

Appendix B

Sampling and Analysis Plan

Appendix B - Table of Contents

1.0 Introduction	4
1.1 Project Objectives	4
2.0 Sampling Rationale.....	4
2.1 Selection of Sampling Locations.....	4
2.2 Selection of Samples for Laboratory Analysis	7
2.3 Selection of Target Metals	7
3.0 Request for Analysis.....	8
3.1 Analyses Narrative	8
3.2 Analytical Laboratory.....	8
3.3 Analytical Methods	9
4.0 Field Methods and Procedures	13
4.1 Team Briefing	13
4.2 Field Equipment	13
4.3 Soil Sample Preparation and Collection	15
4.4 Soil Sample Location Identification.....	16
4.5 Decontamination	16
5.0 Investigation Derived Waste Management	16
5.1 Water	16
5.2 Solid Waste	16
6.0 Sampling Containers, Preservation, and Storage.....	17
7.0 Samples Documentation and Shipment.....	17
7.1 Sample Labeling	17
7.2 Field Notes and Photographs	17
7.3 Sample Chain of Custody Procedures	17
8.0 Quality Assurance/Quality Control	19
8.1 Laboratory QA/QC	19
8.2 Background Samples	19
8.3 Data Analysis	19
8.4 Data Validation	20
9.0 References.....	21

List of Tables

Table B-1: Sampling Design and Rationale.....	7
Table B-2: Analysis Design and Rationale.....	8
Table B-3: XRF Analysis Procedure – EPA Method 6200.....	10
Table B-4: Acid Digestion Procedure for GFAA - EPA Method 3050B.....	11
Table B-5: Acid Digestion Procedure for FIAA - EPA Method 3050B.....	12
Table B-6: Equipment and Supplies for Sampling.....	13
Table B-7: Surface Soil Sampling Procedure.....	15
Table B-8: Decontamination Procedure.....	16

List of Figures

Figure B-1: Grid Sampling for AZ Magma Mine.....	5
Figure B-2: Detail View of Sampling Grid.....	6
Figure B-3: Blank Chain of Custody Form.....	18

List of Abbreviations

AA	Atomic Absorption
BLM	Bureau of Land Management
COC	Contaminant of Concern
FIAA	Flame Ionization Atomic Absorption
GFAA	Graphite Furnace Atomic Absorption
GIS	Geographic Information System
GPS	Global Positioning System
HASP	Health and Safety Plan
HAZWOPER	Hazardous Waste Operations and Emergency Response
IDW	Investigation Derived Wastes
KRMC	Kingman Regional Medical Center
NAU	Northern Arizona University
PA	Preliminary Assessment
PPE	Personal Protective Equipment
QA	Quality Assurance
QC	Quality Control
SAP	Sampling and Analysis Plan
SI	Site Inspection
SRL	Soil Remediation Level
XRF	X-ray Fluorescence

1.0 Introduction

This Sampling and Analysis Plan (SAP) details sampling that will occur at the AZ Magma Mine located on Bureau of Land Management (BLM) land, as well as all analysis that will be completed on the soil samples taken from the site. Selection of sampling locations on site and laboratory analysis is explained and detailed. An aligned grid of the mine is provided, with a discussion of the number and type of samples to be taken in each grid. Field methods are also provided to describe how the team is to collect, preserve, store, document, and transport samples. Quality control (QC) is also discussed, including the use of background samples and data correlation to ensure the results are legitimate.

1.1 Project Objectives

The purpose of this site inspection is to collect and analyze soil samples from AZ Magma Mine located near Chloride, Arizona for possible contaminants of concern (COC). The data obtained from this inspection will be used as screening level data for the BLM to determine if further action should be taken at this site. If the COCs, discussed in Section 2.3 the SAP, are above their respective action levels, further action should be taken.

2.0 Sampling Rationale

This section discusses the sampling locations and the rationale for each, including general samples, hotspot, and background samples. The samples that will be chosen for analysis are also included in this section, as well as the COCs that may be expected at this site.

2.1 Selection of Sampling Locations

The sampling grid that will be used during sampling can be seen below in Figure B-1. A detail view of the sampling grid can be seen in Figure B-2. The large grid spacing is located on the main tailings and is 100 feet by 100 feet. The small grid spacing is within the wash and onto the road near the mine and is 40 feet by 40 feet. The large grid nodes are labeled left-to-right, top-to-bottom. The small grid nodes are labeled left-to-right, top-to-bottom, following the numbering of all large grid nodes. A total of 100 samples will be taken throughout the whole site, including grid, hotspot, and background samples. Hotspot and background samples will be chosen at the site with oversight from supervisor Bridget Bero. Table B-1 lists the sampling location, ID number of the sample, and the rationale for that sample. Information on the ID number format for all samples can be seen in Section 4.4 of the SAP. All sample types will be surface samples to avoid digging into the 10-foot deep tailings.

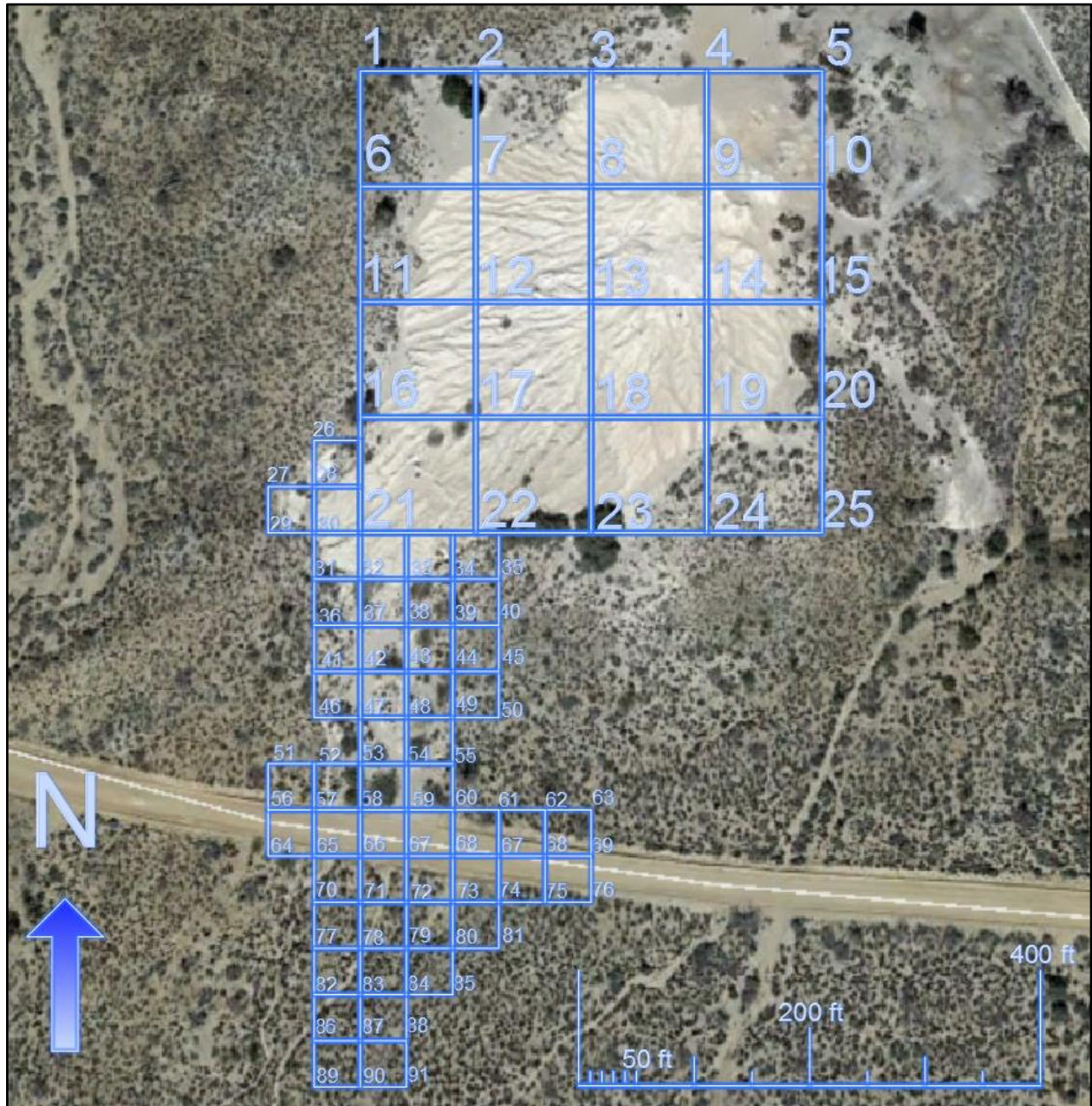


Figure B-1. Grid Sampling for AZ Magma Mine (Google Maps, 2016)



Figure B-2. Detail View of Sampling Grid

Table B-1. Sampling Design and Rationale

Sampling Location	ID Number	Rationale
Main Tailings Pile	GN1 - GN25	Concentrations within the main tailings pile will be consistent, so a larger grid spacing and smaller amount of samples is appropriate.
The Wash	GN26 - GN55, GN77 - GN91	Concentrations within the wash will vary as distance from the main tailings site increases, so a smaller grid and more frequent sampling amount is appropriate.
The Road	GN56 - GN76	Concentrations on the road will vary depending on the side of the road and distance from main tailings.
Hotspot Samples - Various Locations	HS1 - HS5	Hotspot samples will be chosen by Bridget Bero in the field at areas of observed tailings. These samples will be representative of the highest concentrations of COCs at the site.
Background Samples - Various Locations	BG1 – BG4	Background samples will be chosen by Bridget Bero in the field as appropriate. These samples will be used to identify the naturally occurring levels of COCs in the area surrounding the site.

2.2 Selection of Samples for Laboratory Analysis

All samples will be analyzed using X-ray fluorescence (XRF). 20% of these samples will also be analyzed by atomic absorption (AA). Samples with high, low, and average concentrations of the COCs will be selected for AA analysis. Discussion of the methods for these analyses can be seen in Section 3.0.

2.3 Selection of Target Metals

Possible COCs derive from the type of mining that occurred at the site. The AZ Magma Mine was primarily used for silver, gold, and lead mining from the 1890's to the late 1930's (ADMMR, 1995). Thus, potential COCs at the site include but are not limited to lead and arsenic (Zielske, 2016).

3.0 Request for Analysis

This section discusses the analytical support for the project. This includes the analyses requested by BLM, possible turnaround times, available resources and laboratories for the team, and method numbers and descriptions for analyses.

3.1 Analyses Narrative

The required analyses and procedures for this investigation include drying, sieving, XRF, acid digestion, and AA. Drying is required so that all material can be properly sieved. All samples will be dried and sieved prior to XRF analysis. Table B-2 shows how sampling will be dispersed among grid, hotspot, and background samples.

Table B-2. Analysis Design and Rationale

Sampling Location	ID Number	Number of Samples (XRF)
Main Tailings Pile	GN1 - GN25	25
The Wash	GN26 - GN55, GN77 - GN91	45
The Road	GN56 - GN76	21
Hotspot Samples - Various Locations	HS1 - HS5	5
Background Samples - Various Locations	BG1 – BG4	4
Total # of Samples		100

3.2 Analytical Laboratory

All sieving, XRF analysis, and acid digestion will be done in the Environmental Engineering Lab of the Engineering Building. All AA analysis will be done in the NAU Chemistry Lab of the Wettaw Building.

3.3 Analytical Methods

This section identifies and briefly describes the methods used for all preparatory and analytical processes required for this investigation.

3.3.1 Drying and Sieving Procedure

Prior to analysis, all soil samples will be dried for 24 hours at 100°F in a drying oven. All samples will be sieved to a #200 sieve to homogenize the samples. The samples will be put into the sieve from their original storage gallon bag (see Section 4.3). Once in the sieve stack, the sieves will be placed into a shaker for approximately five minutes. After the shaker, the soil will be put back into its gallon bag to be analyzed. The sieves must be thoroughly cleaned with a soft bristle brush between every sample analysis.

3.3.2 X-Ray Fluorescence Spectrometry

The method to be followed for XRF analysis is the Environmental Protection Agency's (EPA) Method 6200. This method is specified for XRF analysis in the field, but all analysis for this investigation will be completed in the Environmental Engineering Lab following sample collection. The procedures for the method are the same despite this difference.

EPA Method 6200 is applicable for 26 analytes, including the potential COCs mentioned in Section 2.3 (EPA, 2007). XRF technology irradiates a soil sample with x-rays and dislodges inner shell electrons, causing outer shell electrons to cascade and fill in their place. This rearrangement results in emissions of x-rays for that atom/contaminant. These x-rays that come back to the XRF facilitate in the measurement of the contaminant in that soil sample (EPA A, 2007). The steps to follow for XRF analysis from EPA Method 6200 are summarized in Table B-3.

Table B-3. XRF Analysis Procedure - EPA Method 6200

Step	Description
1.	Make sure the bag is flat and the soil in the bag is evenly dispersed, then mark the bag with a 3x3 grid to separate it into 9 sections. Each section will be analyzed using the XRF one time (i.e. 9 samples per gallon bag).
2.	Place the gallon bag on the floor of the lab.
3.	Turn the XRF on and ensure it is in in situ mode. Enter the sample name, location, and team member responsible for analysis.
4.	Place the instrument directly on top of the section of the bag containing the soil sample to analyze.
5.	Pull the trigger with the device touching the bag. Keep the trigger pulled down for the entire 90 second time period. After 90 seconds, the light will flash and the shutter will close. Do not remove the XRF from the sample until the shutter is closed. Repeat for each of the 9 sections following a top left to bottom right pattern.
6.	Review the concentrations on the screen and transfer them from the XRF device to a spreadsheet on a laptop or desktop computer.
7.	Discard the single lowest and single highest values from the 9 collected values. Average the remaining for each COC.

3.3.3 Acid Digestion

An acid digestion must be completed before the samples are sent to a lab for AA analysis. If trace metals, like arsenic, lead, nickel, etc. are found, Graphite Furnace Atomic Absorption (GFAA) will be completed (NAU, 2016). If base cations, like sodium and potassium, or major metals, like zinc and iron, are found, Flame Ionization Atomic Absorption (FIAA) will be completed (NAU, 16). Only 20% of the samples (20 samples) will go through AA analysis. The method to be followed for acid digestion is EPA Method 3050B (Acid Digestion of Sediments, Sludges, and Soils). This method, although not a total digestion, is effective and will dissolve almost all elements that could become environmentally available (EPA B, 1996).

The procedure for acid digestion to prepare samples for GFAA from EPA Method 3050B is summarized in Table B-4.

Table B-4. Acid Digestion Procedure for GFAA - EPA Method 3050B

Step	Description
1.	Transfer a 1-2 gram sample (wet weight) or 1 gram (dry weight) to a digestion vessel.
2.	Add 10 mL of 1:1 HNO ₃ to the sample, mix, and cover. Heat the sample to 95°C ± 5°C and reflux, or boil so the vapor returns to the liquid after condensing, for 10 to 15 minutes. Allow the sample to cool, add 5 mL of HNO ₃ , replace cover, and reflux at same temperature for 30 minutes. Repeat this step until no brown fumes are given off by the sample. Allow the solution to evaporate to approximately 5 mL without boiling, or heat at 95°C ± 5°C for two hours. Ensure the solution is covered at all times.
3.	Allow sample to cool. Add 2 mL of water and 3 mL of 30% H ₂ O ₂ . Cover the vessel and return to the heat source to begin peroxide reaction. Heat until effervescence subsides, remove the vessel from the heat source, and let it cool.
4.	Continue to add 30% H ₂ O ₂ in 1-mL increments until effervescence is minimal or until the sample appearance is unchanged. Do not add more than a total of 10 mL.
5.	Cover the vessel and heat until the volume is reduced to 5 mL, or heat at 95°C ± 5°C for two hours. Ensure the solution is covered at all times.
6.	Let the sample cool and then dilute to 100 mL with water. Particulates in the dilute solution should be removed by filtration, centrifugation, or settling. 6a. Filter through Whatman No. 41 filter paper. 6b. Centrifugation at 2,000-3,000 rpm for 10 minutes.
7.	The sample is now ready to be sent to the lab for GFAA.

The procedure for acid digestion to prepare samples for FIAA from EPA Method 3050B is summarized in Table B-5.

Table B-5. Acid Digestion Procedure for FIAA - EPA Method 3050B

Step	Description
1.	Transfer a 1-2 gram sample (wet weight) or 1 gram (dry weight) to a digestion vessel.
2.	Add 10 mL of 1:1 HNO ₃ to the sample, mix, and cover. Heat the sample to 95°C ± 5°C and reflux, or boil so the vapor returns to the liquid after condensing, for 10 to 15 minutes. Allow the sample to cool, add 5 mL of HNO ₃ , replace cover, and reflux at same temperature for 30 minutes. Repeat this step until no brown fumes are given off by the sample. Allow the solution to evaporate to approximately 5 mL without boiling, or heat at 95°C ± 5°C for two hours. Ensure the solution is covered at all times.
3.	Allow sample to cool. Add 2 mL of water and 3 mL of 30% H ₂ O ₂ . Cover the vessel and return to the heat source to begin peroxide reaction. Heat until effervescence subsides, remove the vessel from the heat source, and let it cool.
4.	Continue to add 30% H ₂ O ₂ in 1-mL increments until effervescence is minimal or until the sample appearance is unchanged. Do not add more than a total of 10 mL.
5.	Cover the vessel and heat until the volume is reduced to 5 mL, or heat at 95°C ± 5°C for two hours. Ensure the solution is covered at all times.
6.	Add 10 mL HCl to the sample prepared in previous steps. Cover and place the vessel into the heating source to reflux at 95°C ± 5°C for 15 minutes.
7.	Filter the solution through Whatman No. 41 filter paper (or something equivalent to that) and collect the filtrate in a 100-mL volumetric flask.
8.	The sample is now ready to be sent to the lab for FIAA.

3.3.4 Atomic Absorption

The team will send the 20 samples that experienced acid digestion to the NAU Chemistry Lab located in the Wettaw Building for AA analysis. This analysis is completed as a confirmatory analysis by finding a correlation to confirm the XRF values found in earlier analyses. As the AA is subcontracted, direct methods and procedures will not be found in this SAP. A brief summary of the process is provided below.

A beam of electromagnetic radiation from excited atoms (lead, arsenic, etc.) is passed through the sample (Royal Society of Chemistry). Some of that radiation will be absorbed by the sample. The more of the atoms in the sample, the more radiation is absorbed. A calibration curve is then constructed to compare known atom concentrations with the measured concentration. The concentration of the possible COCs will then be sent back to the team to compare to XRF data.

4.0 Field Methods and Procedures

4.1 Team Briefing

Prior to sampling, all personnel will be briefed on sampling procedures, necessary personal protective equipment (PPE), calibration procedures, logging procedures, and decontamination procedures. No personnel will be permitted to work on-site without full understanding of all procedures and precautionary measures. The Health and Safety Plan (HASP) (Appendix C) details all safety considerations for this investigation.

4.2 Field Equipment

Table B-6 shows the equipment necessary to sample the soil.

Table B-6. Equipment and Supplies for Sampling

General Supplies		
Supply/Equipment	Use	Quantity
Site map and aerial photos	Used to identify sampling locations	6
Black Sharpies	Used to write on sampling containers	5
Field logbooks (Rite in the Rain Environmental)	Used to log field notes and observations	1
Handheld global positioning system (GPS) unit	Used to track sampling coordinates	1
Digital camera	Used to take photographs of sampling locations	2

Sample Collection Supplies		
Supply/Equipment	Use	Quantity
Trowel	Used to collect samples	4
Custody tape	Used to indicate sample tampering, will seal boxes that hold numerous samples	2 rolls
Custody forms	Used to list numerous samples in a single box container, will be placed in box container	8 forms
1-gallon plastic bags	Used to contain samples	250 pack, 1 per sample, spares in case of tears
Sample Location Surveying and Documentations		
Supply/Equipment	Use	Quantity
Compass	Used to determine direction	2
Measuring tape	Used to measure distances between sampling locations	2
Surveying stakes/flags	Used to mark sampling locations	100 total, 1 per sample
Decontamination Supplies		
Supply/Equipment	Use	Quantity
5-gallon decontamination waste bucket	Used to contain waste liquid	3
Bottles for distilled water, 16 oz	Used to clean sampling equipment	4 bottles
Paper towels	Used to dry sampling equipment	3 rolls
Dish soap, 1 oz	Used to clean sampling equipment	1 bottle
Scrub brushes	Used to clean sampling equipment	2

Personal Protective Equipment		
Supply/Equipment	Use	Quantity
Tyvek suits (come with overboots)	Used to protect the sampling team from exposure to contaminants	12 total, 1 per person per day, 2 spares
Nitrile gloves	Used to protect the user from exposure to contaminants, spare gloves will be brought in the event any gloves tear	400 total, 1 per person per sample

4.2.1 Calibration of Field Equipment

The handheld GPS unit will be calibrated in accordance with the frequency and methods suggested by the manufacturer. All calibration procedures will be recorded in the project logbook as deemed necessary. The GPS must be calibrated to $\pm 5\%$ of the manufacturer's standard. Failure to do so will result in invalidation of sampling identification (ID) numbers (AMEC, 2010). Prior to departing for the site, the GPS and the camera will be tested to verify they work.

4.3 Soil Sample Preparation and Collection

Surface soil sampling will be conducted using the procedures detailed in Table B-7.

Table B-7. Surface Soil Sampling Procedure

Step	Description
1.	Remove surface litter (vegetation, large rocks/pebbles).
2.	Collect surface soil sample using trowel. Surface soil samples will be collected at a maximum depth of 3 inches.
3.	Each sample will be placed into a 1-gallon plastic bag and labeled. Approximately half of the bag's volume will be filled with soil. The logbook will record the person taking the sample, location, grid number, and date/time taken.

4.4 Soil Sample Location Identification

Each sample will be assigned a unique ID number that corresponds to the location it was taken from. It will be permanently marked on the sample container with the following format: AZMG-type-#, where:

- AZMG identifies the location (AZ Magma Mine)
- Type of sample = GN for grid node, HS for hotspot, BG for background
- # corresponds to the particular sample for a sample type (1, 2, 3...)

4.5 Decontamination

Sampling equipment intended for reuse shall be decontaminated using the procedures detailed in Table B-8.

Table B-8. Decontamination Procedure

Step	Description
1.	Place equipment in 5-gallon bucket.
2.	Scrub equipment with dish soap and water.
3.	Rinse the equipment with distilled or deionized water in a second clean bucket.
4.	Dry equipment on a clean surface with paper towels and store in a third clean bucket.

5.0 Investigation Derived Waste Management

Whenever possible, generation of investigation derived wastes (IDW) will be minimized. IDW includes, but is not limited to, soil and sediment, decontamination fluids, and disposable sampling equipment/PPE (Stroh, 2012).

5.1 Water

Decontamination fluids will be discharged on-site.

5.2 Solid Waste

Disposable sampling equipment and PPE will be treated as a solid waste and disposed of in appropriate containers (Stroh, 2012). Containers with disposable equipment will be shipped back to NAU for proper disposal.

Soil samples below Arizona’s residential soil remediation levels (SRLs) will be disposed of as solid waste. Soil samples above Arizona’s residential SRLs will be retained for further use in the Environmental Engineering Lab or disposed of as hazardous waste per NAU Risk Management protocols.

6.0 Sampling Containers, Preservation, and Storage

All individual soil samples will be placed in 1-gallon plastic bags, which will be stored in plastic boxes. Samples will be double bagged if leaks/breaks are observed in the original bag. The samples will remain in the boxes until transported to the NAU campus. No preservation additives are necessary for soil samples in this operation. The holding time for all samples is 6 months, after which the samples are no longer valid (MDH, 2013).

7.0 Samples Documentation and Shipment

This section describes procedures for sample labeling, logbook field notes, chain of custody, and sample shipment.

7.1 Sample Labeling

The sequential ID number for each sample will be written on the 1-gallon plastic bag it is collected in. See Section 4.4 of the SAP for further information.

7.2 Field Notes and Photographs

For each sample, the following information will be logged in a field notebook:

- The sample's ID number
- Time at which the sample was taken
- The person who took the sample
- Visual characteristics of sample
- Other relevant observations

A photograph of each sample before and after excavation will be taken.

7.3 Sample Chain of Custody Procedures

The boxes containing numerous bags of samples will be sealed with tape when full. A Chain of Custody form listing all samples in the box will be placed in each box prior to sealing.

For samples sent to the NAU Chemistry Lab for AA analysis, the Chain of Custody form will accompany the samples. This form will inform the lab technician of the analyses requested for each soil sample. A blank version of the Chain of Custody form to be used is presented in Figure B-3.

Team Name:			Billing Information:			Analysis / Container / Preservative	
Magma Consulting							
Report to:		City/State Collected:					
Jessica Szaro, Project Manager email: jas843@nau.edu phone: (520) 519-9370		Chloride, AZ					
Sample ID	Comp/Grab	Matrix	Collected by	Date	Time		
AZMG-	Grab						
AZMG-	Grab						
AZMG-	Grab						
AZMG-	Grab						
AZMG-	Grab						
AZMG-	Grab						
AZMG-	Grab						
AZMG-	Grab						
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AZMG-	Grab						
AZMG-	Grab						
AZMG-	Grab						
AZMG-	Grab						
Relinquished by: (Signature)	Date:	Time:	Received by: (Signature)	Date:	Time:	Temperature Received:	
Relinquished by: (Signature)	Date:	Time:	Received by: (Signature)	Date:	Time:		
Relinquished by: (Signature)	Date:	Time:	Received by: (Signature)	Date:	Time:	COC Seal Intact (Y/N):	
Relinquished by: (Signature)	Date:	Time:	Received for lab by: (Signature)	Date:	Time:		

Figure B-3. Blank Chain of Custody Form

8.0 Quality Assurance/Quality Control

The quality assurance/quality control (QA/QC) plan has been developed to ensure that sample collection and data obtained are reliable and produces trustworthy results. The procedures described in the following sections ensure that field sampling, laboratory analysis, and statistical analyses will meet the data quality objectives. Failure to properly collect samples or failure to properly conduct the laboratory analysis will introduce uncertainty, reducing the reliability of the data (AMEC, 2010).

8.1 Laboratory QA/QC

QA/QC in the laboratory will be ensured through the use of standard operating procedures, frequent inspection, and complete documentation of all laboratory activities. All sample drying will be documented in a lab notebook. If necessary, a dried sample will be placed in a new plastic bag and properly labeled if the original bag contains excessive moisture. For soil sieving, each sieve will be properly cleaned and decontaminated between individual uses. All sieving will be documented in a lab notebook. For XRF analysis, all data will be recorded in a lab notebook. The highest and lowest values from an individual sample (which are excluded from the sample average, see Table B-3) will be identified in the lab notebook.

The analytical laboratory that will be contracted for the AA analysis will be asked to provide the following information in support of QA/QC (AMEC, 2010):

- Analytical method
- Method detection limit
- Practical quantitation limit
- Units of measure
- Sample collection and analysis dates
- Adherence to designated holding time
- Method blank results
- Laboratory duplicate results and relative percent difference
- Initial and continuing calibration checks

8.2 Background Samples

Background sampling will be taken from undisturbed natural areas outside of the waste site. These samples are collected in order to understand what the naturally occurring levels of the COCs are (Innis 24).

8.3 Data Analysis

XRF data will be compared to AA data using least squares linear regression. If any measured concentrations span more than one order of magnitude, the data will be log transformed. The correlation coefficient obtained from the regression analysis must be at least 0.7 for the XRF data to be suitable in a screening level risk assessment.

8.4 Data Validation

Data validation is intended to identify unreliable/invalid laboratory measurements caused by improper sample collection procedures or analytical laboratory methods. Data validation will follow the guidelines set by the EPA for inorganics. The following steps will be taken to validate laboratory analytical data (AMEC, 2010):

- Chain of Custody forms and laboratory reports will be checked to ensure that the samples were analyzed for the correct parameters and methods. Samples that were not analyzed with the correct method or the correct contaminant will be flagged.
- Samples that have a dissolved metal concentration that is larger than the total metal concentration will be flagged because dissolved concentrations should never exceed total concentrations.
- Any data points noted to have potential transcription error, anomalous, and omitted data will be flagged.

All data points that pass the criterion listed above will be entered into the project database. Potential data limitations include, but are not limited to (AMEC 2010):

- Unreported values
- Measurement procedure uncertainty
- Detection limits that change throughout the analytical period
- Small sample size
- Sample outliers
- Improper quality control procedures
- Improper data validation procedures
- Improper data presentation/transcription

Data with limitations are still suitable for analysis given that the user considers the limitations (AMEC, 2010).

9.0 References

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