite sampling, it would represent a change from current sampling practices within the Silver Bow Creek/Butte Area NPL Site. As such, a change could create issues surrounding consistency and comparability with previous RMAP and NPL Site sampling results. In addition to having been used historically within the NPL Site and on the RMAP project specifically, composite sampling is the recommended approach for sampling residential parcels provided in EPA's *Superfund Lead-Contaminated Residential Sites Handbook* (EPA, 2003). For consistency and comparability with previous RMAP and NPL Site sampling results, composite sampling may be the most appropriate sampling method for the project.

X-ray fluorescence (XRF) has been used historically to analyze arsenic and lead concentrations in Butte soils. This method provides a quick output that can be used for immediate decision making. However, it is less sensitive than laboratory analytical methods, and cannot be used for mercury analysis. Because samples must be packaged and shipped to a laboratory for mercury analysis, it may be more practical to have all three metals analyzed by the laboratory via inorganic analyses. Inorganic analyses data from an analytical laboratory can also be validated. If inorganic analyses are used, expedited laboratory analysis (5 to 7 business day turn around on data and level 2 data packages and 10 to 12 business day turn around on data and level 4 data packages) and data validation (7 business day turn around after data packages are received) options should be investigated in order to achieve the project assessment and remediation goals.

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

Specifying the target population. The 2020 RMAP/Program area addressed under this QAPP will include exterior soil of schools, parks, and non-residential daycares identified in Figure 1. Interior assessments and sampling of these properties will be addressed under a subsequent revision to this QAPP. Because of differences in potential soil exposures with depth, and for consistency and comparability with previous RMAP sampling, soil should be sampled separately from discrete depth intervals. For example, EPA recommends sampling soil from the 0 to 2-inch depth interval to assess contact by most activities of children, while some activities may result in contact with deeper soil, and vegetable gardens, which have been observed at some schools in the 2020 RMAP/Program area may involve digging up to 2 feet. Exterior soil sampling should be conducted at multiple depth intervals (0 to 2 inches, 2 to 6 inches, and 6 to 12 inches) to enable assessment of potential health risks under different land uses, and to obtain data that are comparable to those from previous sampling efforts. Flower/vegetable garden components should be sampled at additional depth intervals of 12 to 18 inches and 18 to 24 inches.

Description of what constitutes a sampling unit. Sampling units should be defined based on land use information. Sampling unit extents are defined as the maximum area to be sampled to support decision-making for each of the five specified land-use categories identified for non-residential RMAP properties (see Step 3). The EPA's *Superfund Lead-Contaminated Residential Sites Handbook* (EPA, 2003), previous RMAP QAPP, and procedures for sampling schools in nearby Anaconda were reviewed to inform sampling unit extents appropriate for each land use type. The recommendations below were developed consistent with EPA recommendations, other RMAP sampling efforts, and sampling of schools where similar types of contamination are present. These recommended sampling unit extents should inform development of the sampling plans for each property.

Land Use Category #1 (playground areas): 6,250 square feet.

Land Use Category #2 (highly accessible areas/barren sports fields): 9,375 square feet.

Land Use Category #3 (maintained grass areas/grass sports fields): 10,890 square feet.

Land Use Category #4 (low access areas/low maintenance areas/open space): 21,780 square feet.

Land Use Category #5 (flower/vegetable gardens): 3,125 square feet.

Time frame for collecting data and making the decision. The temporal boundaries of the investigation include the time from when evaluation and sampling actions begin at each property to the time these actions are completed. No temporal variability in soil concentrations is expected, so the sampling effort should be primarily dictated by when it is easiest to conduct sampling, meaning when no snow is present and when school facilities are not in use (i.e., summer). School sampling should be completed prior to when school starts in the fall. Outreach meetings should be conducted with each school to better understand individual schedule restraints (summer activities/camps, construction projects, etc.)

Specifying the scale for decision making. For the non-residential RMAP properties, the sampling unit extent for each land use category should be specified as the maximum area for decision-making by land use type to ensure that any location where arsenic, lead, or mercury concentrations are above health-protective action levels is remediated. Some properties may have multiple land uses and more than one sampling unit. By setting the decision unit equal to the sampling unit, decisions to remediate can be made for subareas of a property, rather than on a property-wide basis, and any subarea with analyte concentrations above action levels can be addressed even if property-wide removal is not warranted.

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

Identification of the population parameters most relevant for making inferences and conclusions on the target population. Arsenic, lead, and mercury concentrations should be measured for each sampling unit as determined by analysis of each corresponding soil sample collected. The concentration measured in each sampling unit is the population parameter that should be used to make inferences and conclusions for each decision unit (i.e., the decision unit should be set equal to the sampling unit to support health-protective decision-making).

Specifying the theoretical decision rule. The theoretical decision rule is as follows.

- If the analyte concentration measured in the sampling unit (i.e., the average concentration within each decision unit for either Arsenic, Lead, or Mercury) exceeds the appropriate Residential Action Level detailed in Table 1, then the soil from the corresponding sampling area will be removed using conventional equipment (such as backhoes, small Bobcat-type loaders, and hand tools), and transported to the Butte Mine Waste Repository using dump trucks.

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

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

Because the goal of the RMAP is to protect human health, the tolerance for making a Type II (false negative) error is lower than the tolerance for making a Type I (false positive) error. Therefore, a sampling design and analysis method that minimizes the potential for false negative decision errors should be selected. Due to the potential for work to occur over more than one season and the need to make decisions on a property-by-property basis, the experiment-wise error rate will likely be difficult to assess and efforts should be made to reduce the Type II error rate at the decision unit, rather than at the project-wide level.

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

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

A composite sampling design would best support the goal of reducing Type II error potential by limiting the size of the decision unit to the extent of the sampling unit. The EPA (2003) handbook states that, "*the overall goals of the sampling effort are to estimate an average soil concentration for risk assessment purposes and to provide information to determine the scope of required cleanup actions.*" The composite sampling method is intended to better approximate potential average exposure to a receptor as they move across an area, rather than remaining at a single spatial point which is less likely to occur. Therefore, collecting a composite sample to estimate the average concentration of each analyte in soil across the extent of each sampling unit is a preferable approach compared to collecting a discrete sample from one location within each area.

In addition to lowering the potential for Type II errors, study error should be minimized through proper training of the field sampling team, sample documentation and handling, the use of appropriate analytical methods that achieve method detection limits below the action levels, analysis of field and analytical QC samples, analysis of precision, accuracy, and other measurement performance criteria (described in detail in Section 2.7.2), and data validation. Decisions should be made using data that meet the performance and acceptance criteria; if these criteria are not met, corrective action steps should be taken.

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

Selecting the sampling design. The data collection scheme is designed to ensure that the information will be of sufficient quality and quantity to determine the component(s) of individual schools, parks, and non-residential daycares requiring remedial action (and the depth to which remedial action is required). The information and outputs generated in Steps 1 through 6 of the DQO process informed selection of the optimized approach for soil sampling and analyses at non-residential RMAP properties described in this final step of the process.

The RMAP sampling plan generally follows the EPA's *Superfund Lead-Contaminated Residential Sites Handbook* (EPA, 2003) composite sampling design (with one composite collected per yard component representing an exposure area that would be remediated). For this reason and because this approach supports the goals of obtaining average concentrations of arsenic, lead, and mercury across each sampling unit and minimizing the potential for false negative conclusions, the schools program is designed to also rely on composites that reflect portions of exposure areas. Arsenic, lead, and mercury concentrations will be determined through composite samples collected from non-residential RMAP properties (schools, parks, and non-residential daycare facilities). The goal of composite soil sample collection and analyses is to obtain a reliable estimate of the average concentration of a COC in soil over a specified area where exposure may occur, for comparison to the appropriate action level for that area.

For each property, sampling unit extents will be defined based on land use types identified at the property, based on the recommendations described in Step 4. Land use should also inform the number of composite subsamples to be collected across each sampling unit. For consistency with the RMAP and with EPA guidance, the same information used to determine appropriate sampling unit extents for each land use category (EPA's lead handbook, previous

RMAP sampling, and Anaconda schools sampling) also informs determination of subsample counts recommended for each land use-specific sampling unit. Details of the extent and number of subsamples to be collected from each area of a non-residential property, based on land use within that area, are provided in Table 1 as well as in Sections 3.2.1.1 through 3.2.1.5. Exterior soil sampling will be conducted at multiple depth intervals (0 to 2 inches, 2 to 6 inches, and 6 to 12 inches) for all five land use categories. Flower/vegetable garden components (Category #5) will be sampled at additional depth intervals of 12 to 18 inches and 18 to 24 inches.

Consistent with prior sampling programs, samples will be sieved to the less than 250 micrometers (µm) fraction, reflecting the fine fraction of soil most likely to adhere to children's hands. More recent EPA guidance (EPA OLEM Directive 9200.1-128) requires sieving to less than 150 µm based on studies that show lead enrichment in very fine soil fractions (e.g., less than 63 µm). There are no data adequate to predict if the less than 150 µm fractions might be detectably enriched as compared with the less than 250 µm fraction. In light of this uncertainty, EPA has agreed with use of the less than 250 µm fraction for the 2021 sampling program while a particle size enrichment demonstration study is planned and conducted.

Based on the assessment of the limitations and benefits of potential sample analyses options completed in Step 3, laboratory analyses was identified as the preferred approach for measurement of arsenic, lead, and mercury concentrations in composite soil samples. Arsenic and lead concentrations will be determined per EPA Method 6010 (inductively-coupled plasma atomic emission spectroscopy [ICP-AES]) or EPA Method 6020 (inductively-coupled plasma mass spectrometry [ICP-MS]). Mercury concentrations will be determined per EPA Method 7471B (Manual Cold-Vapor Technique). The detection limits associated with these methods are expected to be well below the applicable Action Levels (see Table 1).

Decision units will be set equal to the sampling unit. The relationship between the average COC concentration and the action level provides the input needed to resolve the decision statements outlined in Step 2 in order to determine whether abatement is required for non-residential RMAP soil. For each decision unit, the decision question (*Are soil concentrations of arsenic, lead and/or mercury at non-residential properties present at levels that may pose a risk to human health (e.g., above the action levels)?*) will be addressed by comparing the composite soil sample result from each sampled depth interval within each sampling unit to the corresponding action level.

Details on how the design should be implemented together with contingency plans for unexpected events. Soil sampling shall be implemented per the guidelines provided in Section 3.2. 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. Corrective action procedures are outlined in Sections 5.1 and 5.2. Any unexpected/unplanned events not specifically addressed by this QAPP will be discussed with Agency personnel and addressed through forthcoming QAPP revisions.

Specifying the Quality Assurance and Quality Control procedures. Sufficient data quality will be achieved through the field and laboratory quality control measures (Sections 3.6 and 3.8 respectively) including the use of appropriate sample collection, handling, and chain of custody procedures and laboratory analytical methods, quality control sample analysis (field and laboratory), assessment of the performance criteria described in Section 2.7.2, following the corrective action procedures detailed in Sections 5.1 and 5.2, and analytical data validation (Section 6.0).

2.7.2 Measurement Performance Criteria for Data

Measurement performance criteria are established by defining acceptance criteria and quantitative or qualitative goals (e.g., control limits) for precision, accuracy, representativeness, comparability, completeness, and sensitivity (PARCCS) of measurement data. The definitions of precision, accuracy, representativeness, comparability, completeness, and sensitivity are provided below. Acceptance limits are detailed in Section 3.5.2 for each measurement performance criteria. Equations for calculation of precision, accuracy, and completeness are provided in Table 2. Additional QC acceptance criteria are provided in Table 3.

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

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

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

Representativeness
Representativeness is the degree to which sample data represent a characteristic of a population, parameter, or environmental condition. Representativeness is a qualitative parameter that is most concerned with proper design of the sampling and analytical schemes. Representativeness is

achieved by determining the number and locations of samples and the appropriate sampling techniques needed to depict, as accurately and precisely as necessary, the conditions being measured. Representativeness deals with protocols for sample storage, preservation, and transportation; analyzing samples with appropriate methods, techniques, and instrumentation; and using the methods to document these protocols. Representativeness will be achieved through judicious selection of sampling locations and methods. This QAPP requires that samples are representative of the medium being sampled and that there are a sufficient number of samples to meet the project DQOs and satisfy the project remedial action design elements.

Comparability

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

Completeness

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

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

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

Sensitivity

Sensitivity is related to the ability to compare analytical results with project-specific action levels. Analytical quantitation limits for the sample analytes should be below the level of interest to allow an effective comparison.

Method Sensitivity

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

Additional details regarding bias, sensitivity, and QC acceptance criteria are included in Section 3.5.2

Laboratory Analyses

The method sensitivity for laboratory analyses is determined as part of the laboratory's SOPs. A review of these detection limits will be conducted as part of the data validation process.

2.8 Special Training

All RMAP field personnel will review the requirements of this QAPP and receive training on Program-related tasks during a project meeting held prior to the beginning of fieldwork. A review of sampling procedures and requirements will be completed prior to field activities to ensure sample collection and handling methods are according to QAPP requirements. Field personnel will be trained in proper use of field equipment, sample collection tools, etc., and procedures according to field data collection SOPs (Attachment C-1) and methods described in the Program. Field personnel performing sampling activities or members who can potentially contact contaminated materials should receiver hazardous waste operations and emergency response (Hazardous Waste Operations and Emergency Response [HAZWOPER]) training.

The BSB Department of Reclamation and Environmental Services Director is responsible for ensuring field personnel receive appropriate training and will maintain up-to-date training records and/or certifications. The BSB Department of Reclamation and Environmental Services Human Health/RMAP Division Manager will assure that each member of the sampling team obtains and is familiar with the recent version of the QAPP, will maintain signatures of each team member who has read the QAPP (including reviews and addenda, as necessary), and make sure each team member has been trained in the appropriate sample collection methods per the Program. The Human Health/RMAP Division Manager will review the SSHASP 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 also 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. One hard copy of the approved version of this QAPP will be maintained for reference in the field vehicle and/or field office. All field team personnel will have access to Portable Document Format (.pdf) files of the complete QAPP.

2.9 Documents and Records

This section describes procedures for documentation management and record keeping for this QAPP from initial record generation through final data formatting and storage. All sampling data conducted for all media under the Program and records of property access requests are housed within the Program database. The Program database is housed in an Access Structured Query Language (SQL) server database and maintained by BSB. Document backups are contained in the BPSOU Document SharePoint and EPA document repository. Refer to the BPSOU *Final Data Management Plan* (Atlantic Richfield Company, TBD) for additional details regarding data management, backup, and storage. Atlantic Richfield and BSB will coordinate Agency testing of the database with the program architects and primary users in a manner to minimize provision of written comment and the potential misinterpretation of those comments.

2.9.1 Property Access Agreements

An executed sampling access agreement (see Attachment B) must be obtained from the property owner (which for non-residential properties may include BSB or other non-private entities/agencies) before sampling takes place. Similarly, an executed Construction Access Agreement must be obtained before remediation begins. Program access agreements are also described in detail within the *Institutional Controls Implementation and Assurance Plan (ICIAP)* (Atlantic Richfield Company, 2019). The agreements represent a temporary agreement between BSB and the property owner stating that the owner is willing to permit BSB to conduct certain sampling and abatement activities on the specified property. Completed agreements will be photocopied, scanned, and the electronic version stored on a hard drive. The status of property access will be tracked in the Program's database tracking system. A copy of the access agreements (Attachment B) will also be included in the project record files.

2.9.2 Field Documentation

Field documentation provides a description of site conditions during sampling activities and provides a permanent record of all field activities. Field documentation will primarily be achieved through electronic means (i.e., field tablets). Field documentation includes a sample location map of the site that shows property boundaries, structures, driveways, contaminant source material, gardens, and lawns. Field personnel creating the sample location map will delineate property features with an accuracy of approximately plus or minus 2.0 feet. Each property will be divided into components (e.g., play area, high access area, etc.) for sampling, and these areas will be identified on the map.

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

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

For any field sampling work, the field documentation will include all applicable items from the Level A/B Assessment Checklist (see Section 6.1.2.1 and Attachment D). At a minimum this includes documentation of the following:

- Sample team and/or leader.
- Sample location, depth, and traceable sample designation number.
- Sample type collected.
- Date and time of sample collection.
- Samples taken by other parties (note the type of sample, sample location, time/date, sampler’s name, sampler’s company, and any other pertinent information).
- Sampling method, particularly any deviations from the field SOPs (Attachment C).
- 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.
- Collection of field duplicates.
- Decontamination of sampling equipment.
- Sample custody documentation.
- Sample preservation (if used).

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

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

2.9.3 Field Photographs

Field personnel will use a digital camera to take photographs at the site. Photographs may be taken of sampling locations, field activities, and to document site conditions, as necessary. Photographs should include a scale in the picture when practical. Documentation of all photographs taken during sampling activities will be recorded in a bound field logbook or appropriate field collection device and will specifically include the following for each photograph taken:

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

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

2.9.4 Chain of Custody Records

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

A copy of each as-transmitted chain of custody form will be scanned and stored on a hard drive. Chain of custody records will also be copied to the project record files (refer to Section 3.10).

2.9.5 Analytical Laboratory Records

Results received from the laboratories will be documented both in report form and in an electronic format. Laboratory documentation includes laboratory confirmation reports such as information on how samples have been batched, the analyses requested, data packages containing the laboratory report and the electronic data deliverable (EDD), and any change requests or corrective action requests. Section 6.1.3 lists the laboratory reporting requirements in detail. The deliverable (data package or report) issued by the laboratory must include data necessary to complete validation of laboratory results. Original reports and electronic files received from laboratories will be maintained with the project quality records. Refer to the BPSOU *Final Data Management Plan* (Atlantic Richfield Company, TBD) for additional requirements.

2.9.6 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 and submitted to the Agencies for review and approval. For the school sampling portion of this project, these data would be anticipated to be submitted on a per school basis to decrease the turnaround time required for landowner reporting as much as possible. Upon received Agency approval, the sample results (for all analytes) will be reported to individual landowners along with a letter explaining what the results indicate (see result letter templates in Attachment E). The action levels for arsenic, lead, and mercury will be reported along with sample results.

Following landowner notification, sample results will be used to develop an individual site work plan (ISWP) for each parcel where sample results exceeded BPSOU action levels (Table 1). The ISWPs will summarize the number of individual sampling components associated with each property, depth of each sample, and corresponding surface area of each component.

In addition to the “real time” submittals described above, all sampling data will be forwarded to the Agencies for review and approval in the form of an annual DSR. This DSR will include figures displaying location of parcels sampled, analytical results, and copies of all field data. As described above, all sampling data will reside in the project records.

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

2.9.7 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 BSB Department of Reclamation and Environmental Services Data Management Division Manager/QA Manager (or designee) in cooperation with the BSB Department of Reclamation and Environmental Services Director, and will include the following 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, and faxes).
- Copies of completed access agreements (Attachment B) for the individual properties sampled.
- Individual property maps, including any field drawings and field photographs.
- Field documentation forms.
- Copies of all field documentation/records.
- Copies of all sample chain-of-custody forms.
- Copies of all laboratory agreements and amendments.
- Laboratory data packages (printed report and electronic version).
- Documentation of field and/or laboratory audit findings and any corrective actions.
- Draft and final delivered versions of all reports and supporting procedures such as statistical analyses, numerical models, etc.

All project data will be maintained indefinitely in the BPSOU Residential Soils and Attic Dust Global Information System (GIS) database, or similar format. The database has not yet been completely developed, and Atlantic Richfield/BSB will be working with the Agencies to finalize the database. This is a long-term project with access to the database provided to many interested parties. Any addendums or revisions to this QAPP will be electronically distributed to all parties identified on the distribution list.

3.0 MEASUREMENT AND DATA ACQUISITION

This section addresses all aspects of project design and implementation for generating and acquiring data. Adhering to the procedures provided in Attachment C in this QAPP and described in this section ensures that the 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 followed.

3.1 Property Access

Non-residential RMAP sampling occurs at a combination of third-party and BSB-owned properties. Prior to conducting any sampling or cleanup activities at a third-party property, access must be obtained from the property owner in the form of an executed sampling access agreement (see Attachment B). To gain access to these properties, Program representatives will actively pursue access in the form of phone calls, text messaging, and in person visits. As required, up to three documented attempts to gain access will be made. After the third unsuccessful contact attempt, Program representatives will cease actively pursuing sampling access. The owner will still be allowed to request sampling on a test-by-request basis. Transfer of property ownership will reset the Program's attempts to gain access to zero. At that point, Program representatives will start over on documented attempts to gain sampling access with the new property owner. The Program will monitor ownership changes on an annual basis.

The Human Health/RMAP Division Manager (or designee) 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 and the electronic version stored on a hard drive. A copy of the access agreements will also be included in the project record files.

Any dispute concerning access should be brought to the attention of the Agencies. It is essential to begin access procurement as early as possible in the remedial process to avoid potentially lengthy delays. If access for response work cannot be reasonably obtained from a third-party owner, EPA may choose to use its authorities under Comprehensive Environmental Response, Compensation, and Liability Act (CERCLA) to secure access and as provided in the current Unilateral Administrative Order (UAO) (EPA, 2011b) and any updated UAOs.

When access is denied (or the owner is deemed to be unresponsive through three unsuccessful contact attempts), Program representatives will track the attempt to gain access of the property for environmental assessment within the Program database. After three attempts are recorded, the property will be flagged in the database (as either having declined access or becoming non-responsive) and the Agencies will be notified of the property status. At this time, the Agencies may elect to issue the property owner an enforcement letter. A copy of the Agency notice form letter is provided in Attachment B-2. Future changes in ownership will be monitored annually. If ownership changes, the access procurement process will be re-initiated.

3.2 RMAP Soil Sampling (Non-Residential Parcels)

All non-residential RMAP soil sampling work (schools, parks, and non-residential daycares) will be conducted as described below to determine the presence of the COCs listed in Table 1. Field personnel will follow the procedures in the SOPs (Attachment C-1) and will record all information in the field logbook/data collection device. The RMAP non-residential parcels will be broken down into sampling components and characterized by five land use categories:

- Land Use Category #1 – This category consists of playground areas. This will typically be defined as the area around playground equipment such as swings, slides, jungle gyms, and other types of equipment.
- Land Use Category #2 – This category consists of high accessible areas near school buildings such as school courtyards. Also contained within the category will be barren sports areas such as a baseball/softball infield.
- Land Use Category #3 – This category consists of maintained grassy areas such as sodded school grounds and turf covered sports fields.
- Land Use Category #4 – This category consists of low use/low maintenance areas that are rarely accessed by children. Examples include school grounds that are fenced off to restrict access by students.
- Land Use Category #5 – This category consists of vegetable and/or flower gardens.

Sample request paperwork will be pursued by program representatives for all non-residential RMAP parcels. Current school/non-residential daycare parcels are listed in Table 4 and the park/playground/open area parcels are listed in Table 5. Tables 4 and 5 are believed to be comprehensive. If additional relevant parcels are identified through future Stakeholder meetings, these additional parcels will be considered for inclusion on the RMAP sampling list. Butte-Silver Bow County will catalogue action items and document milestones in the Program database. The EPA will be notified prior to sampling any parks and/or schools.

Consistent with how residential sampling logic does not change for parcels within or outside the BPSOU, all non-residential RMAP parcels within the 2020 RMAP Area (see Figure 1) will be characterized and sampled per the requirements of this section regardless of geographic location within the 2020 RMAP Area. This will ensure proper characterization of all non-residential parcels regardless of their location in relation to the BPSOU boundary.

Generally speaking, the property boundary will be used to establish the extent of the sample area. Exceptions to this rule will include, but are not limited to, school areas that are inaccessible to children due to existing fencing, heavy existing cover (e.g., trees), and steep terrain. Field sampling plans will be developed for each parcel and submitted to the Agencies for review and approval prior to beginning sampling work. The procedures for RMAP soil sampling are summarized below.

3.2.1 Sample Density, Location, and Compositing

Sample locations within sampling components will be determined by sampling personnel based upon site-specific conditions. Non-residential RMAP sampling density and compositing decisions will be made dependent upon current land use determinations.

Soil subsamples will not be collected from an area between adjacent structures where the distance between the structures is less than 3 feet.

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. Opportunistic samples will be collected of suspect piles, discolored materials, or notable barren areas greater than approximately 25 feet by 25 feet in area. All opportunistic samples collected will be comprised of a minimum of 3 subsamples.

Soil samples for mercury analysis for this project will be collected by removing a subsample aliquot from the homogenized sample contained in the Ziploc® bag during the sample collection process and placed in glass containers. This process helps to ensure sample representativeness between the sample aliquots. According to Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, US EPA Publication SW 846, the sample jars for mercury analysis will be shipped from the field on ice to the primary laboratory.

The project soil samples collected in Ziploc® bags for arsenic and lead will be shipped from the field and stored by a second laboratory at ambient temperature conditions.

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.

3.2.1.1 Land Use Category #1 (Playground Areas)

For Land Use Category #1 sampling components, subsamples will be collected from a minimum of 3 subsample locations or at a rate of 1 subsample per 625 square feet (ft^2) (25 feet by 25 feet) in surface area per sampling component, whichever is greater. Subsamples from these locations will be composited in the field, and a single composite sample per depth interval will be analyzed for arsenic, lead, and mercury. Each subsample should have similar mass so that each location is equally represented in the total sample mass. The maximum area represented by a single composite sample will be 6,250 ft^2 (meaning a maximum of 10 subsamples will be collected from any single Land Use Category #1 sampling component) (see Table 1).

Samples will be thoroughly mixed in a clean 1-gallon plastic Ziploc® bag or stainless steel bowl to ensure representativeness of the aliquot ultimately submitted for analyses. During this homogenization process, particles greater than 0.5 inches in diameter will be discarded. Sample volumes will consist of approximately 500 to 800 grams of material. Samples will be submitted to the laboratory by the samplers under chain of custody procedures.

3.2.1.2 Land Use Category #2 (Highly Accessible Areas/Barren Sports Fields)

For Land Use Category #2 sampling components, subsamples will be collected from a minimum of 3 subsample locations or at a rate of 1 subsample per 625 ft^2 (25 feet by 25 feet) in surface area per sampling component, whichever is greater. Subsamples from these locations will be composited in the field, and a single composite sample per depth interval will be analyzed for arsenic, lead, and mercury. Each subsample should have similar mass so that each location is equally represented in the total sample mass. The maximum area represented by a single composite sample will be 9,375 ft^2 (meaning a maximum of 15 subsamples will be collected from any single Land Use Category #2 sampling component) (see Table 1).

Samples will be thoroughly mixed in a clean 1-gallon plastic Ziploc® bag or stainless steel bowl to ensure representativeness of the aliquot ultimately submitted for analyses. During this homogenization process, particles greater than 0.5 inches in diameter will be discarded. Sample volumes will consist of approximately 500 to 800 grams of material. Samples will be submitted to the laboratory by the samplers under chain of custody procedures.

3.2.1.3 Land Use Category #3 (Maintained Grass Areas/Grass Sports Fields)

For Land Use Category #3 sampling components, subsamples will be collected from a minimum of 3 subsample locations or at a rate of 1 subsample per 2,200 ft^2 in surface area per sampling component, whichever is greater. Subsamples from these locations will be composited in the field, and a single composite sample per depth interval will be analyzed for arsenic, lead, and mercury. Each subsample should have similar mass so that each location is equally represented in the total sample mass. The maximum area represented by a single composite sample will be 10,890 ft^2 (meaning a maximum of 5 subsamples will be collected from any single Land Use Category #3 sampling component) (see Table 1).

Samples will be thoroughly mixed in a clean 1-gallon plastic Ziploc® bag or stainless steel bowl to ensure representativeness of the aliquot ultimately submitted for analyses. During this homogenization process, particles greater than 0.5 inches in diameter will be discarded. Sample volumes will consist of approximately 500 to 800 grams of material. Samples will be submitted to the laboratory by the samplers under chain of custody procedures.

3.2.1.4 Land Use Category #4 (Low Access Areas/Low Maintenance Areas/Open Space)

For Land Use Category #4 sampling components, subsamples will be collected from a minimum of 3 subsample locations or at a rate of 1 subsample per 2,200 ft^2 in surface area per sampling component, whichever is greater. Subsamples from these locations will be composited in the field, and a single composite sample per depth interval will be analyzed for arsenic, lead, and mercury. Each subsample should have similar mass so that each location is equally represented in the total sample mass. The maximum area represented by a single composite sample will be 21,780 ft^2 (meaning a maximum of 10 subsamples will be collected from any single Land Use Category #4 sampling component) (see Table 1).

Samples will be thoroughly mixed in a clean 1-gallon plastic Ziploc® bag or stainless steel bowl to ensure representativeness of the aliquot ultimately submitted for analyses. During this homogenization process, particles greater than 0.5 inches in diameter will be discarded. Sample volumes will consist of approximately 500 to 800 grams of material. Samples will be submitted to the laboratory by the samplers under chain of custody procedures.

3.2.1.5 Land Use Category #5 (Flower/Vegetable Gardens)

In order to limit disturbance in small components (such as vegetable and flower gardens), only one sample location will be used when the component area is approximately 50 ft^2 or less in area. For Land Use Category #5 sampling components greater than 50 square feet in area, subsamples will be collected from a minimum of 2 subsample locations or at a rate of 1 subsample per 625 ft^2 in surface area per sampling component, whichever is greater. When applicable, subsamples from these locations will be composited in the field, and a single composite sample per depth interval will be analyzed for arsenic, lead, and mercury. Each subsample should have similar mass so that each location is equally represented in the total sample mass. The maximum area represented by a single composite sample will be 3,125 ft^2 (meaning a maximum of 5 subsamples will be collected from any single Land Use Category #5 sampling component) (see Table 1).

Samples will be thoroughly mixed in a clean 1-gallon plastic Ziploc® bag or stainless steel bowl to ensure representativeness of the aliquot ultimately submitted for analyses. During this homogenization process, particles greater than 0.5 inches in diameter will be discarded. Sample volumes will consist of approximately 500 to 800 grams of material. Samples will be submitted to the laboratory by the samplers under chain of custody procedures.

3.2.2 Sample Depths

Three depth samples will be collected from each identified component. There will be 1 surface sample (0 to 2 inches below ground surface [bgs]) along with 2 subsurface samples (2 to 6 and 6 to 12 inches bgs).

Because most of these sampling components are expected to be covered with a turf mat, 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 container 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 material on the surface, the sample will be collected from the surface (0- to 2-inch) layer.

Gardens will be subject to additional subsurface sampling. In addition to the 3 depth samples described above, 2 additional subsurface samples will be collected from the 12 to 18-inch and 18 to 24-inch depth intervals, for a total of 5 depth samples within a vegetable or flower garden.

3.2.3 Previously Sampled Properties

Butte-Silver Bow County will review the Program database to identify properties that were previously sampled but have incomplete data sets. This information will be provided to the Agencies in the form of Field Sampling Plan (FSP) submittals. Property owners of these previously partially sampled properties where remediation was not performed will be contacted to request access to conduct additional sampling to fill the data gaps. The goal will be to produce a complete data set that includes data for all required depth intervals and analytes.

Areas of the property that were sampled at the 0- to 2-inch depth interval and remediated will not be resampled because these components have already been remediated to a 12-inch depth.

3.2.4 Soil Sample Equipment Decontamination

Re-usable equipment will be decontaminated between sampling sites in accordance with manufacturer's recommendations and established SOPs (Attachment C-1) prior to being re-used. Equipment used for sample homogenization or scoops used for sample bagging or subsampling for mercury analysis will be single-use disposable equipment. Decontamination solutions may be disposed of to the ground surface, in the same general area in which soil sampling occurred. Disposable supplies will be collected by the field team leader and disposed of at the BPSOU Mine Waste Repository or local landfill as appropriate.

3.2.5 Soil Sample Preparation Methods

The temperature upon mercury sample receipt is measured and recorded by the laboratory on sample condition upon receipt documentation. The samples will be stored chilled (less than or equal to 6° Celsius [C], but not frozen) in temperature monitored refrigerators prior to laboratory digestion and analysis within 28 days of sample collection. The mercury digestion and analysis will be performed on "wet" sample aliquots and reported on a dry weight basis.

The project soil samples collected in Ziploc® bags for lead and arsenic will be shipped from the field and stored by a second laboratory at ambient temperature conditions. The soil samples will undergo sample drying and sieving (within approximately 5 days of collection) prior to ambient shipment of the dried sample to the primary laboratory for sample digestion and analysis for lead and arsenic.

Sample preparations and analyses will be in accordance with the EPA analytical method specifications provided below as well as standard laboratory practices. Specifically, the soil samples must be measured for percent moisture and prepared for metals analyses. Samples must be sieved using a No. 60 sieve to obtain the fine fraction, less than 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 until the criteria for sample disposal is met (see Section 3.7). The weight of the coarse fraction and the fine fraction will be measured and recorded by the laboratory for each soil sample prepared in this manner. The SOPs addressing soil sieving are included in Attachment C-2.

Consistent with prior sampling programs, samples will be sieved to the less than 250 µm fraction, reflecting the fine fraction of soil most likely to adhere to children's hands. More recent EPA guidance (EPA OLEM Directive 9200.1-128) requires sieving to less than 150 µm based on studies that show lead enrichment in very fine soil fractions (e.g., less than 63 µm). There are no data adequate to predict if the less than 150 µm fractions might be detectably enriched as compared with the less than 250 µm fraction. In light of this uncertainty, EPA has agreed with use of the less than 250 µm fraction for the 2021 sampling program while a particle size enrichment demonstration study is planned and conducted.

3.2.6 Soil Sample Collection Equipment

Soil samples are collected using primarily hand tools and are limited to readily available products. If supplies should be exhausted, replacement supplies can be purchased at nearby retailers. Hand tools may include sampling probe, Sharpshooter® type shovels, and heavy duty 5- to 6-foot steel pry bars. Single-use scoops and protective (latex/nitrile) gloves will be used to collect and mix the samples. Ziploc® bags will be used as sample containers for those samples requiring arsenic and lead analyses. Those samples requiring mercury analysis will use glass sample jars as sample containers.

3.3 Sample Handling and Chain of Custody

After collection and labeling, the samples will be maintained under strict chain of custody protocols, in accordance with the sample packaging SOP (Attachment C-1). The field sampling personnel will complete a chain of custody form for each shipment/delivery (i.e., batch of coolers) of samples to be delivered to the laboratory for analysis. The coolers containing sample jars for mercury analysis will be shipped from the field on ice to the Pace Analytical Laboratories Minneapolis, Minnesota, laboratory (1700 Elm Street SE, Minneapolis, MN 55414) for analysis. The coolers containing project soil samples collected in Ziploc® bags for lead and arsenic will be shipped from the field at ambient temperature conditions to the Pace Analytical Laboratories Green Bay, Wisconsin, laboratory (1241 Bellevue Street, Suite 9, Green Bay, WI 54302) for drying and sieving. Upon completion of drying/sieving activities, these samples will be shipped to the Pace Analytical Laboratories in Minneapolis for analysis. Jennifer Anderson is the Pace Analytical point of contact.

The sampler is responsible for initiating and filling out the chain of custody form. The chain of custody for a shipment/delivery will list only those samples in that shipment/delivery. Any documentation, including chain of custody, should be placed inside a re-sealable plastic bag, within the shipment/delivery container. Coolers which are to be shipped will be custody sealed, securely taped shut, and have a shipping label securely adhered to the cooler. Sample containers hand delivered to the laboratory do not need to be prepared for shipping.

The sampling personnel whose signature appears on the chain of custody form is responsible for the custody of the samples from the time of sample collection until custody of the samples is transferred to a designated laboratory, a courier, or to another project employee for the purpose of transporting the samples to the designated laboratory. Custody is transferred when both parties to the transfer complete the portion of the chain of custody under "Relinquished by" and "Received by”. Signatures, printed names, company names, dates and times are required. Upon transfer of custody, the sampling personnel who relinquished the samples will retain the third sheet (pink copy), photocopy, or electronic copy of the chain of custody. When the samples are shipped by a common carrier, a Bill of Lading supplied by the carrier will be used to document the sample custody, and its identification number will be entered on the chain of custody. Copies, receipts, and carbons of Bills of Lading will be retained as part of the permanent documentation in the project file. It is not necessary for courier personnel to sign the chain of custody.

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

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

3.4 Sample Identification

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

Matrix:

S – Soil

RMAP Database Resident ID: (example of R-00001)

Site Property Codes:
- C – Commercial
- P – Park
- S – School

Resident ID:
- 00001 – associated with a specific address or geocode

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

- PA – Playground Area (Land Use Category #1)
- HA – High Access Area (Land Use Category #2)
- GA – Maintained Grass Area (Land Use Category #3)
- LA – Low Access Area (Land Use Category #4)
- G – Flower/Vegetable Garden (Land Use Category #5)
- OP – Opportunistic Sample
- BA - Bare Area
- SA - Source Area

Quality Control/Quality Assurance Codes:
- D – Field Duplicate

Depth Intervals: Depth intervals are only applicable to soil sampling events.

1. 0 to 2 inches bgs
2. 2 to 6 inches bgs
3. 6 to 12 inches bgs
4. 12 to 18 inches bgs (flower/vegetable gardens only)
5. 18 to 24 inches bgs (flower/vegetable gardens only)

An example sample identification would be: S-S-0001-PA-2. This indicates that the soil sample was collected at the School with the Resident ID S-0001 (corresponding to a physical address and/or geocode) in a playground area at the 2 to 6-inch depth interval. The sample identification for a field duplicate collected at this location would be: S-S-0001-PA-D-2.

Sample identifiers will be documented in field logbooks/data collection device and on the chain-of-custody forms, as required by the RMAP Field SOPs located in Attachment C-1.

3.5 Analyses Methods

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

3.5.1 Soil Sample Analysis Method

All RMAP soil samples will be analyzed to determine metals concentrations via standard laboratory analytical methodologies for arsenic, lead, and mercury. 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 arsenic and lead concentrations will be determined per EPA Method 6010 (inductively-coupled plasma atomic emission spectroscopy [ICP-AES]) or EPA Method 6020 (inductively-coupled plasma mass spectrometry [ICP-MS]). Mercury concentrations will be determined per EPA Method 7471B (Manual Cold-Vapor Technique).

The laboratory SOPs for EPA Methods soil sieving, 3050B, 6010, 6020, and 7471B are included in Attachments C-2.

3.5.2 Laboratory Quality Control Samples

As outlined above in Sections 3.5.1, RMAP soil samples will be analyzed to determine metals concentrations (arsenic, lead, and mercury) via standard laboratory analytical methodologies. Laboratory QC procedures are outlined below.

All analyses will be governed by the appropriate calibration procedures and frequencies that are specified in the laboratory's SOPs (see Attachment C).

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

Laboratory 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. Method blank results outside of specified control limits will be re-run/redigested and re-analyzed with all associated samples and/or flagged by the laboratory per the QC requirements of the analytical method. Initial and continuing calibration blanks are also analyzed every 10 samples and samples are reanalyzed within compliant blank analyses. All elements of interest must be evaluated to +/- the RL for Method 6020.

Laboratory Control Samples

An LCS, or a blank spike, is an aqueous or solid control sample of known composition that is analyzed using the same sample preparation, reagents, and analytical methods employed for the Program samples. The LCS is obtained from an outside source or is prepared in the laboratory by spiking reagent water or a clean solid matrix from a stock solution that is different from that used for 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 LCS is re-analyzed once. If re-analysis of the LCS fails, all samples affected by the failing LCS elements need to be redigested and re-analyzed.

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 (Attachment C). 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.

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 5-fold dilution of a field sample (field blanks may not be used) and calculating the percent difference between the original determination and the serial dilution result. Serial dilutions are only applicable for analyte concentrations that are greater than 50 times the 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 Attachment C.

Matrix Spikes

Laboratory MS samples are used to evaluate potential sample matrix effects on the accurate quantitation of an analyte using the prescribed analytical method. The MS/MSDs are prepared by adding an analyte to a subsample of a field sample before sample preparation and analyses. A percent recovery is calculated from the concentrations of the analyte in the spiked and un-spiked samples. Perform a post digestion spike on any elements that fail to meet criteria If the percent recovery for the MS and MSD falls outside the control limits, the results are flagged by the laboratory that they are outside acceptance criteria along with the parent sample.

Additional Quality Control Samples

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

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

3.6 Field Quality Control Samples

Field QC samples are used to identify any biases from transportation, storage, and field handling processes during sample collection and to determine sampling precision. All field QC samples will be delivered with field samples to the laboratory. This section includes brief descriptions of the QC samples to be collected during sampling activities along with frequency, collection, and analytical instructions.

Sampling protocols will be consistent with the Field SOPs included in Attachment C-1 and will include 1 field duplicate collected for every 20 primary samples or once per sampling event (e.g. once per sampling day), whichever is more frequent (in accordance with Level A/B field screening/data review criteria, Attachment D). 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 is decontaminated and used between samples. Any deviation from the SOPs or this QAPP will be identified in the logbook/data collection device and discussed in the annual DSR.

3.6.1 Field Duplicate (Soil Samples)

A field duplicate consists of one well-mixed and homogenized sample that is split in the field into two samples and placed in different sample containers for separate analyses.

As with all other samples, samples to be split for duplicate samples will be thoroughly mixed in a clean 1-gallon plastic Ziploc® bag or stainless steel bowl to ensure representativeness of the aliquot ultimately submitted for analysis. During this homogenization process, particles greater than 0.5 inches in diameter will be discarded. Once the homogenization process is complete, the natural sample is split into two samples. Each split will have its own sample number. Both split samples will be analyzed for identical chemical parameters. The results of the field duplicate will be compared to determine laboratory and sampling precision. Field duplicate samples will be collected at a frequency of 1 per 20 samples or once per sampling event (e.g., once per sampling day), whichever is more frequent. The RPD field precision goal for soil field duplicates will be 35% for sample pairs with both sample results being greater than 5 times the RL. For soil field duplicate/primary sample pairs with 1 or both sample results being less than 5 times the RL, an absolute difference of less than or equal to 2 times the RL (difference less than or equal to 2 times the RL) will be used as the precision goal. Laboratory precision goals are laboratory specific.

3.7 Sample Disposal

Soil samples shipped to the laboratory for analyses will be held until the laboratory analyses has been completed, the Agencies have reviewed and approved all subsequent project laboratory data and work plans, and the sample hold times have expired. At this point, the laboratory may dispose of samples or return them to BSB for disposal. Any excess soil mass that was not included in the aliquot submitted to the laboratory will be subject to the same disposal criteria.

3.8 Instrument/Equipment Testing, Inspection and Maintenance

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

3.8.1 Field Equipment

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

3.8.2 Laboratory Equipment

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

3.9 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 field team members.

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

3.10 Data Management Procedures

This section describes the management of data for the project including field and laboratory data. The Program quality records will be maintained by the Data Management Division Manager, as described in the BPSOU *Final Data Management Plan* (Atlantic Richfield Company, TBD). These records, either electronic or hard copy in form, may include the following:

- Project work plans with any approved modifications, updates, and addenda.
- Individual property maps (hard copy or scanned field drawings and electronic files).
- Individual property owner result letters (both no action and remedial action required).
- Project QAPP, including this QAPP, with any approved modifications, updates, addenda, and corrective or preventative actions.
- Access agreements from 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).
- Data validation documentation.
- Annual completion report.

Hard copy field and laboratory records will be maintained in the project's central data file, where original field and laboratory documents are filed chronologically for future reference. These records are also scanned to produce electronic copies. The electronic versions of these records are maintained on a central server system with backup scheduled 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 the information is incorporated into the database. Laboratory EDDs and related data packages will be reviewed as part of the internal data review process. The Data Management Division Manager, or designated alternate, will be responsible for ensuring data integrity prior to database uploads. Following these review steps, field and laboratory electronic data files will be imported to the project database.

Standardized data import formats and procedures will be used to upload both field and laboratory data into the electronic database. An existing EDD format will be used to upload into the project 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. Data management activities for the RMAP program will be further defined in the *BPSOU Data Management Plan* (Atlantic Richfield Company, TBD).

3.10.1 Requests for Data

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

4.0 RECLAMATION MATERIAL

Should sample results indicate that removal of soils at a school, park, or non-residential daycare is warranted, a removal work plan will be submitted by BSB and Atlantic Richfield for approval by the Agencies. All materials used for reclamation activities in areas above action levels must meet requirements set forth in the Butte Hill Revegetation Specifications (BHRS) (BPSOU ROD [EPA, 2006b]). The source of all materials used in site reclamations will be provided in writing for approval.

4.1 Backfill

Backfill material (i.e., replacement soil) will be from a pre-approved source and will not contain any trash, debris, or large roots from shrubs or trees. Backfill material for garden areas must be suitable for germination and cultivation of flowers and vegetables with ordinary amendments.

4.1.1 Backfill Testing

A minimum of three soil samples from the source area will be submitted to an approved laboratory for analyses. Samples will be analyzed for the parameters listed below using U.S. Department of Agriculture (USDA) classification and test methods as described in the American Society of Agronomy (ASA)/Soil Science Society of America (SSSA) Monograph No. 9, Methods of Soil Analysis, Parts 1-2, most recent edition.

- Texture class and particle size.
- pH.
- Saturation percent.
- Electrical conductivity in millimhos per centimeter (mmhos/cm).
- Organic matter percent.
- Nitrate Ion - nitrogen.
- Available phosphorus.
- Available potassium.

Samples will also be analyzed for the presence of the following metals in soil: arsenic, cadmium, copper, lead, and zinc. All soil imported to remediation areas must include a Butte Hill Cover Soil Approval Submittal form (Attachment F) and meet the BHRS requirements (EPA, 2006b) prior to placement.

4.2 Engineered Cover Materials

Materials used for engineered covers must also be analyzed for metals described in Section 4.1.1. For driveways and parking areas, a pit-run gravel base will be used, and it will be capped with a 6-inch depth of ¾-inch minus base course “road-mix” gravel material.

Sod must be certified weed free and source areas approved prior to placement. Seed mixtures and sources must be approved prior to placement as described in the BHRS (EPA, 2006b). Copies of seed bag tags and certification must be collected and recorded to be included in the annual construction completion documentation for the specific remediated property (refer to Section 5.3).

5.0 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. All internal audits will be conducted by Atlantic Richfield’s contractor Environmental Standards, Inc. The internal field audit will be conducted during the initial week of sampling activities to ensure compliance with the QAPP and consistency between individual crews. The internal lab audit of the Pace Analytical Services Green Bay, Wisconsin facility will also be conducted during the initial week of sampling activities. The internal lab audit of the Pace Analytical Services Minneapolis, Minnesota facility will follow shortly thereafter. External audits may be performed by the Agencies as necessary.

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

5.1 Corrective Actions

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

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

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

Health/RMAP Division Manager or the Data Management Division Manager/QA Manager. 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, or new samples may be obtained.

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

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

All corrective actions taken by the laboratory will be documented in writing by the laboratory project manager and reported to the BSB QA Manager. 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 quality records.

5.2 Corrective Action During Data Assessment

The need for corrective action may be identified by any member of the project team during data assessment. Potential types of corrective action may include re-sampling by the field team, re-analyses of samples by the laboratory, or re-submitting data packages with corrected clerical errors. The appropriate and feasible corrective actions are dependent upon the ability to mobilize the field team and whether the data to be collected is necessary to meet the required QA objectives (e.g., the holding time for samples is not exceeded). 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 BSB QA Manager on a Corrective Action Report (CAR) and will be included in any subsequent reports.

5.3 Reports to Management

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

After site investigations and remedial actions are complete, the Data Management Division Manager/QA Manager will prepare an annual DSR (Section 2.9.6) summarizing the sampling activities. The laboratory and data validation turnaround times for providing sample results will be expedited in order to achieve project assessment and remediation goals while also allowing timely completion of the annual DSR. This is estimated to be a 5 to 7 business day turnaround time on lab data and level 2 data packages and 10 to 12 business day turn around on lab data and level 4 data packages. Data validation is estimated to be a 7 business day turnaround time after data packages are received from the lab. The report will describe specific field sampling activities performed during implementation of the QAPP. Each annual report will include field documentation, documentation of field QC procedures, results of all field and laboratory data, data validation results, and data usability assessments.

A separate report will be prepared by the BSB QA Manager, as needed, to communicate the results of performance evaluations or program audits to identify specific significant QA issues and provided to the EPA for review. Any corrective action reporting described in Section 5.2 above will be summarized and included as appropriate.

6.0 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 for the express purposes of achieving the stated DQOs (Section 2.7.1). Data review/validation process under this QAPP is streamlined to support the post-BPSOU ROD (EPA, 2006b) decision-making process. The analytical data collected under this QAPP and produced by analytical laboratories will undergo a combination of Stage 4 and 2B data validation. The field documentation will be subject to Level A/B criteria review, and analytical data will be validated per the *Clark Fork River Superfund Site Investigations Data Management/Data Validation Plan* (ARCO, 1992a), the *EPA National Functional Guidelines for Inorganic Superfund Methods Data Review* (EPA, 2020b), and the project DQOs. Data review and validation will be conducted by a qualified technical consultant who is independent from the sampling consultant (i.e., an individual other that the individual who performed sampling).

6.1 Data Review, Verification, and Validation

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

6.1.1 Data Review Requirements

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

6.1.1.1 Field Data Review

Raw field data will be entered in field logbooks/data collection device and reviewed for accuracy and completeness by the Human Health/RMAP Division Manager, QA Manager, or Field Team Leader before those records are considered final. The overall quality of the field data from any given sampling round will be further evaluated during the process of data reduction and reporting. The field data will be reviewed quarterly by the Program QA Manager, or designated alternate.

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

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

6.1.1.2 Laboratory Data Review

Internal laboratory data reduction procedures will be according to each laboratory's quality management plan. At a minimum, paper records will be maintained by the analysts to document sample identification number and the sample tag number with sample results and other details, such as the analytical method used (e.g., method SOP #), name of analyst, the date of analysis, matrix sampled, reagent concentrations, instrument settings and the raw data. These records will be signed and dated by the analyst. Secondary review of these records by the Laboratory Supervisor (or designee) will take place prior to final data reporting. The laboratory is responsible for assigning appropriate flags/qualifiers in accordance with the analytical method and internal laboratory SOPs.

6.1.2 Data Verification Requirements

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

6.1.2.1 Field Data Verification

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

The Level A criteria include:

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

The Level B criteria include:

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

6.1.3 Laboratory Data Verification

The laboratory will prepare Level 3 and Level 4 data packages for transmittal of results and associated QC information to the Human Health/RMAP Division Manager or its designee within a standard turnaround time unless otherwise required.

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

Each data package, as described above, will be accompanied by an EDD prepared by the laboratory. If data qualifiers are required, they will be added to the laboratory EDD and provided for uploading to the database. Additional laboratory QC data can be included in the EDD. The EDDs will be cross checked against corresponding data reports to confirm consistency in results reported in these two separate formats. This cross check will take place as part of the data verification process. All data will be submitted in both Level 3 and Level 4 format.

6.1.3.1 Resolution of Deficiencies

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

6.1.4 Data Validation Requirements

The purpose of analytical data validation is to provide an assessment of data quality. Data validation will be performed by qualified, independent data validation personnel, who are not associated with data collection or sampling responsibilities, and that have applicable training. Data validation categorizes data as acceptable for use, unacceptable for use, or qualified for select use. The validation effort routinely identifies data use limitations and corrects a reporting and quantitation errors. The data packages provided for validation will be evaluated for compliance with respect to the requested analytical methods and/or the QAPP and completeness of requested deliverables. Concurrent with the data validation efforts, analytical data usability will also be assessed. Analytical data usability is the determination of whether or not a data set is sufficiently complete and of sufficient quality for further evaluation by the data user as detailed in Section 6.3 of the QAPP to support a decision or action.

The data will be validated during the data validation process with guidance from the *CFRSSI QAPP* (ARCO, 1992b), the *CFRSSI DM/DV Plan* (ARCO, 1992a), the *CFRSSI DM/DV Plan Addendum* (AERL, 2000), the *National Functional Guidelines for Inorganic Superfund Methods Data Review* (EPA, 2017), laboratory-specific QC criteria, and/or method-specific criteria where applicable. The use of the Functional Guidelines versions listed above is important to maintain consistency between data validation and qualification of data currently being performed and future work to be performed under the RMAP program. It should be noted that the US EPA National Functional Guidelines, which were developed for the validation of data generated in accordance with the Contract Laboratory Program (CLP), are not directly applicable to the type of analyses/protocols associated with the analyses for this project. US EPA National Functional Guidelines qualifies data based on strict contractual CLP method requirements and acceptance criteria which may not be consistent with the requirements and acceptance criteria presented in SW-846 methods. Data validators will apply the US EPA guidelines as appropriate, assess the data relative to method QC protocols and DQOs in this QAPP, and use professional judgment according to the documents listed above.

6.2 Verification and Validation Methods

The Level A/B Assessment checklists included in Attachment D are based on the *CFRSSI DM/DV Plan Addendum* (AERL, 2000) guidance and will be used for Field Data Verification as detailed in Section 6.1.2.1.

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

The Data Validator will be responsible for reviewing field documentation associated with sample collection, conducting the verification and validation of laboratory-produced data, and completing a data validation report, which will be reviewed by the Human Health/RMAP Division Manager and QA Manager.

Qualifiers that may be applied to the data during the data validation process include the following:

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

6.2.1 Differences Between Stage 2B and Stage 4 Validation

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

Stage 2B and Stage 4 data validations and reports are generated by an initial reviewer on a per-SDG or sampling location basis from the complete Level 4 data package to ensure completeness and data usability of data packages. Level 3 data packages are a condensed version of final data prior to completion and receipt of Level 4 data packages. Level 3 data packages contain the same information as the Level 4 data packages with the exception that instrumental QC (i.e., instrument tunes and raw data) to support the sample and the QA/QC results are not provided. Each validation report is reviewed by a senior chemist for accuracy to ensure that the initial reviewer has rigorously evaluated the recoveries/results and applied the applicable qualifiers to the data.

6.2.2 Stage 2B and Stage 4 Validation Procedure

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

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

- Completeness of laboratory data package
- Requested analytical methods performed
- Compliance with the QAPP, analytical method, and analyte list.
- Proper sample collection, custody, preservation, and handling procedures.
- Holding times.
- Reported detection limits.
- Dilution factors.
- Tuning
- Instrument Calibration
- Initial and Continuing Calibration Verification Standards
- Initial and Continuing Calibration Blank Standards
- ICP and ICP/MS interference check samples.
- Method blanks.
- LCSs.
- Reporting Limit Check Standard recoveries.
- Field duplicate results.
- MS/MSDs (pre-digestion and post-digestion for inorganics only).
- ICP/MS internal standard recoveries.
- ICP and ICP/MS serial dilutions.
- Results verification and reported detection limits.
- Sample Preparation and Analytical Run Logs

6.2.3 Data Validation Ratios

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

6.3 Reconciliation and User Requirements

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

Step 1: Review DQOs and sampling design.
Step 2: Conduct preliminary data review.
Step 3: There are no statistical tests that are planned in the interpretation of the non-residential soils results; laboratory results will be compared directly to action limits defined in the DQOs (Section 2.7.1).
Step 4: Verify assumptions.
Step 5: Draw conclusions about the quality of the data (data report will not include interpretation of results but will state conclusions regarding the quality of the results).

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

The PARCCS data quality indicators (Section 2.7.2) will be used when conducting the DQA. If the PARCCS assessment satisfies the project DQOs, then usability of the data will follow the enforcement/screening/unusable data categories as described in the *CFRSSI DV/DM* (ARCO, 1992b):

1. Enforcement Quality (Unrestricted Use). Data enforcement quality data may be used for all purposes under the Superfund program including the following: site characterization, health and safety, Environmental Evaluation/Cost Analysis, remedial investigation/feasibility study, alternatives evaluation, confirmational purpose, risk assessment, and engineering design.

2. Screening Quality (Restricted Use). Data potential uses of screening quality data, depending upon their quality, include site characterization, determining the presence or absence of contaminants, developing or refining sampling and analysis techniques, determining relative concentrations, scoping and planning for future studies, engineering studies and engineering design, and monitoring during implementation of the response action.

3. Unusable Data. These data are not useable for Superfund-related activities.

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

E = Enforcement quality. No qualifiers, U qualifier or J qualifier (see note below) and meets Level A and B criteria.

S = Screening quality. J or UJ qualifier and/or meets only Level A criteria.
R = Unusable. R qualifier and/or does not meet Level A or B requirements.

Enforcement/Screening Designation

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

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

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.

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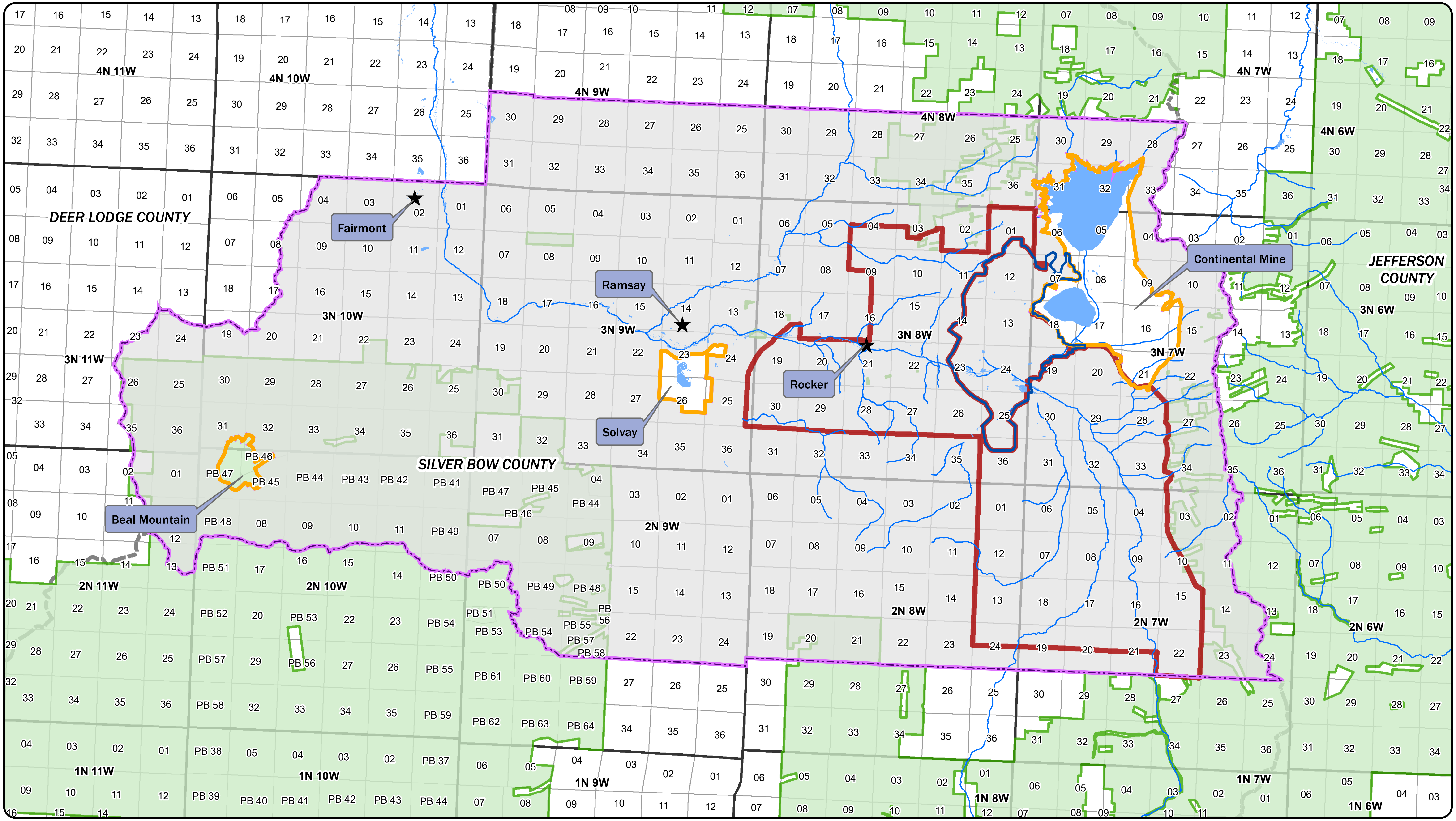
FIGURES



LEGEND

- 🟦 BPSOU BOUNDARY
- ▨ 2020 RMAP AREA BOUNDARY
- 🟧 AREAS EXCLUDED FROM RMAP (CONTINENTAL MINE, SOLVAY, BEAL MOUNTAIN)
- 🟥 2011 RESIDENTIAL METALS EXPANDED AREA
- ▭ COUNTY BOUNDARY
- 🟩 US FOREST SERVICE

N

DISPLAYED AS:
PROJECTION/ZONE: MSP
DATUM: NAD 83
UNITS: INTN'L FT
SOURCE: PIONEET/BSB/NRIS

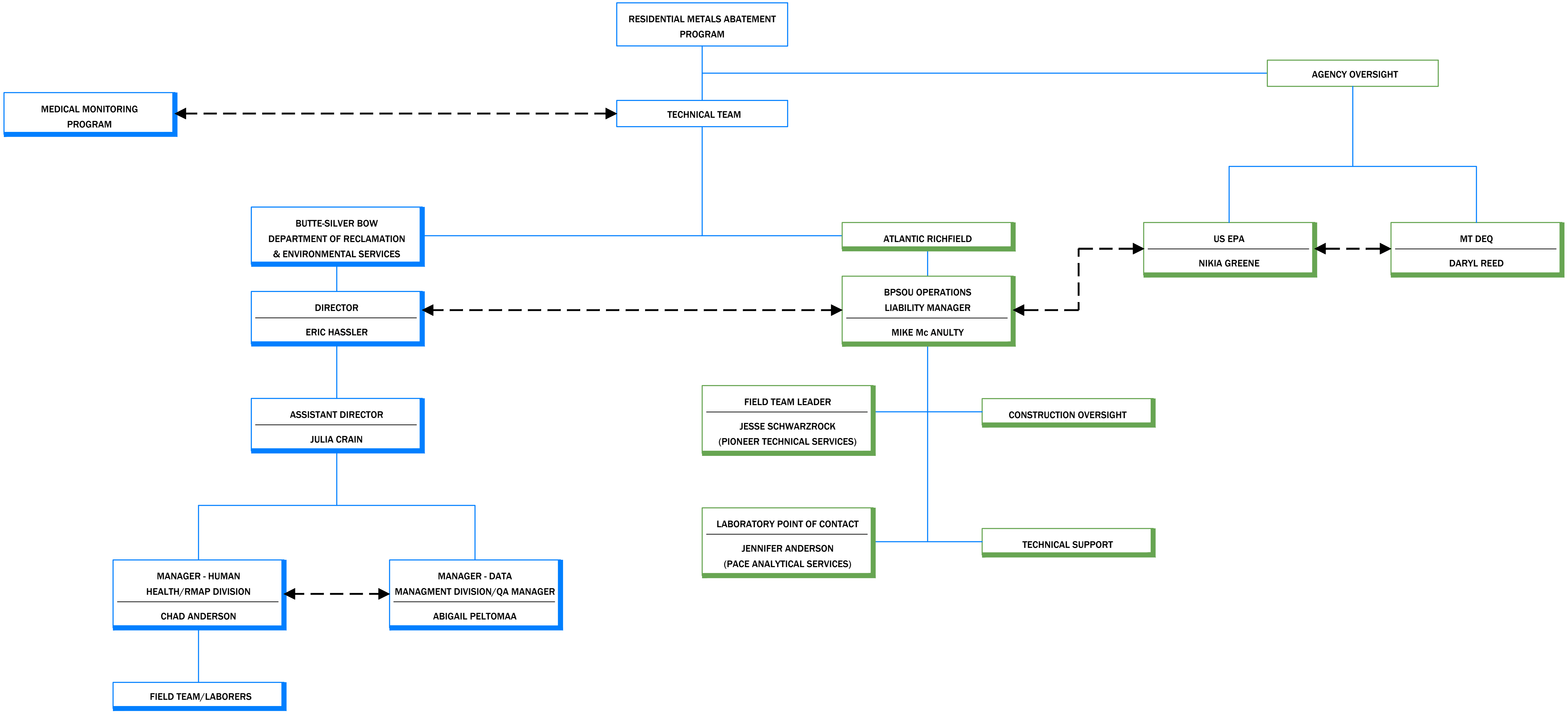
0 1 2 4
Miles

FIGURE 1

2020 RMAP /
BPSOU BOUNDARIES

DATE: 3/16/2021

Path: Z:\Shared\Active Projects\ARCO\BPSOU\RMAP\GIS\RMAP_Bndrys.mxd



—— AUTHORITY

← – – → COMMUNICATION

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

SCALE IN FEET
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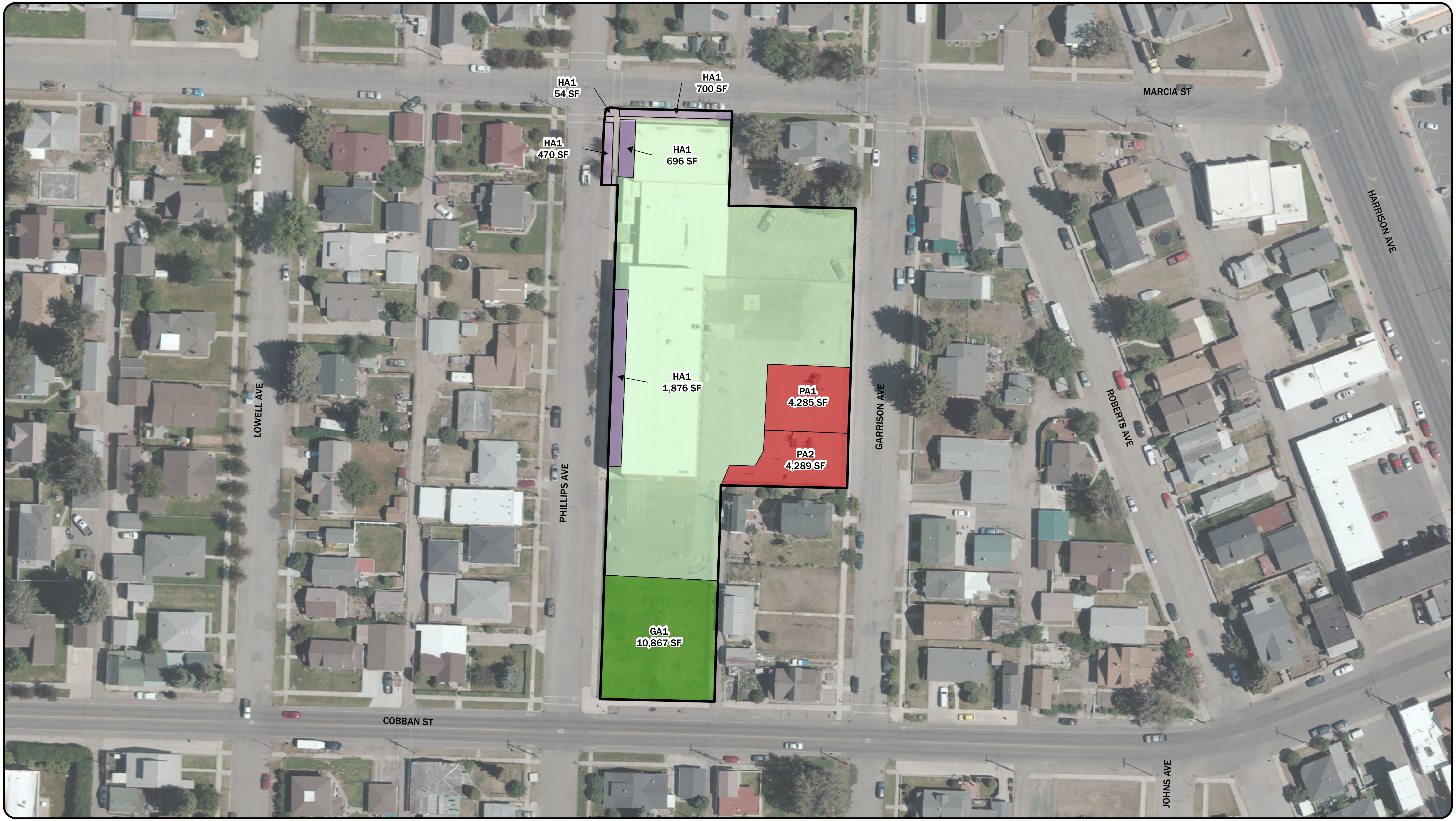
FIGURE 2

RMAP ORGANIZATIONAL & COMMUNICATIONS STRUCTURE

DATE: 6/25/2021

6/28/2021 8:43:46 AM Z:\SHARED\ACTIVE PROJECTS\ARCO\BPSOU\RMAP\CADD\FIGURES\RMAP-OC-FG-PLN-001-21.DWG



LEGEND

🟥 LAND USE CATEGORY #1 (PLAYGROUND AREAS)
🟪 LAND USE CATEGORY #2 (HIGHLY ACCESSIBLE AREAS/BARREN SPORTS FIELDS)
🟩 LAND USE CATEGORY #3 (MAINTAINED GRASS AREAS/GRASS SPORTS FIELDS)
🟦 LAND USE CATEGORY #4 (LOW ACCESS AREAS/LOW MAINTENANCE AREAS/OPEN SPACE)
🟨 LAND USE CATEGORY #5 (GARDEN AREAS)
🟫 OPPORTUNISTIC SAMPLES
🟩 NON-SAMPLING AREAS
▭ SAMPLING POLYGON BOUNDARY
▭ PROPERTY BOUNDARY

N

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PROJECTION/ZONE: MSP
DATUM: NAD 83
UNITS: FEET
SOURCE: PIONEER

0 40 80 160
Feet

FIGURE 3

EXAMPLE
NON-RESIDENTIAL
FIELD SAMPLING PLAN

DATE: 6/25/2021

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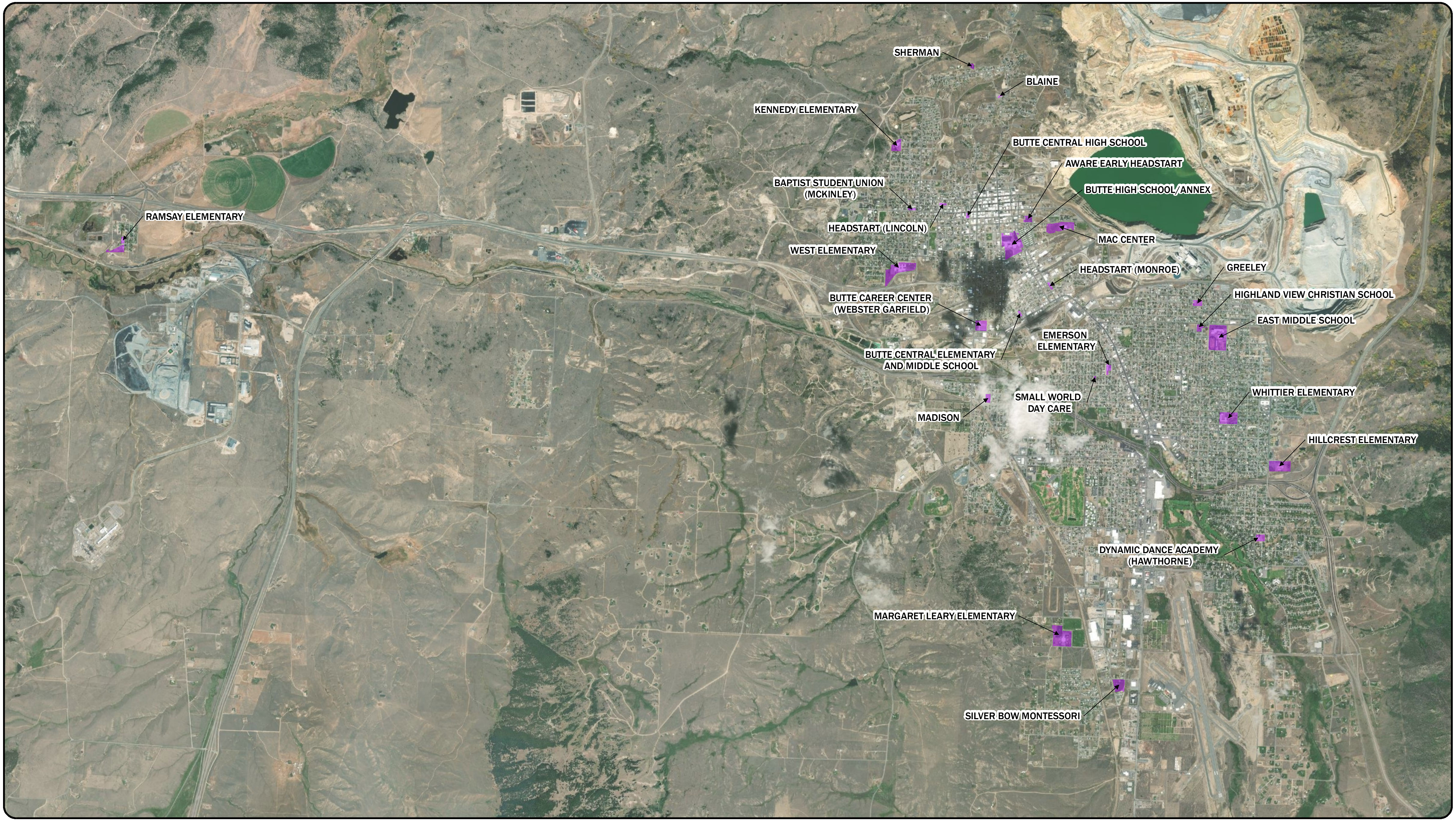

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PROJECTION/ZONE: MSP
DATUM: NAD 83
UNITS: FEET
SOURCE: PIONEER

0 2,000 4,000 8,000
Feet

FIGURE 4 BUTTE-SILVER BOW SCHOOLS & FORMER SCHOOLS

DATE: 6/28/2021

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TABLES

TABLE 1: RMAP ACTION LEVELS AND SAMPLE PROTOCOL
(for RMAP Non-Residential Parcels)

	Contaminant of Concern:	**Lead**			**Arsenic**			**Mercury**					
Matrix	**Exposure Scenario**	**Action Levels Concentration**	**Analytical Methods**	**Method Detection Limits (MDLs)[1]**	**Action Levels Concentration**	**Analytical Methods**	**Method Detection Limits (MDLs)[1]**	**Action Levels Concentration**	**Analytical Methods**	**Method Detection Limits (MDLs)[1]**	**Sample Frequency**	**Sample Depth Intervals**	**Sample Density**
Soil	Land Use Category #1 (Playground Areas)	Residential - 1,200 mg/kg	EPA Methodology (EPA 6010/6020)	0.087 mg/kg (6020)	Residential - 250 mg/kg	EPA Methodology (EPA 6010/6020)	0.156 mg/kg (6020)	Residential - 147 mg/kg	EPA Methodology (EPA 7471B)	0.008 (7471B)	1 composite sample per component per depth interval	0-2 inches; 2 - 6 inches; and 6 - 12 inches	Minimum of 3 subsample locations per component or one subsample location per 625 sf (whichever is greater). Maximum area represented by a single composite sample is 6,250 sf (or 10 subsample locations).
Soil	Land Use Category #2 (Highly Accessible Areas/ Barren Sports Fields)												Minimum of 3 subsample locations per component or one subsample location per 625 sf (whichever is greater). Maximum area represented by a single composite sample is 9,375 sf (or 15 subsample locations).
Soil	Land Use Category #3 (Maintained Grass Areas/ Grass Sports Fields)												Minimum of 3 subsample locations per component or one subsample location per 2,200 sf (whichever is greater). Maximum area represented by a single composite sample is 10,890 sf (or 5 subsample locations).
Soil	Land Use Category #4 (Low Access Areas/ Low Maintenance Areas/ Open Space)												Minimum of 3 subsample locations per component or one subsample location per 2,200 sf (whichever is greater). Maximum area represented by a single composite sample is 21,780 sf (or 10 subsample locations).
Soil	Land Use Category #5 (Flower/Vegetable Gardens)											0-2 inches; 2 - 6 inches; 6 - 12 inches; 12 - 18 inches; and 18 - 24 inches	Minimum of 1 subsample location per component if the area is 50 sf or smaller in area. For components greater than 50 sf in area, a minimum of 2 subsample locations per component or one subsample location per 625 sf (whichever is greater). Maximum area represented by a single composite sample is 3,125 sf (or 5 subsample locations).

[1] - Detection limits will be re-evaluated and may change on a quarterly basis, but will typically be within ±5 mg/kg of the values listed above.

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: QUALITY CONTROL SAMPLE ACCEPTANCE CRITERIA

Analyte	Method	Residential Action Limit (mg/Kg)	Method Detection Limit (MDL) (mg/Kg)[1]	Reporting Limit (RL) (mg/Kg)[1]	Laboratory Control Sample (LCS) Recovery Limits	Matrix Spike/Matrix Spike Duplicate (MS/MSD) Recovery Limits[2]	MS/MSD Relative Percent Different (RPD)[2]	Laboratory Duplicate Precision	Field Duplicate Precision[3]
Arsenic	Method 6020A	1,200	0.156	0.50	70-130%	75-125%	20	20	± 35%
Lead		250	0.0870	0.20	70-130%	75-125%	20	20	± 35%
Mercury	Method 7471A	147	0.00868	0.02	70-130%	75-125%	20	20	± 35%

Notes:

[1] The MDLs and RLs are considered the laboratory base values. Soil samples for arsenic and lead will be dried prior to sample digestion and will not be dry weight corrected. Sample results for mercury will be reported on a dry weight basis, since soil samples will be digested on an “as received basis. MDLs and RLs may also be affected based on the actual weight of sample digested and potential dilutions required for high concentration samples. The BPSOU residential action levels (Arsenic – 250 mg/kg, Lead – 1,200 mg/kg, Mercury – 147 mg/kg) will be utilized for all work completed under this QAPP.

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

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

TABLE 4: BUTTE-SILVER BOW SCHOOLS AND FORMER SCHOOLS

Butte-Silver Bow Schools

Item	Geocode	Name	Construction Date	BPSOU
1	1119713454100000	Butte High School/Annex	1937/1968	Yes
2	1109506106060000	Silver Bow Montessori	1947	No
3	1119724113050000	Butte Career Center (Webster Garfield)	1948	Yes
4	1119713213100000	Butte Central High School	1951	Yes
5	01119614310150000 01119623201050000	Ramsay Elementary	1953	No
6	1119829154010000	Whittier Elementary	1954	No
7	01119820229010000 01119820121010000 01119820122010000 01119820211010000 01119820101010000 01119820109010000 01119820110010000 01119820102010000 01119820103010000	East Middle School	1957	No
8	01119819440340000 01119819440360000 01119819440160000	Emerson Elementary	1957	No
9	01119711410240000 01119711413010000 01119711410180000 01119711410140000 01119711410010000 01119711406380000 01119711406290000 0111971129904MINE	Kennedy Elementary	1958	Yes
10	1119713226010000	Headstart (Lincoln)	1958	Yes
11	1119819243110000	Headstart (Monroe)	1959	Yes
12	1119724117160000	Butte Central Elementary and Middle School	1960	Yes
13	1119828201050000	Hillcrest Elementary	1968	No
14	1119714411010000	West Elementary	1969	Yes
15	01119831302010000 01119831301250000	Margaret Leary Elementary	1973	No
16	1119818387010000	MAC Center	2004	Yes
17	01119820227170000 01119820227240000 01119820227300000 01119820227150000	Highland View Christian School	2010	No
18	01119713106200000 01119713106330000 01119713106300000 01119713106250000	Aware Early Headstart	2001	Yes
19	01119819428120000	Small World Day Care	1920	No

Former Schools

Item	Geocode	Name	Construction Date	BPSOU
20	1119712250010000	Sherman	1902	Yes
21	1119714112010000	Baptist Student Union (McKinley)	1903	Yes
22	1119724403010000	Madison	1904	Yes
23	1119832140010000	Dynamic Dance Academy (Hawthorne)	1918	No
24	1119820264010000	Greeley	1952	No
25	1119712499550000	Blaine	1959	Yes

TABLE 5: BUTTE-SILVER BOW PARKS AND OPEN AREAS

Item	**Geocode**	Name	Owner	Reclaimed Site	Adjacent Reclaimed Sites
1	01119712206016500	Walkerville Park (Walkerville Drive and Ryan Road)	BSB	Yes	
2	01119712213010000	Walkerville Park (5th and Transit)	City of Walkerville		
3	01119712129160000, 01119712129150000, 01119712129200000, 01119712129130000	Walkerville Park (Daly and Dunn)	City of Walkerville	Yes	
4	01119712129200000, 01119712129180000	Walkerville Park (Daly and Dunn)-there is another small parcel here	ARCO		
5	01119712298040000	Walkerville Park (Clark and Academy/Main/Dunn Area)	ARCO		Yes
6	01119712298050000, 01119712298040000	Walkerville Park (Alley of North Main and Alley of West Daly)	ARCO		Yes
7	0111971110205MINE	Walkerville Ball Park			
8	01119713272020000	Antimony Ball Field	BSB		Yes
9	01119819114010000	Clark Park	BSB		
10	01119830303010000	Stodden Park Golf Course	BSB		
11	0111971349904MINE, 0111971329904MINE	Mandan Park	BSB		Yes
12	01119831305010000	Jeremy Bullock Soccer Field	BSB		
13	01119725320010000 01119725321010000 01119725301050000 01119725299080000 01119725327270000 01119725327010000 01119725316510000 01119725301050000 01119725327250000 01119725320480000 01119725321450000 01119725299080000 01119725315390000 01119725315100000 01119725315150000	Copper Mountain	BSB	Yes	
14	01119830303010000	Stodden Park	BSB		
15	01119829202016500	Father Sheehan Park	BSB		
16	01119820225330000, 01119820225170000,	Racetrack Park	BSB		
17	01119818404010000	Skate park	BSB		Yes
18	01119818407010000	McGruff Park	BSB		Yes
19	01119818301150000	Koprivica Park	BSB		Yes
20	01119724125020000	Charlie Judd Park	BSB		Yes
21	01119713365016500	Chester Steele Park	BSB		
22	01119713365980000	Cinders Field	BSB		
23	01119714134150000	Copper/Emmet	BSB		Yes
24	01119714422350000	Gold/Emmett-called Tot Lot	BSB		
25	01119714118340000	Granite/Henry-called Hanna Park	BSB		
26	01119713242120000	Copper/Crystal-called Cherokee Park	BSB		Yes
27	01119713228290000	Broadway/Washington-called Peace Park	BSB		
28	01119830134600000	C Street	BSB		
29	0111971349904MINE	Montana/Woolman-called Souix Park	BSB	Yes	
30	01119714447210000, 01119714447160000	Silver/Girard-called Peoples Park and Skating	BSB	Yes	
31	01119713250010000	Scown Field	BSB		Yes
32	1119712300022010, 0111971239904MINE, 0111971249904MINE	Missoula Ball Fields	BSB	Yes	
33	01119713466010000, 01119713466010000	Emma Park	BSB	Yes	
34	01119820312016500, 01119820312100000	JFK Park	BSB		
35	01119832408010000	Rickey Park	BSB		
36	01119833320206500	Fleecer Drive	BSB		
37	01119833315456500	Blacktail Lane	BSB		
38	01119828201200000	Skyline Park	BSB		
39	01119818205110000, 01119818206010000	Mercury/Shields- Is this Belmont Park	BSB	Yes	
40	01119830416010000	Longfellow Ball Fields	BSB		
41	01119714411010000	West includes Dahlberg Field	School Dist No. 1		Yes
42	01119819440340000, 01119819440360000	Emerson	School Dist No. 1		
43		Skyline	School Dist No. 1		
44	01109521401300000	9 Mile	BSB		
45	0110952810101MINE	Eagles Nest	BSB		

TABLE 5: BUTTE-SILVER BOW PARKS AND OPEN AREAS

Item	**Geocode**	Name	Owner	Reclaimed Site	Adjacent Reclaimed Sites
46	01109534101010000	Lions Den	USFS		
47	01119833305100000	High Altitude Park	BSB		
48	0111971429906MINE	Huron Tennis Courts	BSB		
49	01119711420010000	McGlone Heights Skating Rink	BOARD OF REGENTS OF HIGHER EDUCATION		
50	01119713232090000	Rock Park-N Clark and W Granite	BSB		

ATTACHMENT A

QAPP CROSSWALK

EPA REGION 8 QA DOCUMENT REVIEW CROSSWALK

QAPP/FSP/SAP for: *(check appropriate box)* **GRANTEE** **CONTRACTOR** **EPA** **Other**	**Entity** *(grantee, contract, EPA AO, EPA Program, Other)* BSB County and AR	**Regulatory Authority** **and/or** **Funding Mechanism**	___ **2 CFR 1500 for Grantee/Cooperative Agreements** ___ **48 CFR 46 for Contracts** ___ **Interagency Agreement** ___ **EPA/Court Order** ___ **EPA Program Funding** ___ **EPA Program Regulation** ___ **EPA CIO 2105**
Document Title ***[Note: Title will be repeated in Header]***	BPSOU Draft Final Residential Metals Abatement Program (RMAP) QAPP (Non-Residential Parcels) (7/7/2021)		
QAPP/FSP/SAP Preparer	AR and BSB County		
Period of Performance *(of **QAPP**/FSP/SAP)*	**2021**	**Date Submitted for Review**	7/7/21
EPA Project Officer **EPA Project Manager**	Nikia Greene	**PO Phone #** **PM Phone #**	
QA Program Reviewer or Approving Official	Nikia Greene	**Date of Review**	7/8/21

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

1. QA Document(s) submitted for review:

QA Document	**Document Date**	**Document Stand-alone**	**Document with QAPP**
QAPP	**7/7/21**	**Yes** / No	■
FSP		Yes / No	Yes / No
SAP		Yes / No	Yes / No
SOP(s)	**(attached)**	■	**Yes** / No

2. WP/SOW/TO/PP/RP Date ____________
WP/SOW/TO/RP Performance Period ______________

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

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

Notes for Document Submittals:

1. A QAPP written by a Grantee, EPA, or Federal Partner must include for review: Work Plan(WP) / Statement of Work (SOW) / Program Plan (PP) / Research Proposal (RP) and funding mechanism
2. A QAPP written by Contractor must include for review:
   - **a)** Copy of Task Order Work Assignment/SOW
   - **b)** Reference to a hard or electronic copy of the contractor's approved QMP
   - **c)** Copy of Contract SOW if no QMP has been approved
   - **d)** Copy of EPA/Court Order, if applicable
   - **e)** The QA Review must determine (with the EPA CO or PO) if a QARF was completed for the environmental data activity described in the QAPP.
3. **a**. Field Sampling Plan (FSP) and/or Sampling & Analyses Plan (SAP) must include the Project QAPP ***or*** must be a stand-alone QA document that contain all QAPP required elements (Project Management, Data Generation/Acquisition, Assessment and Oversight, and Data Validation and Usability).
   c. SOPs must be submitted with a QA document that contains all QAPP required elements.

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

1. A QAPP is a formal document describing in comprehensive detail the necessary QA, QC, and other technical activities that must be implemented to ensure the results of the work performed will satisfy the stated performance criteria. The QAPP must be a stand-alone document that specifies the project's technical and quality objectives, the intended measurements, data generation, and data acquisition methods appropriate for achieving project objectives. A few references to

external documents have been made in this version of the QAPP. The information contained in these external references need to be included in the QAPP and not rely on finding or obtaining the external document. Another deficiency is the lack of discussion of field QC measures and sampling. Please eliminate all reference to the CFRSSI documents/standard operating procedures and include stand-alone support documentation specific to these data collection activities. In addition, please include a reference to the BPSOU Data Management Plan, which EPA and DEQ are currently reviewing (note: the review is an annual update review).

Atlantic Richfield Response (6/7/21): Field QC measures and project-specific standard procedures have been included in the revised document. Reference to the CFRSSI documents and SOPs has been removed from the plan. The BPSOU Data Management Plan was submitted to the Agencies for review and comment on December 22, 2017, and later approved in June 26, 2018.

EPA: Resolved. (6/16/21)

2. As a critical component of the ROD, documenting all RMAP activities is important. This includes noting site deficiencies, preparing corrective actions, management of data including the use of existing data, and tracking site progress. This information is critical to EPA's ability to assess whether BPSOU ROD remedial action objectives are being met, and the RMAP must describe how records of these activities will be kept and maintained. Further, EPA must have access to the data collected under this QAPP and the ability to access and determine the status and records of the RMAP.

   Atlantic Richfield Response (6/7/21): Section 2.9 in the revised QAPP describes documents and records. The RMAP database has been developed and is in use. The database is available for Agency access and additional comments and functionality requests. Atlantic Richfield and Butte-Silver Bow would like to coordinate Agency testing of the database with the program architects and primary users in a manner to minimize provision of written comment, and the potential misinterpretation of those comments. Ideally, this coordinated review and testing would occur concurrently with the Agencies review of the two separate RMAP QAPPs (residential and non-residential).

   EPA: Resolved. (6/16/21)

3. In 2011, the Explanation of Significant Differences added the Expanded Area to allow for attic sampling of residential properties outside of the BPSOU as part of the RMAP. With the recent attention given to the West Side Soils Operable Unit, EPA proposes to address contamination concerns outside of the BPSOU to allow for residential yard soil, interior living space dust, and lead-based paint sampling to occur as-needed or by request in the Expanded Area. Sampling adjacent areas outside of the Expanded Area will be permitted on a case-by-case basis. Please modify the RMAP QAPP to include all sampling types within the Expanded Area (and adjacent to the Expanded Area case-by-case) after receiving a request or a development proposal that could lead to a potential exposure pathway at a residential property.

   Atlantic Richfield Response (6/7/21): Atlantic Richfield and Butte-Silver Bow have agreed to expansion of the RMAP to the geographic extent indicated on Figure 1. Testing of properties outside of the BPSOU boundary (see Figure 1), yet falling within the 2020 RMAP Area, will be performed on a "by request" basis as defined in the RMAP Plan and QAPP.

   EPA: Resolved. (6/16/21)

4. As discussed further below, additional sampling efforts are needed for parks and play areas within the BPSOU and to address residential properties or sections where soil sampling may only have occurred in the 0-2 inch depth interval. In addition, updating and clarification on the data validation requirements is needed.

   Atlantic Richfield Response (6/7/21): Section 3.2.3 describes previously sampled properties that were sampled to a 0-2 inch depth. Section 6.0 in the revised QAPP describes the proposed data validation process.

Atlantic Richfield and Butte-Silver Bow have also agreed to include parks, play areas, schools, and commercial properties with residential living spaces within the RMAP. Discussion of these program additions are provided in Section 3.2 (schools, parks, and non-residential daycares). Commercial properties with residential living spaces will be addressed in the forthcoming RMAP Residential Parcels QAPP.

EPA: Resolved. (6/16/21)

5. The QAPP needs to clarify whether XRF or EPA methodologies will be used to analyze soil sampling within the RMAP. The QAPP seems to mostly specify that EPA standard methods will be used to analyze soils except in a few instances. For example, Sections 2.6.2, 3.8.3, and 7 have conflicting statements on which analytical method will be used for soils. Importantly, if XRF will be used to analyze soils, significant additions to the QAPP will be necessary. This includes a procedure for XRF sample preparation and analysis, submission of confirmation samples to establish XRF/wet laboratory correlations, evaluation of calibration verification checks against standard reference materials, and establishing an alternate XRF action level to limit remediation errors.

   Atlantic Richfield Response (6/7/21): XRF is not proposed for non-residential parcel soil analysis. The QAPP has been revised to clarify and eliminate conflicting statements.

   EPA: Resolved. (6/16/21)

6. Further details for the sampling of parks, schools, and commercial properties should be added to the QAPP. EPA anticipates that sampling these locations will be based on site-specific conditions. Please specify that EPA will be notified prior to the sampling of parks, schools, and commercial properties.

   Atlantic Richfield Response (6/7/21): Section 3.2 (schools, parks, and non-residential daycares) in the revised QAPP describes additional sampling in these areas previously excluded from RMAP. Commercial properties with residential living spaces will be addressed in the forthcoming RMAP Residential Parcels QAPP.

   EPA: Resolved. (6/16/21)

7. AR/BSB County should expect that revisions to the RMAP QAPP will be necessary on an annual basis. EPA anticipates that the effort to produce the updated RMAP QAPP will be reduced as refinements are made each year.

   Atlantic Richfield Response (6/7/21): Atlantic Richfield Response: The QAPP will be reviewed annually and updates completed as needed to accurately reflect program needs.

   EPA: Resolved. (6/16/21)

8. The BSB County and AR 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)”. In the crosswalk below, please provide your response in a different text color.
   All EPA crosswalk comments have been resolved (7/8/21)

Element	Acceptable *Yes/No/NA*	Page/ Section	Comments
A. Project Management			
A1. Title and Approval Sheet			

a. Contains project title	Yes	Title page and page i	EPA: No comments.
b. Date and revision number line (for when needed)	Yes	Title page and page i	EPA: Add a revision number line to the title and approval pages. **Atlantic Richfield Response (6/7/21): Text added.** EPA: Resolved. (6/16/21)
c. Indicates organization's name	Yes	Title page	EPA: No comments.
d. Date and signature line for organization's project manager	Yes	Page i	EPA: No comments.
e. Date and signature line for organization's QA manager	Yes	Page i	EPA: Add "Quality Assurance Approval Official" to Nikia Greene's signature line. **Atlantic Richfield Response (6/7/21): Text added.** EPA: Resolved. (6/16/21)
f. Other date and signatures lines, as needed	Yes	Page i	EPA: No comments.
A2. Table of Contents			
a. Lists QA Project Plan information sections	Yes	Pages iii to vi	EPA: No comments.
b. Document control information indicated	Yes	Page v	EPA: No comments.
A3. Distribution List			
Includes all individuals who are to receive a copy of the QA Project Plan and identifies their organization	Yes	Page ii	EPA: No comments.
A4. Project/Task Organization			
a. Identifies key individuals involved in all major aspects of the project, including contractors	Yes	Sections 2.0 through 2.3	EPA: The names of the key individuals need to be provided here in Sections 2.1 through 2.3 or, alternatively, in a new table. EPA realizes periodically there will be personnel changes – these changes can be captured in the annual review and update of the QAPP. **Atlantic Richfield Response (6/7/21): Sections 2.1 through 2.3 address this requirement at the organization level. Specific names, titles, and project roles are provided in the revised RMAP Non-Residential Parcels QAPP. An updated organizational chart is also provided in the revised QAPP Figure 2.** EPA: Resolved. (6/16/21)
b. Discusses their responsibilities	Yes	Sections 2.0 through 2.3	EPA: No comments.

c. Project QA Manager position indicates independence from unit generating data	Yes	Section 2.3, Figure 2	EPA: The QA manager was not specified. The responsibilities of the QA manager need to be added. **Atlantic Richfield Response (6/7/21): Section 2.3 was revised to clearly identify the role of the Superfund Quality Assurance Manager.** EPA: Resolved. (6/16/21)
d. Identifies individual responsible for maintaining the official, approved QA Project Plan	Yes	Section 2.3	EPA: The individual responsible for maintaining the official approved QAPP was not specified. **Atlantic Richfield Response (6/7/21): Section 2.3 was revised to clearly identify that the BSB Department of Reclamation and Environmental Services Director is responsible for maintaining the official approved QAPP, and for ensuring that the work is performed in accordance with the requirements contained in the RMAP QAPP.** EPA: Resolved. (6/16/21)
e. Organizational chart shows lines of authority and reporting responsibilities	Yes	Figure 2	EPA: The figure currently shows responsibilities extraneous to the RMAP program. An organizational chart specific only to the RMAP (with names) should be prepared. Additionally, other stakeholders should be depicted (such as AR, EPA/DEQ, QA Manager). **Atlantic Richfield Response (6/7/21): A revised organizational chart is provided in Figure 2 of the QAPP.** EPA: Resolved. (6/16/21)
A5. Problem Definition/Background			
a. States decision(s) to be made, actions to be taken, or outcomes expected from the information to be obtained	Yes	Sections 1.0 and 2.5 (formerly Section 2.4)	EPA: In Section 1.1, remove the two references to the Uniform Federal Policy for QAPPs (i.e., EPA 2005). This document is not in the format of a UFP-QAPP. Edit the reference section accordingly. Replace the second to last sentience of the first paragraph of Section 1.1 with: “This QAPP has been developed in accordance with the *EPA Requirements for Quality Assurance Project Plans, EPA QA/R-5* (EPA 2001), the *Guidance on Systematic Planning Using the Data Quality Objectives Process, EPA QA/G4* (EPA 2006), and the *EPA Region 8 Quality Assurance Document Review Crosswalk* checklist (EPA 2016).” In Section 2.4, modify the second sentence to read “,,,the soil sampling depth from 0 to 2 inches to the depth intervals provided in Section 3.2; changed the soil removal…” The 0-2, 2-6, and 6-12 depth intervals are discussed in Sections 3.2.1 as well as 3.2.2.

			Atlantic Richfield Response (6/7/21): The requested modifications were made to Section 1.1 and Sections 2.5 (formerly Section 2.4). Section 1.1 was revised to "*This QAPP has been developed in accordance with the EPA Requirements for Quality Assurance Project Plans, EPA QA/R-5 (EPA 2001), the Guidance on Systematic Planning Using the Data Quality Objectives Process, EPA QA/G4 (EPA 2006a), and the EPA Region 8 Quality Assurance Document Review Crosswalk checklist (EPA, 2016) provided in Attachment A.*" **Section 2.5 (formerly Section 2.4 in previous version of the document) was revised to "*This QAPP was developed in response to the EPA and Montana DEQ (the Agencies) 2006 Record of Decision (ROD) (EPA, 2006b) and Explanation of Significant Differences (ESD) to the 2006 Butte Priority Soils Operable Unit Record of Decision (EPA, 2011a). The ESD modified the soil sampling depth from 0 to 2 inches to the depth intervals provided in Section 3.2; changed the soil removal from a minimum depth of 18 inches to the minimum depth of 12 inches or to the soil bedrock interface if less than 12 inches; and extended the project schedule to accommodate expansion of the program.*"** EPA: Resolved. (6/16/21)
b. Clearly explains the reason (site background or historical context) for initiating this project	Yes	Sections 2.5 & 2.6 (Formerly Sections 2.4 & 2.5)	EPA: No comments.
c. Identifies regulatory information, applicable criteria, action limits, etc. necessary to the project	Yes	Section 2.1	EPA: In Section 2.1, modify the last sentence to read: "The Agencies also review sampling results, including those above the action levels listed in Table 1, and project completion reports." **Atlantic Richfield Response (6/7/21): Section 2.1 has been updated as requested.** EPA: Resolved. (6/16/21)
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 projects goals	Yes	Sections 1.0 and 2.6 (formerly Section 2.5)	EPA: In the second paragraph of Section 1.0 and fourth paragraph of Section 2.5, add interior air monitoring for mercury vapor to the list of sampling tasks. **Atlantic Richfield Response (6/7/21): Section 1.0 has been modified to state "*The potential sources of lead, arsenic, and/or mercury exposure addressed in the Program include lead, arsenic, and mercury in yard soil and interior living space dust, lead in interior and/or exterior LBP and drinking water from pipe solder, mercury exposure through attic dust when exposure pathways are identified and/or earthen basement soil, and interior air monitoring for mercury vapor.*"** **Section 2.6 (formerly Section 2.5), fourth paragraph has been modified to state "*The Program stipulates sampling residential soil, interior living space dust and attic dust for all COCs and interior air monitoring for mercury vapor within the BPSOU and by-request environmental sampling and remediation, if necessary, of residential properties outside of BPSOU, but within the 2020 RMAP Area shown in Figure 1*".** EPA: Resolved. (6/16/21)
b. Provides work schedule indicating critical project points, e.g., start and completion dates for activities such as sampling, analysis, data or file reviews, and assessments	Yes	Section 2.6 (formerly Section 2.5)	EPA: No comments.
c. Details geographical locations to be studied, including maps where possible	Yes	Sections 1.0 and 2.6 (formerly Section 2.5), Figure 1	EPA: No comments.
d. Discusses resource and time constraints, if applicable	Yes	Section 2.6.1 (formerly Section 2.5.1)	EPA: No comments.
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	No	Section 2.7.1 (formerly Section 2.6.1)	EPA: In Step 1, Table 1 specifies the analytical method for mercury vapor as "OSHA ID-140"; however, this method apples to the use of sorbent dosimeters analyzed by a laboratory, not a portable instrument like the TRACKER-3000. If sorbent dosimeters are being utilized (e.g., for confirmation measurements), this needs to be discussed further in the QAPP. In Step 2, add study questions for the other types of sampling being conducted (e.g., mercury vapor in air, indoor dust, etc.). Also add text on what actions may result. A table with the study questions and resulting actions may be a good way to provide this information. In Step 3, the text provides a good start describing the information inputs, but is incomplete. A summary of all the inputs needed to resolve the study questions in Step 2 is needed as well as text describing the use of input to resolve each study question. A table with the study question, the input to resolve the question, and the use of the input to resolve the question may be a good way to provide this information. In Step 4, more information on the vertical boundaries should be added, such as the highest point in a residential property and depths of sampling. The temporal boundaries of the investigation include the time from when evaluation and sampling actions begin at each property to the time specific clearance or completion criteria are met. In Step 5, a decision rule for mercury vapor needs to be added. In the third indented paragraph, specify the depth(s) of removal that may be implemented. Also in Steps 2 and 5, provide a definition and usage for the term "outdoor dust". **Atlantic Richfield Response (6/7/21): Project DQOs have been revised per the guidelines provided in the *Guidance on Systematic Planning Using the Data Quality Objectives Process* (EPA QA/G-4) (Feb 2006) per Agency request. Additionally, the term "outdoor dust" is not applicable to the Program and has been removed.** EPA: See comments on the revised DQOs provided in EPA's June 19, 2021 comment memorandum. (6/19/21) **Atlantic Richfield Response (7/2/21): Project DQOs have been updated per EPA's June 19, 2021 comment memorandum.** EPA: Resolved. (7/8/21)

b. Discusses precision	Yes	Sections 2.7.2 (formerly Section 2.6.2) and Section 3.5.2 (formerly Section 3.8.3)	EPA: No comments.
c. Addresses bias	Yes	Sections 2.7.2 (formerly Section 2.6.2) and Section 3.5.2 (formerly Section 3.8.3)	EPA: No comments.
d. Discusses representativeness	Yes	Sections 2.7.2 (formerly Section 2.6.2) and Section 3.5.2 (formerly Section 3.8.3)	EPA: It is not clear in the QAPP which soil samples will be analyzed by XRF and those that will be analyzed by EPA standard laboratory methods. For example, here in Section 2.6.2 discussing representativeness it is stated that "…in-place soils and backfill material will be analyzed by laboratory-grade XRF…" However, later in Section 3.8.1 it states that "…analyses will be in accordance with the EPA analytical method specifications…". Please clarify in the document when the different analytical methods will be used for soil analyses. **Atlantic Richfield Response (6/7/21): The referenced text has been removed from Section 2.7.2 (formerly Section 2.6.2) in the revised document. Standard methods referenced in the document will be used.** EPA: Resolved. (6/16/21)
e. Identifies the need for completeness	Yes	Sections 2.7.2 (formerly Section 2.6.2) and Section 3.5.2 (formerly Section 3.8.3)	EPA: No comments.

f. Describes the need for comparability	Yes	Sections 2.7.2 (formerly Section 2.6.2) and Section 3.5.2 (formerly Section 3.8.3)	EPA: No comments.
g. Discusses desired method sensitivity	No	Add to Section 2.7.2 (formerly Section 2.6.2)	EPA: Add a discussion regarding data sensitivity. For example, a discussion of the sensitivity of the TRACKER-3000 compared to the mercury vapor action level is needed. **Atlantic Richfield Response (6/7/21): Sensitivity is related to the ability to compare analytical results with project-specific action levels. Analytical quantitation limits for the sample analytes should be below the level of interest to allow an effective comparison.** **Indoor assessments/sampling such as mercury vapor are not addressed under this version of the QAPP and will be addressed through forthcoming annual QAPP revisions. A mercury vapor method detection limit will be addressed at that time.** EPA: Resolved. As EPA understands it, indoor mercury vapor investigations are usually only implemented if soil sample results indicate the presence of mercury. Because the focus of this QAPP is on exterior evaluations, no further modifications are necessary. However, the interior Non-Residential QAPP Amendment will need to document the process for mercury vapor investigation. (6/19/21)
A8. Special Training/Certifications			

a. Identifies any project personnel specialized training or certifications	Yes	Section 2.8 (Formerly Section 2.7)	EPA: In the first paragraph, make sure it is clear that this is RMAP training. Note any special training requirements for use of the XRF and/or mercury vapor analyzer. Also, all field personnel should have HAZWOPER training. **Atlantic Richfield Response (6/7/21): Section 2.8 (formerly Section 2.7) has been updated as "*All RMAP field personnel will review the requirements of this QAPP and receive training on Program-related tasks during a project meeting held prior to the beginning of fieldwork. A review of sampling procedures and requirements will be completed prior to field activities to ensure sample collection and handling methods are according to QAPP requirements. Field personnel will be trained in proper use of field equipment, sample collection tools, etc., and procedures according to field data collection SOPs (Attachment C-1) and methods described in the Program. Field personnel performing sampling activities or members who can potentially contact contaminated materials should receiver hazardous waste operations and emergency response (HAZWOPER) training.*"** EPA: Resolved. (6/16/21)
b. Discusses how this training will be provided	Yes	Section 2.8 (Formerly Section 2.7)	EPA: No comments.

c. Indicates personnel responsible for assuring training/certifications are satisfied	Yes	Section 2.8 (Formerly Section 2.7)	EPA: The personnel responsible for this element need to be identified. **Atlantic Richfield Response (6/7/21): Section 2.8 (formerly Section 2.7), second paragraph has been updated to state "*The BSB Department of Reclamation and Environmental Services Director is responsible for ensuring field personnel receive appropriate training and will maintain up-to-date training records and/or certifications. The BSB Department of Reclamation and Environmental Services Human Health/RMAP Division Manager will assure that each member of the sampling team obtains and is familiar with the recent version of the QAPP, will maintain signatures of each team member who has read the QAPP (including reviews and addenda, as necessary), and make sure each team member has been trained in the appropriate sample collection methods per the Program. The Human Health/RMAP Division Manager will review the SSHASP 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 also 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.*"** EPA: Resolved. (6/16/21)
d. identifies where this information is documented	Yes	Section 2.8 (Formerly Section 2.7)	EPA: No comments.
A9. Documentation and Records			
a. Identifies report format and summarizes all data report package information	Yes	Section 2.9 (formerly Section 2.8)	EPA: No comments.
b. Lists all other project documents, records, and electronic files that will be produced	Yes	Section 2.9 (formerly Section 2.8)	EPA: No comments.

c. Identifies where project information should be kept and for how long	No	Section 2.9 (formerly Section 2.8)	EPA: Add text on how the project information described in Section 2.8 can be obtained, where it is being stored, and for how long. **Atlantic Richfield Response (6/7/21): Section 2.9 (formerly Section 2.8) has been revised to the include additional details regarding project storage, backup and retention. All sampling data conducted for all media under the RMAP, including soils, attic dust, indoor dust and basement soils within the BPSOU and records of property access requests are housed within the RMAP database. The RMAP database is housed in an Access SQL server database and maintained by BSB. Document backups are contained in the BPSOU Document SharePoint and EPA document repository. Refer to the BPSOU Data Management Plan for additional details regarding data management, backup, and storage.** EPA: Resolved. (6/16/21)
d. Discusses back up plans for records stored electronically	No	Section 2.9 (formerly Section 2.8)	EPA: Add more detail on how the data and information is backed up. **Atlantic Richfield Response (6/7/21): Refer to response above.** EPA: Resolved. (6/16/21)

e. States how individuals identified in A3 will receive the most current copy of the approved QA Project Plan, identifying the individual responsible for this	No	Section 2.8 (formerly Section 2.7)	EPA: Clarify how the QAPP will be distributed and identify the individual responsible for this. **Atlantic Richfield Response (6/7/21): Atlantic Richfield will be responsible for distributing the original Agency approved QAPP to the individuals on the QAPP distribution list. Subsequent annual revisions will be distributed by the Butte Silver Bow Department of Reclamation and Environmental Services QA Manager. This is documented in text directly below the QAPP distribution list.** **Section 2.8 (formerly Section 2.7) has been revised to state "*The BSB Department of Reclamation and Environmental Services Director is responsible for ensuring field personnel receive appropriate training and will maintain up-to-date training records and/or certifications. The BSB Department of Reclamation and Environmental Services Human Health/RMAP Division Manager will assure that each member of the sampling team obtains and is familiar with the recent version of the QAPP, will maintain signatures of each team member who has read the QAPP (including reviews and addenda, as necessary), and make sure each team member has been trained in the appropriate sample collection methods per the Program.*"** EPA: Resolved. (6/16/21)

B. Data Generation/Acquisition

B1. Sampling Process Design (Experimental Design)

a. Describes and justifies design strategy, indicating size of the area, volume, or time period to be represented by a sample	Yes	Section 3.0	EPA: Given recent park sampling efforts in Anaconda, a fresh assessment of environmental conditions of all parks and play areas in the BPSOU and surrounding area is needed. A new section titled "Parks & Play Areas Sampling" needs to be added describing the compiling of existing park data, cataloguing response actions taken to date at parks and recreation sites, plans to fill data gaps with supplemental sampling, and the preparation of a data summary report for this sampling effort. **Atlantic Richfield Response (6/7/21): Section 3.2 details the sampling plan for schools, parks, and non-residential daycares (which includes the delineation of the 5 land use categories). Existing park data will be reviewed but is not anticipated to be particularly useful for future remedial decision making due to current RMAP sampling guidelines (particularly current guidance around delineation of sampling polygons based upon land use categories).** **BSB will include cataloguing of action items in development of RMAP database.** EPA: Resolved. (6/16/21)
b. Details the type and total number of sample types/matrix or test runs/trials expected and needed	Yes	Sections 3.2, 3.3, 3.4, and 3.5	EPA: No comments. **Atlantic Richfield Response (6/7/21): Former sections 3.3 (attic and crawl spaces), 3.4 (interior dust sampling), and 3.5 (interior air monitoring) are not addressed in this version of the QAPP. They will be addressed through forthcoming annual QAPP revisions.**

c. Indicates where samples should be taken, how sites will be identified/located	Yes	Section 3.2.1	EPA: As originally stated in the BPSOU ROD, soil was to be sampled from the 0-2 inch depth interval, at a minimum. As modified in the 2011 BPSOU ESD, the residential yard sampling described in Section 3.2.1 calls for sampling from the 0-2 inch, 2-6 inch, and 6-12 inch depth intervals. EPA understands there may be properties where only the 0-2 inch depth interval was sampled in prior sampling and evaluation events. Please add text describing the identification of those properties where only 0-2 inch sampling has occurred and the plans to complete the sampling from the 2-6 and 6-12 inch depth intervals and taking appropriate follow-up action is action levels are exceeded. **Atlantic Richfield Response (6/7/21): Section 3.2.3 (Previously Sampled Properties) has been added to address this sampling scenario. BSB will review the RMAP database to identify properties that were previously sampled to the 0-2 inch depth interval. Property owners of previously sampled properties where remediation was not completed will be contacted to request access to repeat the sampling to appropriate depth intervals. Sampling protocol described previously will be followed for the 2-6 inch and 6-12 inch depth intervals.** **Properties that were sampled at the 0-2 inch depth interval and remediated will not be resampled.** EPA: Resolved. (6/16/21)

d. Discusses what to do if sampling sites become inaccessible	Yes	Section 3.1	EPA: This item refers to sampling sites that become inaccessible due, for example, to weather conditions, etc. Physically, access is not an anticipated issue during the RMAP sampling. However, EPA understands there may be property owners who refuse to participate in the RMAP. The Agencies will assist AR and BSB in these cases. An addendum to the RMAP will be provided, describing the assistance that will be taken. **Atlantic Richfield Response (6/7/21): Additional language has been included in Section 3.1 regarding three documented attempts to gain access, and a reset of attempts with a change in property ownership. *"After three attempts are recorded, EPA and DEQ will be notified of the property status. At this time, EPA and/or DEQ may elect to issue the property owner an enforcement letter. A copy of the Agency notice form letter is provided in Attachment B-2. Future changes in ownership will be monitored annually. If ownership changes, the access procurement process will be re-initiated."*** EPA: Resolved. (6/16/21)
e. Identifies project activity schedules such as each sampling event, times samples should be sent to the laboratory, etc.	Yes	Sections 3.2, 3.3, 3.4, and 3.5	EPA: No comments. **Atlantic Richfield Response (6/7/21): Former sections 3.3 (attic and crawl spaces), 3.4 (interior dust sampling), and 3.5 (interior air monitoring) are not addressed in this version of the QAPP. They will be addressed through forthcoming annual QAPP revisions.**
f. Specifies what information is critical and what is for informational purposes only	Yes	Sections 3.2, 3.3, 3.4, and 3.5	EPA: No comments. **Atlantic Richfield Response (6/7/21): Former sections 3.3 (attic and crawl spaces), 3.4 (interior dust sampling), and 3.5 (interior air monitoring) are not addressed in this version of the QAPP. They will be addressed through forthcoming annual QAPP revisions.**
g. Identifies sources of variability and how this variability should be reconciled with project information	Yes	Step 6	EPA: No comments.
B2. Sampling Methods			

a. Identifies all sampling SOPs by number, date, and regulatory citation, indicating sampling options or modifications to be taken	Yes	Sections 3.3 and 3.4	EPA: As noted in comments in other recent documents, the CFRSSI SOPs cited are out of date and need updating. The cited SOP from the *Interior and Attic Dust Sampling and Analysis Plan* (Atlantic Richfield, 2007) must be attached to this QAPP. Alternatively, an SOP for dust sampling could be prepared. QAPPs are intended to be stand-alone documents with all sampling information contained therein. The first sentence of the second paragraph in Section 3.3 is confusing as there is not a CFRSSI SOP for attic dust sampling. In Section 3.4, the HVS3 manual should be attached to the QAPP. **Atlantic Richfield Response (6/7/21): The appropriate SOPs have been included in the revised QAPP.** EPA: Resolved. (6/16/21)
b. Indicates how each sample/matrix type should be collected	Yes	Sections 3.2, 3.3, 3.4, and 3.5	EPA: No comments. **Atlantic Richfield Response (6/7/21): Former sections 3.3 (attic and crawl spaces), 3.4 (interior dust sampling), and 3.5 (interior air monitoring) are not addressed in this version of the QAPP. They will be addressed through forthcoming annual QAPP revisions.**
c. If in situ monitoring, indicates how instruments should be deployed and operated to avoid contamination and ensure maintenance of proper data	NA	NA	EPA: No in-situ instruments will be deployed.
d. If continuous monitoring, indicates averaging time and how instruments should store and maintain raw data, or data averages	NA	NA	EPA: No continuous monitoring instruments will be deployed.
e. Indicates how samples are to be homogenized, composited, split, or filtered, if needed	Yes	Section 3.8.2	EPA: Verify the sieve size needed for dust samples. Section 3.8.2 specifies a No. 18 sieve size, whereas the HVS3 method specifies and No. 100 sieve size. **Atlantic Richfield Response (6/7/21): Former section 3.8.2 (Dust Analyses Methods has been removed from this QAPP. Interior assessments/sampling are not addressed in this version of the QAPP and will be addressed through forthcoming annual QAPP revisions.** EPA: Resolved. (6/16/21)

f. Indicates what sample containers and sample volumes should be used	Yes	Sections 3.2, 3.3, 3.4, and 3.5	EPA: In Section 3.6.1, please add the container type and sample volume requirement for the non-metals analysis. **Atlantic Richfield Response (6/7/21): Former section 3.6.1 (Residential Lead Paint Sampling) has been removed from this QAPP. This initial version of the QAPP focuses solely on non-residential parcel soil sampling.** EPA: Resolved. (6/16/21)
g. Identifies whether samples should be preserved and indicates methods that should be followed	Yes	Section 3.6.2	EPA: Regarding residential water sampling, add information to this section regarding the analytes to be requested (is it just lead?), bottle size required, and preservative. **Atlantic Richfield Response (6/7/21): Former section 3.6.2 (Residential Water Sampling) has been removed from this QAPP. This initial version of the QAPP focuses solely on non-residential parcel soil sampling.** EPA: Resolved. (6/16/21)
h. Indicates whether sampling equipment and samplers should be cleaned and/or decontaminated, identifying how this should be done and by-products disposed of	Yes	Section 3.2.4, SOP G-8, Manuals	EPA: No comments except suggest adding notes that sampling equipment (e.g., the HSV3) will be decontaminated per manufacturer requirements. **Atlantic Richfield Response (6/7/21): Section 3.2.4 has been revised to include the following text "*Re-usable equipment may be decontaminated between sampling sites in accordance with manufacturer's recommendations and established SOPs (Attachment C-1) and prior to being re-used.*"** EPA: Resolved. (6/16/21)
i. Identifies any equipment and support facilities needed	Yes	TBD	EPA: Specify in the document where the sample preparation and XRF analytical work, if used, will be performed. **Atlantic Richfield Response (6/7/21): This version of the QAPP deals solely with soil sampling. As detailed in Section 3.5.1, *"All RMAP soil samples will be analyzed to determine metals concentrations via standard laboratory analytical methodologies for arsenic, lead, and mercury as appropriate."* Interior assessments/sampling will be addressed through forthcoming annual QAPP revisions.** EPA: Resolved. (6/16/21)

j. Addresses actions to be taken when problems occur, identifying individual(s) responsible for corrective action and how this should be documented	Yes	Section 5.0	EPA: No comments.
B3. Sample Handling and Custody			
a. States maximum holding times allowed from sample collection to extraction and/or analysis for each sample type and, for in-situ or continuous monitoring, the maximum time before retrieval of information	Yes	Section 3.3 (formerly Section 3.7.1)	EPA: No comments.
b. Identifies how samples or information should be physically handled, transported, and then received and held in the laboratory or office (including temperature upon receipt)	Yes	Section 3.3 (formerly Section 3.7.1)	EPA: No comments.
c. Indicates how sample or information handling and custody information should be documented, such as in field notebooks and forms, identifying individual responsible	Yes	Section 2.9.4 (formerly Section 2.8.4)	EPA: No comments.
d. Discusses system for identifying samples, for example, numbering system, sample tags and labels, and attaches forms to the plan	Yes	Section 3.4 (formerly Section 3.7)	EPA: No comments.
e. Identifies chain-of-custody procedures and includes form to track custody	Yes	Section 2.9.4 (formerly Section 2.8.4)	EPA: No comments.
B4. Analytical Methods			
a. Identifies all analytical SOPs (field, laboratory and/or office) that should be followed by number, date, and regulatory citation, indicating options or modifications to be taken, such as sub-sampling and extraction procedures	Yes	Section 3.5 (formerly Section 3.8), Table 1, Attachment C (formerly Attachment 2), Former Attachment 3 (Manufacturer Procedures) has been removed from this QAPP	EPA: Table 1 needs to make clear which analytical method will be used to analyze soils (i.e., XRF or EPA Methodology). Additionally, Ashe Analytics should be removed as a laboratory services provider. **Atlantic Richfield Response (6/7/21): Table 1 has been updated to reflect that EPA Methodology will be used to analyze all soil samples. Interior assessments/sampling will be addressed through forthcoming annual QAPP revisions.** **The revised QAPP does not include reference to Ashe Analytics.** EPA: Resolved. (6/16/21)

b. Identifies equipment or instrumentation needed	Yes	Section 3.5 (formerly Section 3.8)	EPA: No comments.
c. Specifies any specific method performance criteria	Yes	Sections 2.7.2 (formerly Section 2.6.2) and Section 3.5.2 (formerly Section 3.8.3)	EPA: No comments.
d. Identifies procedures to follow when failures occur, identifying individual responsible for corrective action and appropriate documentation	Yes	Section 5.0	EPA: No comments.
e. Identifies sample disposal procedures	Yes	Section 3.7 (formerly Section 3.9)	EPA: No comments.
f. Specifies laboratory turnaround times needed	Yes	Section 5.3	EPA: No comments.
g. Provides method validation information and SOPs for nonstandard methods	Yes	Section 6.0	EPA: No comments.
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	No	Sections 3.2, 3.3, 3.4, and 3.5	EPA: Field QC measures and sampling (e.g., duplicates) for each type of sampling need to be discussed in these sections. **Atlantic Richfield Response (6/7/21): Field duplicate samples are described in Section 3.6 in the revised QAPP.** **Former sections 3.3 (attic and crawl spaces), 3.4 (interior dust sampling), and 3.5 (interior air monitoring) are not addressed in this version of the QAPP. They will be addressed through forthcoming annual QAPP revisions.** EPA: See additional comments regarding field and laboratory QC provided in EPA's June 19, 2021 comment memorandum. (6/19/2021) **Atlantic Richfield Response (7/2/21): Field and laboratory QC information has been updated per EPA's June 19, 2021 comment memorandum.**

b. Details what should be done when control limits are exceeded, and how effectiveness of control actions will be determined and documented	Yes	Section 5.0	EPA: No comments.
c. Identifies procedures and formulas for calculating applicable QC statistics, for example, for precision, bias, outliers and missing data	Yes	New section similar to Section 3.8.3	EPA: A new section similar to Section 3.8.3 discussing field QC activities and QC samples needs to be added. This new section needs to discuss, for example, field duplicate samples, the results of duplicate sampling, QC measurements during XRF paint analysis, results of decon blanks (e.g., after sieve decon), QC checks needed for the TRACKER-3000, etc. **Atlantic Richfield Response (6/7/21): Section 3.6 Field Quality Control Samples has been added to the Revised QAPP.** EPA: Resolved. (6/16/21)
B6. Instrument/Equipment Testing, Inspection, and Maintenance			
a. Identifies field and laboratory equipment needing periodic maintenance, and the schedule for this	Yes	Section 3.8 (formerly Section 3.10)	EPA: No comments.
b. Identifies testing criteria	Yes	Section 3.8 (formerly Section 3.10)	EPA: No comments.
c. Notes availability and location of spare parts	Yes	Section 3.8 (formerly Section 3.10)	EPA: No comments.
d. Indicates procedures in place for inspecting equipment before usage	Yes	Section 3.8 (formerly Section 3.10)	EPA: No comments.
e. Identifies individual(s) responsible for testing, inspection and maintenance	Yes	Section 3.8 (formerly Section 3.10)	EPA: No comments.
f. Indicates how deficiencies found should be resolved, re-inspections performed, and effectiveness of corrective action determined and documented	Yes	Section 3.8 (formerly Section 3.10)	EPA: No comments.
B7. Instrument/Equipment Calibration and Frequency			

a. Identifies equipment, tools, and instruments that should be calibrated and the frequency for this calibration	Yes	Sections 2.8 (former Section 2.7), 2.9.2 (former Section 2.8.2), 3.8 (former Section 3.10)	EPA: No comments.
b. Describes how calibrations should be performed and documented, indicating test criteria and standards or certified equipment	Yes	Sections 2.8 (former Section 2.7), 2.9.2 (former Section 2.8.2), 3.8 (former Section 3.10)	EPA: No comments.
c. Identifies how deficiencies should be resolved and documented	Yes	Section 5.0	EPA: No comments.
B8. Inspection/Acceptance for Supplies and Consumables			
a. Identifies critical supplies and consumables for field and laboratory, noting supply source, acceptance criteria, and procedures for tracking, storing and retrieving these materials	Yes	Section 3.9 (former Section 3.11)	EPA: No comments.
b. Identifies the individual(s) responsible for this	Yes	Section 3.9 (former Section 3.11)	EPA: No comments.
B9. Use of Existing Data (Non-direct Measurements)			
a. Identifies data sources, for example, computer databases or literature files, or models that should be accessed and used	Yes	Section 6.0	EPA: No comments.
b. Describes the intended use of this information and the rationale for their selection, i.e., its relevance to project	Yes	Section 6.0	EPA: No comments.
c. Indicates the acceptance criteria for these data sources and/or models	Yes	Section 6.0	EPA: No comments.
d. Identifies key resources/support facilities needed	Yes	Section 6.0	EPA: No comments.
e. Describes how limits to validity and operating conditions should be determined, for example, internal checks of the program and Beta testing	Yes	Section 6.0	EPA: No comments.
B10. Data Management			

a. Describes data management scheme from field to final use and storage	Yes	Section 3.10 (formerly Section 3.12)	EPA: No comments.
b. Discusses standard record-keeping and tracking practices, and the document control system or cites other written documentation such as SOPs	Yes	Section 3.10 (formerly Section 3.12)	EPA: No comments.
c. Identifies data handling equipment/procedures that should be used to process, compile, analyze, and transmit data reliably and accurately	Yes	Section 3.10 (formerly Section 3.12)	EPA: No comments.
d. Identifies individual(s) responsible for this	Yes	Section 3.10 (formerly Section 3.12)	EPA: Add the individuals responsible for data management and/or add text clarifying this in Section 2.3. **Atlantic Richfield Response (6/7/21): The Superfund Quality Assurance Manager has been identified as the responsible party for data management in Section 2.3.** EPA: Resolved. (6/16/21)
e. Describes the process for data archival and retrieval	Yes	Section 3.10 (formerly Section 3.12)	EPA: Summarize the process where entities such as EPA can request or review data and information from the RMAP. **Atlantic Richfield Response (6/7/21): Subsection 3.10.1 Requests for Data has been added to the revised QAPP.** EPA: Resolved. (6/16/21)
f. Describes procedures to demonstrate acceptability of hardware and software configurations	Yes	Section 3.10 (formerly Section 3.12)	EPA: No comments.
g. Attaches checklists and forms that should be used	Yes	Section 3.10 (formerly Section 3.12)	EPA: Please provide a copy of the BSB Data Management Plan (BSB, 2016) with the next submittal of this QAPP. **Atlantic Richfield Response (6/7/21): The appropriate reference is Final Draft BPSOU Data Management Plan, Rev. 1 submitted 12/22/2017.** EPA: Resolved. (6/16/21)
C. Assessment and Oversight			
C1. Assessments and Response Actions			
a. Lists the number, frequency, and type of assessment activities that should be conducted, with the approximate dates	Yes	Section 5.0	EPA: No comments at this time.

b. Identifies individual(s) responsible for conducting assessments, indicating their authority to issue stop work orders, and any other possible participants in the assessment process	Yes	Section 5.0	EPA: No comments at this time.
c. Describes how and to whom assessment information should be reported	Yes	Section 5.1 and 5.2	EPA: No comments at this time.
d. Identifies how corrective actions should be addressed and by whom, and how they should be verified and documented	Yes	Section 5.1 and 5.2	EPA: No comments at this time.
C2. Reports to Management			
a. Identifies what project QA status reports are needed and how frequently	Yes	Section 5.3	EPA: No comments at this time.
b. Identifies who should write these reports and who should receive this information	Yes	Section 5.3	EPA: No comments at this time.
D. Data Validation and Usability			
D1. Data Review, Verification, and Validation			

Describes criteria that should be used for accepting, rejecting, or qualifying project data	Yes	Section 6.0	EPA: 1) There is reference made to the CFRSSI Data Management/Data Validation Plan Addendum (AERL 2000) in Section 6. It is EPA's understanding that this QAPP updates the validation process and is not following the older documents but developing an updated approach to validation while maintaining the critical elements of the previous historical documents. Clearly, it is time to update the 2000 DM/DV Plan and Pilot Data Report or take the steps needed to incorporate the necessary information from these documents into the BPSOU QMP and this QAPP. **Atlantic Richfield Response (6/7/21): The critical elements of the CFRSSI documents, including the data validation checklists, Level AB assessment checklist, definitions of enforcement / screening / rejected data quality, and the data quality assessment process, have been included with the appropriate references in the revised QAPP.** **All samples analyzed for metals at a commercial laboratory will be validated following the CFRSSI documents and the EPA National Functional Guidelines for Inorganic Superfund Methods Data Review, January 2017.** EPA: Resolved. (6/16/21) 2) Information needs to be provided on what level of quality the data needs to be that is being collected (enforcement versus screening). **Atlantic Richfield Response (6/7/21): Section 6.0 has been modified to describe enforcement quality data.** EPA: Resolved. (6/16/21) 5) Update the reference for the EPA National Functional Guidelines to the current version: National Functional Guidelines for Inorganic Superfund Methods Data Review (January 2017). **Atlantic Richfield Response (6/7/21): The EPA National Functional Guidelines has been updated as recommended.** EPA: Resolved. (6/16/21) 6) Update the reference for the EPA CLP SOW for Inorganic Superfund Methods from 2010 to the October 2016 version. The

			current ISM SOW is ISM02.4. **Atlantic Richfield Response (6/7/21): The EPA CLP SOW for Inorganic Superfund Methods has been updated as recommended.** EPA: Resolved. (6/16/21)
D2. Verification and Validation Methods			
a. Describes process for data verification and validation, providing SOPs and indicating what data validation software should be used, if any	Yes	Section 6.0	EPA: See applicable comments from D1. **Atlantic Richfield Response (6/7/21): Section 6.0 has been updated.** EPA: Resolved. (6/16/21)
b. Identifies who is responsible for verifying and validating different components of the project data/information, for example, chain-of-custody forms, receipt logs, calibration information, etc.	Yes	Section 6.0	EPA: See applicable comments from D1. **Atlantic Richfield Response (6/7/21): Section 6.0 has been updated.** EPA: Resolved. (6/16/21)
c. Identifies issue resolution process, and method and individual responsible for conveying these results to data users	Yes	Section 6.0	EPA: See applicable comments from D1. **Atlantic Richfield Response (6/7/21): Section 6.0 has been modified to describe responsibilities.** EPA: Resolved. (6/16/21)
d. Attaches checklists, forms, and calculations	Yes	Section 6.0	EPA: See applicable comments from D1. **Atlantic Richfield Response (6/7/21): Section 6.0 has been updated.** EPA: Resolved. (6/16/21)
D3. Reconciliation with User Requirements			
a. Describes procedures to evaluate the uncertainty of the validated data	Yes	Section 6.0	EPA: See applicable comments from D1. **Atlantic Richfield Response (6/7/21): Section 6.0 has been updated.** EPA: Resolved. (6/16/21)
b. Describes how limitations on data use should be reported to the data users	Yes	Section 6.0	EPA: See applicable comments from D1. **Atlantic Richfield Response (6/7/21): Section 6.0 has been updated.** EPA: Resolved. (6/16/21)

ATTACHMENT B

ACCESS FORMS

ATTACHMENT B-1

EXAMPLE ACCESS AGREEMENT FORM

ACCESS AGREEMENT

BUTTE SILVER BOW ("OWNER"), whose mailing address is, 155 Granite Street, Butte, MT 59701 and **Atlantic Richfield Company** ("Atlantic Richfield"), whose mailing address is 317 Anaconda Road, Butte, MT 59701, enter into this Access Agreement ("Agreement") this ______ day of _______________, 2021 and agree as follows:

1. GRANT OF ACCESS. OWNER hereby grants to Atlantic Richfield, including its authorized representatives (and, as may be appropriate, to EPA and/or the State of Montana and the authorized representatives of each) the right to enter OWNER's real property, as described in Exhibit A, which is attached hereto and incorporated herein by reference (the "Property"), to conduct all activities related to sampling of interior/attic dust and/or soils (collectively referred to as "Sampling"). OWNER represents to Atlantic Richfield that, to the best of OWNER's knowledge, OWNER possesses ownership interests in the Property sufficient to grant access to Atlantic Richfield to conduct the Sampling.

2. ATLANTIC RICHFIELD REPRESENTATIONS. Atlantic Richfield or its representative will notify OWNER, either in writing or verbally, at least 24 hours prior to first commencing Sampling on the Property. Atlantic Richfield will make every reasonable effort to minimize any inconvenience to OWNER during its Sampling on the Property, to return the Property to the condition it was in at the time Atlantic Richfield first entered the Property under this Agreement, and to consult with OWNER to address any concerns OWNER may have about the Sampling activity.

3. SPLIT SAMPLE. Atlantic Richfield agrees to use its best efforts to provide, upon OWNER's prior written request a portion of any sample taken on OWNER's Property for subsequent laboratory analysis, provided that a sufficient quantity of the materials to be sampled are available on the day of sampling, and provided further that the sampling requirements of Atlantic Richfield are satisfied.

4. TERMINATION. This Access Agreement will terminate thirty (30) days following receipt of the written notice from Atlantic Richfield stating the Sampling activities on your Property have been completed.

IN WITNESS WHEREOF, OWNER and Atlantic Richfield Company have executed this Agreement effective as of the date first written above.

OWNER:	ATLANTIC RICHFIELD COMPANY
By: ______________________________	By: __________________________
Title (If other than Home Owner): ____________________	Title: Project Manager
Telephone Contact No. _______________	

EXHIBIT A

For the purposes of this Access Agreement, the term Property refers to the following described real estate, situated in the County of Silver Bow, State of Montana:

Sample Identification: P-00001

Property Address: No Physical Address (Jeremy Bullock Soccer Fields), Butte, MT 59701

Property Geocode: 01119831305010000

Legal Description: S31, T03 N, R07 W, POR SW4 AKA ALL BLKS 6, 7 VAC OREGON AVE BETWEEN SUB TRACTS

ATTACHMENT B-2

EPA NOTICE FORM LETTER

UNITED STATES ENVIRONMENTAL PROTECTION AGENCY
REGION 8, MONTANA OFFICE
FEDERAL BUILDING, 10 West 15TH Street, Suite 3200
Helena, MT 59626-0096
Phone 866-457-2690
www.epa.gov/region8

Ref: 8 ORC-LEP/MO

DRAFT 9/16/2019 EPA DATE

URGENT: FINAL OPPORTUNITY. PLEASE READ AND RESPOND.

Ref: 8EPR-SR

NAME
ADDRESS
CITY, STATE, ZIP

Re: PROPERTY LEGAL DESCRIPTION:______________________

Dear Property Owner:

The U.S. Environmental Protection Agency (EPA) requests access to your property for environmental assessment, including the collection and analysis of samples of exterior yard soils, interior living space dust and attic dust if exposure pathways are identified. These activities are components of the Multi-Pathway Residential Metals Abatement Program (RMAP) which is designed to mitigate potentially harmful residential exposures to sources of lead, arsenic and mercury contamination. The RMAP is being implemented pursuant to EPA's authority under the federal Superfund law known as the Comprehensive Environmental Response, Compensation and Liability Act (CERCLA).

You were previously contacted by (Butte Silver Bow County) *or* (the Atlantic Richfield Company) for such access in letters dated _________________. An affirmative reply to those requests has not been received.

This is your final opportunity to provide voluntary access to your residential property so that the environmental assessment and abatement activities, if required, can occur as required by CERCLA. If you do not provide access to your property by ______________, you may be responsible for any future assessment and cleanup of your property.

Assessment and abatement actions, if indicated by the sampling results collected during the initial assessment, will protect human health and meet objectives of the final remedy as defined in the EPA's Butte Priority Soils Record of Decision, as amended. If the EPA is unable to complete the investigation of your property, be advised that EPA or the State of Montana have authority to and will consider recording a copy of this letter in the chain of title for your property in the Butte-Silver Bow County real property records. The purpose of such recording is to inform future potential owners of your property that your property has not been assessed and appropriately remediated, as indicated by the results of sampling conducted in the course of the RMAP assessment.

To grant access for assessment of your property, please call an EPA representative at ___________ or return the enclosed access form in the postage-paid return envelope **to the EPA by ____________. We will attempt to schedule the RMAP inspection and future abatement activities, if required based upon the results of the initial environmental assessment activities, at a time that is convenient for you; however, the assessment and sampling of your property must be scheduled by _______________.**

After the inspection and assessment of your property is complete, including the receipt of any sampling results, you will receive a letter from Butte Silver Bow County documenting the results of the environmental assessment. Thank you for considering this opportunity. Please contact the Nikia Greene at 406 457-5019 if you have any questions or concerns.

Sincerely,

Site Attorney, BPSOU

Enclosures: Access form and return envelope

ATTACHMENT C

STANDARD OPERATING PROCEDURES

ATTACHMENT C-1

FIELD SOPs

Attachment C-1
Field SOPs
Index

RESIDENTIAL METALS ABATEMENT PROGRAM

STANDARD OPERATING PROCEDURE
RMAP-SOP-1A
NON-RESIDENTIAL PARCEL 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 areas that may require remediation to protect the public health. This SOP is applicable to non-residential parcels within the Residential Metals Abatement Program (RMAP) such as schools, parks, and non-residential daycares.

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 component specific analytical data from which remedial action decisions can be made.

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

COMPOSITE SAMPLING

Sample collection devices include disposable plastic scoops. The following procedure is designed to be used to collect soil samples from the 0-12 inch horizon. These procedures may be modified in the field based on field and site conditions after appropriate annotations have been made in the field log book.

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 12 inches. The size and depth of the sample pit required would depend on the amount of material needed for sample analysis and the interval to be sampled. If a sod mat is present, it shall be separated from the mineral soil surface with the chosen sampling tool. The removed sod mat shall be shaken and scraped over the sample collection bowl to dislodge any mineral soil particles. All dislodged particles shall be placed in the sample. 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.

4. Measure the interval to be sampled (0-12 inches) with a stainless steel tape measure, a ruler or other calibrated marking device and mark the appropriate interval.

5. Scrape the walls of the sample pit within the marked interval with 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, a new cleaned foil pan or gallon Ziploc bag.

Each subsample test hole will be prepared and sampled in the manner discussed above.

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 or gallon Ziploc 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 and labeled. Any remaining sample material will be returned to the sample holes. A sufficient quantity of soil will be collected in each sample container to provide for analysis 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 container. Mix the remaining material in the container with the sampling tool.

3. Transfer the soil sample directly into the appropriate sample container according to Standard Operating Procedure (*Soil and Water Sample Packaging and Shipping*) (SOP-SA-01).

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

5. Decontaminate sampling tools according to procedures outlined in Standard Operating Procedure (*Equipment Decontamination*) (SOP-DE-01).

COMPOSITE SAMPLE AREAS

Composite sample areas within a parcel will be developed prior to sampling. These sampling areas are determined based upon land use. Depending upon the area of each sample area, some composite sample areas will require multiple composite samples (see below). The following land use areas are considered separate composite sample areas.

- Land Use Category #1 – This category consists of playground areas. This will typically be defined as the area around playground equipment such as swings, slides, jungle gyms, and other types of equipment.
- Land Use Category #2 – This category consists of high accessible areas near school buildings such as school courtyards. Also contained within the category will be barren sports areas such as a baseball/softball infield.
- Land Use Category #3 – This category consists of maintained grassy areas such as sodded school grounds and turf covered sports fields.
- Land Use Category #4 – This category consists of low use/low maintenance areas that are rarely accessed by children. Examples include school grounds that are fenced off to restrict access by students.
- Land Use Category #5 – This category consists of vegetable and/or flower gardens.

Land Use Category #1 (Playground Areas)

For Land Use Category #1 sampling components, subsamples will be collected from a minimum of 3 subsample locations or at a rate of 1 subsample per 625 square feet (ft^2) (25 feet by 25 feet) in surface area per sampling component, whichever is greater. Subsamples from these locations will be composited in the field, and a single composite sample per depth interval will be analyzed for arsenic, lead, and mercury. Each subsample shall have similar mass so that each location is equally represented in the total sample mass. The maximum area represented by a single composite sample will be 6,250 ft^2 (meaning a maximum of 10 subsamples will be collected from any single Land Use Category #1 sampling component). See Table 1.

Samples will be thoroughly mixed in a clean 1-gallon plastic Ziploc® bag or stainless steel bowl to ensure representativeness of the aliquot ultimately submitted for analysis. During this homogenization process, particles greater than 0.5 inches in diameter will be discarded. Sample volumes will consist of approximately 500 to 800 grams of material. Samples will be submitted to the laboratory by the samplers under chain of custody procedures.

Land Use Category #2 (Highly Accessible Areas/Barren Sports Fields)

For Land Use Category #2 sampling components, subsamples will be collected from a minimum of 3 subsample locations or at a rate of 1 subsample per 625 ft^2 (25 feet by 25 feet) in surface area per sampling component, whichever is greater. Subsamples from these locations will be composited in the field, and a single composite sample per depth interval will be analyzed for arsenic, lead, and mercury. Each subsample shall have similar mass so that each location is equally represented in the total sample mass. The maximum area represented by a single composite sample will be 9,375 ft^2 (meaning a maximum of 15 subsamples will be collected from any single Land Use Category #2 sampling component). See Table 1.

Samples will be thoroughly mixed in a clean 1-gallon plastic Ziploc® bag or stainless steel bowl to ensure representativeness of the aliquot ultimately submitted for analysis. During this homogenization process, particles greater than 0.5 inches in diameter will be discarded. Sample volumes will consist of approximately 500 to 800 grams of material. Samples will be submitted to the laboratory by the samplers under chain of custody procedures.

Land Use Category #3 (Maintained Grass Areas/Grass Sports Fields)

For Land Use Category #3 sampling components, subsamples will be collected from a minimum of 3 subsample locations or at a rate of 1 subsample per 2,200 ft^2 in surface area per sampling component, whichever is greater. Subsamples from these locations will be composited in the field, and a single composite sample per depth interval will be analyzed for arsenic, lead, and mercury. Each subsample shall have similar mass so that each location is equally represented in the total sample mass. The maximum area represented by a single composite sample will be 10,890 ft^2 (meaning a maximum of 5 subsamples will be collected from any single Land Use Category #3 sampling component). See Table 1.

Samples will be thoroughly mixed in a clean 1-gallon plastic Ziploc® bag or stainless steel bowl to ensure representativeness of the aliquot ultimately submitted for analysis. During this homogenization process, particles greater than 0.5 inches in diameter will be discarded. Sample volumes will consist of approximately 500 to 800 grams of material. Samples will be submitted to the laboratory by the samplers under chain of custody procedures.

Land Use Category #4 (Low Access Areas/Low Maintenance Areas/Open Space)

For Land Use Category #4 sampling components, subsamples will be collected from a minimum of 3 subsample locations or at a rate of 1 subsample per 2,200 ft^2 in surface area per sampling component, whichever is greater. Subsamples from these locations will be composited in the field, and a single composite sample per depth interval will be analyzed for arsenic, lead, and mercury. Each subsample shall have similar mass so that each location is equally represented in the total sample mass. The maximum area represented by a single composite sample will be

21,780 ft^2 (meaning a maximum of 10 subsamples will be collected from any single Land Use Category #4 sampling component). See Table 1.

Samples will be thoroughly mixed in a clean 1-gallon plastic Ziploc® bag or stainless steel bowl to ensure representativeness of the aliquot ultimately submitted for analysis. During this homogenization process, particles greater than 0.5 inches in diameter will be discarded. Sample volumes will consist of approximately 500 to 800 grams of material. Samples will be submitted to the laboratory by the samplers under chain of custody procedures.

Land Use Category #5 (Flower/Vegetable Gardens)

In order to limit disturbance in small components (such as vegetable and flower gardens), only one sample location will be used when the component area is approximately 50 ft^2 or less in area. For Land Use Category #5 sampling components greater than 50 square feet in area, subsamples will be collected from a minimum of two subsample locations or at a rate of 1 subsample per 625 ft^2 in surface area per sampling component, whichever is greater. When applicable, subsamples from these locations will be composited in the field, and a single composite sample per depth interval will be analyzed for arsenic, lead, and mercury. Each subsample shall have similar mass so that each location is equally represented in the total sample mass. The maximum area represented by a single composite sample will be 3,125 ft^2 (meaning a maximum of 5 subsamples will be collected from any single Land Use Category #5 sampling component). See Table 1.

Samples will be thoroughly mixed in a clean 1-gallon plastic Ziploc® bag or stainless steel bowl to ensure representativeness of the aliquot ultimately submitted for analysis. During this homogenization process, particles greater than 0.5 inches in diameter will be discarded. Sample volumes will consist of approximately 500 to 800 grams of material. Samples will be submitted to the laboratory by the samplers under chain of custody procedures.

SOURCE AREA COMPOSITE

A composite sample is collected in potential source areas (waste rock piles, mine dumps, etc.). 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 surrounding parcel through runoff.

In cases where a potential source area is contained within two or more 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 SOP.

OPPORTUNITY COMPOSITE

Subsamples are collected in the areas within a parcel 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 lot soils.

SAMPLE COLLECTION

Samples will be collected based upon land 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 LOCATIONS

Sample locations within sampling components will be determined by sampling personnel based upon site-specific conditions.

SAMPLING DEPTH INTERVALS BY COMPOSITE AREA

This SOP addresses soil sampling to decide whether a remedial action is required in non-residential RMAP parcels.

All subsample locations will be plotted on the map representing each parcel sampled. 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.

SAMPLE DEPTHS

Samples from all non-garden components will be collected from the following depth intervals: 0 to 2 inches bgs, 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.

Most areas are expected to be covered with grass; consequently, surface samples will be collected from immediately beneath the vegetative mat, or in the absence of vegetation, in the 0 to 2 inch bgs and 2 to 6 inch bgs intervals. 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 container 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 or similar surface. If the surface material is coarse-grained material free of intermixed materials, the samples will be collected from the 0 to 2 inch, 2 to 6 inch, and 6 to 12 inch soil layers immediately beneath the coarse-grained material. However, if the graveled driveway or similar surface contains soil/dust material on the surface, the samples will be collected from the surface, in the 0 to 2 inch, 2 to 6 inch, and 6 to 12 inch layers.

Subsurface samples from vegetable and flower gardens will be collected from the following depth intervals: 0 to 2 inches bgs, 2 to 6 inches bgs; 6 to 12 inches bgs; 12 to 18 inches bgs; and

18 to 24 inches bgs.

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 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.

RESIDENTIAL METALS ABATEMENT PROGRAM

STANDARD OPERATING PROCEDURE
RMAP SOP-1B
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 (e.g. in this case, one sampling day). 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 Standard Operating Procedure (*Soil Sample Packaging and Shipping*) (SOP-SA-01).

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

> A field duplicate consists of one well-mixed and homogenized sample that is split in the field into two samples and placed in different sample containers for separate analyses. Each duplicate shall be analyzed for identical chemical parameters.
>
> As with all other samples, samples to be split for duplicate samples will be thoroughly mixed in a clean 1-gallon plastic Ziploc® bag or stainless steel bowl to ensure representativeness of the aliquot ultimately submitted for analysis. During this homogenization process, particles greater than 0.5 inches in diameter will be discarded. Once the homogenization process is complete, the natural sample is split into two samples.

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 logbook.

RESIDENTIAL METALS ABATEMENT PROGRAM

STANDARD OPERATING PROCEDURE
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 0-6 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.

4. Measure the interval to be sampled (0-6 inches) with a stainless steel tape measure, a ruler or other calibrated marking device 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, a new cleaned foil pan or gallon Ziploc bag.

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 Standard Operating Procedure (*Soil and Water Sample Packaging and Shipping*) (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 Standard Operating Procedure (*Equipment Decontamination*) (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 or gallon Ziploc 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 and labeled. Any remaining sample material will be

returned to the sample holes. A sufficient quantity of soil will be collected in each sample container to provide for analysis 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 container. Mix the remaining material in the container with the sampling tool.

3. Transfer the soil sample directly into the appropriate sample container according to Standard Operating Procedure (*Soil and Water Sample Packaging and Shipping*) (SOP-SA-01).

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

5. Decontaminate sampling tools according to procedures outlined in Standard Operating Procedure (*Equipment Decontamination*) (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 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 (0-6 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 4ºC or less.

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

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

RESIDENTIAL METALS ABATEMENT PROGRAM

STANDARD OPERATING PROCEDURE
SOP-DE-01
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 (SSHASP). 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 SSHASP.

<u>INORGANIC CONTAMINANTS - HEAVY METALS:</u>

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:

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.

RESIDENTIAL METALS ABATEMENT PROGRAM

STANDARD OPERATING PROCEDURE
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. The samples will be surrounded with non-contaminating packaging materials to reduce movement.

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.

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

RESIDENTIAL METALS ABATEMENT PROGRAM

STANDARD OPERATING PROCEDURE
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.

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. If volatile compounds are sampled then samples will be transported in insulated coolers with ice ('blue ice' is acceptable) as necessary to maintain temperature at 4^{o} C+/- 2^{o}C until receipt by the analytical laboratory otherwise storage at room temperature is acceptable.

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;
- 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.

RESIDENTIAL METALS ABATEMENT PROGRAM

STANDARD OPERATING PROCEDURE
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 Residential Metals Abatement Program (RMAP) environmental investigations. These procedures apply from the time field work begins until site activities are completed.

RESPONSIBILITIES:

A designated field logbook or electronic device will be used for each field project. If field logbooks are utilized, 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.

The following information will be documented:

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.
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. The following items shall be recorded for each photograph taken:

1. 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. Alternatively, electronic field data collection can be utilized provided the data collected meets the requirements of this SOP and the applicable QAPP.

ATTACHMENT C-2

SOIL LABORATORY SOPs

Attachment C-2
Soil Laboratory SOPs
Index

Laboratory	SOP Number	Revision #	Effective Date	SOP Title	# Pages
Pace	ENV-SOP-GBAY-0164	0	04/12/21	Soil Sieve	13
Pace	ENV-SOP-MIN4-0056	3	02/22/21	Metals Preperation of Solid Samples for Analysis by ICP and ICP-MS by 3050B	11
Pace	ENV-SOP-MIN4-0052	5	07/31/20	Metals Analysis by ICP - Method 6010 and 200.7	22
Pace	ENV-SOP-MIN4-0043	4	02/22/21	Metals Analysis by ICP/MS - Method 6020 and 200.8	24
Pace	ENV-SOP-MIN4-0054	4	07/31/20	Mercury in Liquid and Solid/Semi-Solid Waste by 7470A, 7471, 7471B, and 245.1	20

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ENVIRONMENTAL SCIENCES

Document Information

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

Document Title: Soil Sieve

Department(s): Wet Chemistry

Date Information

Effective Date: 12 Apr 2021

Notes

Document Notes:

All Dates and Times are listed in: Central Time Zone

Signature Manifest

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

All dates and times are in Central Time Zone.

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

QM Approval

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

Management Approval

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

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

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1.0 SCOPE AND APPLICATION

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

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

1.2 Applicable Matrices: Soils and sediments.

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

2.0 SUMMARY OF METHOD

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

3.0 INTERFERENCES

Not applicable to this SOP.

4.0 DEFINITIONS

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

5.0 HEALTH AND SAFETY

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

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

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

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

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solvents (evaporation/concentration processes) must be in a hood or apparatus that prevents employee exposure.

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

6.0 SAMPLE COLLECTION, PRESERVATION, HOLDING TIME, AND STORAGE

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

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

Requirements for container type, preservation, and field quality control (QC) for the common list of test methods offered by Pace are included in the laboratory's quality manual.

General Requirements

Matrix	Routine Container	Minimum Sample Amount[1]	Preservation	Holding Time
Hg Samples	Ziplock Bag	2 cups	Thermal: ≤ 6°C Chemical: None	28 Days
All Other Metals	Ziplock Bag	2 cups	Thermal: ≤ 6°C Chemical: None	6 Months
Organic Parameters	16 oz glass jar	2 cups	Thermal: ≤ 6°C Chemical: None	VOA 14 Days SVOA 7 Days

[1] Minimum amount needed for each discrete analysis.

Thermal preservation is checked and recorded on receipt in the laboratory in accordance with laboratory SOP ENV-SOP-GBAY-0006 *Sample Management* (current revision or replacement). Chemical preservation is checked and recorded at time of receipt or prior to sample preparation.

After receipt, samples are stored at ambient temperature until sample preparation. Prepared samples (extracts, digestates, distillates, other) are stored at ambient temperature until sample analysis.

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

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7.0 EQUIPMENT AND SUPPLIES

7.1 Equipment

Equipment*	Manufacturer / Vendor*	Catalog #*
Sieve Shaker	RO-TAP®	RX-29
Sieve Shaker	Gilson	SS-15
Sieve Shaker	Endecotts	Minor 200
Sieve Shaker	Endecotts	Octagon 200
Sieve	Gilson or equivalent	Stainless steel, #10, #60, or other as needed
Sieve catch pans and lids	Gilson or equivalent	Stainless steel
Bakers' racks	Restaurant Supply	To hold 18" x 26" trays
Drying fan	Various	Local Store
Mortar ceramic/porcelain	Cole-Parmer	60322
Pestle ceramic/porcelain	Cole-Parmer	60323

*Or Equivalent

7.2 Supplies

Supplies	Vendor	Model/Version
Aluminum Foil Cake Pan	Durable Packaging / Webstaurant	612604245
8x8 Ziploc Bags	Fisher Scientific	23700218
12x12 Ziploc Bags	Uline	S-14416
Freezer Paper	Fisher Scientific or equivalent	50-200-5215
Wooden Rolling Pin	Restaurant Supply	Local Store
Rubber Mallet	Various	Local Store
Scissors	Various	Local Store

*Or Equivalent

8.0 REAGENTS AND STANDARDS

Not applicable to this SOP.

9.0 PROCEDURE

9.1 Balance calibration must be verified daily prior to use. Refer to SOP ENV-SOP-GBAY-0115 *Support Equipment* (current revision or replacement).

9.2 For any USDA marked samples, refer to SOP ENV-SOP-GBAY-0121 *Regulated Soil Handling* (current revision or replacement). Containers will be labeled with a pink Regulated Soil sticker.

9.3 Pulling Samples

9.3.1 Batch the samples in the LIMS.

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9.3.2 Pull the samples from either the soil Walk-In Cooler or from the ambient storage area in the Physical Testing Lab and organize them in the order to be processed. Their location will be dependent on the analytical work, if any, that will be done after the sieving.

9.4 Create a new Dry Sieve Worksheet File.

9.4.1 Use the Dry Sieve Template in the Dry Sieve folder, and make sure to "Save As", using the Horizon Batch Number (HBN).

9.4.2 Fill in the drying information for each sample on the Worksheet.

9.5 Air Dry Samples

9.5.1 Wearing gloves, line a tray with freezer paper wax side down. Fold the sides of freezer paper up about 1- 1 1/2" on each side to form a "boat".

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

9.5.3 Entire sample does not need to be dried if excess volume was received. Sample must be homogenized before splitting. Return undried portion to original container.

9.5.4 Weigh and document remaining sample mass. Some projects may require this to be labeled as "Archive".

9.5.5 Spread the soil evenly. Break up all clumps into about 1/2" or less size pieces. This will speed the drying process and ease the disaggregation process prior to sieving. Continue this process for all samples in the set. Change gloves between each sample.

9.5.6 In the drying logbook record the sample numbers, date, time, temperature, and humidity when the samples are placed in the drying cabinet. Place the entire set in a drying cabinet to air dry overnight. Longer drying may be required for wetter samples.

9.6 Soil Disaggregation

9.6.1 After the samples are dried remove them from the drying cabinets. Record the date, time, temperature, and humidity in the drying logbook.

9.6.2 Place a tray on the counter. Pick any rocks, twigs or other foreign matter and set to the side of the freezer paper boat.

9.6.3 Disaggregate the soil. Disaggregation is the process of loosening the clumped soil. It is not meant to crush or reduce the natural particle size of the soil. Place a sheet of paper, wax side up, over the sample. Using a rolling pin, roll over the dried soil for 1-2 minutes. A rubber mallet or pestle may be used to disaggregate soil clumps. Take care that the sample remains on the freezer paper. If sample is hard clay, a porcelain pestle may be used to break up chunks, being careful not to crush rocks.

9.7 Soil Sieve Procedure Using #10 Sieve

9.7.1 Place sieve on catch pan or clean freezer paper, wax side down. Pour sample into #10 sieve and sift. Gently rub the sample remaining on the sieve to break up clumps. When no more sample passes through sieve, dump all remaining sample on top of sieve onto

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a separate sheet of freezer paper. If large clumps are still present, repeat disaggregation and sieve until no clumps remain.

9.7.2 The sample portion remaining in the #10 sieve is then weighed, documented, and bagged with the sample number and a "Coarse Fragments" label on it.

9.7.3 Weigh, document, and place all the sample passing through the #10 sieve into a labeled Ziploc bag with the sample number and a "Fines" label on it. Add any organic matter that had been removed previously. This Organic matter may need to be cut up into smaller pieces using clean scissors.

9.7.4 Change gloves between samples.

9.8 Soil Sieving Procedure using sieves other than #10

9.8.1 Determine the sieve sizes and process to be used to meet project specifications.

9.8.1.1 Check with the project manager or lab manager for project specifications.

9.8.1.2 If multiple sieve portions are to be obtained, stack the set of sieves in the with the largest size openings on top to the smallest on the bottom, with a catch pan at the base.

9.8.1.3 If sieve sizes smaller than a #10 sieve are being used, the #10 sieve can be used to not plug up the smaller sieve. Anything retained by the #10 sieve must be considered part of the biggest sieve portion.

9.8.2 Pour the dried and disaggregated soil onto top sieve.

9.8.3 Record the sample number on the side of the catch pan. An abbreviated number may be used such as 407-1.

9.8.4 All dried contents are poured onto the sieve including the rocks and foreign matter that had been set to the side. The organic foreign matter may need to be cut up into smaller pieces using clean scissors.

9.8.5 Place the set of sieves on a mechanical shaker. Tighten the mechanical shaker adjustments so that the sieves fit tightly and securely in the mechanical shaker. Set the timer for 10 minutes and begin the sieve shaking.

9.8.6 After 10 minutes remove the sieves from the mechanical shaker.

9.8.7 Weigh, document, and place all the sample contents in the catch pan into a labeled Ziploc bag with the sample number and a "Fines" label on it.

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

9.8.9 Certain projects may require that the portion of sample above the sieve be retained. If this is required pour the sample remaining on top of the sieve(s) into a second bag, label with the lab number and mark "Coarse Fragments". Zero the balance with the same bags used, then weigh and document the mass of this portion.

9.8.10 Record the sieve date, analyst, shaker ID, and sieve size used on the Soil Sieve Prep Log. Note if coarse fragments were retained.

9.9 Pulverization - Some projects or methods may require that the sieved sample be further pulverized prior to analysis. The sample may be pulverized with a motor and pestle or other method.

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

9.10.1 Place the sieves in the sink and scrub with a brush or green scrubbie and running hot water to remove any soil particles embedded in the mesh. Rinse well with tap water then rinse with deionized water. Soap is not used as it is very difficult to rinse from the sieves.

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

10.0 DATA ANALYSIS AND CALCULATIONS

Not applicable to this SOP.

11.0 QUALITY CONTROL AND METHOD PERFORMANCE

11.1 Quality Control - Not applicable to this SOP.

11.2 Instrument QC - Not applicable to this SOP.

11.3 Method Performance

11.3.1 Method Validation

11.3.1.1 Detection Limits - Not applicable to this SOP.

12.0 ANALYST QUALIFICATIONS AND TRAINING

Employees that perform any step of this procedure must have a completed Read and Acknowledgment Statement for this version of the SOP in their training record. In addition, prior to unsupervised (independent) work on any client sample, analysts that prepare or analyze samples must have successful initial demonstration of capability (IDOC) and must successfully demonstrate on-going proficiency on an annual basis. Successful means the initial and on-going DOC met criteria, documentation of the DOC is complete, and the DOC record is in the employee's training file. Refer to laboratory SOP ENV-SOP-GBAY-0094 *Training and Employee Orientation* (current revision or replacement) for more information.

13.0 DATA REVIEW AND CORRECTIVE ACTION

Data Review

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

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

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

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

Refer to laboratory SOP ENV-SOP-GBAY-0120 *Data Review and Final Report Processes* (current revision or replacement) for specific instructions and requirements for each step of the data review process.

13.1 Corrective Action

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

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

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

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

There is no QC performed with this analysis.

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14.0 POLLUTION PREVENTION AND WASTE MANAGEMENT

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

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

15.0 MODIFICATIONS

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

16.0 RESPONSIBILITIES

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

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

17.0 ATTACHMENTS

Attachment I: Sieve prep log (Example)

Attachment II: Dry Sieve Flow Chart

18.0 REFERENCES

18.1 Pace Quality Assurance Manual - most current version.

18.2 The NELAC Institute (TNI); Volume 1, "Management and Technical Requirement for Laboratories Performing Environmental Analysis" - most current version.

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19.0 REVISION HISTORY

This Version: ENV-SOP-GBAY-0164-Rev.00

Section	Description of Change
All	First Issue of SOP.

This document supersedes the following document(s):

Document Number	Title	Version

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Attachment I: Sieve Prep Log

Work Order		**Date/Time In**		**Humidity In (%)**		**Temp In (°C)**		**Reviewed by**
Batch		**Date/Time Out**		**Humidity Out (%)**		**Temp Out (°C)**		

Samples		**Sieve Date**	**Shaker ID**	**Analyst**	**Archive Weight (g)**		**Analyst**	**Weight of >60 Mesh (g)**		**Analyst**	**Weight of <60 Mesh (g)**		**Analyst**
					Balance ID:	**40BALW**		**Balance ID:**	**40BALX**		**Balance ID:**	**40BALX**	
	-001		40SKR3										
	-002		40SKR4										
	-003		40SKR5										
	-004		40SKR3										
	-005		40SKR4										
	-006		40SKR5										
	-007		40SKR4										
	-008		40SKR6										
	-009		40SKR3										
	-010		40SKR4										
	-011		40SKR6										
	-012		40SKR7										
	-013		40SKR8										
	-014		40SKR3										
	-015		40SKR4										
	-016		40SKR5										
	-017		40SKR6										
	-018		40SKR7										
	-019		40SKR8										
	-020		40SKR4										

A similar version including the same information may be used.

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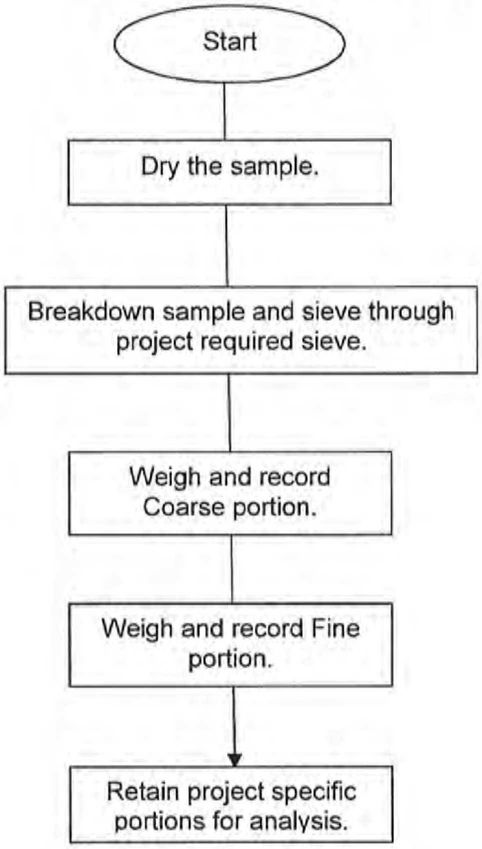
Attachment II: Dry Sieve Flow Chart



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ENVIRONMENTAL SCIENCES

Document Information

Document Number: ENV-SOP-MIN4-0056 **Revision:** 03

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

Department(s): Metals

Date Information

Effective Date: 22 Feb 2021

Notes

Document Notes:

All Dates and Times are listed in: Central Time Zone

Signature Manifest

Document Number: ENV-SOP-MIN4-0056 **Revision:** 03

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

All dates and times are in Central Time Zone.

ENV-SOP-MIN4-0056

QM Approval

Name/Signature	Title	Date	Meaning/Reason
Janielle Ward (007319)	Manager - Quality	22 Feb 2021, 11:06:07 AM	Approved

Management Approval

Name/Signature	Title	Date	Meaning/Reason
Andrew Mickelson (009792)	Manager	18 Feb 2021, 08:49:18 AM	Approved
Krista Carlson (004514)	Project Manager 1	18 Feb 2021, 10:35:56 AM	Approved
Adam Haugerud (005828)	General Manager 2	22 Feb 2021, 09:08:26 AM	Approved

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1.0 SCOPE AND APPLICATION

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

1.1 Target Analyte List and Limits of Quantitation (LOQ)

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

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

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

1.2 Applicable Matrices

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

2.0 SUMMARY OF METHOD

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

3.0 INTERFERENCES

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

4.0 DEFINITIONS

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

5.0 HEALTH AND SAFETY

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

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

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

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

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

6.0 SAMPLE COLLECTION, PRESERVATION, HOLDING TIME, AND STORAGE

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

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

General Requirements

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

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

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

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

7.0 EQUIPMENT AND SUPPLIES

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7.1 Equipment

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

7.2 Supplies

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

8.0 REAGENTS AND STANDARDS

8.1 Reagents

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

8.2 Standards

Standard	Concentration/Description	Requirements/Vendor/Item #
Metals Spike - Stock solution standards for LCS and MS/MSD	The solution identifications are PACE-67Aand Pace-67B. See Appendix A for composition	Purchased from Inorganic Ventures (or equivalent). Store at room temperature. Expires as specified by manufacturer.
Mercury Spike – Stock solution standards for LCS and MS/MSD	10 µg/mL Hg-STK Stock	Purchased from Spex Certiprep. Store at room temperature. Expires as specified by manufacturer.

9.0 PROCEDURE

9.1 Equipment Preparation

9.1.1 Support Equipment

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

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

9.1.2 Equipment

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

9.2 Sample Preparation

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

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

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

9.2.2.2 Spike the LCS, MS/MSD using 0.25 mL of each PACE-67A and PACE-67B. If mercury is requested spike 0.40 mL of Hg-STK Stock.

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

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

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

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

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

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

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

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

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

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

9.3 Documentation

9.3.1 Digestion Records

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

10.0 DATA ANALYSIS AND CALCULATIONS

10.1 Calculations

Refer to associated analytical SOP for equations and common calculations.

11.0 QUALITY CONTROL AND METHOD PERFORMANCE

11.1 Quality Control

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

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

11.2 Method Performance

11.2.1 Method Validation

11.2.1.1 Detection Limits

Detection limits (DL) and limits of quantitation (LOQ) are established at initial method setup and verified on an on-going basis thereafter. Refer to Pace ENV corporate SOP ENV-SOP-CORQ-0011 *Method Validation and Instrument*

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Verification and to the laboratory's SOP ENV-SOP-MIN4-0163 *Determination of LOD and LOQ* (or equivalent replacement) for these procedures.

11.3 Analyst Qualifications and Training

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

12.0 DATA REVIEW AND CORRECTIVE ACTION

12.1 Data Review

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

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

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

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

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

12.2 Corrective Action

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

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

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

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

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

13.0 POLLUTION PREVENTION AND WASTE MANAGEMENT

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

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

14.0 MODIFICATIONS

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

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

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

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

15.0 RESPONSIBILITIES

Pace ENV employees that perform any part this procedure in their work activities must have a signed Read and Acknowledgement Statement in their training file for this version of the SOP. The employee

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is responsible for following the procedures in this SOP and handling temporary departures from this SOP in accordance with Pace's policy for temporary departure.

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

16.0 ATTACHMENTS

Appendix A – Stock Standard Summary

17.0 REFERENCES

Pace Quality Assurance Manual- most current version.

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

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

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

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

18.0 REVISION HISTORY

This Version:

Section	Description of Change
All	Updated SOP references.
6.0	Updated from 45 to 21 days for sample retention.
9.2.3	Updated DI addition from "Add 10 mL DI.." to "Add DI to the 10 mL marking…".
Appendix A	Updated standard composition – to ZPACEMN-105 from PACE-67B and to ZPACEMN-106 from PACE-67A. Updated elements and concentrations accordingly.

This document supersedes the following document(s):

Document Number	Title	Version
ENV-SOP-MIN4-0056	Metals Preparation of Solid Samples for Analysis by ICP and ICPMS by EPA Method 3050B	02

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Appendix A: Metals Standard Reference

Stock standards used for solid sample preparation

ZPACEMN-105		ZPACEMN-106		Hg-STK Stock	
Element	(mg/L)	Element	(µg/L)	Element	(µg/L)
Ca	2000	Si	500	Hg	10000
Fe	2000	Sb	100		
Mg	2000	Mo	100		
K	2000	Sn	100		
Na	2000	Ti	100		
Al	2000	S	2000		
Ba	100	As	100		
Be	100	Pd	20		
Bi	100	Pt	20		
B	100	Se	100		
Cd	100				
Cs	100				
Cr	100				
Co	100				
Cu	100				
Li	100				
P	100				
Mn	100				
Pb	100				
Ni	100				
Ag	50				
Sr	100				
Tl	100				
V	100				
Zn	100				
U	100				
Th	100				

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ENVIRONMENTAL SCIENCES

Document Information

Document Number: ENV-SOP-MIN4-0052 **Revision:** 05

Document Title: Metals Analysis by ICP - Method 6010 and 200.7

Department(s): Metals

Date Information

Effective Date: 31 Jul 2020

Notes

Document Notes:

All Dates and Times are listed in: Central Time Zone

Signature Manifest

Document Number: ENV-SOP-MIN4-0052 **Revision:** 05

Title: Metals Analysis by ICP - Method 6010 and 200.7

All dates and times are in Central Time Zone.

ENV-SOP-MIN4-0052 - ICP

QM Approval

Name/Signature	Title	Date	Meaning/Reason
Janielle Ward (007319)	Manager - Quality	30 Jul 2020, 05:05:44 PM	Approved

Management Approval

Name/Signature	Title	Date	Meaning/Reason
Andrew Mickelson (009792)	Manager	20 Jul 2020, 02:32:20 PM	Approved
Krista Carlson (004514)	Project Coordinator 1	20 Jul 2020, 04:50:45 PM	Approved
Adam Haugerud (005828)	General Manager 2	31 Jul 2020, 11:01:55 AM	Approved

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1.0 SCOPE AND APPLICATION

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

1.1 Target Analyte List and Limits of Quantitation (LOQ)

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

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

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

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

1.2 Applicable Matrices

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

2.0 SUMMARY OF METHOD

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

3.0 INTERFERENCES

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

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

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

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

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

4.0 DEFINITIONS

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

5.0 HEALTH AND SAFETY

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

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

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

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

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

6.0 SAMPLE COLLECTION, PRESERVATION, HOLDING TIME, AND STORAGE

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

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

General Requirements

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

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

[2] *Samples must equilibrate for a minimum of 24 hours if acidification is performed in the lab.*

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

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

After analysis, unless otherwise specified in the analytical services contract, samples are retained for 45 days from date of final report and then disposed of in accordance with Federal, State, and Local regulations.

7.0 EQUIPMENT AND SUPPLIES

7.1 Equipment

Equipment	Description
ICPOES (Inductively Coupled Plasma Optical Emiison Spectrometer)	Agilent 720 or 5110 ICP instrumentation equipped with an CCD Detector, full wavelength region. Each instrument has an associated auto-sampler and recirculating chiller.
Centrifuge	Thermo Sorvall Legend XT
Analytical Balance	Sartoriius or equivalent, capable of weighing to 0.01g
Mechanical pipettors	Eppendorf, Fisher brand or equivalent replacement, various sizes
Glassware	Class A or B volumetric flasks and graduated cylinders of various sizes

7.2 Supplies

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

8.0 REAGENTS AND STANDARDS

8.1 Reagents

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

8.2 Standards

Reagent	Description
Calibration Stock Standards	Custom blend of elements. See Appendix D for the standard information
Initial Calibration Verification (ICV) Stock Standard solutions	Custom blend. Must be separate stock from the calibration standards. Spex Certiprep or equivalent. See Appendix D for the standard information
Cesium Ionization Buffer for use with Agilent 720	50,000 PPM, High Purity Standards P/N 1B-CS-B5 or equivalent.
Wavelength Cal Solution - Agilent	Various analytes, Agilent P/N 6610030100
Internal Standards	Yttrium, Inorganic Ventures or equivalent

9.0 PROCEDURE

9.1 Equipment Preparation

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

- Verify the level of nebulizer waste and rinse waste, if more than half full, empty it into the acid waste stream
- Ar/O pressure - The argon supply pressure should be set at about 80-100psi. If the supply argon pressure falls below about 80psi, a safety interlock automatically shuts off the torch.
- Wash solution level - The wash solution supply is maintained in a 4-liter carboy. Ensure that there is sufficient volume present for the analytical sequence.

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- Peristaltic pump tubing - Change the sample and internal standard tubing, spray chamber drain tubing and the rinse station tubing as needed. Signs of degradation include flattened sections and hazy appearance. Allow at least 30 minute for break-in period
    - Adjust the pump-tubing in such a way to ensure proper flow prior to igniting the plasma. Decrease flow to where flow of bubble actually stops or barely moves. Turn knob 2 full turns.

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

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

9.1.1 Support Equipment

Chiller temperature, pressure and water level - The temperature should be regulated at 17 ± 1ºC. Check the current temperature on the chiller to ensure it is within this range. Check the inlet cooling water pressure that must be between 55 and 60psi. Check to ensure that chiller water level is full. If it is not, fill with Polyclear 30.

9.1.2 Instrument

9.1.2.1 Routine Instrument Operating Conditions

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

9.2 Initial Calibration

9.2.1 Calibration Design

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

9.2.2 Calibration Sequence

Example Analytical Sequence

CAL0
CAL1
ICV
ICB
CRDLA
ICSA
ICAB
Fe 2000 SIC
Ca 2000 SIC/LDR
Al 1000 SIC/LDR
Mg 1000 SIC/LDR
Mn 100 SIC
CCV

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CCB
Ba 20 SIC/LDR
Cr 50 SIC/LDR
Co 20 SIC/LDR
As 10 SIC
V 20 SIC/LDR
Cu 20 SIC/LDR
Ni 50 SIC/LDR
Ti 30 SIC/LDR
Mo 10 SIC/LDR
Zr 20 SIC
CCV
CCB
P 50 SIC
Ce 10 SIC
LDR A
LDR B
LDR C
CCV
CCB
CLIENT SAMPLES
CCV
CCB
CRDLA

9.2.3 ICAL Evaluation

9.2.3.1 Curve Fit

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

9.2.3.2 Relative Standard Error (RSE)

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

9.2.3.3 Initial Calibration Verification

In addition to meeting the linearity requirement, any new calibration curve must be assessed for accuracy in the values generated. To assess the accuracy, a single standard from a secondary source must be analyzed and the results obtained must be compared to the known value of the standard. This step is referred to as Initial Calibration Verification. The ICV, followed by an ICB, is analyzed immediately following an initial calibration curve.

9.2.4 Continuing Calibration Verification

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

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9.3 Sample Preparation

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

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

9.3.3 Centrifuge soil samples to minimize need for filtering.

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

9.3.5 Use Horizon Uploader to copy labels into the sequence.

10.0 DATA ANALYSIS AND CALCULATIONS

10.1 Quantitative Identification

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

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

10.1.2.1 Export data from instrument to CSV file.

10.1.2.2 Open LIMSLINK

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

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

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

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

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

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

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

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

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

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

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

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

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

10.2 Calculations

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

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

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

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

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

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

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

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

11.0 QUALITY CONTROL AND METHOD PERFORMANCE

11.1 Quality Control

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

QC Item	Frequency
Method Blank (MB)	1 per batch of 20 or fewer samples.

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

11.2 Instrument QC

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

QC Item	Frequency
Initial Calibration	Daily
Initial Calibration Verification (ICV)	Immediately after each initial calibration.
Spectral Interference Check Solutions (SIC)	Immediately after each ICV/ICB.
Initial Calibration Blank	Immediately after each ICV.
Continuing Calibration Verification (CCV)	Prior to the analysis of any samples and after every 10 injections thereafter. Samples must be bracketed with a closing CCV standard.
Continuing Calibration Blank	Following every CCV injection
CRDL / LLCCV verification	At the beginning of each run for 6010B/C/D/200.7 and at a minimum of once at the end of each run for 6010C.
ICSA verification	At the beginning of each sample run sequence after the CRDL.
ICSAB verification	This is analyzed following the ICSA when requested. This is required by certain clients. It is not a method requirement and need be analyzed only for clients specifying this in the QAPP.
Internal Standard	An appropriate internal standard is required.

11.3 Method Performance

11.3.1 Method Validation

11.3.1.1 Detection Limits

Detection limits (DL) and limits of quantitation (LOQ) are established at initial method setup and verified on an on-going basis thereafter. Refer to Pace ENV corporate SOP ENV-SOP-CORQ-0011 *Method Validation and Instrument Verification* and to the laboratory's SOP ENV-SOP-NW-0018 *Determination of LOD and LOQ* for these procedures.

11.3.2 Linear Dynamic Range (LDR)

Method 6010D requires that a LDR check sample be analyzed daily. Because of this requirement for 6010D, the LDR is established daily for all methods. For some elements a single element standard is used to establish the LDR while in other cases a mixed standard is used to establish the LDR. If an LDR standard is not analyzed for a particular analyte then the LDR defaults to the highest calibration point in the calibration curve.

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Data is reported up to 90% of the LDR. When evaluating interferences use values up to the full LDR for the interferent. The LDR may be established at higher or lower levels on a daily basis based on expected levels of samples being tested that day. The LDR may vary daily depending on slight changes in instrument performance (things like pump tubing wear, etc.). Refer to Attachment VII for default linear ranges and the typical standards used to establish them

11.4 Analyst Qualifications and Training

Employees that perform any step of this procedure must have a completed Read and Acknowledgment Statement for this version of the SOP in their training record. In addition, prior to unsupervised (independent) work on any client sample, analysts that prepare or analyze samples must have successful initial demonstration of capability (IDOC) and must successfully demonstrate on-going proficiency on an annual basis. Successful means the initial and on-going DOC met criteria, documentation of the DOC is complete, and the DOC record is in the employee's training file. Refer to laboratory SOP ENV-SOP-NW-0025 *Training and Orientation Procedures* for more information.

12.0 DATA REVIEW AND CORRECTIVE ACTION

12.1 Data Review

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

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

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

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

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

12.2 Corrective Action

Corrective action is expected any time QC or sample results are not within acceptance criteria. If corrective action is not taken or was not successful, the decision/outcome must be documented in the analytical record. The primary analyst has primary responsibility for taking corrective action

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when QA/QC criteria are not met. Secondary data reviewers must verify that appropriate action was taken and/or that results reported with QC failure are properly qualified.

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

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

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

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

13.0 POLLUTION PREVENTION AND WASTE MANAGEMENT

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

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15.0 RESPONSIBILITIES

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16.0 ATTACHMENTS

Appendix A – Target Analyte List and Routine LOQ

Appendix B – QC Summary

Appendix C – Working Standard Summary

Appendix D – Stock Standard Summary

Appendix E – Check Standard Summary

17.0 REFERENCES

Pace Quality Assurance Manual- most current version.

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

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

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

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

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

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

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

18.0 REVISION HISTORY

This Version:

Section	Description of Change
17.0	Added years to 6010B & 200.7 references, updated formatting.
Appendix B	Updated MB Acceptance Criteria and Corrective Action for all methods. Updated Post Digestion Spike Acceptance Criteria for 6010B and 6010D.

This document supersedes the following document(s):

Document Number	Title	Version
ENV-SOP-MIN4-0052	Metals Analysis by ICP – Method 6010 and 200.7	04

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Appendix A: Target Analyte List and Routine LOQ

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

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

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

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Appendix B: QC Summary

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

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	10, and at end of analytical window	RL for 200.7, 6010B, 6010C and 6010D. Depending on the data quality objective of individual clients different criteria may apply.	it can be demonstrated that the CCB exceedance has no impact on analytical measurements. For example, the CCB has detections and the analyte is not detected in sample(s).	CCB out of criteria.
Internal Standards	Every field sample, standard and QC sample	70-125% of its true concentration	Troubleshoot instrument performance. Reanalyze samples and dilute if needed.	Qualify outages and explain in case narrative.
Interference check solution (ICSA)	A mixed solution containing concentrations of Al, Ca, and Mg at 500 PPM and Fe at 200 PPM is analyzed at the beginning of each sample run sequence. In some specific client requirements the ICSA must bracket the run or the analytical batch.	Acceptance criteria for the spiked analytes are 80-120%. Unspiked analytes must have an absolute value less than the RL.	Identify and correct source of problem, repeat performance verification(s). Note: The ICSA can be re-processed after appropriate SIC solutions are analyzed and the IECs are recalculated. If ICSA passes, continue.	None. Do not proceed with analysis for elements that cannot be verified.
Interference check solution (ICSAB)	A solution containing concentrations of Al, Ca, and Mg at 500 PPM and Fe at 200 PPM with low to mid-range concentrations of target analytes as outlined in ILM5.3. This is analyzed following the ICSA when requested. This is required by certain clients. It is not a method requirement and need be analyzed only for clients specifying this in the QAPP	The acceptance criteria are 80-120% for all spiked analytes.	Identify and correct source of problem, repeat performance verification(s). Note: The ICSAB can be re-processed after appropriate SIC solutions are analyzed and the IECs are recalculated. If ICSAB passes, continue.	None. Do not proceed with analysis for elements that cannot be verified.
Spectral Interference Check Solutions (SIC)	SIC solutions are single-element solutions used to evaluate and correct IEC factors. Specific elements evaluated are listed in specific instrument methods.	Unspiked analytes must have an absolute value less than the RL.	If SIC fails, re-calculate IEC and re-process data. If a sample level exceeds an SIC level and the interfering element affects target analytes, then: a) run a higher SIC or b) dilute the sample.	None. Do not proceed with analysis for elements that cannot be verified.
Method Blank	One per 20 samples	Method 200.7: The method blank is considered to be acceptable if it does not contain the target analytes that exceed 1/2 LLOQ or project-specific DQOs.	Identify source of problem, re-analyze. If reanalysis of the MB fails, all samples affected by the failing MB elements need to be re-digested and re-analyzed.	Qualify outages and explain in case narrative.

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		Method 6010B, 6010C and 6010D: The method blank is considered to be acceptable if it does not contain the target analytes that exceed the LLOQ or project-specific DQOs. WIDNR and West Virginia require samples to be reported to the MDL. The blanks must be clean to the data quality objectives.	If the method blank exceeds the criteria, but the associated samples are either below the reporting level or other DQOs, or detections in the sample are >10x MB detections then the sample data may be reported. J-flag qualification will be applied for blank detections between the LOQ and LOD when DQOs require evaluation to the MDL.	
LCS	One per 20 samples	80-120% for 6010B,6010C and 6010D 85-115% for 200.7	Identify source of problem, re-analyze. If reanalysis of the LCS fails, all samples affected by the failing LCS elements need to be re-digested and re-analyzed. If LCS recovery is > QC limits and these compounds are non-detect in the associated samples	Qualify analytes with LCS out of criteria.
LCSD	An LCSD must be substituted in the event of insufficient sample volume for a matrix spike duplicate sample.	80-120% for 6010B,6010C and 6010D 85-115% for 200.7 %Diff ≤ 20%	Identify source of problem, re-analyze. If reanalysis of the LCS fails, all samples affected by the failing LCS elements need to be re-digested and re-analyzed. If LCS recovery is > QC limits and these compounds are non-detect in the associated samples	Qualify analytes with LCS out of criteria.
MS/MSD	One per 20 samples for 6020 / 6020A / 6020B One per 10 samples for 200.8	75-125% for 6010B, 6010C, and 6010D 70-130% for 200.7 % RPD: 20%	Perform a SD and PDS on any elements that fail to meet criteria for method 6020(A)(B).	Qualify analytes with MS out of criteria.
Sample Duplicate	Per client request	%Diff ≤ 20%	Qualify outages	Qualify outages.
Serial Dilution	One SD per batch. Method suggestion / Pace Policy, if reporting by 6010B, 6010C, or 6010D.	6010B/C: 1:5 dilution of sample, SD RPD should agree within +/- 10% of the original result when the original sample is greater than 10x the RL. 6010D: 1:5 Dilution of sample or MS, for concentrations 25x > LLOQ in parent sample, resultant RPD should agree within +/- 20%.	Data is qualified.	Qualify outages.
Post Digestion Spike	Method suggestion / Pace policy if reporting by 6010B, 6010C, 6010D and MS/MSD fail outside 75-125%	80-120% for 6010C 75-125% for 6010B and 6010D.	Data is qualified.	Qualify outages.

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Laboratory Filter Blank (FB)	Analyzed only with batches of lab filtered dissolved metals, one per batch of 20 or less.	All elements of interest must be evaluated to a criteria of +/- ½ the RL for method 6010D. All elements of interest must be evaluated to a criteria of +/- the RL for method 60106010B,6010C and 200.7. If the FB does not contain target analytes at a level that interferes with project-specific DQOs, then the FB would be considered acceptable.	Identify source of problem, re-analyze. If reanalysis of the MB fails, all samples affected by the failing MB elements need to be re-digested and re-analyzed. If sample(s) non-detect, report the data. If sample result >10x MB detections, report the data.	Qualify outages and explain in case narrative.
Linear Dynamic Range	If a SIC/LDR standard is not analyzed for any specific element, the highest standard in the calibration becomes the linear range. See Appendix C.	The standard must recover within 10% of the true value, and if successful, establishes the linear range. In each scenario, the data reporting range is established using 90% of the highest calibration level or LDR sample.	The linear range of the instrument must be adjusted until 90% recovery of the reference standard can be achieved.	N/A

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

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Appendix C: Linear Range Reference Table

Wavelength	LDR (PPM)	Standard	Type
Ag 328	2	CAL1	LDR
Al 237	1000	Al 1000 SIC/LDR	SIC/LDR
As 188	10	As 10 SIC	SIC
As 188	20	LDR B	LDR
B 249	20	LDR A	LDR
Ba 455***/Ba 585**	20	Ba 20 SIC/LDR	SIC/LDR
Ba 585*	50 0	Ba 50 SIC	SIC/
Be 234	4	CAL1	LDR
Ca 370	2000	Ca 2000 SIC/LDR	SIC/LDR
Cd 214	20	LDR B	LDR
Co 228	50	Co 50 SIC/LDR	SIC/LDR
Cr 267	20	Cr 20 SIC/LDR	SIC/LDR
Cr 267	50	Cr SIC/LDR	50
Cu 327	20	Cu 20 SIC/LDR	SIC/LDR
Cu 327	50	Cu 50 SIC/LDR	SIC
Fe 261	200	LDR C	LDR
Fe 273*	2000	Fe 2000 SIC	SIC
K 766***	200	LDR C	LDR
K 766**	20	CAL1	LDR
Li 670	4	CAL1	LDR
Mg 383	1000	Mg 1000 SIC/LDR	SIC/LDR
Mn 257	20	LDR B	LDR
Mn 293*	100	Mn 100 SIC	SIC
Mo 204	10	Mo 10 SIC/LDR	SIC/LDR
Na 589***	200	LDR C	LDR
Na 589**	20	CAL1	LDR
Ni 231	50	Ni 50 SICLDR	SIC/LDR
P 213	20	LDR B	LDR
Pb 220	100	LDR A	LDR
S 181	200	LDR C	LDR
Sb 206	20	LDR A	LDR
Se 196	20	LDR B	LDR
Si 251	20	CAL1	LDR
Sn 189	20	LDR A	LDR
Sr 421	4	CAL1	LDR
Ti 334	20	LDRA	LDR
Ti 334	30	Ti 30 SIC	SIC
Tl 190	20	LDR B	LDR
U	4	CAL1	LDR
V 292	20	V 20 SIC/LDR	SIC/LDR
Zn 206	50	LDR A	LDR

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

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Appendix D: Standard Reference Tables

	ICP Working Calibration Standard				**ICP Calibration Verification Standard**			
Element	Stock Conc. (mg/L)	Aliquot (mL)	Final Volume (mL)	Cal STD Final Conc. (mg/L)	Stock Conc. (mg/L)	Aliquot in (mL)	Final Volume (mL)	Final Conc. (mg/L)
Ag	100	1.0	50	2	50	1.0	50	1
Al	2,000	0.5	50	20	1000	0.5	50	10
As	200	1.0	50	4	100	1.0	50	2
Ba	200	1.0	50	4	100	1.0	50	2
Be	200	1.0	50	4	100	1.0	50	2
Ca	2000	0.5	50	20	1000	0.5	50	10
Cd	200	1.0	50	4	100	1.0	50	2
Co	200	1.0	50	4	100	1.0	50	2
Cr	200	1.0	50	4	100	1.0	50	2
Cu	200	1.0	50	4	100	1.0	50	2
Fe	2000	0.5	50	20	1000	0.5	50	10
K	2000	0.5	50	20	1000	0.5	50	10
Mg	2000	0.5	50	20	1000	0.5	50	10
Mn	200	1.0	50	4	100	1.0	50	2
Na	2000	0.5	50	20	1000	0.5	50	10
Ni	200	1.0	50	4	100	1.0	50	2
Pb	200	1.0	50	4	100	1.0	50	2
S	10000	0.1	50	20	10000	0.05	50	10
Sb	200	1.0	50	4	100	1.0	50	2
Se	200	1.0	50	4	100	1.0	50	2
Tl	200	1.0	50	4	100	1.0	50	2
V	200	1.0	50	4	100	1.0	50	2
Zn	200	1.0	50	4	100	1.0	50	2
Mo	200	1.0	50	4	100	1.0	50	2
B	200	1.0	50	4	100	1.0	50	2
Sn	200	1.0	50	4	100	1.0	50	2
Ti	200	1.0	50	4	100	1.0	50	2
Si	1000	1	50	20	500	1	50	10
Li	200	1	50	4	100	1	50	2
P	200	1	50	4	100	1	50	2
Sr	200	1	50	4	100	1	50	2
U	1000	0.2	50	4	1000	0.1	50	2

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Appendix E: Interference Check Standard Reference Tables

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

ICSAB				
Element	Stock Conc. (mg/L)	Aliquot in (mL)	Final Volume (mL)	Final Conc. (ug/L)
Ag	20	1.0	100	200
Al	5000	5.0	100	500000
As	10	1.0	100	100
Ba	50	1.0	100	500
Be	50	1.0	100	500
Ca	5000	5.0	100	500000
Cd	100	1.0	100	1000
Co	50	1.0	100	500
Cr	50	1.0	100	500
Cu	50	1.0	100	500
Fe	2000	5.0	100	200000
Mg	5000	5.0	100	500000
Mn	50	1.0	100	500
Ni	100	1.0	100	1000
Pb	5	1.0	100	50
Sb	60	1.0	100	600
Se	5	1.0	100	50
Tl	10	1.0	100	100
V	50	1.0	100	500
Zn	100	1.0	100	1000

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Document Information

Document Number: ENV-SOP-MIN4-0043 **Revision:** 04

Document Title: Metals Analysis by ICP/MS - Method 6020 and 200.8

Department(s): Metals

Date Information

Effective Date: 22 Feb 2021

Notes

Document Notes:

All Dates and Times are listed in: Central Time Zone

Signature Manifest

Document Number: ENV-SOP-MIN4-0043 **Revision:** 04

Title: Metals Analysis by ICP/MS - Method 6020 and 200.8

All dates and times are in Central Time Zone.

ENV-SOP-MIN4-0043

QM Approval

Name/Signature	Title	Date	Meaning/Reason
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Management Approval

Name/Signature	Title	Date	Meaning/Reason
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Andrew Mickelson (009792)	Manager	18 Feb 2021, 08:49:25 AM	Approved
Krista Carlson (004514)	Project Manager 1	18 Feb 2021, 10:54:23 AM	Approved

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1.0 SCOPE AND APPLICATION

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

1.1 Target Analyte List and Limits of Quantitation (LOQ)

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

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

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

1.2 Applicable Matrices

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

Dissolved elements are determined after suitable filtration and acid preservation. In order to reduce potential interferences, dissolved solids should not exceed 0.2 % (w/v).

For the determination of total recoverable analytes in aqueous samples containing particulate and suspended solids a digestion step is required prior to analysis.

2.0 SUMMARY OF METHOD

Prior to analysis, samples must be solubilized or digested using appropriate sample preparation methods. For the total recoverable determination of analytes in drinking water by 200.8 where sample turbidity is < 1 NTU, the sample is made ready for analysis by the appropriate addition of nitric acid, mixed, and allowed to equilibrate for the required time prior to analysis.

Sample solutions are introduced by pneumatic nebulization into a plasma, in which desolvation, atomization and ionization occurs. Ions are extracted from the plasma through a differentially pumped vacuum interface and sorted on the basis of their mass-to-charge ratio. The ions transmitted through the quadrupole are detected by an electron multiplier. Ion intensities at each mass are recorded and compared to those obtained from external calibration standards to generate concentration values for the samples. Results are corrected for instrument drift and matrix effects using internal standards.

3.0 INTERFERENCES

Isobaric Elemental Interferences – Isobaric elemental interferences result when isotopes of different elements have the same nominal mass-to-charge ratio and cannot be resolved with the instruments spectrometer. One way to solve this problem is to measure a different isotope for which there is no

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interference. Alternatively, one can monitor another isotope of the element and subtract an appropriate amount from the element being analyzed, using known isotope ratio information. Corrections for most of the common elemental interferences are programmed into the software.

Isobaric Polyatomic Interferences – Isobaric polyatomic interferences result when ions containing more than one atom have the same nominal mass-to-charge ratio as an analyte of interest and cannot be resolved by the instrument's spectrometer. An example includes ClO+ (mass 51), which interferes with V, and must be corrected by measuring ClO+ at mass 53. When possible an interference free isotope should be chosen for measurement.

Physical interferences are associated with the sample nebulization and transport processes as well as with ion-transmission efficiencies. Nebulization and transport processes can be affected if a matrix component causes a change in surface tension or viscosity. Changes in matrix composition can cause significant signal suppression or enhancement. Dissolved solids can deposit on the nebulizer tip of a pneumatic nebulizer and on the interface skimmers (reducing the orifice size and the instrument performance). Total solid levels below 0.2% (2,000 mg/L) have been currently recommended to minimize solid deposition. An internal standard can be used to correct for physical interferences, if it is carefully matched to the analyte so that the two elements are similarly affected by matrix changes.

Memory interferences can occur when there are large concentration differences between samples or standards, which are analyzed sequentially. Sample deposition on the sampler and skimmer cones, spray chamber design, and the type of nebulizer affects the extent of the memory interferences, which are observed. The rinse period between samples must be long enough to eliminate significant memory interference.

4.0 DEFINITIONS

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

5.0 HEALTH AND SAFETY

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

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

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

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

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solvents (evaporation/concentration processes) must be in a hood or apparatus that prevents employee exposure.

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

6.0 SAMPLE COLLECTION, PRESERVATION, HOLDING TIME, AND STORAGE

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

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

General Requirements

Matrix	Routine Container	Minimum Sample Amount[1]	Preservation	Holding Time
Aqueous	250 mL Plastic	25 mL	Acidified[2] with nitric acid to pH<2, stored ambient	Must be analyzed within 180 days of collection. If mercury is requested, analysis must occur within 28 days of sample collection.
Solid	8 oz glass jar	1 gram	<6°C, but above freezing	

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

[2] *Samples must equilibrate for a minimum of 24 hours following acidification. Lead and Copper Rule Monitoring and Reporting Guidance for Public Water Systems, EPA 816-R-10-004, March 2010, Exhibit II-9, Samples must stand in the original container used for sampling for at least 28 hours after acidification.*

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

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

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

7.0 EQUIPMENT AND SUPPLIES

7.1 Equipment

Equipment	Description
ICPMS (Inductively Coupled Plasma Mass Spectrometer)	Agilent 7700, 7800 7900 ICPMS instrumentation equipped with interference reduction technology. Each instrument has an associated auto-sampler, rough pump and recirculating chiller.

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Centrifuge	Thermo Sorvall Legend XT
Analytical Balance	Sartoriius or equivalent, capable of weighing to 0.01g
Mechanical pipettors	Eppendorf, Fisher brand or equivalent replacement, various sizes
Glassware	Class A volumetric flasks and graduated cylinders of various sizes

7.2 Supplies

Supply	Description
Argon gas	Praxair or equivalent, High purity grade, 99.99%
Collision Gas	Praxair or equivalent, Ultra high purity He, Ultra high purity H_2,
Analytical Balance	Sartoriius or equivalent, capable of weighing to 0.01g
Auto-sampler tubes	Moldpro or equivalent, 15 mL metals free auto-sampler tubes
Digestion cups	Moldpro or equivalent, 50 mL disposable digestion cups
Data-Uploading Software	Pace internal software used to transfer data from the instrument to the LIMS

8.0 REAGENTS AND STANDARDS

8.1 Reagents

Reagent	Description
Reagent water	ASTM Type II
Nitric Acid (HNO_3)	Fisher Scientific, A-509-P212 or equivalent replacement
Hydrochloric acid (HCl)	Fisher Scientific, A-508-P212 or equivalent replacement
2% (v/v) Nitric Acid/1% (v/v) Hydrochloric Acid Solution	Used for instrument blanks, standards and dilutions. Prepared in 1 L increments utilizing a volumetric flask and transferring into a C&G narrow mouth storage bottle. This is measured by mixing 20 mL of HNO_3 trace metals grade acid and 10 mL of HCl trace metals grade acid and DI H2O, and bringing to volume of 1 L.
Rinse Blank	2-5% (v/v) Nitric Acid solution for rinsing between runs. Combine76 mL of HNO_3 trace metals grade acid and 38 mL of HCl trace metals grade and DI H2O, and bringing to volume of 1 G.

8.2 Standards

Reagent	Description
Calibration Stock Standards	Custom blend of elements. See Appendix D for the standard information
Agilent Tune Solution	Purchased multi-element standard from a qualified vendor, 10ug/mL.
EPA Tune solution	Purchased multi-element standard from a qualified vendor, 10ug/mL.
Internal Standard Stock Solution	Various suppliers; single element standards to be mixed prior to use with concentrations of 1,000 and 10,000 ug/mL
Working Standards	See Appendix C

9.0 PROCEDURE

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9.1 Equipment Preparation

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

9.1.1 Support Equipment

- Vacuum pump oil - Examine the sight glasses of the vacuum pump. Oil should be no darker than a light brown color. If it is, change the oil in the pump according to the directions in the manufacturer's guide.
- Chiller temperature, pressure and water level - The temperature should be regulated at 17 ± 1ºC. Check the current temperature on the chiller to ensure it is within this range. Check the inlet cooling water pressure that must be between 55 and 60psi. Check to ensure that chiller water level is full. If it is not, fill with Polyclear 30.
- Verify the level of nebulizer waste and rinse waste, if more than half full, empty it into the acid waste stream.
- Ar/O pressure - The argon supply pressure should be set at about 80psi. If the supply argon pressure falls below about 45psi, a safety interlock automatically shuts off the torch.
- Helium / Hydrogen pressure - The helium and hydrogen supply pressure should be set at about 15 and 9 psi respectively.
- Wash solution level - The wash solution supply is maintained in a 4-liter carboy. Ensure that there is sufficient volume present for the analytical sequence.
- Peristaltic pump tubing - Change the sample and internal standard tubing, spray chamber drain tubing and the rinse station tubing as needed. Signs of degradation include flattened sections and hazy appearance. Allow at least 30 minutes for break-in period.
- Interface cones - Remove and inspect the outside of the sampling and skimmer cones around the orifice. Install a new set of cones if needed or clean the existing cones using the following procedure: Carefully polish each cone with silver polish and cotton swabs dampened with deionized water. Rinse cones with deionized water and blow-dry with house air supply, being careful not to damage the cones. After the cones are fully dry, replace them in the instrument. Allow for conditioning of the cones with a solution containing sufficient concentrations of major cations. The orifice should be circular and about 1mm in diameter. Examine the orifice periodically with a magnifier to determine if there are irregularities that may impair instrument performance. DO NOT use a cone with a significantly degraded tip.

9.1.2 Instrument

Lighting Torch and Warm-Up: After all pre-start checks pass inspection, perform the following steps:

- Torch Ignition - Click on the Plasma icon to open the Instrument window, and then click on the plasma on button to light the plasma. This takes a little over a minute to complete. (See instrument software guide.)
- Warm-up- Instrument is allowed to warm-up 30 minutes. Instrument has a timer to let you know when it is ready to move on to the next step.
- Check peristaltic pump flow by monitoring bubble movement in the pump tubing. Adjust tension as needed to achieve a smooth flow.

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- Start-up Configuration - Once the analysis tubing is placed in the Agilent tune solution and stable signal is achieved, the start-up configuration can be initiated. See section 9.1.2.1 for Agilent tune performance monitoring and criteria.
- Create New Experiment File – Open template from the drive. Apply the proper run name for the day (MMDDYYICPMS#). Introduce EPA tune solution and allow signal to stabilize. Initiate performance verification for each mode of analysis. Save each performance report to the network drive. See section 9.1.2.1 for EPA tune acceptance criteria.

9.1.2.1 Routine Instrument Operating Conditions

The instrument is configured to go through the manufacturer recommended startup tune procedure which includes; Torch Alignment, Axis/Resolution, EM settings, Plasma Correction, Standard Lenses tune, and standard mode performance verification. The measured ratios of oxides 156/140 and doubly charged 70/140 should be <3%. The measured masses of ^{7}Li, ^{89}Y, ^{205}Tl are monitored for initial resolution/axis tuning. EPA Performance verification is later performed for each cell condition used for sample analysis.

EPA Tune Verification - The EPA tuning standard must be analyzed in each mode of analysis to verify resolution and mass calibration are within the required specifications. The tuning standard is analyzed in each mode of analysis at least five times and the relative standard deviation (RSD) must be <5% for all analytes contained in the tuning standard. Conduct mass calibration and resolution checks in the mass regions of interest. If the mass calibration differs more than 0.1 amu from the true value, then the mass calibration must be adjusted to the correct value. The resolution must also be verified to be <0.9 amu full width at 5% peak height.

Pace Minneapolis maintains approval for the analysis of up to 35 elements by the EPA Methods 200.8, 6020, 6020A, 6020B for water and soil matrices. All target analytes are analyzed either in a Helium mode (Collision Cell), hydrogen (Collision Cell), or No gas mode on the Agilent instruments depending on the sample matrix type. The use of interference reduction technologies (Collision Cell) is not allowed for drinking water analysis. Separate calibrations are performed for samples reporting by regulation of the SDWA.

9.2 Initial Calibration

9.2.1 Calibration Design

The calibration curve must consist of a minimum of a calibration blank and five non-zero standards for each mode of analysis. Use the average of at least three integrations for both calibration and sample analyses. Using the instrumentation software, prepare a standard curve for each element by plotting absorbance versus concentration. The working range varies with each analyte, see appendix C for summary. The calibration is a linear regression using equation; $y = mx + b$ The analyst may employ a regression equation that does not pass through the origin, however forcing through zero is not allowed. Additional calibration specifications may be referenced in ENV-POL-CORQ-0005 *Acceptable Calibration Practices for Instrument Testing*, or equivalent replacement.

9.2.2 Calibration Sequence

Calibration Blank (CAL0)

CAL1
CAL2
CAL3
CAL4
CAL5
CAL6 (optional)
CAL7 (optional)
ICV
ICB
CRDL
ICSA
ICSAB
CCV
CCB
Client samples
CCV
CCB
CRDL (Optional)

9.2.3 ICAL Evaluation

9.2.3.1 Curve Fit

With a multi-point calibration, the regression calculation will generate a correlation coefficient (r) that is the measure of the “goodness of fit” of the regression line to the data. In order to be used for quantitative purposes, the correlation coefficient must be > 0.998.

9.2.3.2 Relative Standard Error (RSE)

%RE is measured at the lowest calibration level and at a point near the mid-level of the calibration (the continuing calibration verification level is recommended). In order for a standard curve to be acceptable, the correlation coefficient/coefficient of determination criterion specified in the method must be met **and** both the low-level and mid-level %RE measures must meet the acceptance criteria. The low-level %RE acceptance criteria is 60%-140% and the mid-level is 90-110%.

9.2.3.3 Initial Calibration Verification

In addition to meeting the linearity requirement, any new calibration curve must be assessed for accuracy in the values generated. To assess the accuracy, a single standard from a secondary source must be analyzed and the results obtained must be compared to the known value of the standard. This step is referred to as Initial Calibration Verification. The ICV is analyzed immediately following an initial calibration curve.

9.2.4 Continuing Calibration Verification

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

9.3 Digestate Preparation

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9.3.1 Homogenization and Subsampling

All solid matrices are subject to centrifuge at a rate of 1000 rpm for 15 minutes or allowed to settle overnight prior to analysis. Once samples have been centrifuged or allowed to settle, an initial dilution of 20 fold is performed on each sample. This is completed by taking 4.75mL of 2% HNO3 / 1% HCL diluent and mixing with a 0.25mL aliquot of sample by means of vortex.

Aqueous samples are inverted multiple times and poured without initial dilution unless historical data demonstrates otherwise.

9.4 Analysis

The instrument performs sample analysis by executing 100 mass sweeps per replicate. Three replicates are utilized for an average result which must fall within a 20% RSD for the replicate values. If any sample or QC is found to have a concentration of >5x the RL and >20% RSD it must be evaluated for interference. If a matrix interferent is determined to be the cause, dilute the sample by 5x and re-analyze. Perform further dilutions if necessary.

The instrument(s) have been setup and configured in conjunction with manufacturer specifications. Masses were carefully selected to avoid and/or minimize interferences. Internal standard selection was based on performance for the appropriate mass range. Internal standard association must remain within 50 amu of targeted analyte.

The total recoverable sample digestion procedure is suitable for the determination of silver in aqueous samples containing concentrations up to 0.1 mg/L. For the analysis of wastewater samples containing higher concentrations of silver, succeeding smaller volumes of well mixed sample aliquots must be prepared until the analysis solution contains < 0.1 mg/L silver.

10.0 DATA ANALYSIS AND CALCULATIONS

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

10.1 Hardness as CaCO3 in mg/L = 2.497 * [Ca in mg/L] + 4.118 * [Mg in mg/L]

10.2 Concentration of lead = summation of signals at 206, 207, and 208 m/z.

10.3 Silica (SiO2) (µg/L) = Silicon (Si) (µg/L) * DF * 60.09 amu (SiO2 molecular weight) / 28.09 amu (Si atomic weight)

Where: DF is the sample Dilution Factor

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

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

Where, c = concentration on instrument, µg/L

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v_f = final volume, L
wt_i = initial weight, g

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

10.5 Calculate the Relative Percent Difference (RPD) between the matrix spike and matrix spike duplicate using Equation 1:

Equation 1

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

Where, S = Sample result, mg/L or mg/kg

D = Duplicate sample result, mg/L or mg/kg

11.0 QUALITY CONTROL AND METHOD PERFORMANCE

11.1 Quality Control

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

QC Item	Frequency
Method Blank (MB)	1 per batch of 20 or fewer samples.
Laboratory Control Sample (LCS)	1 per batch of 20 or fewer samples.
Laboratory Control Sample Duplicate (LCSD)	As needed
Matrix Spike (MS)	1 per batch of 20 or fewer samples for 6020 (A)(B). 1 per batch of 10 or fewer samples for 200.8
Matrix Spike Duplicate (MSD)	1 per batch of 20 or fewer samples.
Sample Duplicate	Performed at client request.
Serial Dilution	1 per batch of 20 or fewer samples.
Post Digestion Spike	1 per batch of 20 or fewer samples for method 6020(A)(B).
Internal Standard	An appropriate internal standard is required for each analyte and sample determined by ICP-MS.

Internal Standard	Associated element
Scandium 45	Li, Be, B, Na, Mg, Al, Si, K, Ca, Ti, V, Cr, Mn, Fe, Se
Germanium 72	Co, Ni, Cu, Zn, As, Sr
Indium 115	Mo, Pd, Ag, Cd, Sn, Sb
Terbium 159	Ba, Pt, Hg, Tl, Pb, Bi
Iridium 193	U Th

11.2 Instrument QC

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

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QC Item	Frequency
Tune	Daily prior to any calibration
Initial Calibration	Daily
Initial Calibration Verification	Immediately after each initial calibration
Initial Calibration Blank	Immediately after each initial calibration
Continuing Calibration Verification	Prior to the analysis of any samples and after every 10 injections thereafter. Samples must be bracketed with a closing CCV standard.
Continuing Calibration Blank	Following every CCV injection
CRDL / LLCCV verification	At the beginning of each run for 6020/6020B/200.8 and must be analyzed at the beginning of each run, and once at the end of each analytical batch for 6020A.
ICSA verification	At the beginning of each sample run sequence after the CRDL. 6020A and 6020B requires the ICSA/AB be analyzed every 12 hours thereafter.
ICSAB verification	At the beginning of each sample run sequence after the ICSA. 6020A and 6020B requires the ICSA/AB be analyzed every 12 hours thereafter.

11.3 Method Performance

11.3.1 Method Validation

11.3.1.1 Detection Limits

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

11.4 Analyst Qualifications and Training

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

12.0 DATA REVIEW AND CORRECTIVE ACTION

12.1 Data Review

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

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

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

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

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

12.2 Corrective Action

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

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

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

Sample results at concentrations above the upper limit of quantitation must be diluted and reanalyzed. The result in the diluted samples should be near the midpoint of the calibration range. If dilution is not performed, any result reported above the upper range is considered a qualitative measurement and must be qualified as an estimated value.

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

13.0 POLLUTION PREVENTION AND WASTE MANAGEMENT

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

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

14.0 MODIFICATIONS

A modification is a change to a reference test method made by the laboratory. For example, changes in stoichiometry, technology, quantitation ions, reagent or solvent volumes, reducing digestion or

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extraction times, instrument runtimes, etc. are all examples of modifications. Refer to Pace ENV corporate SOP ENV-SOP-CORQ-0011 *Method Validation and Instrument Verification* for the conditions under which the procedures in test method SOPs may be modified and for the procedure and document requirements.

14.1 Tuning criteria observed is more stringent than required by the SW846 methods so that the same criteria can be used for both methods 6020 and 200.8.

14.2 The following elements are not listed in the method 6020A recommended analyte list; bismuth, boron, lithium, molybdenum, palladium, platinum, silica, silicon, strontium, tin, titanium, thorium, and uranium. The accuracy and precision for the analysis of these analytes have been demonstrated in the matrices of interest, at the concentration of interest, and in the same manner as the elements recommended in the method.

14.3 The following elements are not listed in the method 200.8 recommended analyte list: bismuth, boron, calcium, iron, lithium, magnesium, palladium, platinum, potassium, silica, silicon, sodium, strontium, tin, and titanium. The accuracy and precision for the analysis of these analytes have been demonstrated in the matrices of interest, at the concentration of interest, and in the same manner as the elements recommended in the method.

14.4 The following elements are not listed in the method 6020B recommended analyte list: bismuth, boron, lithium, molybdenum, palladium, platinum, silica, silicon, strontium, tin, titanium and uranium. The accuracy and precision for the analysis of these analytes have been demonstrated in the matrices of interest, at the concentration of interest, and in the same manner as the elements recommended in the method.

15.0 RESPONSIBILITIES

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

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

16.0 ATTACHMENTS

Appendix A – Target Analyte List and Routine LOQ

Appendix B – QC Summary

Appendix C – Working Standard Summary

Appendix D – Stock Standard Summary

17.0 REFERENCES

Pace Quality Assurance Manual- most current version.

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

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

U.S. Environmental Protection Agency. Method 200.8, Determination of Trace Elements in Waters and Wastes by Inductively Coupled Plasma – Mass Spectrometer, Revision 5.4, EMMC Version, May 1994.

U.S. Environmental Protection Agency. SW846 Method 6020, Inductively Coupled Plasma – Mass Spectrometry, Revision 0, 9/94.

U.S. Environmental Protection Agency. SW846 Method 6020A, Inductively Coupled Plasma – Mass Spectrometry, Revision 1, 02/2007.

U.S. Environmental Protection Agency. SW846 Method 6020B, Inductively Coupled Plasma – Mass Spectrometry, Revision 2, 7/2014.

Test Methods for Evaluating Solid Waste Physical/Chemical Methods, SW-846, Third Edition. Method 3020A.

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

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

18.0 REVISION HISTORY

This Version:

Section	Description of Change
6.0	Updated sample retention from 45 to 21 days.
8.2	Internal Standard Stock Solution – added “1,000 and”
9.2.1	Updated 3 to 5 non-zero standards. Added “The working range…C for summary.”
9.2.2	Added “(optional)” to CAL6. Added “CAL7 (optional)”.
10.0	Added sections 10.4 and 10.5.
11.1	Updated Thoridium 232 to Iridium 193.
14.0	14.2 & 14.4: removed “-238” from uranium. 14.2: added thorium.
17.0	Removed references for Fisions and Region 9 Laboratory SOP.
Appendix A	Added Thorium. Updated Silica and Silicon entries. Removed Mercury NPW and potable water entries.
Appendix B	Updated ICAL Acceptance Criteria. Updated methods referenced in MB Acceptance Criteria. Added LDR acronym to QC Item.
Appendix C & D	Re-formatted tables.

This document supersedes the following document(s):

Document Number	Title	Version
ENV-SOP-MIN4-0043	Metals Analysis by ICP/MS – Method 6020 and 200.8	03

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Appendix A: Target Analyte List and Routine LOQ[1]

Analyte	Non-Potable Water (ug/L)	Potable Water (ug/L)	Soil (mg/kg)
Aluminum	20.00	20.0	20.00
Antimony	0.50	0.50	0.50
Arsenic	0.50	0.50	0.50
Barium	0.30	0.30	0.30
Beryllium	0.20	0.20	0.20
Bismuth	0.50	-	0.50
Boron	10.00	-	10.00
Cadmium	0.08	0.08	0.08
Calcium	40.00	-	40.00
Chromium	0.50	0.50	0.50
Cobalt	0.50	-	0.50
Copper	1.00	1.00	1.00
Iron	50.00	-	50.00
Lead	0.10	0.10	0.20
Lithium	0.50	-	0.50
Magnesium	10.00	-	10.00
Manganese	0.50	0.50	0.50
Mercury	-	-	0.20
Molybdenum	0.50	-	0.50
Nickel	0.50	0.50	0.50
Palladium	0.50	-	-
Platinum	0.50	-	-
Potassium	100.00	-	100.00
Selenium	0.50	0.50	0.50
Silica	214.00	-	214.0
Silicon	100.00	-	100.00
Silver	0.50	0.50	0.50
Sodium	50.00	-	50.00
Strontium	0.50	-	0.50
Thallium	0.10	0.10	0.10
Thorium	0.50	-	0.50
Tin	0.50	-	2.000
Titanium	1.00	-	1.00
Vanadium	1.00	1.00	1.00
Uranium-238	0.50	0.50	0.50
Zinc	5.00	5.00	5.00

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

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Appendix B: QC Summary

QC Item	Frequency	Acceptance Criteria	Corrective Action	Qualification
Tune	Daily prior to any calibration	Adjust spectrometer resolution to produce a peak width of approximately 0.75 amu at 5% peak height. This must be completed using 5 replicates with a resulting RSD of <5%.	Adjust mass calibration if it has shifted by more than 0.1 amu from unit mass. Identify and correct source of problem, repeat performance verification(s).	None. Do not proceed with analysis.
ICAL	Daily	r ≥ 0.998 a Midlevel (recommended near ICV/CCV concentrations) %RE 90-110% Low-Level (Cal1) %RE 60-140%	Identify and correct source of problem, repeat.	None. Do not proceed with analysis.
ICV	After Each ICAL	All analytes must be within ± 10% of the true value. (%R)	Identify source of problem, re-analyze. If repeat failure, repeat ICAL. Analysis may proceed if it can be demonstrated that the ICV exceedance has no impact on analytical measurements. For example, the ICV %R is high, CCV is within criteria, and the analyte is not detected in sample(s).	Qualify analytes with ICV out of criteria.
ICB	Immediately after the initial calibration verification	All elements of interest must be evaluated to a criterion of +/- ½ of the RL for method 6020 (A)(B) and samples originating from NC. All elements of interest must be evaluated to +/- the RL for method 200.8, and 6020. WIDNR and West Virginia require samples to be reported to the MDL. The blanks must be clean to the data quality objectives.	Identify source of problem, re-analyze. Analysis may proceed if it can be demonstrated that the ICB exceedance has no impact on analytical measurements. For example, the ICB has detections and the analyte is not detected in sample(s).	Qualify analytes with ICB out of criteria.
CRDL / LLCCV	At the beginning of each run for 6020/6020B/200.8 and must be analyzed at the beginning of each run, and once at the end of each analytical batch for 6020A.	For 6020/200.8: The acceptance criteria are ± 40% (or specified by the client). For 6020A: The acceptance criteria are ± 30% (or specified by the client). 6020B: The acceptance criteria is ± 20% (or specified by the client).	Identify source of problem, re-analyze. Analysis may proceed if it can be demonstrated that the CRDL exceedance has no impact on analytical measurements. For example, the CRDL %R is high and the analyte is not detected in sample(s). For example, the CRDL %R is high and the analyte detections exceed the continuing calibrations verification level (midpoint of the curve).	Qualify outages and explain in case narrative.

			If the CRDL is biased low, no data can be reported for the target elements failing criteria.	
CCV	Daily, before sample analysis, after every 10, and at end of analytical window.	All analytes must be within ± 10% of the true value. (%R): %RSD between multiple integrations must be ≤ 5%	Identify source of problem, re-analyze. Analysis may proceed if it can be demonstrated that the CCV exceedance has no impact on analytical measurements. For example, the CCV %R is high, and the analyte is not detected in sample(s).	Qualify analytes with CCV out of criteria.
CCB	Daily, before sample analysis, after every 10, and at end of analytical window	All elements of interest must be evaluated to a criterion of +/- ½ of the RL for method 6020 (A) and samples originating from NC. All elements of interest must be evaluated to +/- the RL for method 200.8, and 6020 (B). WIDNR and West Virginia require samples to be reported to the MDL. The blanks must be clean to the data quality objectives.	Identify source of problem, re-analyze. Analysis may proceed if it can be demonstrated that the CCB exceedance has no impact on analytical measurements. For example, the CCB has detections and the analyte is not detected in sample(s).	Qualify analytes with CCB out of criteria.
Internal Standards	Every field sample, standard and QC sample	For method 6020, the intensity of internal standard in the ICB/CCB and ICS (ICSA/AB) standards must not deviate more than 80-120% from its original intensity in the associated calibration blank. The intensity of internal standard in the samples and remaining QC must not deviate more than 30-120%. For method 6020A/B, the intensity of the internal standard must not fall below 70% and not exceed 130% from its original intensity in the associated calibration blank. For Method 200.8 the intensity of internal standard in the samples and QC must not deviate more than 60-125% from its original intensity in the associated calibration blank.	Troubleshoot instrument performance. Reanalyze samples and dilute if needed.	Qualify outages and explain in case narrative.
Interference check solutions	ICSA containing high concentrations of C, Cl, Al, Ca, Fe, K, Mg, Mo, Na, P, S and Ti is analyzed at the beginning of each sample run sequence after the CRDL. ICSAB containing high concentrations of	ICSA all spiked elements are to be within 20% of the expected true value. The non-spiked elements are to be below the RL. ICSAB all spiked elements are to be within 20% of the expected true value.	Identify and correct source of problem, repeat performance verification(s).	None. Do not proceed with analysis for elements that cannot be verified.

	C, Cl, Al, Ca, Fe, K, Mg, Mo, Na, P, S and Ti and mid-range concentrations of the remaining elements is analyzed at the beginning of each sample run sequence following the ICSA. 6020A and 6020B requires the ICSA/AB be analyzed every 12 hours thereafter.			
Method Blank (MB)	One per 20 samples	Method 200.8: The method blank is considered to be acceptable if it does not contain the target analytes that exceed 1/2 LLOQ or project-specific DQOs. Method 6020, 6020A and 6020B: The method blank is considered to be acceptable if it does not contain the target analytes that exceed the LLOQ or project-specific DQOs.	Identify source of problem, re-analyze. If reanalysis of the MB fails, all samples affected by the failing MB elements need to be re-digested and re-analyzed. If the method blank exceeds the criteria, but the associated samples are either below the reporting level or other DQOs, or detections in the sample are >10x MB detections then the sample data may be reported. J-flag qualification will be applied for blank detections between the LOQ and LOD when DQOs require evaluation to the MDL.	Qualify outages and explain in case narrative.
LCS	One per 20 samples	6020/6020A/6020B: 80-120% 200.8: 85-115%	Identify source of problem, re-analyze. If reanalysis of the LCS fails, all samples affected by the failing LCS elements need to be re-digested and re-analyzed. If LCS recovery is > QC limits and these compounds are non-detect in the associated samples	Qualify analytes with LCS out of criteria.
LCSD	An LCSD must be substituted in the event of insufficient sample volume for a matrix spike duplicate sample.	6020/6020A/6020B: 80-120% 200.8: 85-115% %Diff ≤ 20%	Identify source of problem, re-analyze. If reanalysis of the LCS fails, all samples affected by the failing LCS elements need to be re-digested and re-analyzed. If LCS recovery is > QC limits and these compounds are non-detect in the associated samples	Qualify analytes with LCS out of criteria.
MS/MSD	One per 20 samples for 6020 / 6020A / 6020B One per 10 samples for 200.8	6020/6020A/6020B: 75-125% 200.8: 70-130%	Perform a SD and PDS on any elements that fail to meet criteria for method 6020(A)(B).	Qualify analytes with MS out of criteria.
Sample Duplicate	Per client request	%Diff ≤ 20%	Qualify outages	Qualify outages.
Serial Dilution[1]	One per batch of 20 samples or less		If criteria is not met, original sample and dilution shall be	Qualify outages.

		6020/6020A fivefold dilution must agree within ± 10% of the original determination if analyte concentration is >50x MDL. 6020B 1:5 dilution of sample 25x > LLOQ or 1:5 dilution of MS since reasonable concentrations are present, results to agree to ± 20%.	reanalyzed. If reanalysis fails, it is determined to be matrix interference.	
Post Digestion Spike[2]	One per batch if there is a MS failure.	6020/ 6020A 80-120% 6020B applicable to elements failing MS, results to agree to +/- 25%. Recommended if high concentration sample not available for dilution test.	If the element fails to meet the recovery criteria, reanalyze. If reanalysis fails, it is determined to be matrix interference.	Qualify outages.
Laboratory Filter Blank (FB)	Analyzed only with batches of lab filtered dissolved metals, one per batch of 20 or less.	Target analytes must be less than reporting limit. NC samples are required to be < ½ RL for target analytes. WIDNR and West Virginia require samples to be reported to the MDL. The blanks must be clean to the data quality objectives.	Identify source of problem, re-analyze. If reanalysis of the MB fails, all samples affected by the failing MB elements need to be re-digested and re-analyzed. If sample(s) non-detect, report the data. If sample result >10x MB detections, report the data.	Qualify outages and explain in case narrative.
Linear Dynamic Range (LDR)	For method 6020B: Following calibration, the laboratory may choose to analyze a standard at a higher concentration than the high standard in the calibration. If a linear range standard is not analyzed for any specific element, the highest standard in the calibration becomes the linear range.	The standard must recover within 10% of the true value, and if successful, establishes the linear range. In each scenario, the linear range is established using 90% of the highest calibration level or LDR sample.	The linear range of the instrument must be adjusted until 90% recovery of the reference standard can be achieved as well as maintaining the minimum number of calibration standard requirements.	N/A

[1] To prepare a 5-fold dilution: take a 1 mL aliquot from the sample and add to 4 mL of diluent. Note: this is a typical process for 200.8 and 6020W. It can be replicated for the preparation of highly concentrated samples by starting with a diluted "parent" sample and then performing the stepwise dilution process.

[2] To Prepare a Post Digestion Spike: An aliquot of the parent sample used for the MS, prepared at the same dilution as the parent sample. The spike addition should produce a minimum level of 10 times the lower limit of quantitation; routine spike volume is 0.020 mL of 20/250 mg/L and 1mg/L mercury stock concentration(s).

Appendix C: Working Standard Summary

Standard	Standard(s) Used	Standard(s) Amount (mL)	Diluent	Diluent Volume (mL)	Final Total Volume[1] (mL)	Final Concentration (ug/L)
Internal Standard	6020-Ge	1	See table 8.1	495	500	2000
	6020-Sc	1				
	6020-Tb	1				
	6020-In	1				
	6020-Ir	1				
Bi/Th primary (Intermediate)	6020-Th	0.5		49.5	50	1,000
	6020-Bi	0.5				
Bi/Th secondary (Intermediate)	6020-Th	0.5		49.5	50	1,000
	6020-Bi	0.5				
Hg 10ppb (intermediate)	HG-LL Stock	0.05		49.95	50	10
6020 Hg-SPK	MERC-STK1	0.05		49.95	50	1000
Hg (Intermediate) C	MERC-STK2	0.25		249.75	250	1000
6020-SPK (intermediate)	Bi-STK	0.2		4.6	10	20,000 / 250,000 / 500,000
	Th-STK	0.2				
	HP7375	5				
6020-SPK2 (intermediate)	HP7376	1		9	10	20,000
6020-SPK3 (intermediate)	HP7379	1		9	10	20,000 / 10,000
CAL-SPK1 (intermediate)	HP7375	0.25		9.5	10	25000/12500/1000/500/10
	HP7379	0.05				
	HP7376	0.05				
	6020Hg-SPK	0.1				
	Bi/Th Intermediate	0.05				
Cal 0	N/A	N/A		50	50	0
Cal 1	ZPACEMN103	0.1		9.7	10	Varied
	ZPACEMN104	0.1				
	Hg 10ppb (intermediate)	0.1				0.1
Cal 2	CAL-SPK1	0.1		9.9	10	250/125/10/5/0.1
Cal 3	CA:L-SPK1	0.5		9.5	10	1250/625/50/25/0.5
Cal 4	CAL-SPK1	1		9	10	2500/1250/100/50/1

Cal 5	CAL-SPK1	2.5		7.5	10	6250/3120/250/125/2.5
Cal 6	CAL-SPK1 (intermediate)	5		-	5	25000/12500/1000/500/10
CRDL	ZPACEMN-103	0.1		9.6	10	varied
	ZPACEMN-104	0.1				
	6020 Hg-SPK	0.2				0.2
ICS-A	ICS-ICPMS	0.25		9.75	10	25000/500
ICS-AB	ICS-ICPMS	0.25		9.56	10	27500/26200/1250/600/100/50/4
	6020-SPK	0.05				
	6020-SPK2	0.05				
	6020-SPK3	0.05				
	6020Hg-SPK	0.04				
ICV / CCV add Hg	XPACEMN-75	0.05		49.31	50	4/80/1000
	XPACEMN-76	0.02				
	Bi/Th Intermediate	0.4				
	XPACEMN-77	0.02				
	Hg Intermediate C	0.2				

[1] Alternate final volumes may be prepared at the discretion of the scientist, so long as the concentrations specified above are maintained.

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Appendix D: Stock Standard Summary

Stock Standard Concentrations

	HP7379	HP7376	HP7375	XPACEMN 77	XPACEMN 76	XPACEMN 75	ZPACEMN 103	ZPACEMN 104	ICS-ICPMS	Agilent Tune	EPA Tune
Analyte	(ug/mL)	(ug/mL)	(ug/mL)	(ug/mL)	(ug/mL)	(ug/mL)	(ug/mL)	(ug/mL)	(ug/mL)	(ug/mL)	(ug/mL)
Aluminum	-		1000			1000	2		1,000		
Antimony		200		200				0.005			
Arsenic	200				200			0.05			
Barium	200				200		0.03				10
Beryllium	200				200		0.02				10
Bismuth							0.05				
Boron		200		200			1				
Cadmium	200				200		0.008				
Calcium			1000			1000	4		1,000		
Chromium	200				200		0.05				
Cobalt	200				200		0.05			10	10
Copper	200				200		0.1				
Iron			500			500	5		1,000		
Lead	200				200		0.01				
Lithium	200				200		0.05			10	10
Magnesium			1000			1000	1		1,000		10
Manganese	200				200		0.05				
Molybdenum		200		200				0.05	20		
Nickel	200				200		0.05				
Palladium		200		200				0.05			
Platinum		200		200				0.05			
Potassium			1000			1000	10		1,000		
Selenium	200				200			0.05			
Silicon			500			500		10			
Silver	100				100		0.05				
Sodium			1000			1000	5		1,000		
Strontium	200				200		0.05				
Thallium					100		0.01			10	10

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Tin		200		200		20		0.05			
Titanium		200		200		20		0.1	20		
Vanadium	200				200		0.1				
Zinc	200				200		0.5				
Uranium	200						0.05				10
Indium											10
Cesium					200						10
Cerium										10	
Yttrium										10	10
Rhodium											10
Thorium							0.05				

Single Element Stock Standard Concentrations

	Bi-STK (Spex)	Bi-STK (Agilent)	6020-Th (Spex)	6020-Th (Agilent)	MERC-STK1	MERC-STK2	HG-LL Stock	6020-Ge	6020-Sc	6020-Tb	6020-In	6020-Ir
Analyte	(ug/mL)	(ug/mL)	(ug/mL)	(ug/mL)	(ug/mL)	(ug/mL)	(ug/mL)	(ug/mL)	(ug/mL)	(ug/mL)	(ug/mL)	(ug/mL)
Bismuth	1000											
Bismuth		1000										
Thorium			1000									
thorium				10000								
Mercury					1000							
Mercury						1000						
Mercury							10					
Germanium								1000				
Scandium									10000			
Terbium										1000		
Indium											1000	
Iridium												1000

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ENVIRONMENTAL SCIENCES

Document Information

Document Number: ENV-SOP-MIN4-0054 **Revision:** 04

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

Department(s): Metals

Date Information

Effective Date: 31 Jul 2020

Notes

Document Notes:

All Dates and Times are listed in: Central Time Zone

Signature Manifest

Document Number: ENV-SOP-MIN4-0054 **Revision:** 04

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

All dates and times are in Central Time Zone.

ENV-SOP-MIN4-0054 - Mercury

QM Approval

Name/Signature	Title	Date	Meaning/Reason
Janielle Ward (007319)	Manager - Quality	30 Jul 2020, 05:04:19 PM	Approved

Management Approval

Name/Signature	Title	Date	Meaning/Reason
Krista Carlson (004514)	Project Coordinator 1	20 Jul 2020, 11:18:09 AM	Approved
Andrew Mickelson (009792)	Manager	20 Jul 2020, 11:31:19 AM	Approved
Adam Haugerud (005828)	General Manager 2	31 Jul 2020, 10:38:58 AM	Approved

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1.0 SCOPE AND APPLICATION

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

1.1 Target Analyte List and Limits of Quantitation (LOQ)

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

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

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

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

1.2 Applicable Matrices

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

2.0 SUMMARY OF METHOD

2.1 The method, a CVAA technique, is based on the absorption of radiation at the characteristic wavelength of 253.7 nm by mercury vapor. The mercury is reduced to the elemental state and aerated from solution in a closed system. The mercury vapor passes through a cell positioned in the light path of an atomic absorption spectrophotometer. Absorbance is measured as a function of mercury concentration.

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

3.0 INTERFERENCES

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

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

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

4.0 DEFINITIONS

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

5.0 HEALTH AND SAFETY

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

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

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

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

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

6.0 SAMPLE COLLECTION, PRESERVATION, HOLDING TIME, AND STORAGE

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

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

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

General Requirements

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

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

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

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

After analysis, unless otherwise specified in the analytical services contract, samples are retained for 45 days from date of final report and then disposed of in accordance with Federal, State, and Local regulations.

7.0 EQUIPMENT AND SUPPLIES

7.1 Equipment

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

7.2 Supplies

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

8.0 REAGENTS AND STANDARDS

8.1 Reagents

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

8.2 Standards

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

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	See appendix B for composition.

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

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

9.0 PROCEDURE

9.1 Water

9.1.1 Sample Preparation

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

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

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

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

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

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

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

9.1.1.8 Loosely cap each cup and place into the digestion block, maintained at a temperature of 95°C ± 2°C and heat for two hours. Observe the initial temperature and time in the block.

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

9.1.1.10 To all samples (including QC) add 1.8 mL of hydroxylamine hydrochloride to reduce the excess permanganate. The permanganate is reduced when the purple color dissipates. If the purple color does not dissipate, add additional hydroxylamine

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hydrochloride until the color dissipates. Note this on the preparation log and adjust in LIMS. For example: if an additional mL is needed, then add 1 mL to the final volume.

9.1.2 Documentation – Digestion Records

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

9.2 Solid/Semi-Solid

9.2.1 Sample Preparation

9.2.1.1 Prepare a MB by weighing 0.3 g of resin pellets in a 50 mL cup.

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

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

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

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

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

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

9.2.1.8 Remove from hot block and allow to cool.

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

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

9.2.1.11 Loosely cap each cup and return samples to hot block digester, maintained at a temperature of 95°C ± 2°C and heat for 30 minutes.

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

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

9.2.2 Documentation – Digestion Records

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

9.3 Equipment Preparation & Analysis

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

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

9.3.3 Check instrument waste and empty as needed.

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

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

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

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

9.3.8 Place the $SnCl_2$ line in DI water.

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

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

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

9.3.12 Fill the stannous chloride bottle with stannous chloride.

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

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

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

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

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

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

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

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

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

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

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

9.3.18.3 Turn off the gas and the lamp.

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

9.4 Routine Instrument Operating Conditions

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

9.5 Initial Calibration

9.5.1 Calibration Design

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

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9.5.1.2 Additional calibration specifications may be referenced in ENV-SOP-NW-0027 *Calibration Procedures*, or equivalent replacement.

9.5.2 Calibration Sequence

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

9.5.3 ICAL Evaluation

9.5.3.1 Curve Fit

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

9.5.3.2 Relative Standard Error (RSE)

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

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

9.5.3.3 Initial Calibration Verification

In addition to meeting the linearity requirement, any new calibration curve must be assessed for accuracy in the values generated. To assess the accuracy, a single standard from a secondary source must be analyzed and the results obtained must be compared to the known value of the standard. This step is referred to as Initial Calibration Verification. The ICV is analyzed immediately following an initial calibration curve.

9.5.4 Continuing Calibration Verification

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

10.0 DATA ANALYSIS AND CALCULATIONS

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

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Equation 1

$$\%\,Recovery = \frac{SR}{SA} x100$$

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

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

Equation 2

$$\%\,Recovery = \frac{(SSR - SR)}{SA} x100$$

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

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

Equation 3

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

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

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

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

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

$$\%Dry\ weight = \frac{Sample\ Dry\ Weight}{Sample\ Wet\ Weight} x100$$

11.0 QUALITY CONTROL AND METHOD PERFORMANCE

11.1 Quality Control

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

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

11.2 Instrument QC

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

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

11.3 Method Performance

11.3.1 Method Validation

11.3.1.1 Detection Limits

Detection limits (DL) and limits of quantitation (LOQ) are established at initial method setup and verified on an on-going basis thereafter. Refer to Pace ENV corporate SOP ENV-SOP-CORQ-0011 *Method Validation and Instrument Verification* and to the laboratory's SOP ENV-SOP-NW-0018 *Determination of LOD and LOQ* for these procedures.

11.4 Analyst Qualifications and Training

Employees that perform any step of this procedure must have a completed Read and Acknowledgment Statement for this version of the SOP in their training record. In addition, prior to unsupervised (independent) work on any client sample, analysts that prepare or analyze samples must have successful initial demonstration of capability (IDOC) and must successfully demonstrate on-going proficiency on an annual basis. Successful means the initial and on-going DOC met criteria, documentation of the DOC is complete, and the DOC record is in the employee's training file. Refer to laboratory SOP ENV-SOP-NW-0025 *Training and Orientation Procedures* for more information.

12.0 DATA REVIEW AND CORRECTIVE ACTION

12.1 Data Review

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

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

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

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

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

12.2 Corrective Action

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

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

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

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

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

13.0 POLLUTION PREVENTION AND WASTE MANAGEMENT

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

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

14.0 MODIFICATIONS

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

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

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

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

15.0 RESPONSIBILITIES

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

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

16.0 ATTACHMENTS

Appendix A – QC Summary

Appendix B – Working Standard Summary

17.0 REFERENCES

Pace Quality Assurance Manual- most current version.

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

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

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

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

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

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

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

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

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

18.0 REVISION HISTORY

This Version:

Section	Description of Change
Appendix A	Updated MB Acceptance Criteria and Corrective Action.

This document supersedes the following document(s):

Document Number	Title	Version
ENV-SOP-MIN4-0054	Mercury in Liquid and Solid/Semi-Solid Waste by 7470A, 7471, 7471B, and 245.1	03

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Appendix A: QC Summary

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

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CCB	Daily, before sample analysis, after every 10, and at end of analytical window	Result must be less than the absolute value of the Reporting Limit (LOQ). NC requires blanks to be clean to ½ RL. WIDNR and West Virginia require samples to be reported to the MDL.	Identify source of problem, re-analyze. Analysis may proceed if it can be demonstrated that the CCB exceedance has no impact on analytical measurements. For example, the CCB has detections and the analyte is not detected in sample(s).	Qualify analytes with CCB out of criteria.
Method Blank	One per 20 samples	Method 7470/7471: The method blank is considered to be acceptable if it does not contain the target analytes that exceed the LLOQ or project-specific DQOs. Method 245.1: The method blank is considered to be acceptable if it does not contain the target analytes that exceed 1/2 LLOQ or project-specific DQOs.	Identify source of problem, re-analyze. If reanalysis of the MB fails, all samples affected by the failing MB elements need to be re-digested and re-analyzed. If the method blank exceeds the criteria, but the associated samples are either below the reporting level or other DQOs, or detections in the sample are >10x MB detections then the sample data may be reported. J-flag qualification will be applied for blank detections between the LOQ and LOD when DQOs require evaluation to the MDL.	Qualify outages and explain in case narrative.
LCS	One per 20 samples	80-120% for 7470/7470A and 7471/7471B. 85-115% for 245.1.	Identify source of problem, re-analyze. If reanalysis of the LCS fails, all samples affected by the failing LCS elements need to be re-digested and re-analyzed. If LCS recovery is > QC limits and these compounds are non-detect in the associated samples	Qualify analytes with LCS out of criteria.
LCSD[1]	An LCSD must be substituted in the event of insufficient sample volume for a matrix spike duplicate sample.	80-120% for 7470/7470A and 7471/7471B. 85-115% for 245.1 % RPD ≤ 20%	Identify source of problem, re-analyze. If reanalysis of the LCS fails, all samples affected by the failing LCS elements need to be re-digested and re-analyzed. If LCS recovery is > QC limits and these compounds are non-detect in the associated samples	Qualify analytes with LCS out of criteria.
MS/MSD[2,3]	One per 20 samples for 7470/7470A and 7471/7471B. One per 10 samples for 200.8	80-120% for 7470/7470A[3] and 7471/74/1B. 245.1: 70-130% %RPD: 20%	If the percent recovery for the MS and MSD fall outside the control limits, the results are flagged that they are outside acceptance criteria along with the parent sample. If the RPD exceeds the acceptance criteria,	Qualify analytes with MS out of criteria.

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			the MSD sample and associated parent sample need to be flagged. If MS or MSD fails and spike amount is less than 4 times the native concentration in the sample, remove M1 flag and replace with P6 flag. If the RPD is outside the limit, report the data and footnote the samples with precision outliers. The footnote only applies to samples within the same batch containing the sample used for the MS and MSD analyses.	
Sample Duplicate	Per client request	%Diff ≤ 20%	Qualify outages	Qualify outages.
Serial Dilution	Per client request	Refer to project specific technical specifications.	Qualify outages	Qualify outages.
Post Digestion Spike	Per client request	Refer to project specific technical specifications.	Qualify outages	Qualify outages.
Laboratory Filter Blank (FB)	Analyzed only with batches of lab filtered dissolved metals, one per batch of 20 or less.	Result must be less than the absolute value of the Reporting Limit (LOQ). NC requires blanks to be clean to ½ RL.	Identify source of problem, re-analyze. If reanalysis of the MB fails, all samples affected by the failing MB elements need to be re-digested and re-analyzed. If sample(s) non-detect, report the data. If sample result >10x FB detections, report the data.	Qualify outages and explain in case narrative.

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

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

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

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

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

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Appendix B: Working Standard Summary

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

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ATTACHMENT D

LEVEL A/B ASSESSMENT CHECKLIST

Level A/B Assessment Checklist

1. General Information

Site:
Project:
Client:
Sample Matrix:

2. Screening Result

Data are:

1. Unusable ____
2. Level A ____
3. Level B ____

I. **Level A**

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

II. **Level B**

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

ATTACHMENT E

EXAMPLE RESULT LETTER TEMPLATES

ATTACHMENT E1

EXAMPLE NO ACTION RESULT LETTER

Atlantic Richfield Company

317 Anaconda Road
Butte, MT 59701
Main: (406) 723-1822

June 5, 2021

Mr. Eric Hassler
Butte-Silver Bow
155 W Granite St
Butte, MT 59701

Dear Mr. Hassler:

This letter is in response to Residential Metals Abatement Program (RMAP) soil sampling activities conducted by Atlantic Richfield Company on your property. Soil sampling was conducted pursuant to the Silver Bow Creek/Butte Area National Priorities List (NPL) Site, Butte Priority Soils Operable Unit (BPSOU) Unilateral Administrative Order (UAO) Amendment issued by the U.S. Environmental Protection Agency (EPA) in August 2020 (UAO Amendment) and under the direct supervision of the EPA. On behalf of the EPA and Atlantic Richfield Company, we would like to provide you the results from the sampling that was conducted on your property.

The arsenic, lead, and mercury concentrations for soil samples collected from your property are attached to this letter. Your results are below the action levels established by the EPA for RMAP soils within the Silver Bow Creek/Butte Area NPL Site. Therefore, further sampling or remediation is not required on your property.

We would like to thank you for your cooperation during this effort. If you have any questions or require further explanation concerning the above information, please give me a call at the number listed below. Alternatively, you may also call Nikia Greene with the EPA (406-457-5019) or Daryl Reed with the MDEQ (406-444-6433) with any questions or concerns.

Sincerely,

Mike McAnulty

Mike Mc Anulty
Liability Manager
Remediation Management Services Company
An affiliate of Atlantic Richfield Company
(406) 723-1822

Attachment: Analytical Soil Sampling Results

cc: Nikia Greene/EPA
Daryl Reed/MDEQ

File: MiningSharePoint@bp.com

ANALYTICAL RESULTS FROM SOIL SAMPLING
CONDUCTED ON YOUR PROPERTY

Geocode: 01119831305010000, 01119831303010000

Physical Address: No Physical Address

Legal Description: -S31, T03 N, R07 W, POR SW4 AKA ALL BLKS 6, 7 VAC OREGON AVE BETWEEN SUB TRACTS
-S31, T03 N, R07 W, LTS 1-10, TRACT D (AKA LTS 90,91) BLK 12, SUBURBAN TRACTS, SW4

Residential ID: P-0001

Resident ID	SAMPLING COMPONENTS	COMPONENT SURFACE AREA (Square Feet)	COMPONENT ARSENIC CONCENTRATION (mg/kg)					COMPONENT LEAD CONCENTRATION (mg/kg)					COMPONENT MERCURY CONCENTRATION (mg/kg)				
P-0001			0-2"	2-6"	6-12"	12-18"	18-24"	0-2"	2-6"	6-12"	12-18"	18-24"	0-2"	2-6"	6-12"	12-18"	18-24"
P-0001-GA1	Grass Area (GA)	10,500	150	88	75	N/A	N/A	343	315	425	N/A	N/A	18	25	12	N/A	N/A
P-0001-GA2	Grass Area (GA)	10,500	142	95	65	N/A	N/A	422	366	358	N/A	N/A	55	38	33	N/A	N/A
P-0001-GA3	Grass Area (GA)	10,500	88	62	105	N/A	N/A	707	255	243	N/A	N/A	23	17	33	N/A	N/A
	Total:	31,500															

🟥	Component Arsenic Concentration is ≥ 250 mg/kg.
🟨	Component Lead Concentration is ≥ 1,200 mg/kg.
🟦	Component Mercury Concentration is ≥ 147 mg/kg.
N/A	= Not applicable per 2021 RMAP Quality Assurance Project Plan.

EPA Action Levels to Determine the Need for Additional Testing or Remediation in RMAP Soils:

Arsenic: Any Component ≥ 250 ppm

Lead: Any Component ≥ 1,200 ppm

Mercury: Any Component ≥ 147 ppm

Definitions of words and abbreviations used above:

COMPONENT CONCENTRATION - The concentration of arsenic, lead, or mercury within a sampling component at a given depth interval.

PARTS PER MILLION (PPM) – Parts per million, an expression of concentration. A good analogy: If you had 20ppm, it would be like having 20 white marbles and 999,980 black marbles in a group of 1,000,000 total marbles.

N/A – Not applicable per the 2021 RMAP Quality Assurance Project Plan (QAPP)

ATTACHMENT E2

EXAMPLE REMEDIAL ACTION RESULT LETTER

317 Anaconda Road
Butte, MT 59701
Main: (406) 723-1822

Atlantic Richfield Company

June 5, 2021

Mr. Eric Hassler
Butte-Silver Bow
155 W Granite St
Butte, MT 59701

Dear Mr. Hassler:

This letter is in response to Residential Metals Abatement Program (RMAP) soil sampling activities conducted by Atlantic Richfield Company on your property. Soil sampling was conducted pursuant to the Silver Bow Creek/Butte Area National Priorities List (NPL) Site, Butte Priority Soils Operable Unit (BPSOU) Unilateral Administrative Order (UAO) Amendment issued by the U.S. Environmental Protection Agency (EPA) in August 2020 (UAO Amendment) and under the direct supervision of the EPA. On behalf of the EPA and Atlantic Richfield Company, we would like to provide you the results from the sampling that was conducted on your property.

You will see that one or more of the samples contained arsenic, lead, or mercury above the Residential Metals Abatement Program (RMAP) soil action levels established by the U.S. Environmental Protection Agency (EPA) for this area. EPA has determined that such soil should be removed from the surface of your property and replaced with clean soil and new vegetation.

This letter describes the work that is proposed for your property and asks you for permission to complete that work at Atlantic Richfield Company's expense. The proposal is described in more detail below, and in the proposed access agreement and work plan attached to this letter.

Sample Results

Soil sampling was conducted pursuant to the Silver Bow Creek/Butte Area National Priorities List (NPL) Site, Butte Priority Soils Operable Unit (BPSOU) Unilateral Administrative Order (UAO) Amendment issued by the U.S. Environmental Protection Agency (EPA) in August 2020 (UAO Amendment) and under the direct supervision of the EPA.

The arsenic, lead, and mercury concentrations for soil samples collected from your property are attached to this letter. Your sample results, which have been reviewed and approved by EPA, indicate that the concentrations of arsenic, lead, and/or mercury detected within your property exceed the RMAP soil action level(s) established by EPA within the Silver Bow Creek/Butte Area National Priorities List (NPL) Site. Therefore, some or all of your property is eligible for soil remediation.

Proposed Remedy and Access Agreement

Atlantic Richfield Company requests your permission to complete the soil remedy that EPA has selected for your property, at Atlantic Richfield's own expense. In order to move forward with soil remediation on your property, you will need to provide us with an access agreement that allows us to complete that work.

An Individual Site Work Plan (ISWP) for your property is attached as Exhibit B to the Access Agreement. The ISWP, which also has been approved by EPA, describes the details of the soil remediation work proposed for your property.

Next Steps

Atlantic Richfield respectfully asks that you review the attached Access Agreement and ISWP. If you concur with these documents and would like to proceed with the proposed soil remediation, please sign the Access Agreement. If you return the fully executed Access Agreement to me in the enclosed self-addressed stamped envelope, I will countersign the Access Agreement and provide you with a copy for your records. Once we receive your executed Access Agreement, we will contact you to schedule the remediation work.

We would like to thank you for your cooperation during this effort. If you have any questions or would like further explanation concerning the above, please call me at **406-723-1822**.

Sincerely,

Mike McAnulty

Mike Mc Anulty
Liability Manager
Remediation Management Services Company
An affiliate of Atlantic Richfield Company
(406) 723-1822

Attachments: Analytical Soil Sampling Results
Construction Access Agreement
Individual Site Work Plan (ISWP)

cc: Nikia Greene/EPA
Daryl Reed/MDEQ

File: MiningSharePoint@bp.com

ANALYTICAL RESULTS FROM SOIL SAMPLING
CONDUCTED ON YOUR PROPERTY

Geocode: 011198313050100000, 011198313030100000

Physical Address: No Physical Address

Legal Description: -S31, T03 N, R07 W, POR SW4 AKA ALL BLKS 6, 7 VAC OREGON AVE BETWEEN SUB TRACTS
-S31, T03 N, R07 W, LTS 1-10, TRACT D (AKA LTS 90,91) BLK 12, SUBURBAN TRACTS, SW4

Residential ID: P-0001

Resident ID	**SAMPLING COMPONENTS**	**COMPONENT SURFACE AREA (Square Feet)**	**COMPONENT ARSENIC CONCENTRATION (mg/kg)**					**COMPONENT LEAD CONCENTRATION (mg/kg)**					**COMPONENT MERCURY CONCENTRATION (mg/kg)**				
P-0001			0-2"	2-6"	6-12"	12-18"	18-24"	0-2"	2-6"	6-12"	12-18"	18-24"	0-2"	2-6"	6-12"	12-18"	18-24"
P-0001-GA1	Grass Area (GA)	10,500	150	88	75	N/A	N/A	1,217	315	425	N/A	N/A	18	25	12	N/A	N/A
P-0001-GA2	Grass Area (GA)	10,500	142	255	65	N/A	N/A	422	366	358	N/A	N/A	55	38	33	N/A	N/A
P-0001-GA3	Grass Area (GA)	10,500	88	62	105	N/A	N/A	707	255	243	N/A	N/A	23	174	33	N/A	N/A
	Total:	31,500															

🟥 Component Arsenic Concentration is ≥ 250 mg/kg.

🟨 Component Lead Concentration is ≥ 1,200 mg/kg.

🟦 Component Mercury Concentration is ≥ 147 mg/kg.

N/A = Not applicable per 2021 RMAP Quality Assurance Project Plan.

EPA Action Levels to Determine the Need for Additional Testing or Remediation in RMAP Soils:

Arsenic: Any Component ≥ 250 ppm

Lead: Any Component ≥ 1,200 ppm

Mercury: Any Component ≥ 147 ppm

Definitions of words and abbreviations used above:

COMPONENT CONCENTRATION - The concentration of arsenic, lead, or mercury within a sampling component at a given depth interval.

PARTS PER MILLION (PPM) – Parts per million, an expression of concentration. A good analogy: If you had 20ppm, it would be like having 20 white marbles and 999,980 black marbles in a group of 1,000,000 total marbles.

N/A – Not applicable per the 2021 RMAP Quality Assurance Project Plan (QAPP)

ACCESS AGREEMENT

BUTTE-SILVER BOW ("Owner") and **Atlantic Richfield Company** ("Atlantic Richfield") enter into this Access Agreement ("Agreement") this __________ day of ____________________, 2021.

1. Atlantic Richfield is conducting certain remedial activities on properties in and near Butte.

2. Access to property owned by Owner and as described in Exhibit A is needed to conduct this remedial work.

3. Owner agrees to permit Atlantic Richfield to conduct such work on Owner's property.

Therefore, in the mutual interest of Owner and Atlantic Richfield, Owner and Atlantic Richfield further agree as follows:

1. GRANT OF ACCESS. Owner hereby grants to Atlantic Richfield, Environmental Protection Agency ("EPA") and the State of Montana ("State"), including the authorized representatives of each, the right to enter Owner's real property described in Exhibit A hereto (the "Property"), to conduct all activities described in the Individual Site Work Plan attached as Exhibit B hereto, including without limitation, excavation and/or removal of soils, removal of attic dust, monitoring and sampling (or to receive split samples) of environmental media, ingress and egress of equipment, machinery and personnel, staging and temporary storage of equipment, and conducting other information gathering activities such as field investigation, data collection, surveys and testing (collectively referred to as "Work"). Owner warrants and represents to Atlantic Richfield that, to the best of Owner's knowledge, Owner possesses ownership interests in the Property sufficient to grant access to Atlantic Richfield to conduct the Work. Atlantic Richfield shall provide Owner, either in writing or verbally, with at least 24 hours notice prior to first commencing the Work on the Property. Atlantic Richfield will make every reasonable effort to minimize any inconvenience to Owner during its Work on the Property, and will work closely with Owner to address any concerns Owner may have about the Work.

2. INDEMNIFICATION OF OWNER. Atlantic Richfield agrees to indemnify and hold harmless Owner from any and all actions, claims, damages, losses, liabilities, or expenses, including damage to property or for loss of use of property ("Liabilities"), which may be imposed on or incurred by Owner as a result of Atlantic Richfield's negligent, wrongful acts or omissions while on the Property to conduct the Work, except to the extent that such liabilities result from the acts or omissions of Owner. Provided that the Work is conducted without negligence or wrongful acts or omissions by Atlantic Richfield, Owner and Atlantic Richfield agree that the Work conducted pursuant to this Agreement shall not give rise to a claim for indemnification under this provision.

3. NOTICE. All written notices pertaining to this Agreement shall be sent to Owner and Atlantic Richfield at the respective addresses below. Either Owner or Atlantic Richfield may

designate a different address for receipt of notice by providing written notice of such change to the other.

TO Atlantic Richfield: Mike Mc Anulty
317 Anaconda Road
Butte, MT 59701
(406) 723-1822

TO OWNER: Butte-Silver Bow
155 W GRANITE STREET
BUTTE, MT 59701

4. CONDITION OF THE PROPERTY. If the Work entails the excavation and removal of soils and/or the removal of attic dust, Atlantic Richfield may photograph the Property prior to and upon completion of the excavation and removal of soils to document and obtain a fair and accurate representation of the condition of the Property.

5. RESTORATION OF PROPERTY. Upon completion of the Work, Atlantic Richfield will use its best efforts to return the Property to the condition it was in at the time Atlantic Richfield first entered the Property under this Agreement, provided such restoration is not inconsistent with the Work conducted pursuant to this Agreement.

6. MISCELLANEOUS.

a. Effect of Agreement. This Agreement and the rights and obligations created hereby shall be binding upon and inure to the benefit of Owner and Atlantic Richfield and their respective assigns and successors in interest.

b. Negation of agency relationship. This Agreement shall not be construed to create, either expressly or by implication, the relationship of agency or partnership between Owner and Atlantic Richfield. Neither Owner nor Atlantic Richfield is authorized to act on behalf of the other in any manner relating to the subject matter of this Agreement.

c. Termination. Except with respect to paragraphs 2, 3 and 6.a of this Agreement, this Agreement will terminate thirty (30) days following Atlantic Richfield's written notification to Owner that the Work is complete.

d. Governing Law. This Agreement shall be governed by and construed in accordance with the laws of the State of Montana.

e. Construction. The invalidity or unenforceability of any provision of this Agreement shall not affect the validity or enforceability of any other provision.

f. Entire Agreement. This Agreement embodies the entire agreement of Owner and Atlantic Richfield with respect to the subject matter hereof, and no prior oral or written representation shall serve to modify or amend this Agreement. This Agreement may be modified only by a written agreement signed by Owner and Atlantic Richfield.

IN WITNESS WHEREOF, Owner and Atlantic Richfield have executed this Agreement effective as of the date first written above.

OWNER	Atlantic Richfield Company
BUTTE-SILVER BOW	
By: ______________________________	By: _________________________
Title (If other than Owner): ______________________	Title: Liability Manager__________
Telephone Contact No. _______________	

EXHIBIT A
(Legal Description of the Property)

For the purposes of this Access Agreement, the term Property refers to the following described real estate, situated in the County of Silver Bow, State of Montana:

Name	Geocode	Legal Description
Jeremy Bullock Soccer Fields	01119831305010000 01119831303010000	-S31, T03 N, R07 W, POR SW4 AKA ALL BLKS 6, 7 VAC OREGON AVE BETWEEN SUB TRACTS -S31, T03 N, R07 W, LTS 1-10, TRACT D (AKA LTS 90,91) BLK 12, SUBURBAN TRACTS, SW4

EXHIBIT B

(Individual Site Work Plan)

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FOR THIS TEMPLATE**

ATTACHMENT F

BUTTE HILL COVER SOIL

APPROVAL SUBMITTAL FORM

BUTTE HILL COVER SOIL APPROVAL SUBMITTAL

3/16/2021

Source:
Sample #:

Description	Specification	Sample	Specification Met	
			Yes	No
Chemical (mg/kg)				
As	< 97			
Cd	< 4			
Cu	< 250			
Pb	< 100			
Zn	< 250			
pH (s.u.)				
	> 5.5			
	< 8.5			
SAR				
	< 12			
Saturation (%)				
	< 85			
	> 25			
EC (mmhos/cm)				
	< 4			
Textural Classification (USDA) <2.0 mm				
	Loam			
	Sandy loam			
	Sandy clay loam			
	Sandy clay			
	Clay loam			
	Silty clay			
	Silty clay loam			
	Silt loam			
	Silt			
	*Per EPA Approval (Loamy sand)			
Rock Content (%) (by volume)	< 45			

Other Information Requested
Organic Matter (%)
WB
Soil Nutrients
NO_3 (ug/g)
P (ug/g)
K (ug/g)
Particle Size
Sand (%)
Silt (%)
Clay (%)

Legend:
🟩 # Value - Criteria met
🟥 **# Value** - Does not meet Criteria

B-SB Representative ____________________ Date: ____________________

EPA Representative: ____________________ Date: ____________________

ATTACHMENT G

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:____________________________

Corrective Action Report/ Corrective Action Plan

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

ATTACHMENT H

DATA VALIDATION CHECKLIST

Data Validation Checklist for Metals Sample Analysis

Site: **Case No:** **Laboratory:**

Project: **Sample Matrix:** **Analyses:**

Sample Date(s): **Analysis Date(s):**

Data Validator: **Validation Date(s):**

1. Holding Times

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

*Reference for Holding Times –

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

Describe Any Actions Taken:

Comments:

2. Instrument Calibration

Was the Tune analysis performed? Y ☐ N ☐ N/A ☐
Was the peak widths and resolution of the masses within the required control limits? Y ☐ N ☐ N/A ☐
Was the percent relative standard deviation ≤ 5% for all analytes in the Tune solutions? Y ☐ N ☐ N/A ☐
Was Instrument successfully calibrated at the correct frequency? Y ☐ N ☐
Was Instrument calibrated with appropriate standards and blanks? Y ☐ N ☐
Were Initial Calibration Verification (ICV) and Continuing Calibration Verification (CCV) samples analyzed? Y ☐ N ☐
Were ICV and CCV results within the control window? Y ☐ N ☐
Were any data flagged because of calibration problems? Y ☐ N ☐

Describe Any Actions Taken:

Comments:

3. Blanks

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

Describe Any Actions Taken:

Comments:

4. Interference Check Samples

Were ICP Interference Check Samples (ICS) within the control limits? Y ☐ N ☐
Were any data flagged because of ICS problems? Y ☐ N ☐

Describe Any Actions Take:

Comments:

5. Laboratory Control Samples

Were Laboratory Control Samples (LCS) analyzed at the frequency of 1 per batch? Y ☐ N ☐

What was the source of the LCS?

Were LCS results within the control window? Y ☐ N ☐

Were any data flagged because of LCS problems? Y ☐ N ☐

Describe Any Actions Taken:

Comments:

6. Duplicate Sample Results

Were Laboratory Duplicate Samples (LDS) analyzed at the frequency of 1 per batch? Y ☐ N ☐

Were LDS results within the control window? Y ☐ N ☐

Were any data flagged because of LDS problems? Y ☐ N ☐

Describe Any Actions Taken:

Comments:

7. Matrix Spike Sample Results

Were Laboratory Matrix Spike Samples (LMS) analyzed at the frequency of 1 per batch? Y ☐ N ☐

Were LMS percent recovery (%R) results within the control window? Y ☐ N ☐

Were any data flagged because of LMS problems? Y ☐ N ☐

Describe Any Actions Taken:

Comments:

8. ICP Serial Dilutions

Were ICP Serial Dilutions (SD) analyzed at the frequency of 1 per batch? Y ☐ N ☐

Were SD percent differences (%D) results within the control window? Y ☐ N ☐

Were any data flagged because of SD problems? Y ☐ N ☐

Describe Any Actions Taken:

Comments:

9. Internal Standards

Were internal standards added to each sample in the analytical batch? Y ☐ N ☐

Were the percent relative recoveries (%RI) within the control window? Y ☐ N ☐

Were any data flagged because of internal standard problems? Y ☐ N ☐

Describe Any Actions Taken:

Comments:

10. Field Blanks

Were field blanks submitted as specified in the Sampling Analysis Plan (SAP)? Y ☐ N ☐ N/A ☐

Were field blanks within the control window? Y ☐ N ☐ N/A ☐

Were any data qualified because of field blank problems? Y ☐ N ☐ N/A ☐

Describe Any Actions Taken:

Comments:

11. Field Duplicates

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

Describe Any Actions Taken:

Comments:

12. Overall Assessment

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

If so, explain:

Comments:

13. Authorization of Data Validation

Data Validator **Name:**	**Reviewed by:**
Signature: ____________________	____________________
Date: ____________________	____________________

ATTACHMENT I

ANNUAL QAPP REVISION SUMMARIES

Attachment I
Annual RMAP QAPP Revision Summary Page

Date	Revision #	Summary of Changes

ATTACHMENT B

DRAFT BUTTE HIGH SCHOOL

INDIVIDUAL SITE WORK PLAN (ISWP)

ADDRESS: 401 S WYOMING STREET
PROPERTY ID: S-0009

S-0009

LEGEND

- 🟩 No Action Required
- 🟪 12" Removal
- ▤ 24" Removal
- ▨ Un-Samplable Area

NOTES:

1. LOOK ON BACK OF SHEET FOR DATA TABLE.

BUTTE HIGH SCHOOL
INDIVIDUAL SITE WORK PLAN

RESIDENTIAL METALS ABATEMENT PROGRAM (RMAP)

BUTTE, MONTANA
SHEET 1 OF 2

Boundaries on this site work plan DO NOT represent a legal survey. These boundaries are to be used for general reference only. No liability is assumed by Atlantic Richfield Company or Pioneer Technical Services for the accuracy of these.

DRAFT
DATA VALIDATION NOT YET COMPLETE

150 75 0 150
Feet

Atlantic Richfield Company
A BP affiliated company

BY:
PIONEER TECHNICAL SERVICES, INC.

Date: 9/6/2021
Revision#: 0
File Name: RMAP_ISWP_ButteHighSchool

REMEDIAL ACTION SUMMARY TABLE

Resident ID	**SAMPLING COMPONENTS**	**COMPONENT SURFACE AREA (Square Feet)**	**COMPONENT ARSENIC CONCENTRATION (mg/kg)**					**COMPONENT LEAD CONCENTRATION (mg/kg)**					**COMPONENT MERCURY CONCENTRATION (mg/kg)**					**ESTIMATED QUANTITIES**		
S-0009			0-2"	2-6"	6-12"	12-18"	18-24"	0-2"	2-6"	6-12"	12-18"	18-24"	0-2"	2-6"	6-12"	12-18"	18-24"	Excavation (Cubic Yards)	General Backfill (Cubic Yards)	Sod (Square Feet)
S-0009-HA1	High Access Area 1 (HA1)	9,181	43	58	67	N/A	N/A	97	275	657	N/A	N/A	0.05	0.23	0.22	N/A	N/A	0.0	0.0	0
S-0009-HA2	High Access Area 2 (HA2)	7,928	47	54	55	N/A	N/A	238	660	1,070	N/A	N/A	0.07	0.14	0.18	N/A	N/A	0.0	0.0	0
S-0009-HA3	High Access Area 3 (HA3)	6,577	59	52	49	N/A	N/A	160	362	529	N/A	N/A	0.07	0.12	0.36	N/A	N/A	0.0	0.0	0
S-0009-GA1	Grass Area 1 (GA1)	9,951	27	32	48	N/A	N/A	281	262	375	N/A	N/A	0.15	0.09	0.33	N/A	N/A	0.0	0.0	0
S-0009-GA2	Grass Area 2 (GA2)	9,959	36	32	50	N/A	N/A	482	207	721	N/A	N/A	0.14	0.09	0.17	N/A	N/A	0.0	0.0	0
S-0009-GA3	Grass Area 3 (GA3)	9,953	32	76	82	N/A	N/A	194	1,520	5,220	N/A	N/A	0.27	0.39	1.10	N/A	N/A	368.6	368.6	9,953
S-0009-GA4	Grass Area 4 (GA4)	9,955	35	51	52	N/A	N/A	268	186	573	N/A	N/A	0.18	0.10	0.25	N/A	N/A	0.0	0.0	0
S-0009-GA5	Grass Area 5 (GA5)	7,871	64	80	180	N/A	N/A	1,840	1,790	2,370	N/A	N/A	0.64	1.40	0.82	N/A	N/A	291.5	291.5	7,871
S-0009-GA6	Grass Area 6 (GA6)	9,467	36	53	35	N/A	N/A	273	158	227	N/A	N/A	0.18	0.08	0.04	N/A	N/A	0.0	0.0	0
S-0009-GA7	Grass Area 7 (GA7)	9,470	39	39	86	N/A	N/A	370	2,090	731	N/A	N/A	0.31	0.13	0.12	N/A	N/A	350.7	350.7	9,470
S-0009-GA8	Grass Area 8 (GA8)	10,405	58	72	62	N/A	N/A	154	700	758	N/A	N/A	0.07	0.14	0.15	N/A	N/A	0.0	0.0	0
S-0009-GA9	Grass Area 9 (GA9)	10,402	30	27	9	N/A	N/A	92	124	69	N/A	N/A	0.04	0.05	0.05	N/A	N/A	0.0	0.0	0
S-0009-GA10	Grass Area 10 (GA10)	10,405	42	57	54	N/A	N/A	68	384	357	N/A	N/A	0.10	0.17	0.05	N/A	N/A	0.0	0.0	0
S-0009-GA11	Grass Area 11 (GA11)	10,406	35	60	57	N/A	N/A	123	310	891	N/A	N/A	0.05	0.20	1.20	N/A	N/A	0.0	0.0	0
S-0009-GA12	Grass Area 12 (GA12)	10,405	43	53	54	N/A	N/A	104	257	467	N/A	N/A	0.07	0.16	0.40	N/A	N/A	0.0	0.0	0
S-0009-GA13	Grass Area 13 (GA13)	10,405	28	43	22	N/A	N/A	153	251	130	N/A	N/A	0.05	0.33	0.09	N/A	N/A	0.0	0.0	0
S-0009-GA14	Grass Area 14 (GA14)	10,402	40	53	55	N/A	N/A	229	683	671	N/A	N/A	0.10	0.17	0.24	N/A	N/A	0.0	0.0	0
S-0009-GA15	Grass Area 15 (GA15)	10,407	64	52	35	N/A	N/A	66	262	344	N/A	N/A	0.04	0.18	0.21	N/A	N/A	0.0	0.0	0
S-0009-GA16	Grass Area 16 (GA16)	10,404	62	39	40	N/A	N/A	167	299	370	N/A	N/A	0.05	0.17	0.12	N/A	N/A	0.0	0.0	0
S-0009-GA17	Grass Area 17 (GA17)	7,240	41	70	52	N/A	N/A	131	254	1,130	N/A	N/A	0.05	0.13	2.50	N/A	N/A	0.0	0.0	0
S-0009-GA18	Grass Area 18 (GA18)	7,394	43	88	86	N/A	N/A	296	250	1,040	N/A	N/A	0.06	0.14	0.57	N/A	N/A	0.0	0.0	0
S-0009-G1	Garden Area 1 (G1)	127	28	33	35	54	55	53	55	60	656	766	0.22	0.25	0.17	1.00	0.48	0.0	0.0	0
		Max:	64	88	180	54	55	1,840	2,090	5,220	656	766	0.64	1.40	2.50	1.00	0.48	1,010.9	1,010.9	27,294.0
🟧	Component Arsenic Concentration is ≥ 250 mg/kg.																			
🟨	Component Lead Concentration is ≥ 1,200 mg/kg.																			
🟩	Component Mercury Concentration is ≥ 147 mg/kg.																			
N/A	= Not applicable per 2021 RMAP Quality Assurance Project Plan.																			

BUTTE HIGH SCHOOL
INDIVIDUAL SITE WORK PLAN

RESIDENTIAL METALS
ABATEMENT PROGRAM (RMAP)

BUTTE, MONTANA
SHEET 2 OF 2

DRAFT
DATA VALIDATION
NOT YET COMPLETE

Atlantic Richfield Company
A BP affiliated company
bp

BY:
PIONEER
TECHNICAL SERVICES, INC.