

Sampling and Analysis Plan for the Viburnum Trend Lead Mining District Transition Zone Assessment Study

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DRAFT

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**MISSOURI
DEPARTMENT
OF NATURAL
RESOURCES**



Southeast Missouri Lead Mining District

Natural Resource Damage Assessment and Restoration

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Appendix A – Health and Safety Plan

Appendix B – Sampling Area Field Sheet & Soil Sample Log Form

Appendix C – XRF Standard Operating Procedures

LIST OF ACRONYMS

CERCLA	Comprehensive Environmental Response Compensation and Liability Act
COC	Chain of Custody
CSM	Conceptual Site Model
DI	Deionized Water
DQO	Data Quality Objectives
ESP	Environmental Services Program
HWP	Hazardous Waste Program
ICS	Incremental Composite Sampling
IDW	Investigation Derived Waste
MDNR	Missouri Department of Natural Resources
MS&T	Missouri University of Science and Technology
NRDA	Natural Resources Damage Assessment
OPA	Oil Pollution Act
PPE	Personal Protective Equipment
QA/QC	Quality Assurance / Quality Control
QAPP	Quality Assurance Project Plan
SEMOLMD	Southeast Missouri Lead Mining District
SOP	Standard Operating Procedure
SU	Sampling Unit
UCL	Upper Confidence Limit
US FWS	United States Fish and Wildlife Service
VTLMD	Viburnum Trend Lead Mining District
XRF	X-ray Fluorescence

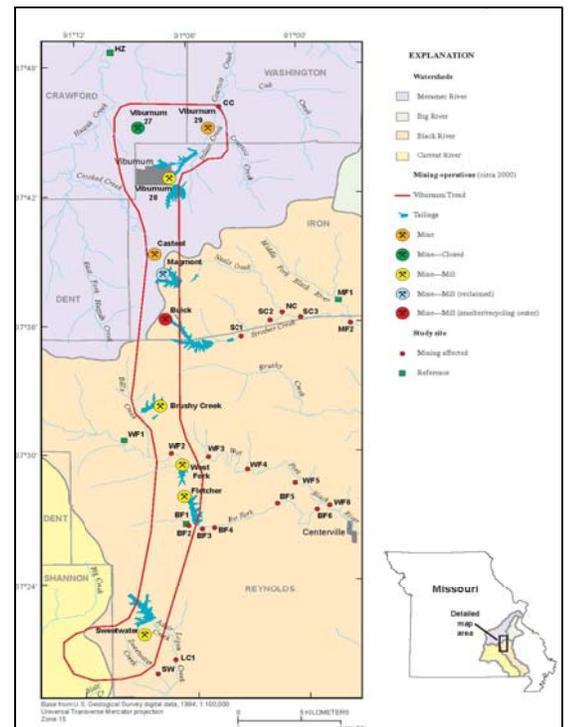
1.0 Introduction

The Comprehensive Environmental Response, Compensation, and Liability Act of 1980 (CERCLA), as amended, and the Oil Pollution Act (OPA) provide for the restoration of natural resources lost or injured by hazardous substance releases (§107(f) and discharges of oil (§1006) and require the designation of certain Federal and State officials to act on behalf of the public as Trustees for natural resources. The Missouri Department of Natural Resources (MDNR) together with the U.S. Fish and Wildlife Service (USFWS), acting as Trustees, are conducting a Natural Resource Damage Assessment (NRDA) in the Viburnum Trend Lead Mining District (VTLMD) in Southeast Missouri. The purpose of conducting a NRDA is to identify and document the extent of injuries to natural resources, quantify the injuries and all associated losses to the public and select restoration projects.

2.0 Site Information

2.1 Location

The VTLMD, also known as the New Lead Belt of Southeast Missouri is a narrow band oriented north-south across parts of Washington, Crawford, Iron, Reynolds, and Shannon Counties in southeast Missouri (Figure 1). The VTLMD lies on the western edge of the Ozark Dome in Missouri and is part of the greater Southeast Missouri Lead Mining District (Figure 2).



2.2 Description

The VTLMD is an active lead (Pb) and zinc (Zn) mining district within the Southeast Missouri Lead Mining District (SEMOLMD). Mining in VTMD is conducted by the Doe Run company, the largest producer of Pb in the nation (Wikipedia, 2012). Topography in the New Lead Belt is characterized by rolling hills dissected by narrow floodplain, creek, and river valleys. Hills and ridges are generally steep sided with flat tops consisting of thin mantles of clayey soils. The major physical features of the area are the St. Francois Mountains in the east, and the dissected topography of the Salem Plateau in the northern and central portions. The principal drainage systems for the VTLMD are the north flowing Courtois and Huzzah Creeks and their tributaries and the east and south flowing Black River and its tributaries.

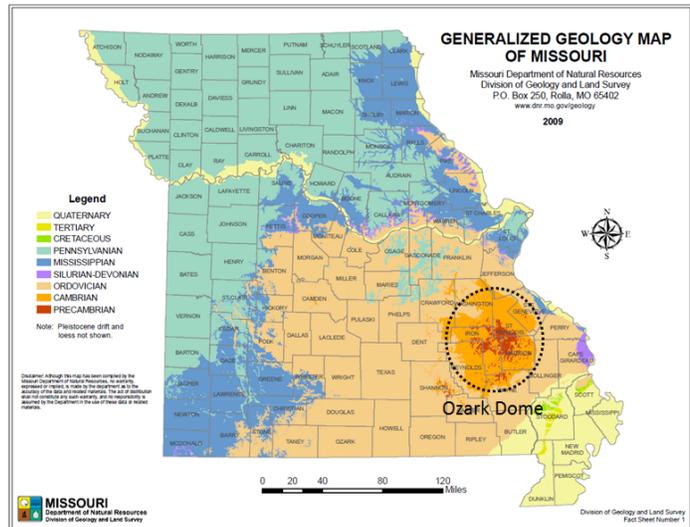


Figure 2. Ozark Dome

Mining in the VTLMD is ongoing, and the district remains a major producer of metals: Missouri's mines have yielded much of the United States' national production of lead, and since 1997, all the lead produced in Missouri originated in The Doe Run Company's Viburnum Trend mines. In addition to lead, the mines produce substantial amounts of zinc and lesser quantities of copper and silver (Seeger 2008).

Mining operations within the VTLMD have been conducted at a number of facilities including Buick, Fletcher, Magmont, West Fork, Bushy Creek, Sweetwater, and in the "new" Viburnum Trend area. Tailings impoundments associated with each of these areas are shown in Figure 3.

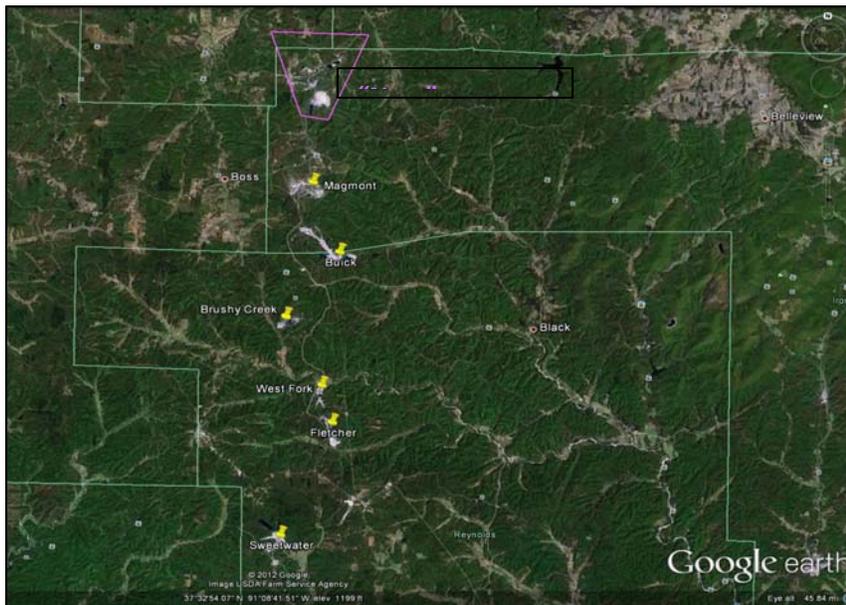


Figure 3. Mining Operations and Associated Tailings Impoundments Within the VTLMD.

Mining in the Viburnum Trend has consisted entirely of the room and pillar method along the ore trend (Seeger 2008). Ore is first crushed within the mine, and then removed to the surface for beneficiation, including further crushing. Following the crushing, the slurried fine material is sent to flotation cells and separated into mineral concentrates through circuits specific to each metal (lead and zinc at all mills, copper as well at all except Sweetwater and West Fork). The mineral concentrates are settled and dewatered before further action (either transport to the smelter or to a buyer). The resultant waste is collected in tailings ponds along with all process water. Unlike earlier operations in the SEMOLMD, the VTLMD operations never used density separation methods, which result in chat piles (Seeger 2008).

2.3 History/Contaminants of Concern

Releases of heavy metals from the tailings impoundments, mines, and smelter in the VTLMD have been documented by previous investigations. Elevated levels of cadmium, copper, lead, nickel, and zinc have been measured in surface and shallow subsurface soil at facilities within the VTLMD. Based on comparison of the concentrations measured relative to common screening benchmarks discussed below, the primary contaminant of concern for this NRDA will

be lead. The Missouri Trustees for Natural Resource Damage Assessment and Restoration (Trustees) believe that there may be potential injuries to natural resources in parts of Crawford, Washington, Reynolds and Iron Counties, Missouri resulting from releases of hazardous substances from the VTLMD. Consequently, the Trustees have jointly designed a plan to study the extent of the potential release of heavy metals from the tailings impoundments in the VTLMD.

3.0 Data Quality Objectives

To help ensure precise, accurate, representative, complete, and comparable data, all field work and analyses will be conducted in accordance with the Quality Assurance Project Plan (QAPP) for Natural Resource Damages, Revision 3, April, 2011 (MDNR, 2011). The QAPP describes the general data quality objectives (DQO) for site assessment investigations conducted by the Trustees. Those DQOs specific to this project are described below.

3.1 Problem Statement

Previous investigations have measured levels of lead exceeding 3,000 mg/kg in surface soil at tailings impoundments in the VTLMD. These levels far exceed most ecological screening benchmarks for lead in soils including USEPA's Ecological Soil Screening Level of 120 mg/kg and remedial action level for residential properties of 400 mg/kg (USEPA, 2005). Therefore, there is concern that lead released from the tailings impoundments has resulted in damages to terrestrial natural resources in the VTLMD area.

3.2 Planning Team

The planning team includes staff from the MDNR Hazardous Waste Program (HWP), Environmental Services Program (ESP), and USFWS, Columbia, Missouri Field Office.

3.3 Conceptual Site Model (CSM)

Mining-related metals can be released from the tailings impoundments and other mining/milling activities in a number of ways. The primary transport mechanism for metals to the terrestrial environment surrounding the tailings impoundments is assumed to be via wind. Therefore a higher soil concentrations and a further extent of influence is expected in the predominant downwind directions at each impoundment. Landscape features, surface water runoff, soil type and vegetative cover will also affect the deposition and fate of mine waste released from the tailings impoundments.

3.4 Study Questions

The area of metals-contaminated soil around tailings impoundments within which metal concentrations transition either to values expected for uncontaminated native soils or to relevant ecological soil screening levels of concern is referred to as the “transition zone”. For this project, an estimate of lead concentrations in native uncontaminated soils will not be made. Rather, the lowest identified relevant ecological soil screening level of 94 mg/kg Pb (Mosby, 2012) will be used to define the outer boundary of the transition zone. Thus, the transition zone will be defined as the land area surrounding each tailings impoundment within which the lead concentration in the <2 mm particle size fraction of the upper 5 centimeter (cm) of surface soil exceeds 94 mg/kg. It is also of interest for this study to identify the area around each impoundment where the lead concentration exceeds a 490 mg/kg level of concern (Sample *et al.*, 2011). The principle study questions are:

1. What is the average distance outward from each tailings impoundment at which the average lead concentration in the <2mm soil particle fraction of the top 5 cm drops below 490 mg/kg;
2. What is the average distance outward from each tailings impoundment at which the average lead concentration in the <2mm soil particle fraction of the top 5cm drops below 94 mg/kg; and

3. What is the mean lead concentration in the <2mm particle fraction of the top 5 cm of soil in a.) the area bounded by the edge of the tailings impoundment and 490 mg/kg isoconcentration contour, and b.) the area between the 490 mg/kg and 94 mg/kg isoconcentration contours.

3.5 Inputs to Study Questions

The following lists the primary inputs required to address the principal study questions.

Results of lead analyses will be compared to various ecological soil screening levels including:

- 94 mg/kg: Calculation of soil concentration that exceeds Lowest Observed Adverse Effect Level to mean Toxicity Reference Value for biochemical endpoint for American Robin (*Turdus migratorius*) (Mosby 2012)
- 490 mg/kg: Lowest ground-feeding songbird preliminary remedial goals (Sample *et al.*, 2011).
- Estimates of mean lead concentrations
 - In surface soil at various distances away from each tailings impoundment along transects established perpendicular to the impoundment (via in-situ XRF analysis);
 - In the fine fraction (<2 mm of soil from the top 5 cm within sampling units (SUs) established at each tailings impoundment (via ex-situ XRF & lab analysis);
- Estimates of variability in lead concentrations in each SU sampled

3.6 Study Boundary

The VTLMD tailings impoundments to be assessed are shown in Figure 3. An attempt will be made to conduct soil sampling at each of the active and historic tailings impoundments within

the VTLMD, excluding the Magmont Joint Venture tailings impoundment. That impoundment has been closed and covered with a cap of soil from the surrounding hillsides. Impoundment closing activities associated with creating the cap have likely altered the metals content of surrounding transition zone soils; thus the Magmont impoundment will be excluded from this study.

Terrestrial ecological receptors may be exposed to contaminated soils at different depths depending on the organism, habitat use and life cycle. For this assessment, the focus will be on lead contamination in the upper 5 cm of soil because wind borne transport results in surface deposition and the presumption is that it has been going on sufficiently long that the top 5 cm will be representative of the deposition. Generally, particles greater than 2 mm (rocks, organic duff layer, etc.) are not considered to be part of 'soil', and will not be included in the assessment.

Soil sampling will take place in 2012 over the course of two to three potentially non-consecutive weeks.

3.7 Tolerable Limits on Decision Error

The hypothesis is that soil lead concentrations taper off with distance from the tailings impoundments to a point where they approach either the native background soil level or the identified ecological screening level of concern. Measures of the extent of this influence will be used to estimate affected areas and mean concentrations which serve as input to the habitat equivalency analysis model; the tool used to monetize natural resource damages. Therefore, accurately identifying the extent of influence from each tailings impoundment is critical. Underestimating the true extent would lead to underestimating resource damage and potentially to inadequate allocation of funding and restoration resources. Overestimating the extent of influence would lead to an unrealistically high estimate of resource damage, potentially causing allocation of more funding and resources than actually necessary.

A sampling design has been chosen to control overall sampling error and minimize the likelihood of decision error. In-situ X-ray fluorescence (XRF) analyses will be used to obtain a high density data set about concentration gradients for lead with distance from each impoundment. The use of field analytical techniques such as XRF allows much higher density data sets than is practical with conventional discrete soil sample/laboratory analysis. Large data sets will increase confidence about conclusions drawn from the data and decrease the likelihood of committing either type of error. In-situ sampling density will be increased when results indicate that concentrations are approaching important decision criteria, further increasing decision quality and reducing likelihood of decision error.

An incremental composite sampling (ICS) approach will be used to collect soil samples within sampling units (SUs) established in the field based on the in-situ XRF results. ICS is designed to obtain a single soil sample that contains contaminants in the same proportion in the sample as they are present in the volume of soil of interest (i.e. are representative samples). This is achieved through the collection of many increments of adequate-mass systematically across each SU from throughout the volume of interest. Representative sampling will decrease the likelihood of committing either type of error.

The ICS sampling procedure will be replicated and results of the replicates will be used to provide a measure of variability required to calculate a conservative estimate (95% Upper Confidence Level (UCL)) of the true mean lead concentration. By using a conservative estimate of mean concentration, we will protect against underestimating the true mean, and therefore of underestimating the extent of natural resource damages.

3.8 Sampling Design

A combination of in-situ XRF analysis and ICS will be used to estimate the extent of influence away from the tailings impoundments and to estimate mean concentrations within specific isoconcentration contours. Since it is not feasible to sample the entire area surrounding each impoundment, smaller sampling areas will be established around each impoundment and

sampled. The results from these sampling areas will be used to represent conditions in the larger areas around the impoundments. For this project, rectangular 20-meter (m) wide sampling areas will be established at each impoundment. The number of sampling areas will depend on the size of the impoundment as shown in Table 1. Fewer sampling areas may be used at each impoundment depending on field conditions and access considerations.

Table 1. Permitted Acreages of Tailings Impoundments and Sampling Area Goals

Tailings Impoundment	County	Permitted Acreage	Estimated # Sampling Areas
“New” Viburnum Tailings	Washington, Iron, and Crawford	1008 acres	8
Buick Mine	Reynolds and Iron	1015 acres	8
Brushy Creek Mine	Reynolds	242 acres	4
West Fork Mine	Reynolds	264 acres	4
Fletcher Mine	Reynolds	435 acres	6
Sweetwater Mine	Reynolds	850 acres	8

A two-step sampling design will be used as shown in Figure 4. First, sampling areas will be positioned to avoid other anthropogenic influences that may affect the results such as roads, buildings, or drainage culverts. The locations of the 490 mg/kg and 94 mg/kg isoconcentration

contours will be estimated within each sampling area using in-situ XRF analyses conducted at various distances along a transect established within each area perpendicular to the impoundment. The density of XRF readings will be increased as concentrations near the two isoconcentration contour targets are observed. Once the isoconcentration contours are established, they will be used to divide the sampling area up into two SUs. In the second step, an ICS consisting of at least 30 increments will be collected from equally-spaced locations across each SU. If an SU area exceeds ½ acre (60 m x 200 m), then 50 increments will be collected. Replicate ICS will be collected as described in Section 5.2.4. Each SU ICS will be air dried, disaggregated, sieved to <2 mm, and analyzed by XRF. The SU ICS results will be used to calculate an estimate of mean lead concentration for the area within each of the 2 isoconcentration contours of interest.

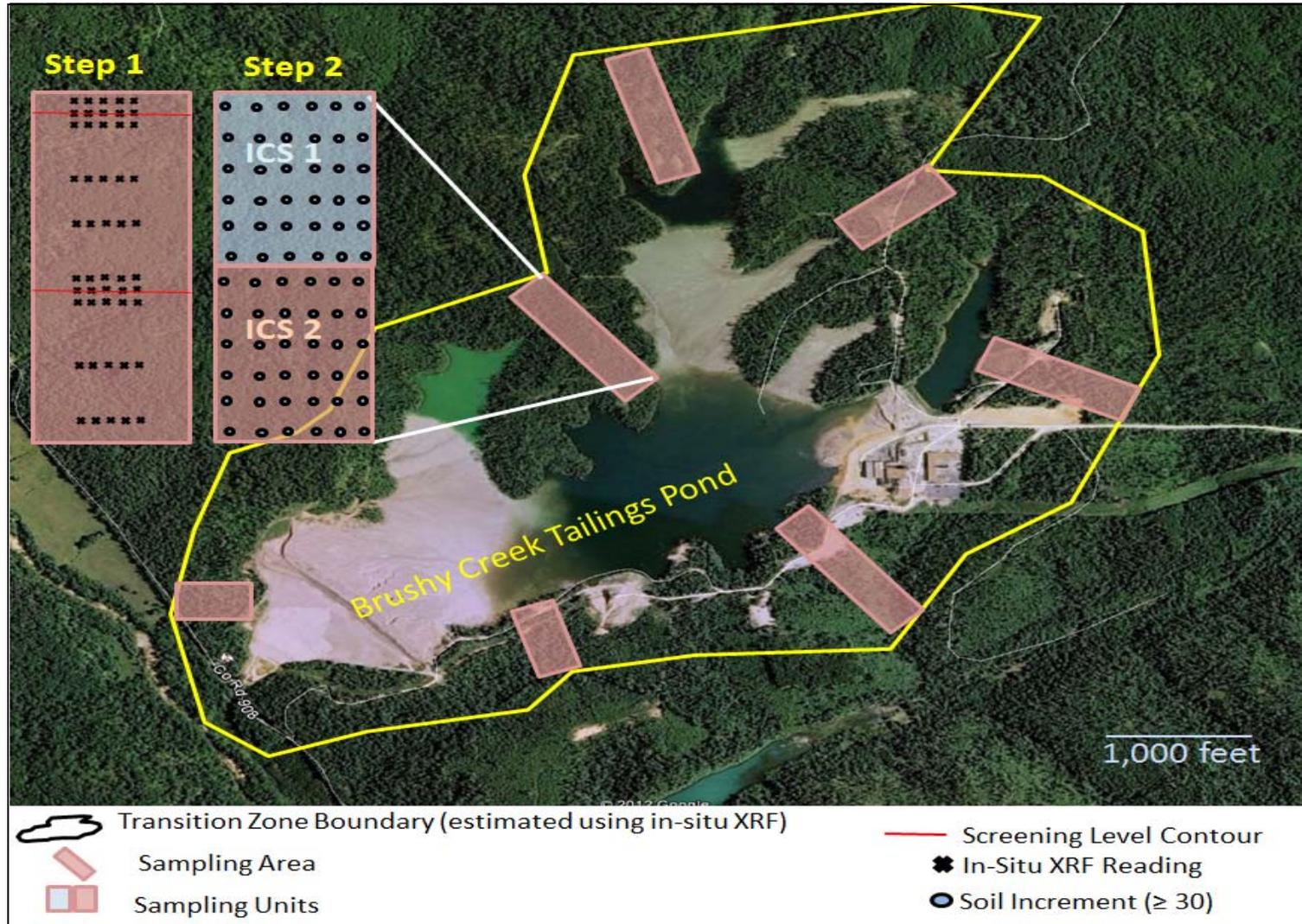
4.0 Field Activities

Sampling will be conducted in three teams of 2-3 personnel. In-situ XRF analysis for lead will be performed along transects perpendicular to the impoundments at various distances in order to identify spatial trends in lead concentration. Locational point data will be collected at each distance interval along each transect. Approximately 90 ICS will be collected from SUs established at the impoundments. Each IC sample will be conditioned and analyzed for lead by XRF as describe in 4.3. Confirmatory laboratory analyses will be conducted on a subset of samples.

4.1 Soil Sampling

Except as otherwise noted, all aspects of sampling shall be performed using standard operating procedures (SOPs) referenced in the QAPP (MDNR, 2011). Modifications to the following sampling methods are described below and others may be made in the field based upon conditions encountered. Any modifications to the methods will be noted on the field sheets and in the Study Report.

Figure 4. Sampling Design



4.1.1 In-Situ XRF Analysis

The XRF analyzer will be calibrated and standardized as per the manufacturer's instruction. Known reference standards containing certified concentrations of lead at various levels will be analyzed prior to initiating field work as specified in the XRF SOP (Appendix C). Rectangular 20-meter wide sampling areas will be established perpendicular to the impoundment based on field observations, accessibility, landscape features, roadways, and other practical considerations. A single transect will be established in the approximate center of the sampling areas extending outward perpendicular to the impoundment. At each distance interval any surface vegetation or duff layer will be removed. Any soil clinging to the vegetation roots will be shaken back out over the bare soil. The soil will be flattened out, large debris (rocks, sticks) removed, and a 30 second in-situ analysis will be performed. The impoundment name, sampling area #, transect distance from impoundment and analyst name will be entered into the XRF prior to each analysis. Four additional in-situ analyses will be conducted at the same distance interval perpendicular to the transect at 5 foot spacings as shown in Figure 4.

The results will be recorded on the field sheet provided in Appendix B, and the averages for each distance interval will be calculated. When average lead concentration along a transect drops below the level of concern for lead (490 mg/kg or 94 mg/kg), additional readings at transect distances slightly closer to and further from the impoundment will be made in order to better identify the distance at which to establish the isoconcentration contour (and therefore the SU boundary). Flags will be placed to mark the boundary of the SU for that transect. The spacing of XRF readings along each transect and the density of in-situ readings may be modified based on field conditions and access considerations.

4.1.1 Surface Soil Sampling

Once the boundaries of each SU in the sampling area has been determined using in-situ XRF analysis, Surface soil ICS samples will be collected as follows. An EVS™ or EnviroStat™ stainless steel incremental sampling tool will be used to collect equal-mass increments of soil at equal spacing across each SU. A 4-cm diameter, 5-cm long stainless steel sampling core will be used. The sampling core will be advanced into the soil and ejected into a 2-gallon size heavy duty resealable plastic bag. Alternative sampling tools may be used as soil conditions warrant and will be documented. This process will be repeated at each increment collection location, and all increments will be combined together into one bag for each SU.

Increments will be collected using a systematic grid approach. The soil increments forming the ICS will be collected from equally spaced locations across the entire SU. Table 2 will be used to aid in determining increment spacing. Distance between increments may be measured simply by pacing approximate distances along rows and columns set up along the SU boundaries. Bags will be labeled with the sampling area #, SU# date, time, and sampler collector's initials. Each sample will be recorded on the soil sample log form shown in Appendix B. Replicate ICS will be collected in identical fashion except that increments will be located in independent systematically determined locations as shown in Figure 5.

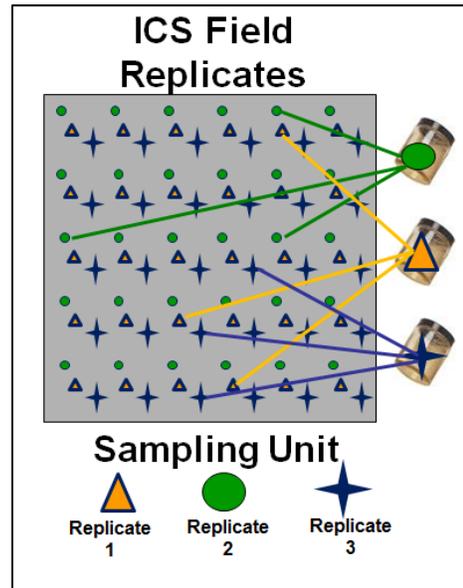


Figure 5. ICS Field Replicates

Table 2. Increment Spacing Vs. SU Area

SU Area (ft ²)	*Approximate Increment Spacing vs Desired Number of SU Increments		
	30 Increments	40 Increments	50 Increments
100	2.2	1.9	1.6
200	3.2	2.7	2.3
300	3.9	3.3	2.9
400	4.5	3.8	3.3
500	5.0	4.2	3.7
1,000	7.1	5.9	5.2
2,000	10	8.4	7.4
3,000	12	10	9.0
4,000	14	12	10
5,000	16	13	12
10,000	22	19	16
20,000	32	27	23
30,000	39	33	29
40,000	45	38	33

* To assist in even increment spacing for rectangular SU. Actual spacing will depend on SU shape and field conditions . Values based on formula:

$$IncrementSpacing = \sqrt{\frac{DU Area}{(\# Increments - 1)^2}}$$

ICS samples will be conditioned as described in Section 4.3 and analyzed for lead by XRF. A portion of the samples will also be submitted for laboratory confirmation analysis.

4.2 Sample Conditioning and Analysis

Soil and sediment samples will be returned to the laboratory, manually disaggregated and air dried in aluminum pans lined with wax paper to a soil moisture level that allows samples to pass through sieve without clinging to sieve mesh. Throughout the drying process, staff will periodically manually disaggregate clumps of soil to aid in drying and prevent the formation of dense clumps of dried soil. The air dried samples will be returned to their original bags, placed inside an additional bag, and then further disaggregated by striking the sample 30 times with a rubber mallet. The soil sample will then be passed through a 2mm sieve to obtain the target particle size. The soil passing through the sieve will be placed inside a thin-walled resealable plastic baggie. Sieves will be decontaminated between samples by scrubbing with a nylon bristle brush and wiping with a damp paper towel. XRF analysis will be conducted on the dried/sieved soil samples following the XRF SOP in Appendix C. Note that sample conditioning may occur either at the MDNR laboratory, or this work may be subcontracted out to the Missouri University of Science and Technology (MS&T) laboratory in Rolla.

4.3 Number of Samples, and Container and Preservation

The actual number of soil samples to be collected will depend on observations made in the field, but is anticipated to be approximately 90 incremental composite samples will be collected from the impoundment sampling areas. Of those, approximately 10%, or approximately 10 samples will be submitted to the laboratory for confirmatory analysis of lead, cadmium, copper, nickel, and zinc following XRF analysis.

The soil samples will initially be collected in 2-gallon resealable plastic bags for transport to the lab. Soil metal concentrations from mining are stable relative to time frames measured in months. Time considerations at this scale should not have a significant effect on results. Samples will therefore not be maintained on ice in the field or refrigerated during sample conditioning or XRF analysis.

4.4 Chain-of-Custody

The ICS soil samples will be stored in the plastic bags in which they were collected. Each bag will be labeled with the impoundment name, sampling area #, SU #, date, time, and collector initials using permanent marker. The samples will be recorded on the Soil Sample Log Form shown in Appendix B. The samples will remain in the custody of ESP and/or MS&T personnel during sample processing and XRF analysis. Those samples identified for laboratory analysis will be placed into appropriate sample containers and entered onto an ESP chain of custody form to be relinquished to a sample custodian at the MDNR laboratory for analysis.

5.0 Quality Control

5.1 Field Decontamination

Clean disposable latex gloves will be worn by sampling personnel and clean or field decontaminated equipment will be utilized for each separate SU to minimize the possibility of cross-contamination. Reusable soil sampling equipment will be cleaned between sampling units as follows:

- Scraping with putty knife or similar tool to remove soil clumps;
- Brushing with stiff-bristle nylon brush to remove visible soil debris;
- Cleaning with soapy water and further brushing;
- Rinsing tool with deionized (DI) water;
- Wiping dry with clean paper towels

5.2 Quality Assurance/Quality Control (QA/QC) Samples

The following samples will be collected as part of the quality control/quality assurance procedures for the investigation.

5.2.1 Equipment Rinsate Blank

An equipment rinsate blank will be collected after decontaminating the soil coring tool between SUs once per day of sampling. Following decontamination of the tool, the core cylinder will be rinsed with DI water into a sample container which will travel with the other samples back to the laboratory for analysis.

5.2.4 Replicate Field ICS

Replicate ICS will be collected to measure precision of the overall soil sampling and analysis process, and to provide data for calculating a conservative estimate of the mean concentration. Based on the CSM, it is assumed that the release mechanism among the various impoundments is similar, and therefore we expect similar levels of contamination and contaminant variability within the SU types across different impoundments. Therefore, rather than collect replicate ICS in each SU to measure variability, we will collect 3 replicate ICS in 20% of the SUs sampled at each impoundment. After the initial ICS is collected, two additional ICS will be collected in an identical manner, except the increment locations for each replicate ICS will be collected from a different set of evenly spaced locations across the SU. The replicate samples will be labeled as “replicate 1”, “replicate 2”, and “replicate 3”. SUs will be chosen for replicate sampling based on in-situ XRF results, SU size, terrain features, and other field observations. One set of replicates will be collected at each impoundment in the SUs within the sampling area oriented closest to the predominant downwind direction from the impoundment. Emphasis will also be placed on selecting SUs for replicate sampling that contain a mixture of upland and swale or floodplain features, and on obtaining replicates from both relatively small and large SUs. The replicate data from each SU will then be examined using ProUCL and spreadsheet statistical tools. As deemed appropriate, variability will then be extrapolated from SUs with replicate samples to those without for purposes of estimating uncertainty and upper confidence limits.

A relative standard deviation less than 30% will generally indicate acceptable precision; however the degree of precision required depends on how close the estimated mean concentration is to the screening level. Thus, more data variability may be tolerable if the concentrations measured are either well above or well below the screening levels.

5.2.5 XRF Precision Samples

The precision of bagged sample XRF analyses will be evaluated by conducting multiple analyses of selected samples at a frequency of 5% for bagged sample analysis and once per day per analyzer for in-situ analysis. For bagged samples, the precision samples will be selected based on lead concentrations. Samples will be chosen to reflect the full range of concentrations observed, however, special emphasis will be placed on selecting samples near the screening levels if possible. The selected sample will be analyzed seven separate times without moving the bagged sample (without moving the analyzer for in-situ analysis) between each analysis. The relative standard deviation among the multiple analyses will be assessed as an indication of instrument precision.

The precision of in-situ XRF analyses will be evaluated by conducting multiple analyses at selected locations at a frequency of once per day per instrument. At the selected location, the analyzer will be placed on the soil surface, and seven consecutive 30-second XRF analyses will be conducted without moving the analyzer between tests. The relative standard deviation among the multiple analyses will be assessed as an indication of instrument precision.

5.2.6 Laboratory Subsampling Replicates

Laboratory subsample replicates will be requested on selected ICS samples submitted to the laboratory. The laboratory will subsample the container two times and conduct two separate analyses. This procedure measures the within-sample container matrix heterogeneity and the

error associated with subsampling the container. Samples will be identified for replicate subsampling by indicating in the "comments" field of the ESP COC form.

5.2.7 Laboratory QC

Laboratory precision and accuracy will be assessed as described in the Quality Assurance Project Plan (QAPP) for Natural Resource Damages, Revision 3, April, 2011.

6.0 Investigation Derived Wastes (IDW) Plan

Efforts will be made to minimize IDW generation. IDW may include soil, sediment, decontamination fluids, disposable sampling equipment, and disposable personal protective equipment (PPE).

Field personnel will attempt to return unused soils to their source immediately after generation or, if warranted, containerize and return to the ESP laboratory for proper disposal. Disposable PPE and disposable sampling equipment will generally be handled as solid waste, containerized, and properly disposed. Wash and rinse waters generated during equipment decontamination will generally be discharged to the ground on-site or, if warranted, containerized and returned to the ESP laboratory for proper disposal.

7.0 Site Safety

A safety briefing will be held on-site prior to initiating field activities and field personnel will be required to read and sign the site-specific health and safety plan. The site safety plan is attached as Appendix A.

8.0 Reporting

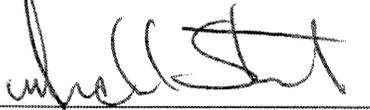
VTLMD Transition Zone Sampling and Analysis Plan

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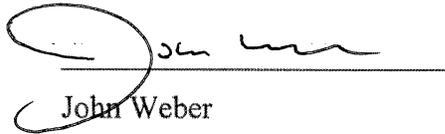
MDNR ESP will provide a copy of the chain of custodies and laboratory result sheets. MDNR HWP and US FWS will prepare a Study Report and post it to each agency's websites.

SIGNATURES

Prepared by:

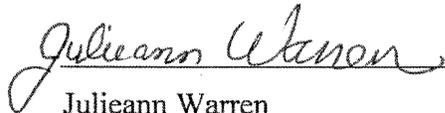


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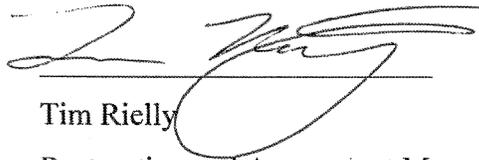


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U.S. Fish and Wildlife Service
Columbia Missouri Field Station

Approved By:



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APPENDIX A
Health & Safety Plan

MISSOURI DEPARTMENT OF NATURAL RESOURCES
DIVISION OF ENVIRONMENTAL QUALITY
ENVIRONMENTAL SERVICES PROGRAM

SITE HEALTH AND SAFETY PLAN

1.0 INTRODUCTION

This plan has been prepared for implementation by DNR employees, using operating procedures for which they are specifically trained. Any use of the plan by other agencies, organizations, or private individuals is at their own risk.

2.0 KEY PERSONNEL

MDNR OSC: Kenneth Hannon SAFETY OFFICER: Kenneth Hannon

OTHER MDNR PERSONNEL/TITLE:

Frances Klahr, Natural Resource Damages Coordinator

Amy Wright, Natural Resource Damages Analyst

Meagan Prestegard, Environmental Engineer

Sean Counihan, Environmental Specialist

Ben Frissel, Environmental Specialist

3.0 SITE INFORMATION

Site name Viburnum Trend Lead Mining Sites (VTLMS) County/City: Iron, Crawford, Washington, Reynolds, Dent, Shannon

Sampling date: 8/29/11 Through 9/2/11

Site Description: Historical mining activities impacting streams

3.1 Overall Incident Risk/Hazard Analysis

Chemical: Serious Moderate XX Low Unknown

Physical: Serious Moderate XX Low Unknown

3.2 Contaminant(s) of Concern: Lead (Pb), Cadmium (Cd), Copper (Cu), Nickel (Ni), Zinc (Zn), and Arsenic (As).

3.2.1 Physical State: XX Liquid XX Solid Sludge Gas/Vapor

Chemical Characteristics: (check all that apply)

- a. ___ carcinogen ___ b. biological ___ c. corrosive ___ d. combustible
 e. ___ explosive ___ f. flammable ___ g. volatile XX h. poison
 i. ___ radioactive ___ j. reactive k. other: _____

Physical Hazards: (check all that apply)

- a. ___ overhead ___ b. below grade ___ c. confined space* ___ d. noise
 e. XX splash ___ f. fire/burn ___ g. puncture XX h. heat stress
 i. ___ cut XX j. slip/trip/fall ___ k. cold stress ___ l. electrical
 m. ___ mechanical/heavy equipment XX n. other: animals, insects, plants.

* The need for confined space entry by ESP personnel shall be evaluated on a site-by-site basis. A confined space entry permit must be signed by the appropriate Unit or Section Chief prior to ESP employees entering a confined space (29 CFR 1910.146). Confined space entry shall be screened in at least Level B prior to downgrade. **Adequate resources must be available and specific planning and tasks determined before confined space entry is initiated.**

3.3 Task-Specific Risk Analysis (attach additional sheets as necessary)

Task Description	Chemical Hazards	Physical Hazards	Level of Protection
Sample Collection	h	e, h, j, n	D

4.0 MEDICAL SURVEILLANCE AND PERSONNEL TRAINING REQUIREMENTS

All ESP field personnel participate in a medical monitoring program and are trained at least to the level of "Hazardous Substance Emergency Response-Technician" as required and specified in the department's written health and safety program located in Section 2 of the MDNR-Hazardous Substances Emergency Response Plan

(HSERP). The written policy satisfies requirements set out in 29 CFR 1910.120. MDNR ESP's respiratory protection program meets the requirements of 29 CFR 1910.134.

ESP personnel will ascertain as much information as possible regarding health and safety issues associated with the site prior to initial entry. Information shall include chemical and physical hazards as listed above, types and amounts of materials involved, and citizens/areas threatened by the incident.

5.0 PERSONAL PROTECTIVE EQUIPMENT

ESP shall utilize the Protection Level categories defined in 29 CFR 1910.120, Appendix B, and known as Levels A, B, C, and D. Refer to Section 2 of the MDNR-HSERP for definitions of Protection Levels. ESP personnel shall inspect APRs and SCBAs at least monthly and maintain a record of such to ensure equipment is functional.

Levels of protection shall be reassessed and upgraded as conditions change and information is updated to comply with worker safety while performing site activities.

Action Levels for evacuation of work zone pending reassessment of conditions:

Level D: O₂ < 19.5% or > 25%; explosive atmosphere > 10% LEL; organic vapors > background levels; other_____.

Level C: O₂ < 19.5% or > 25%; explosive atmosphere > 20% LEL; organic vapors (in breathing zone) > 5 m.u.; other_____.

Level B: O₂ Explosive atmosphere > 20% LEL; unknown organic vapors (in breathing zone) > 500 m.u.; other_____.

Level A: ESP personnel shall evaluate the need for entry on a site-specific basis and may utilize its emergency response contractor for Level A situations which may arise.

6.0 FREQUENCY AND TYPE OF AIR MONITORING/SAMPLING

Instrument	Contaminant of Concern	Sample Location (Area/Source)	Frequency	Odor Threshold/ Description
NA				

7.0 SITE CONTROL MEASURES

7.1 The "Buddy-System": ESP personnel performing any work activities within the exclusion zone shall employ the "buddy-system" at all times, as required and defined in Section 2 of the MDNR-HSERP. The "buddy-system" may not be required while an ESP staff member is observing or providing oversight of cleanup activities performed by a contractor or responsible party.

7.2 Safe work Practices: Refer to Section 2 of the MDNR-HSERP for written safety practices to be followed at all times by ESP personnel while on-site at an incident.

7.3 Site Communications: The use of two-way radios or establishment of hand signals for communications shall be determined prior to entering the work zone and followed by ESP personnel.

7.4 Radiation Safety: Due to the possibility of an unknown radiation hazard being present on a site, ESP personnel shall be required to wear radiation indicator badges (TLD badges) while on-site.

7.5 Work Zones: ESP personnel shall ensure work zones are established and be aware of their locations.

8.0 DECONTAMINATION PROCEDURE/SOLUTIONS:

Personnel: Gloves and clothing will be placed in a garbage bag and returned to Jefferson City for proper disposal.

Equipment: Returned to Jefferson City for proper decontamination.

Instruments: Returned to Jefferson City for proper decontamination or disposal.

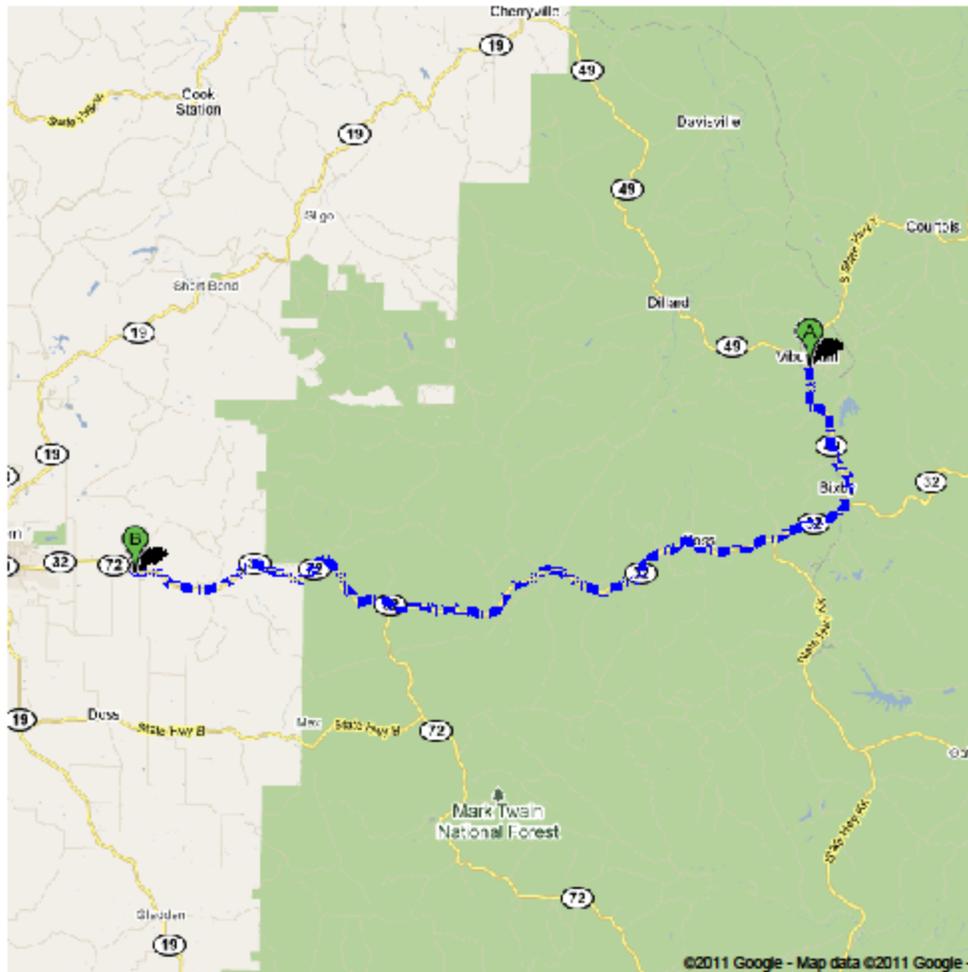
Decontamination fluids/materials may be containerized for proper disposal.

9.0 EMERGENCY INFORMATION:

In the event of an emergency, notify the MDNR Environmental Emergency Response Office at 573/634-2436. The Duty Officer will make the appropriate notifications.



Directions to Missouri 72, Salem, MO 65560
27.5 mi – about 32 mins



APPENDIX B

Field Sheets:

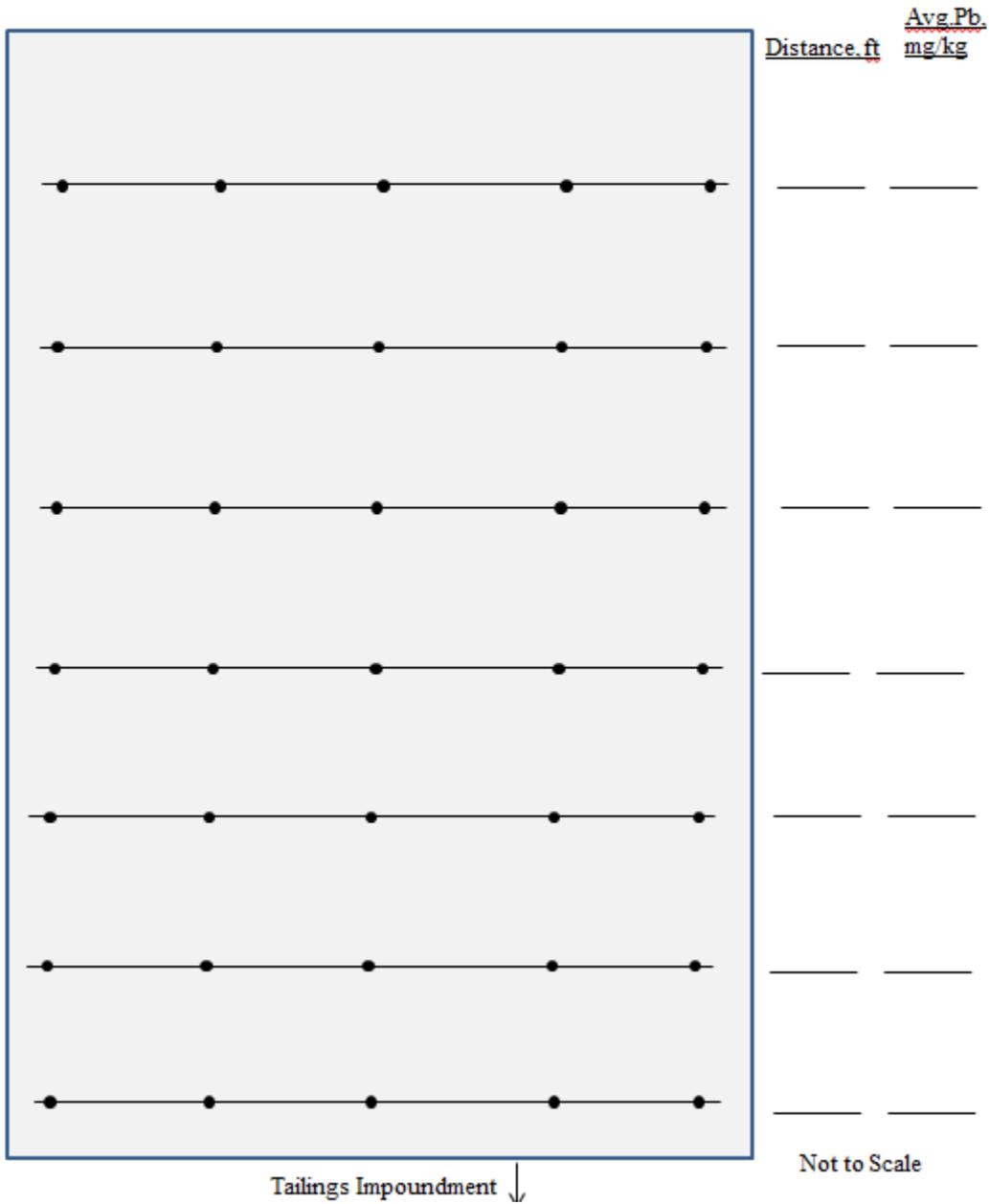
Sampling Area Field Sheet

Soil Sample Log Form



**Viburnum Trend Lead Mining District
Transition Zone NRD Assessment
Sampling Area Field Sheet**

Tailings Impoundment: _____ Date Sampled: _____
Sampling Area# : _____ Sampled By: _____





SOIL SAMPLE LOG

VTLMD Transition Zone Study

	Impoundment Name	Sampling Area #	SU #	Date/Time Collected	Collector's Name	Comments
1						
2						
3						
4						
5						
6						
7						
8						
9						
10						
11						
12						
13						
14						
15						
16						
17						
18						
19						
20						
21						
22						
23						
24						
25						

	Impoundment Name	Sampling Area #	SU #	Date/Time Collected	Collector's Name	Comments
26						
27						
28						
29						
30						
31						
32						
33						
34						
35						
36						
37						
38						
39						
40						
41						
42						
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44						
45						
46						
47						
48						

*Samples collected in Ziploc bags and stored at ambient temperatures.

Relinquished by: _____ Received by: _____ Date: _____ Time: _____

Relinquished by: _____ Received by: _____ Date: _____ Time: _____

APPENDIX C
XRF Standard Operating Procedures

Standard Operating Procedure for XRF Analyzers

VTLMD Transition Zone Study

In-situ surface soil analyses will be conducted in the field along transects established perpendicular to the impoundments. Bagged soil samples will be analyzed with the analyzer in the testing stand and controlled from a laptop PC. A 30-second analysis time will be used for in-situ testing and 45 second analysis time for all bagged samples. Note, do not operate the laptop PC software with the laptop connected to the network servers.

All XRF analyses will be recorded either on field sheets or in a written log book for each instrument. The analyst will record the date, the XRF run number (automatically generated by the XRF), sample ID information, and the total Pb result in mg/kg.

I. Startup

- Power up the analyzer and start the InnovX PC software.
- The instrument will automatically perform an initialization procedure, which lasts for 1-2 minutes.
- Following initialization, place the stainless steel standardization disc over the instrument's sampling window in the test stand and close the stand cover.
- Click the "Standardize" button from the upper left window titled "Soil" in the PC software. The instrument will perform an internal 60-second standardization procedure. During standardization, and any other time the x-ray tube is on, the red light on top of the test stand will flash. When the x-ray tube is off, the red light will remain on solid. **Do not open the test stand lid when the light is flashing.**
- Following standardization, an information window will pop up displaying the analyzer resolution. Record the resolution in the XRF Log Book along with the Run number automatically assigned by the analyzer.
- The analyzer is now ready to analyze standard reference materials (SRMs).

II. Calibration Check

- The NIST 5861 (41ppm Pb), 4315 (244ppm), 2586 (431ppm), 2711 (1162), and 2710 (5532ppm), and Blank SRMs will be analyzed at the beginning of each use. Selected

SRMs will also be reanalyzed periodically during the day based on site findings and length of time in the field.

- On the PC software main menu bar, select “Edit” and then chose “Edit Test Information”. A data entry window will pop up allowing input of information about the next test.
- Select your name from the “Analyst” dropdown menu & select the check sample from the “Chk_Sampl” dropdown list.
- Click the “Start” button in the Soil Window in the upper left corner of the screen to initiate the test.
- Assess instrument calibration by comparing the measured values to the control chart prepared for each analyzer for each SRM. Verify that the result is within 2SD of the control chart mean, if so, continue to In-situ or Bagged Sample Analysis sections below.
- If values outside 2SD of control chart mean are observed, re-analyze the calibration check sample, or if there are more than one SRM sample cup, analyze another one. If the measured value is still outside 2SD, re-standardize as described above, and re-analyze the standard(s). If the result is still outside 2SD, note the failure in the logbook. If more than one check sample fails this procedure on a given day (particularly if the exceedances are in the same direction – e.g. both 2SD above or below the mean), it may be necessary to update the control charts, manually enter new calibration response factors into the analyzers, or both.

III. Sample Analysis

All XRF analyses will be conducted using InnovX X-ray tube analyzers. In the event of instrument malfunction in the field, in-situ analyses may also be conducted using Niton Xli700 Americium²³² isotope analyzers.

In-Situ Analysis

- Following successful calibration check, click Edit from the main menu bar and select Edit sample Information.
- Enter all applicable information about the first in-situ analysis into the iPAQ using the dropdown and direct edit fields.
- Remove any vegetation from the soil surface exposing an area of approximately five square inches. Shake out any soil clinging to vegetation roots back onto the cleared spot and use a stainless steel spoon or other tool to prepare a one square inch area on which to place the XRF analyzer window. Place a thin-walled plastic baggie over the analyzer snout to protect it from the soil.
- Initiate a 30 second analysis. The analyzer can be operated hands-free using the tripod attachment.
- Record the information in columns on the XRF Data Field Sheet, and continue with additional analyses.
- When data from a given distance for a transect is collected, calculate and record the average on the field sheet.

- Periodically and at the end of the day, re-analyze the SRMs as described in the Calibration Check section above.
- Once per day per analyzer, conduct a precision check by repeating an in-situ analysis 7 times without moving the analyzer between tests. The precision check should be conducted in an area found to contain elevated lead, preferably a concentration near an action/screening level.

Bagged Sample Analysis (InnovX analyzers)

- Following successful calibration check, click Edit from the main menu bar and select Edit Sample Information.
- Enter all applicable information about the first sample to be analyzed from the bag label, using the dropdown menus and direct edit fields
- Gently roll the sieved soil around inside the bag to homogenize;
- Place the sample over the analyzer's sampling window ensuring that the soil and bag are in as close contact with the window as possible.
- Close the stand cover.
- Click the Start button from the Soil window to initiate the test.
- The data being acquired will appear in the Chemistry window in the lower center of the PC screen during analysis.
- After analysis, the results will appear in the Results window on the PC.
- A running list of the analyses will appear in the window at the lower left of the PC screen.
- The sample information will remain from the previous test, so no changes are necessary for subsequent replicate analyses on a given sample bag.
- Roll the sieved soil around inside the bag, and re-analyze. Repeat analysis 2 more times.
- Calculate a mean and 95%UCL for the replicate analyses.

If the mean and the 95% UCL lead concentrations straddle a screening level, conduct 3 additional replicate analyses, recalculate the mean and UCL and reassess. This process may need to be repeated for another round of 3 analyses in an effort to get both the mean and UCL on one side of the screening level. It may not be possible in all cases to do this (e.g. when the mean and UCL are very close to the screening level). If after 10 replicate analyses, the mean and UCL still straddle the screening level, the sample will be considered to contain lead above the screening level

- After completing replicate analysis on a bagged sample, click the Edit Sample Information again and enter information for the next bagged sample as above.
- Place the second sample in the test stand, close the cover and initiate the analysis.
- Repeat for remaining samples

- An instrument precision check will be conducted at a frequency of 5%. This will consist of analyzing a sample seven separate times without moving the sample in between each analysis. The %RSD on the replicate analyses should not exceed 15%. Select a bagged sample for the precision check that has elevated lead levels, preferably a concentration near a screening/action level if possible.

IV. Data Downloading

- After the last analysis for the day, select Readings from the main menu bar, and chose Export Readings.
- In the Export pop up box, verify that the “Export readings on date” radio button is selected, the Mode to export is “All”, and today’s date is circled on the calendar.
- Click OK.
- Insert a USB thumb drive in the laptop, download data to it, and then move data onto network server. Select the directory and file name for the downloaded data. For this project, file naming convention is date & XRF serial number (e.g. 10_14_11_5434)
- Verify that the file type is “Comma Separated Values”, and click Save.
- A message will pop up indicating a successful download, and asking whether you would like to open the file. Select Yes, and file will open in Excel. Verify that the data appears correct. Make any corrections you had noted in the run log book.
- Choose Save As from the File menu, and select File Type “Microsoft Excel 97 Workbook.”
- Close the InnovX software, power down the analyzer, and shut down the laptop PC.
- Copy the file from thumb drive to the network as soon as possible after analyses. Files will be stored in the H:/Sections/Superfund/SiteFiles/Viburnum Trend Transition Zone/XRF directory.

Note: For any operation that requests a password, the administrator password is lower case z, and the factory password is 1234.