

**Sampling and Analysis Plan for the 2009
Tri-State Mining District Transition Zone
Assessment Study
Kansas, Missouri and Oklahoma**

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Prepared by:

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1.0 Introduction

1.1 Sampling and Analysis Plan for the 2009 Tri-State Transition Zone Assessment Study

A sampling and analysis plan (SAP) integrates the three primary elements into one plan including, a Quality Assurance Project Plan (QAPP), a Field Sampling Plan (FSP), and Health and Safety Plan (HSP). The QAPP describes the policy, organization, functional activities, quality assurance and quality control protocols necessary to achieve project data quality objectives (DQOs) dictated by the intended use of the data. The FSP provides guidance for all fieldwork by detailing the sampling and data gathering methods to be used on the project. This document includes the essential elements of both a QAPP and FSP. This document describes the sampling and analysis that will be conducted in November/December 2009 to assess soil quality conditions in the Transition Zones of the Tri-State Mining District (TSMD). More specifically, this document includes the following components:

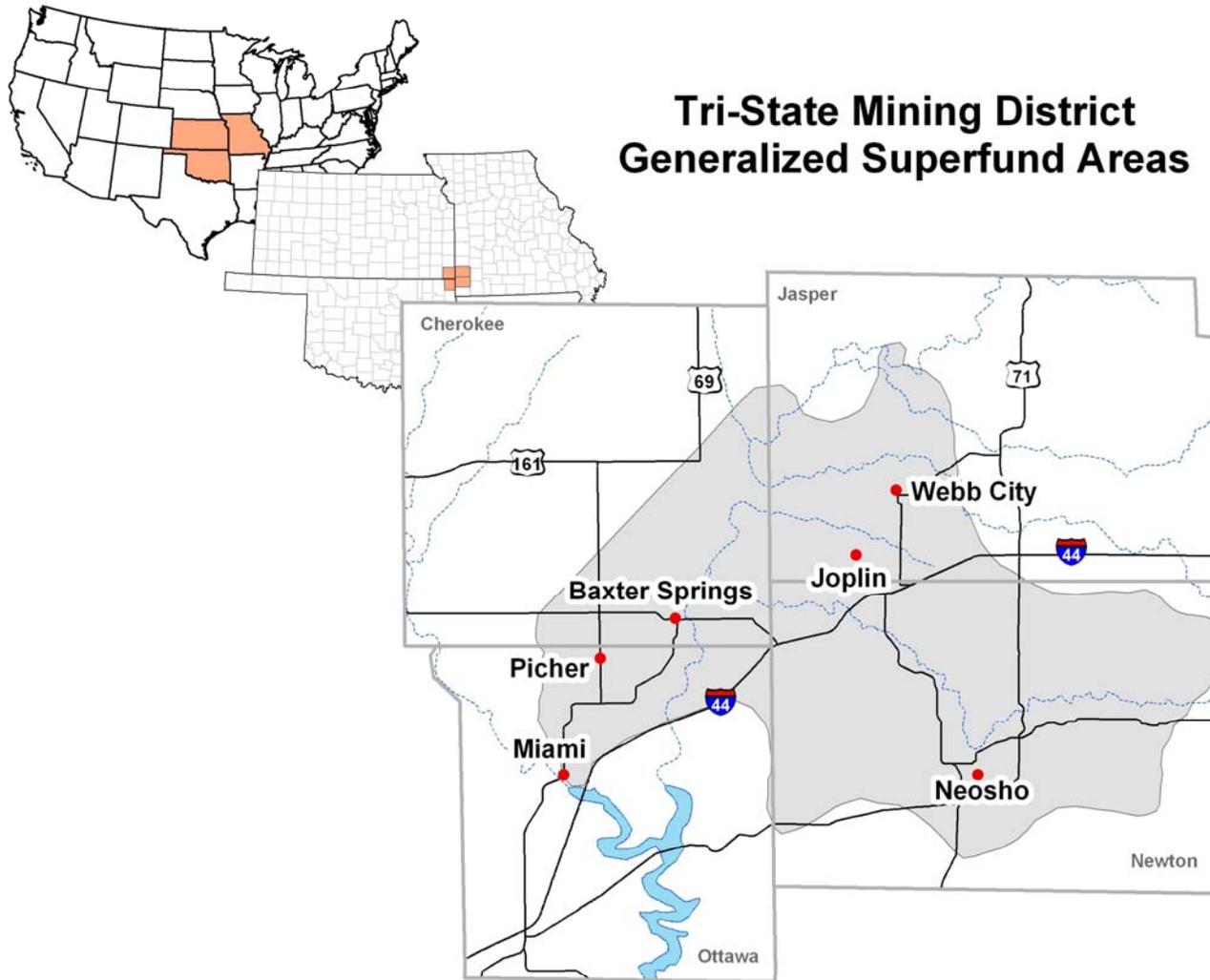
- Introduction (Section 1);
- Sampling objectives (Section 2);
- Sampling program design (Section 3);
- Sampling locations and frequency (Section 4);
- Sampling timing (Section 5);
- Sample designation (Section 6);
- Sampling equipment and procedures (Section 7);
- Sample handling and analysis (Section 8)
- Contaminants of potential concern (Section 9);
- Roles and responsibilities of the sampling team (Section 10);
- Quality assurance (Section 11);
- Examples of forms and instructions for completing paperwork (Section 12);
- References (Section 13).

2.0 Sampling Objectives

The 2009 Tri-State Mining District Transition Zone Assessment Study is intended to provide the information needed to determine the extent of soil degradation from historic mining operations in the TSMD (Figure 1). More specifically, the objectives of the SAP are to:

1. Obtain data on target metal concentrations for approximately 250 -300 soil samples collected from Cherokee County, Kansas; Jasper and Newton Counties, Missouri and Ottawa County, Oklahoma in potential depositional areas resulting from historic mining operations. The samples collected will be dried and an X-ray fluorescence (XRF) analysis performed in accordance with EPA Method 6200.
2. Confirm results of XRF-based metal analyses by submitting a minimum of 5% of the collected samples for laboratory analysis. Samples will be sieved to <250 microns, digested following EPA Method 3052, and analyzed by Inductively Coupled Plasma/Atomic Emission Spectrometry (ICP-AES, EPA Method 6010B).

Figure 1 – Tri-State Mining District



3.0 Sampling Program Design

Soil sampling will take place in November and December 2009 over the course of two to three weeks. Three sampling teams comprised of two to three people will collect approximately 250-300 samples. Each sample will be analyzed using XRF technology, as indicated in Section 7.0. A Minimum of 5% of the samples will be corroborated through laboratory analysis. In-situ will be performed to determine when background concentrations are achieved in the field.

4.0 Sampling Locations and Frequency

The sampling locations (i.e. chat piles and/or bases transition zones) for the 2009 Tri-State Mining District Transition Zone Assessment Study will be selected based on the following parameters:

- Surrounding land use (i.e. agriculture, prairie, wooded, roads etc);
- Height of chat pile (i.e. chat base vs. standing chat);
- Remediation status of the chat pile;
- Proximity of other chat piles/bases;
- Previously sampled chat piles for comparison; and
- Chat pile ownership.

Sampling locations will be identified based on the parameters outlined above. Landowner permission will be obtained for property access. Samples will be collected at the identified locations unless unsafe conditions exist or landowner permission is not received. In these situations, alternative sampling locations (i.e. another chat pile/base) will be identified. If an acceptable alternative location cannot be identified, then no sample will be collected. GPS coordinates will be taken for all sampling points/transects.

5.0 Sampling Timing

The soil samples for the 2009 Tri-State Mining District Transition Zone Assessment Study will be collected in late November to early December 2009. Approximately 250-300 transition zone soil samples will be collected. Soil metal concentrations from historic mining are stable relative to time frames measured in months. Soil sampling within this short timeframe should not have a significant affect on results. One exception could be in the case of anthropogenic land disturbance, which is taken into account as described in section 4.0 above.

6.0 Sample Designation and Procedures

6.1 Sample Collection Procedures

A map of current and historic chat piles and/or bases will be used to indicate where to begin each transect. Sampling locations (i.e. chat piles and/or bases) will be identified using the parameters outlined in Section 4.0. Transects will be extended from the chat piles and/or bases in four opposing directions (i.e. north, south, east, west, or northeast, southwest, northwest, southeast) if possible. If it is not possible to sample in four opposing directions, a minimum of two directions is acceptable.

Each transect will start at the edge of the chat pile/base delineated from current and historic maps. Samples will be collected at 50 foot intervals from the chat pile and/or bases until two consecutive samples are at or below background concentrations (Tidball 1984, Dames & Moore, 1995). A portable XRF unit will be used to determine when background concentrations have been reached or unless a stream, another chat pile or other physical barrier is encountered that would preclude sampling. In-situ XRF analyses will be conducted just below the vegetated zone and/or duff layer.

At each sampling collection point, all plant material and organic detritus will be removed from the surface, the sampler (e.g. sharp shooter, shovel, and trowel) will be used to gather the sample from a depth of 1-6 inches. A description of whether the soil is native soil, vegetated chat or tailings will be logged in the data collection form.

The soil sample will be placed into a 1 liter zip lock bag and labeled appropriately. Split samples will be used for quality assurance and confirmatory laboratory analysis, requiring two bags (a minimum of 5% of all samples will be sent to the laboratory for confirmatory analysis). All samples will be dried and sieved to <250 microns. The split sample will be sent to a qualified lab for analysis by EPA Method 3052 to confirm the results of the field chemistry results. The remaining samples will be analyzed with the XRF using EPA Method 6200 by FWS personnel.

Before a new transect is sampled, the sampling equipment will be cleaned by removing any excess soil and inserting into the soil near the sample location. Between each transect, the sampler will be rinsed with deionized water, followed by Alconox, and deionized water, then placed in a stainless steel tray for transport between transects. Alternatively, a pre-cleaned dedicated sampling spoon or scoop will be used at each sampling location.

Table 1 lists the equipment that will be used to collect samples for the 2009 Tri-State Mining District Transition Zone Assessment Study (see Appendix A). The following information will be recorded following retrieval of the sample:

- Unusual events that occurred during sampling ;
- Sample depth;
- Description of sample (i.e. sandy, clay etc);
- Description of sample color (i.e., black, brown, etc.); and
- Sample processing procedure used (i.e., total volume collected, type of containers used).

6.2 Precautions to Avoid Exposure to Contaminated Soil

It is anticipated that contaminated soil will be routinely encountered during sampling throughout much of the study area. As such, the sampling crew should take precautions to minimize exposure to potentially toxic substances. At a minimum, steps that should be taken include:

- Handling sampling equipment and samples carefully;
- Avoiding direct dermal contact with samples; and,
- Wearing protective equipment, such as gloves, safety glasses, long-sleeved shirts, long pants, rubber boots, and/or rain gear.

More detailed guidance on avoiding hazards during sampling and minimizing the potential for personal injury is provided in the Health and Safety Plan (Appendix A)

6.3 Other Precautions to Avoid Sample Contamination

Generation of reliable data for soil quality conditions is the primary objective of this sampling event. As such, all reasonable efforts should be made to minimize the potential for sample contamination during the sample collection, handling, and processing. At a minimum, steps that should be taken to avoid sample contamination include:

- Ensuring that samples do not come in contact with any item that has not undergone the approved decontamination process;
- Ensuring that any utensils that are used in the sampling process do not come in contact with any item that has not undergone the approved decontamination process;
- Decontaminating all sampling equipment fully after sampling is completed; and,
- Prohibiting any activity that could result in sample contamination (e.g., smoking, consumption of food or drinks during the sampling event). Note: A separate cooler for food and drinks will be available.

6.4 Information to be Collected at Sampling Locations

The following basic information will be collected and recorded at each sampling location on the data collection form:

- Sample location name and number;
- Sampling date and time;
- Latitude and longitude coordinates in WGS84 datum using decimal degrees;
- Weather conditions, including precipitation, temperature etc.;
- Type of sampler used;
- Names of sampling personnel;
- Soil type/soil genesis (i.e. native soil or formed over mine waste); and
- In-situ XRF results for Pb, Zn, and Cd.

7.0 Sample Handling and Preparation

Procedures for handling and preparing samples for metal analysis should follow the procedures described in ASTM (2004; also Appendix A -Table 2). Samples should be carefully packed and transferred to the appropriate laboratory for storage along with the appropriate chain of custody forms. If samples are shipped, an inventory must be maintained of all samples that are shipped each day to facilitate confirmation of receipt the following business day. All samples will be dried and sieved to <250 microns. The samples selected for confirmatory chemical analysis (i.e. split samples) will digested using EPA Method 3052 (Microwave assisted acid digestion of siliceous and organically based matrices) and analyzed by ICP-AES using EPA Method 6010B. The remaining samples will be analyzed with the XRF using EPA Method 6200.

8.0 Chemicals of Potential Concern

Previous studies in the Tri-State Mining District have identified cadmium, lead, and zinc as the primary contaminants elevated due to mining activity (Dames & Moore, 1995). The adverse biological effects of these metals are well documented (Eisler 2004). In order to identify potential injury from mining activity, samples will be analyzed for these target metals. The list of priority analytes and associated data quality objectives for the chemical analyses are presented in Appendix A - Table 3.

9.0 Roles and Responsibilities of Sampling Team

Samples supporting the Tri-State Mining District Transition Zone Assessment Study will be systematically collected within the sampling area. The sampling teams will be comprised of two to three individuals with responsibility for collecting and preparing samples, conducting on-site XRF analyses of samples, and preparing and shipping samples for lab analysis. Quality assurance and control (QA/QC) for the field portion of this investigation will be directed by Suzanne Dudding, John Miesner and Dave Mosby of the U.S. Fish and Wildlife Service.

The sample collection teams will:

- ensure that all necessary sampling equipment and supplies are loaded into the sampling vehicle(s) each day,
- verify the sample locations (using handheld GPS),
- collect sufficient soil volumes for analyses of soil chemistry,
- prepare and label samples,
- decontaminate the samplers between transects,
- and follow the completion of sampling activities at each location.

One team member will perform the XRF analysis; a second team member will collect the soil samples and decontaminate the sampler; and the third team member will collect the GPS readings and take field notes.

Samples will be transported in a secure manner to avoid damage to the sample. Any samples lost or damaged during transport must be identified by sampling location and documented. If a commercial shipping service is used, soil samples may be shipped on Monday, Tuesday, Wednesday, Thursday, and Sunday only (to avoid weekend delivery issues). Any soil samples not shipped on the date of collection will be held and shipped on the next appropriate shipping day. Unused portions of samples will be disposed of at the sampling location where collected.

Sample collection and disposition will be clearly documented. At each sampling location, the data collection form will be filled out (see example in Addendum A). Samples will be stored in appropriate containers. Samples transported for laboratory analysis will be packaged with the chain of custody (COC) manifest will be prepared (see example in Appendix A). A copy of the COC will be maintained and the initial disposition will be noted on the data collection form. The laboratory will also return a copy of the signed and dated COC to the sender upon receipt and acceptance.

10.0 Quality Assurance

Generation of quality chemistry data is essential for supporting the 2009 Tri-State Mining District Transition Zone Assessment Study. To avoid problems associated with data reliability, it is necessary to implement adequate quality assurance measures in the sampling program, during data collection and analysis. In this study, the quality of data analyzed by portable XRF will be evaluated by conducting laboratory analysis of a minimum of 5% of the field-collected samples. Samples for XRF analysis will be dried to a constant weight at the U.S. Fish and Wildlife Service (FWS) field office in Tulsa, Oklahoma. The dried samples will be sent to the FWS field office in Columbia, Missouri. The samples will be sieved to <250 microns and analyzed with the XRF using EPA Method 6200 by FWS personnel. FWS personnel in the Missouri office will direct quality assurance and control during XRF analysis.

Split samples, discussed in section 6.1 above, will be collected by placing alternating spoonfuls of soil into different bags, or placing half of each scoop into each bag. A minimum of one split sample will be collected per day of sampling.

The samples selected for confirmatory chemical analysis will digested using EPA Method 3052 (Microwave assisted acid digestion of siliceous and organically based matrices) and analyzed by ICP-AES using EPA Method 6010B.

11.0 Examples of Forms for Sampling Event

Examples of forms that will be used for this sampling event are presented as addenda, including the data collection form and chain of custody form (Appendix B).

12.0 References

Dames & Moore, 1995, Final Remedial Investigation: Neck/Alba, Snap, Oronogo/Duenweg, Joplin, Thoms, Carl Junction, and Waco Designated Areas, Jasper County Site, Jasper County, Missouri.

Eisler, R. 2000. Handbook of chemical risk assessment Health hazards to humans, plants, and animals. Volume 1.

EPA. 1996 Standard Method 3052: Microwave Assisted Acid Digestion of Siliceous and Organically Based Matrices.

EPA. 1996 Standard Method 6010B: Inductively Coupled Plasma-Atomic Emissions Spectrometry

EPA. 1998 Standard Method 6200: Field Portable X-Ray Fluorescence Spectrometry for the Determination of the Elemental Concentrations in soil and Sediment. 31pp

Tidball, R.R., 1984. Geochemical Survey of Missouri, U.S. Geol. Survey Professional Paper - .954-8.1.

Appendix A – Health and Safety Plan

**TRI-STATE MINING DISTRICT TRANSITION ZONE STUDY
HEALTH AND SAFETY PLAN**

October 2008

Prepared by:

Suzanne Dudding, Fish and Wildlife Service, Oklahoma Field Office

Introduction

This Health and Safety Plan has been developed as part of the Tri-State Mining District Transition Zone Study soil sampling effort planned for November/December 2009. The sampling effort will include collection of 250-300 soil samples in Cherokee County, Kansas; Jasper and Newton Counties, Missouri and Ottawa County, Oklahoma. Additional details about the sampling effort are provided in the document in the main body of this document. **A copy of this Health and Safety Plan should be kept with each sampling team during this sampling effort.**

DIAL 911 IN ALL LOCATIONS IN CASE OF EMERGENCIES

Sampling Operations

Sampling operations: Sampling operations and plans will be discussed at a meeting prior to sampling. Each day prior to commencing work a meeting will be held by the team leader informing all members of the type and location of safety gear, as well as the anticipated activities for the day, during a pre-departure safety “tailgate” meeting.

Sampling Locations: Soil sampling will take place throughout the Tri-State Mining district. Sampling locations have been determined prior to sampling and locations have been loaded into GPS units.

Weather: Cold weather conditions are expected in the mornings with warmer weather expected in the afternoons. It is recommended that personnel dress in layers and bring sunscreen to avoid sun exposure. Inclement weather severe enough to disrupt sampling operations is not anticipated. However, National Weather Service broadcasts will be monitored. Sampling activities may be modified based on weather reports or weather conditions experienced while on-site. In all cases, sampling will occur only under conditions deemed safe by Suzanne Dudding and/or team leader

Hazards: There are many hazards associated with working outdoors including slips, trips, and falls; sun exposure, poison ivy and cuts and scraps from vegetation.

Safety Equipment

All vehicles will have a first aide kit and at least one cell phone to contact emergency personnel

Safety Rules

- No sampling will occur after before sunrise or after dark
- No sampling will occur without landowner permission
- No illegal drugs or alcohol are allowed on board the vessel; and

Communications

Teams will exchange cell phone numbers before embarking into the field. Teams will check in every four hours with Suzanne Dudding. In case of inclement weather, teams will contact each other and proceed to the boat ramp.

Emergency phone numbers

Telephone numbers of local emergency service providers in the vicinity of sampling locations are listed below. For contact information of field personnel, see the Communications section of this plan.

DIAL 911 IN ALL LOCATIONS IN CASE OF EMERGENCIES

NEAREST HOSPITALS/ CLINICS

Miami, Oklahoma

Integris Baptists Regional Hospital
200 Second Avenue SW
Miami, OK 74354
Telephone Number: (918) 542-6611

Joplin, Missouri

St. John's Regional Medical Center
2727 McClelland Blvd.
Joplin, MO 64804
Telephone Number: (417) 781-2727

Appendix B – Sampling Forms

Table 1. Sampling Equipment Required to Support the Tri-State Mining District Transition Zone Assessment Study	
Cleaning supplies	
<ul style="list-style-type: none"> Alconox solution Deionized/reverse osmosis water 	<ul style="list-style-type: none"> Polyethylene squeeze bottles or spray bottles Long bristle scrub brush
Sampling supplies	
<ul style="list-style-type: none"> Maps of the study area Zip Lock bags - Liter Sharp shooter Stainless steel trowel Sample collection forms 	<ul style="list-style-type: none"> Latex gloves - non-powdered Stainless steel spoons Stainless steel homogenization buckets - 2 x 1 L; 2 x 5 L Sharpie pens
Measurement supplies	
<ul style="list-style-type: none"> GPS (handheld) 	
Shipping and storage supplies	
<ul style="list-style-type: none"> 48-L plastic coolers FedEx labels for shipments - as needed Paper towels 	<ul style="list-style-type: none"> Shipping manifests - as needed Electrical tape
Personal supplies	
<ul style="list-style-type: none"> Gloves Rubber boots Cooler for onsite food and beverage consumption Tick spray and poison ivy lotion 	<ul style="list-style-type: none"> Long pants Rain/ foul-weather gear
Miscellaneous supplies	
<ul style="list-style-type: none"> Data collection forms Field book (Rite in the Rain) 	<ul style="list-style-type: none"> Digital camera with accessories (spare batteries; extra memory chips)

Table 2. Volume, Container Material, Preservation Specifications, and Holding Times for Samples Collected for Tri-State Mining District Transition Zone Assessment Study.

Parameter Analyzed	Laboratory ¹	Approximate Volume	Container Material	Preservation Method	Holding Time
Metals (by XRF)	FWS facility	125 mL	Zip Lock bag	room temp	28-d
Metals (by EPA Method 6010B)	Approved FWS lab - TBD	125 mL	Zip Lock bag	room temp	28-d

d = day; XRF = X-Ray fluorescence;

Table 3. Contaminants of Potential Concern and Data Quality Objectives for the Tri-State Mining District Transition Zone Assessment Study (Applicable to confirmatory chemistry by Method 6010B only.)

Contaminant of Potential Concern	Target Detection Limit (as μL in digestate)	Target Mean Accuracy (Average % Recovery)	Target Precision (Relative Standard Deviation %)	Target Completeness
<i>Metals (mg/kg)</i>				
Cadmium	2.3	75-125	20	100%
Lead	28	75-125	20	100%
Zinc	1.2	75-125	20	100%

Example Chain of Custody form

Company Name: U.S Fish and Wildlife Service						Billing Address: Same					
Address: 9014 East 21st Street						Catalog #:					
City: Tulsa		State: OK		Zip Code: 74129		Project Name:					
Telephone: (918) 581-7458		FAX #:		(918) 581-7467		Contract Order #:					
Report To: Suzanne Dudding		Sampled by:				Order #:					
						Manager:					
Turnaround time:			Sample type:			Analysis Requested					
15 working days .. 2 working days ..			Potable Water .. Vegetation ..			XRF					
10 working days .. 24 hours ..			Waste Water .. Air ..								
5 working days .. Same day ..			Soil/Sediment x Other ..								
	Client Sample ID	Date/Time Sampled	Matrix Type	# of Cont.	Cont. Size/Type	Sample Log-in #					Comments
1			Sediment	1			x				
2			Sediment	1			x				
3			Sediment	1			x				
4			Sediment	1			x				
Relinquished by: _____ Date: _____ Time: _____						Received by: _____ Date: _____ Time: _____					
Relinquished by: _____ Date: _____ Time: _____						Received by: _____ Date: _____ Time: _____					
Relinquished by: _____ Date: _____ Time: _____						Received by: _____ Date: _____ Time: _____					
Relinquished by: _____ Date: _____ Time: _____						Received by: _____ Date: _____ Time: _____					
Sample conditions upon receipt:						Other Information: (e.g. sample pre-treatment, holding time, storage, deficiencies, etc.)					
Frozen: ..		Method of Shipment: _____									
Cold: ..		_____									
Ambient: x		Shipment condition: (e.g. breakage, leakage): _____									
N/A ..		_____									

Data Collection Form

Sample Site Name:
Sample Site Number:
Names of Sampling Personnel:
Sampling Date:
Sampling Time:
Latitude and longitude :
(including instrumentation used and any problems encountered)
Weather conditions:
Precipitation
Wind speed and direction
Type of sampler used:
Sample depth:
Unusual events during sampling:
(i.e., sampler did not close completely, etc.)
Description of sample:
(i.e. sand, clay)
Description of sample colour:
(i.e., black, brown, etc.)

