Sampling and Analysis Plan
West Oakland
Residential Lead Sampling
Oakland, Alameda County, California

TDD No.: TO2-09-09-09-0001
Job No.: 002693.2052.01RA

September 2009

Prepared for:

U.S. ENVIRONMENTAL PROTECTION AGENCY
Region IX

Prepared by:

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Superfund Technical Assessment and Response Team

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1 Introduction

The United States Environmental Protection Agency (U.S. EPA) tasked Ecology and Environment, Inc.'s (E & E's) Superfund Technical Assessment and Response Team (START) to support a U.S. EPA funded Removal Assessment of the South Prescott Residential Neighborhood, in West Oakland, California. In order to support the U.S. EPA's environmental data collection activities, the START has identified project data quality objectives and developed this Sampling and Analysis Plan (SAP).

In response to community concern, the U.S. EPA, Region IX Emergency Response Section (ERS) will conduct an assessment of lead contamination in surface soils within the South Prescott residential neighborhood in West Oakland, California. Community concerns precipitated from the 2007 AMCO Chemical Corporation (AMCO) National Priorities List (NPL) Site and subsequently the DC Metals, Inc. scrap metals (DC Metals) Site, remedial investigation. The Remedial Investigation conducted by the U.S. EPA identified elevated levels of lead in soils adjacent to the former AMCO/DC Metals facility located at 1414 Third St., Oakland, California. This Assessment Action will focus on the potential dispersion of lead-contamination into adjacent residential surface soils, in connection with the former AMCO and DC Metals facility operations, and identify potential removal actions.

The scope of work and objectives outlined in this SAP are derived from direction from the U.S. EPA. This SAP describes the project and data use objectives, data collection rationale, quality assurance goals, and requirements for sampling and analysis activities. It also defines the sampling and data collection methods that will be used for this project. This SAP is intended to accurately reflect the planned data-gathering activities for this support activity; however, site conditions, budget, and additional U.S. EPA direction may warrant modifications. All significant changes are to be documented in site records.

The specific field sampling and chemical analysis information in this SAP was prepared in accordance with the following U.S. EPA documents: EPA Requirements for Quality Assurance Project Plans (EPA QA/R 5, March 2001, EPA/240/B 01/003); Guidance on Systematic Planning Using the Data Quality Objectives Process (EPA QA/G 4, February 2006, EPA/240/B-06/001); Guidance on Choosing a Sampling Design for Environmental Data Collection (EPA QA/G 5S, December 2002, EPA/240/R 02/005); and Uniform Federal Policy for Implementing Environmental Quality System (EPA/505/F-03/001, March 2005).

1.1 Project Organization

U.S. EPA Federal On-Scene Coordinator (FOSC) – The U.S. EPA On-Scene Coordinators will be Steve Calanog and Chris Reiner. Mr. Calanog and Mr. Reiner are the primary decision-makers and will direct the project, specify tasks, and ensure that the project is proceeding on schedule and is within budget. Additional duties include coordination of communication with the START Project Manager, U.S. EPA Quality Assurance (QA) Office, and community residents.

START Project Manager (PM) – Mr. David Neil Ellis of START is the PM. The PM manages the project's data collection efforts and is responsible for implementing the SAP, coordinating project tasks and field sampling, managing field data, and completing all preliminary and final reporting.

Principal Data Users – Data generated during the implementation of this SAP will be utilized by the FOSC to make decisions regarding potential removal activities.

START Quality Assurance Coordinator – Mr. Howard Edwards is responsible for the development of this SAP. Specifically, Mr. Edwards is responsible for the documentation of project objectives and for preparation and review of the draft and final SAP document. Mr. Edwards will coordinate with the U.S. EPA's Quality Assurance Office as needed.

Sample Analysis and Laboratory Support – The U.S. EPA's Region IX laboratory in Richmond, California, will be responsible for sample analysis by definitive analytical methodologies. The START will be responsible for field sample analysis by non-definitive analytical methodologies.

1.2 Distribution List

Copies of the final SAP will be distributed to the following persons and organizations:

- Steve Calanog and Chris Reiner, U.S. EPA, Region IX
- U.S. EPA, Region IX, Quality Assurance Office
- E & E START Field Team
- E & E START project files

1.3 Statement of the Specific Problem

There is public and regulatory concern as to whether residents adjacent to the former AMCO/DC Metals facility may be currently exposed to or have potential for exposure to lead contamination documented as being present along the eastern side of Center Street in West Oakland. Lead-contaminated soils documented at residential homes in this area led the U.S. EPA to perform Remedial Removal Actions at several residences in 2007, to mitigate contaminant migration and exposure pathways. Since 2007, public concern has arisen over the potential for exposure and extent of lead dispersion originating from the former AMCO/DC Metals facility at residential properties west of Center Street. The U.S. EPA has determined that surface soil sampling on residential properties adjacent to the former AMCO/DC Metals facility and previously remediated homes is necessary to assess the lateral extent of contamination and magnitude of potential human lead exposure. To mitigate the threat posed by soil contamination, if present, the U.S. EPA will evaluate removal and/or remedial options.

2 Site Background

2.1 Site Location and Description

The West Oakland residential study area is located in the City of Oakland, Alameda County, California. The site is situated approximately 1 mile west of Downtown Oakland and immediately south and southwest of the West Oakland Bay Area Rapid Transit (BART) rail line.

The study area is comprised of approximately 6 blocks of residential houses and yards (approximately 23 acres); bounded by Center Street to the east, Third Street to the south, Peralta Street to the west, and the BART rail line to the north. (Figure 2-1, Site Location Map). The geographic coordinates for the approximate center of the area of concern are Longitude 122°17'49.692"West and Latitude 37°48'14.958"North.

2.2 Site History

The historic NPL site located at 1414 Third Street was formerly a chemical distribution facility owned by AMCO Chemical Corporation (AMCO) between the 1960's and 1989. The AMCO facility included a railroad spur, above ground tanks and drums, and underground storage tanks used to transfer and store raw materials. All above ground tanks and drums were removed from the site by AMCO in 1989, although there is no record of the removal of underground storage tanks. From 1989 to November 1998, the site was operated as a scrap metals yard by DC Metals, Inc. In November 1998, all metal scrap and equipment was removed from the site by the operator and all site operations ceased. The site has subsequently been leased to Cable Moore, Inc. and is being used for cable storage.

Both the former AMCO/DC Metals site and the nearby Southern Pacific Transportation Company (SPTC) property at 1401 Third Street are known or suspected hazardous release sites. Numerous site investigations at and around the former AMCO/DC metals and SPTC properties have documented the presence of chlorinated solvents (including 1,1-dichloroethane; 1,1,2- and 1,2,2-trichloroethane; 1,1- and cis-1,2-dichloroethene; methylene chloride, and vinyl chloride), petroleum hydrocarbons (including benzene, toluene, ethyl benzene and xylenes), and metals in soil (URS Corporation 2007, ER-QASP).

In 2007 the U.S. EPA performed an assessment of lead in residential soils at properties located on Third and Center Streets that are immediately adjacent to the former AMCO/DC Metals property. This investigation revealed concentrations of lead in bordering residential soils of up to 2700 parts per million (ppm). This prompted the U.S. EPA to conduct Removal Actions at eight (8) residential properties containing lead contaminated soils. According to the available documentation, this investigation was the extent of the most recent investigation conducted for lead-contaminated soils within the above-mentioned residential neighborhood. Complete background reports/ information for the 2007 assessment and removal actions are not available to START at this time. However, small amounts of information from these actions are available (contaminant concentration maps, laboratory data, and ER-QASP) and are referenced for preparation of this SAP.

Based on available conditions documented by the 2007 investigation and removal action, the U.S. EPA is conducting an assessment to address data gaps described later in this document.

The objectives discussed herein are for the approximate 6 block residential neighborhood area located immediately west and northwest of the formerly assessed/remediated properties, known as the West Oakland Residential Lead Assessment (the site).

Source: U.S. Census TIGER

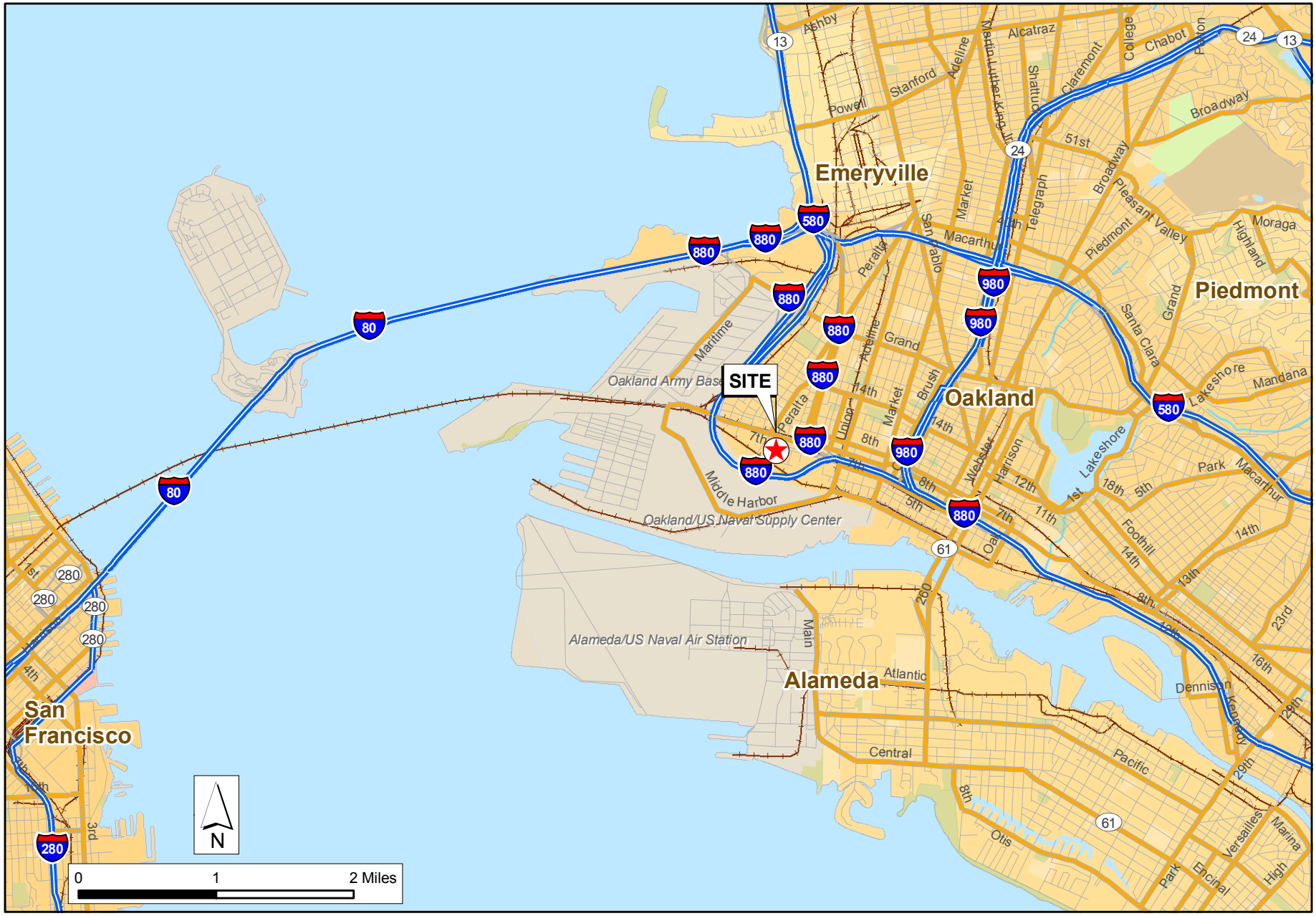

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Figure 2-1

West Oakland Residential Lead Sampling Site Location Map

West Oakland, California

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Source: DigitalGlobe, Image date 2009-04-01

N

0 250 500 Feet

LEGEND

Former AMCO chemical facility

Area of Concern

Figure 2-2

West Oakland Residential Lead Sampling Operations Area Map

West Oakland, California

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Source: Team 9 - Joint Venture URS and AECOM
Aerial Photo - DigitalGlobe, Image date 2009-04-01

LEGEND

- Excavation Site
- Former AMCO chemical facility
- Area of Concern

Figure 2-3

Location of 2007 Residential Soil Removal Actions

West Oakland, California

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3 Project Objectives

3.1 Project Task/Sampling Objectives

The data generated by implementing this SAP will be used to evaluate contamination in surface soils and human health hazards associated with lead contamination at residential properties within the study area of West Oakland. The sampling results will be reviewed to identify and delineate areas above site specific action levels.

The U.S. EPA tasked the START to prepare this SAP to support the environmental data collection activities needed to support assessment of the site.

Soil sampling followed by immediate field analysis will be implemented to accomplish the project objectives. Definitive laboratory sample analysis will be performed in order to document and validate the field analysis data. Sampling objectives include the following:

- Identify lead-contaminated surface soils potentially associated with the former adjacent hazardous waste operations site.
- Document the concentrations of lead in surface soils at all sampling locations.
- Identify whether an area requires additional assessment and/or remedial excavation.

3.2 Action Levels

The site action levels are based on human health threats and exposure resulting from lead contaminant migration from the adjacent hazardous waste operations facility. Action levels for this assessment were developed to provide a maximum concentration of lead at the site which would not adversely impact humans. These criteria will be used to calculate the acceptable concentration of lead in soils.

The site-specific action level for lead during this assessment is 400 mg/kg for delineation of surface soil concentrations.

The following benchmarks were considered prior to establishment of the site-specific action levels:

- U.S. EPA Regional Screening Levels (RSLs) for Residential Soil, April 2009
- Lawrence Berkeley National Laboratory; Analysis of background distributions of metals in the soil, 2009

Benchmarks for lead are presented in Table 3-1 and Table 3-2.

Table 3-1 Benchmarks and Data Quality Indicator Goals Definitive Data for EPA Method 6010C

Chemical of Potential Concern	Upper Estimated Regional Background Concentration (mg/kg)	Site-Specific Action Level for Assessment	U. S. EPA RSL (mg/kg)	U. S. EPA Region IX Laboratory Reporting Limits (mg/kg) and SW-846 Method	Accuracy (% Recovery for MS/ MSD)	Precision (RPD from MS/MSD and Duplicates)	Percent Completeness
Lead	48	400	400	4	75 - 135	<35%	> 90%[1]

Notes:
mg/kg = milligrams per Kilogram
RSL = Regional Screening Level (U. S. EPA April, 2009)
MS/MSD = Matrix Spike/Matrix Spike Duplicate
RPD = Relative Percent Difference
[1] Should also be greater then ten percent of all samples analyzed by EPA Method 6200.

Table 3-2 Benchmarks and Data Quality Indicator Goals Non-Definitive Data for EPA Method 6200

Chemical of Potential Concern	Upper Estimated Regional Background Concentration (mg/kg)	Site-Specific Action Level for Assessment	Innov-X XRF Site Specific MDL SW-846 Method 6200 (mg/kg)	Accuracy (% Recovery of Check Standards) SW-846 Method 6200	Precision (RPD from Duplicates)	Percent Completeness
Lead	48	400	10	65 – 135	<35%[2]	> 90%

Notes:
mg/kg = milligrams per Kilogram
RPD = Relative Percent Difference
MDL = Method Detection Limit
[2] Should not exceed 25% for analytical duplicates.

The U.S. EPA RSLs combine current U.S. EPA toxicity values with standard exposure factors to estimate contaminant concentrations in environmental media (soil, air, and water) that are considered protective of humans, including sensitive groups, over a lifetime. Chemical concentrations above these levels would not automatically designate a site as contaminated or trigger a response action; however, exceeding the RSLs suggests that further evaluation of the potential risks that may be posed by site contaminants is appropriate.

3.3 Data Quality Objectives

3.3.1 Data Quality Objective (DQO) Process

The DQO process, as set forth in the U.S. EPA document, *Guidance on Systematic Planning Using the Data Quality Objectives Process (EPA/240/B-06/001),* (U.S. EPA 2006), was followed to establish the data quality objectives for this project. An outline of the process and the outputs for this project are included in Appendix A. The following sections outlines and summarized the outputs of seven step DQO process completed in accordance with the guidance.

3.3.2 Step 1 – State the Problem

The following paragraphs outline Step 1 of the DQO process. A concise description of the problem is given in Section 1.3, Statement of the Specific Problem.

Planning Team

Planning Team members have been identified in Section 1.2, Project Organization. Planning and scoping meetings were held with the U.S. EPA beginning on August 20, 2009.

START will be responsible for data generation, collection and dissemination; report preparation, and quality assurance/quality control. During the field effort the START will report field data to the FOSC and distribute to GIS support as required for map generation.

Exposure Scenario

Migration of lead from the former adjacent hazardous waste operations site may be impacting residential soils to the west and northwest. If the contamination is not identified and delineated, it could potentially impact humans.

Available Resources

The current budget for the START activities includes the planning, coordination, development and implementation of the SAP, and post sampling activities. U.S. EPA resources to be used include laboratory analytical services and field analytical instruments.

Other Considerations and Constraints

The scheduling of data collection activities is dictated by the U.S. EPA funded assessment schedule. Mobilization to the site for assessment activities is scheduled to begin on October 26, 2009. START field work is not expected to exceed five field days.

Soil analyses available for assessment are not always useful for determining disposal and remediation costs. Additional waste testing of excavated soil is usually necessary to determine disposal requirements.

3.3.3 Step 2 – Identify the Decision

This section describes the decision that requires new data to address the potential contamination problem. The principal study questions and alternative actions are outlined below.

Principal Study Questions: What are lead concentrations in exposed residential soils within the study area (West Oakland Residential site)? What is lead concentration distribution in individual residential properties within the study area? What is the estimated volume of contaminated soil that is above the action level?

Alternative Action 1a: If it is determined that exposed soils at individual residential properties have lead in soil concentration greater then the action limit, then the information may be used to determine what will need to be excavated/remediated or it may be determined that additional investigation of the property is required.

Alternative Action 1b: If it is determined that exposed soils at individual residential properties have lead in soil concentration less then the action limit, then no further action regarding the property will be required.

Decision Statement

Analytical data will be used to evaluate if soil contains lead concentrations greater than the site-specific action levels in order to determine whether additional investigations is necessary and to assist with determining areas and the quantity of soil that might need to be excavated/ remediated.

3.3.4 Step 3 – Inputs to the Decision

The following paragraphs describe inputs required to make the decision.

Information Currently Available

A review of available files was conducted while preparing this SAP and is summarized in Sections 2.2 and 2.3. Historic soil data from residential homes immediately adjacent to the former hazardous waste operations site indicates that lead is present in residential soil up to 2700 ppm.

New Data Required

The following data are required to resolve the decision statement.

- Field analytical sampling data that will be generated within several hours of sampling.
- Physical site data that will be generated in the field with the GPS mapping, photography, and physical observations.
- Definitive confirmation data that will be generated from samples collected by START and submitted to the U.S. EPA Region IX Laboratory for analysis by U.S. EPA SW-846 methods.

Basis for Determining the Action Levels

The basis for determining the action levels is discussed in Section 3.3.

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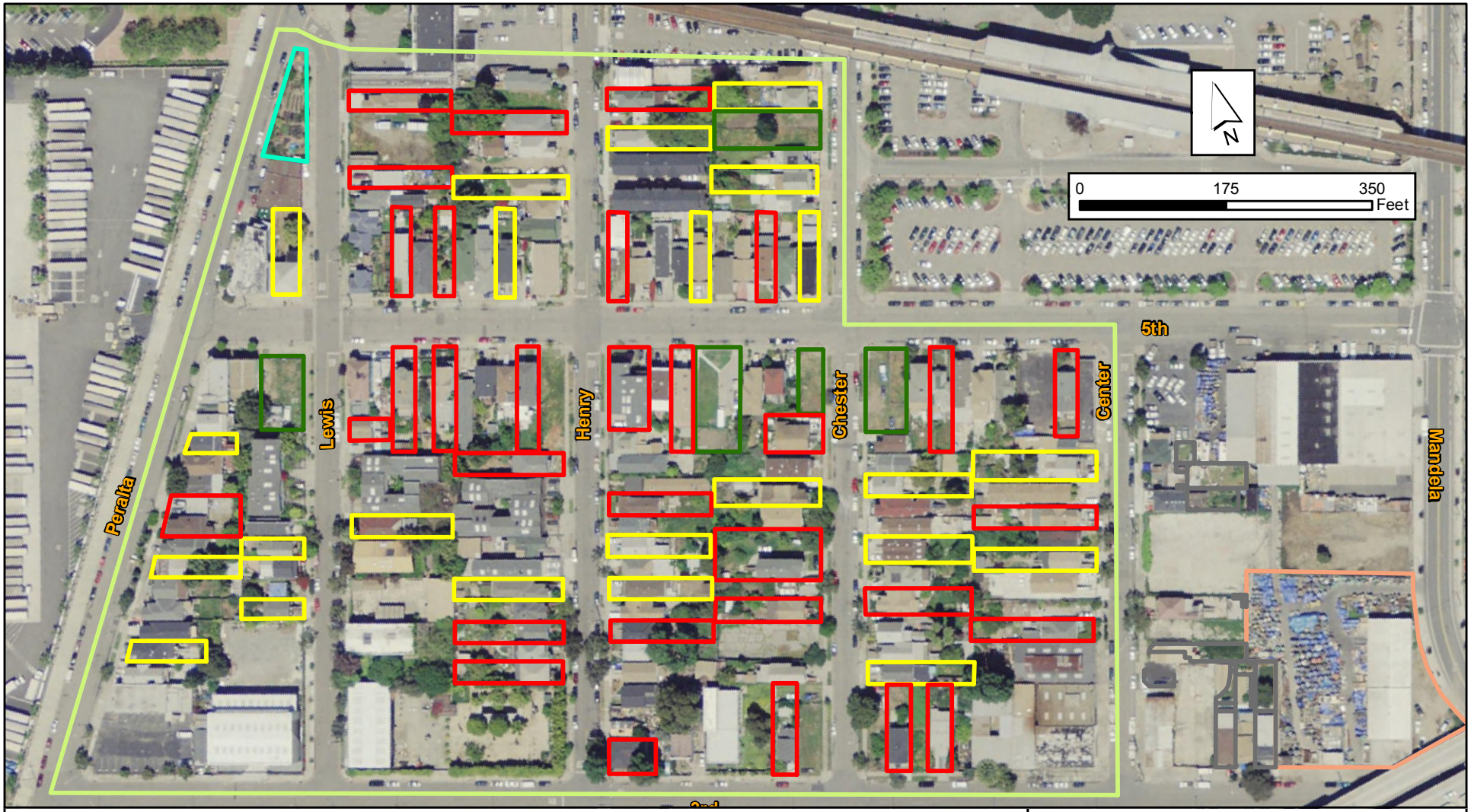
Source: DigitalGlobe, Image date 2009-04-01



LEGEND

- Potential Sample Location, Vacant Lot
- Potential Sample Location, Community Garden
- Potential Sample Location, Residential, Front and Back Yard
- Potential Sample Location, Residential, Back Yard
- Former AMCO chemical facility
- Area of Concern

Figure 4-1

West Oakland Residential Lead Sampling Sample Location Map

West Oakland, California

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Data Collection Methods
Planned sampling techniques are described in Section 6.2 of this SAP.

Data Measurement Methods
The site-specific measurement methods are described in Section 5 of this document. The screening-level methods of analyses to determine lead concentrations are outlined in Section 6.3.

3.3.5 Step 4 – Define the Boundaries of the Study
The specific characteristic that define the population being studied is the lead concentrations in residential soils within the specified spatial boundaries.

Spatial Boundaries
New data will be generated from samples collected from the areas as designated below and shown on Figure 2-2.

West Oakland Residential Study Area
The boundary will encompass residential homes located to the west and northwest of the former AMCO and DC Metals hazardous waste operations site located at 1414 Third Street in Oakland, California. The area can be described as approximately 6 blocks of residential properties (approximately 23 acres); bounded by Center Street to the east, Third Street to the south, Peralta Street to the west, and the BART rail line to the north, with depth of 0 to 6 inches bgs.

Temporal Boundaries
The decisions will apply to determinations of risk associated with long-term exposure to contaminated surface soil from direct exposure. However, decisions may also apply to short-term (acute) exposure to contaminated soil due to potential development activities.

Lead is environmentally persistent and migrates slowly, so soil concentrations generally do not vary greatly over time. Given the location, human accessibility, and the existing community, potential threats are expected to be immediate or imminent.

Thus, the following assessment time-frame has been proposed:

- October 13, 2009 – The Draft SAP will be submitted to U.S. EPA and should be reviewed, revised and finalized.
- October 26, 2009 – Proposed sample collection will take place following SAP approval by the U.S. EPA.
- October 26 – October 30, 2009 – Field analytical sampling data that will be generated within several hours of sampling.
- Preliminary analytical data should be available within 3 weeks of sample delivery to the laboratory.
- Data packages and final data should be reported to project management approximately 5 weeks after sample delivery to the laboratory.
- Decision statement resolutions are expected to occur approximately 6 weeks after sampling and should take place prior to development decisions.

Scale of Decision-Making

A decision unit will be a sampling location (i.e. Such as the back yard at a specific address). The distribution of lead through out the entire study area will also be evaluated. The sampling at residential properties is described in Section 7.

3.3.6 Step 5 – Develop Decision Rules

Site Action Level

The site action levels are specified in Table 3-1 and Table 3-2. The action level for lead is 400 mg/kg for delineation of contamination associated with the former adjacent hazardous waste operations site.

Decision Rule for Decision Units at Individual Residences

1. If the new data indicate that a decision unit is not contaminated above the applicable action-level, then that decision unit will not be considered in need of further investigation or soil removal/ remediation.

2. If the new data indicate that a decision unit is contaminated above an applicable action level, then that decision unit will be considered in need of further investigation or soil removal/ remediation.

Decision Rule for the West Oakland Study Area

1. If the new data for entire study area does not resolve the lead distribution for the study area, then the decision-maker will report data and make recommendations on additional sampling.

.

2. If the new data for entire study area does resolve the lead distribution for the study area, then the decision-maker will report data and considered in need of further investigation or soil removal/ remediation.

3.3.7 Step 6 – Specify Tolerable Limits on Decision Errors

Range of the parameter(s) of interest

For all investigation areas and parameters, the range of interest for lead is from ½ the action level to anything above the action levels. Quantitatively precise and accurate determinations of contaminant concentrations that are significantly above (i.e., >100 times) the action level are not necessary.

Based on past investigations, the soil contaminant concentrations are expected to be above action levels in some residential properties.

Baseline Condition *(the Null Hypothesis)*

The contaminant concentrations in soils are greater than or equal to the action level.

Alternative Condition *(the Alternative Hypothesis)*

The contaminant concentrations in soils are less than the action level.

Decision Error
Decision error and error limit goals are discussed in Appendix A.

3.3.8 Step 7 – Optimize the Design for Obtaining Data
To optimize the sampling design, U. S. EPA and START will use a dynamic sampling design that uses composite sampling, on-site analysis, and on-site sampling related decision making.

3.4 Data Quality Indicators (DQIs)
Data quality indicators (DQIs) are defined as: precision, accuracy, representativeness, completeness, comparability, and method detection limits. The DQIs for this project were developed following the guidelines in *U.S. EPA Guidance for Quality Assurance Project Plans, EPA QA/G 5 Final*. All sampling procedures are documented in Sections 6.2 and 6.3. Standard operating procedures will be followed to ensure representativeness of sample results by obtaining characteristic samples. Approved U.S. EPA methods and standard reporting limits will be used. All data not rejected will be considered complete. Table 3-1 and Table 3-2 documents the site-specific DQI goals for lead.

3.5 Schedule of Sampling Activities
The field sampling activities are schedule to commence on October 26, 2009. Samples will be submitted for field analysis on October 30, 2009, and laboratory analysis beginning on November 2, 2009.

3.6 Special Training Requirements/Certifications
The operation of the field analytical instruments requires specialized training that will be administered, prior to mobilization, to all START personnel scheduled to be onsite.

Data validation requires specialized training and experience. Project management must determine and verify a qualified data validation resource prior to data validation.

Field sampling personnel should be trained and have experience with soil sampling at hazardous waste sites while wearing appropriate protective equipment. One field sampler should be trained and familiar with Global Positioning System (GPS) data collection. All sampling personnel must have appropriate training that complies with 29 Code of Federal Regulations 1910.120. The site-specific health and safety plan for this project is to be appended to this plan by project management (Appendix B).

4 Sampling Rationale and Design

As discussed in previous sections of this SAP, the START reviewed available site information including recent sampling data and the U. S. EPA FOSC's objectives for the Assessment Action to determine the specific sampling design

Identification of lead contamination in residential soils within the West Oakland study area is the principal focus of this assessment operation.

The sampling design and rationale for the area of concern (AOC) is discussed below. Figure 2-2 shows the West Oakland residential AOC while Figure 4-1 indicated the tentative sample locations and decision units. Table 4-1 summarizes the identified decision unit and unique sampling locations by residential blocks and gives and estimate of total number of samples to be collected. Additional samples may be identified and investigated if directed by the U.S. EPA FOSC.

The Superfund Program Representative Sampling Guidance, Volume 1: Soil (OSWER Directive 9360.4-10, EPA 540/R-95-141, December 1995) was referenced during development of the sampling design. Table 4-1 provides a sampling summary of the AOC. After collection, samples will be handled and analyzed according to Sections 5.1, 6.2, and 6.3 of this SAP. Sample locations will be recorded in the field logbook as sampling is completed. Individual sample locations will be recorded using GPS equipment. The GPS location of the center point sample will be recorded where five point composite samples are collected.

The sampling design will generate samples that may have contaminants from leaded paint, areal lead deposition, naturally occurring lead and lead for source related to this investigation.

4.1 Selection of Decision Units

An aerial photograph review and site reconnaissance performed on September, 29 2009 was used to identify available sampling locations. Based upon this information, approximately one hundred unique areas that are not covered by concrete, asphalt or structure were identified. These areas of exposed soil, generally back yards, side yards, or front yard are the decision units for this investigation. A more detailed inspection of the AOC will be done once property access has been granted. Additional decision units are expected to be identified at this time.

To maintain the initial budgetary goal of one hundred sampling locations the decision units will be prioritized based upon physical and legal accessibility and distance from the AMCO NPL site.

4.2 Composite Sampling

Each decision unit will be sampled at five random systematic points. Equal volumes of soil from each sampling point will be composited into a single composite sample for anaylsis. The field X-ray fluorescence (XRF) operated in situ at the sampling points within the decision units may be used to generate data that can be used to calculate an estimation of standard deviation of the measurement mean to verify that it is less the 65 mg/kg needed to support a five point composite.

If the standard deviation of the mean for measurements is greater than expected the composite number can be increased to meet the situation.

4.3 Analyte of Concern

The analyte of concern is lead. All samples collected in the field will be field analyzed for lead using the XRF (U.S. EPA Method 6200C. Ten percent of the samples collected in the field will be sent to a laboratory for definitive analysis for lead (U.S. EPA Method 6010C).

The selected analyses will generate lead concentration data that may include lead from leaded paint, areal lead deposition from fuel combustion, naturally occurring lead and lead for source related to this investigation.

Table 4-1 Sampling Summary

Area	Identified Properties	Identified Decision Units and Unique Samples
Center Street West	4	6
Chester Street East	4	5
Chester Street West	7	11
Henry Street East	6	11
Henry Street West	6	10
Third Street North	3	6
Fifth Street South	11	22
Fifth Street North	8	12
Lewis Street East	4	7
Henry Street West	3	4
Paralta Street West	4	5
Estimated Total Unique Sample	60	99

[1] Note: Block 7 is three lots between Peralta and Lewis Streets

Source: 2009 Ecology and Environment, Inc.

5 Request for Analyses

Samples will be analyzed in the field for lead by U.S. EPA Method 6200. Samples will be analyzed at the U.S. EPA Region IX Laboratory in Richmond, California by U.S. EPA SW-846 method 6010C for lead.

5.1 Field Analysis

All samples collected will be analyzed in the field by START using the XRF. The manufacturer's guidance and SW-846 Method 6200 (Appendix C) will be used to conduct analysis.

To provide analytical quality control for the field analytical effort, the following measures will be utilized:

- Analytical precision and sensitivity of the XRF instrument in the determination of lead concentrations in site specific samples will be determined during the initial days of field analysis.
- The correlation between field lead data and data generated by standard U.S. EPA SW-846 method 6200 methodology will be determined during the initial days of field analysis.
- The START will submit a minimum of 10 percent of the soil samples analyzed in the field to an off-site laboratory for confirmation analysis of lead. At least 10 samples from the AOC will be submitted and represent the following ranges: less than the instrument detection limit, just below action level, just above the action level, and high lead concentrations, as determined by the field analysis, will be submitted to the laboratory for data correlation purposes.

5.2 Laboratory Analysis

A minimum of ten and a maximum of twenty percent of field-screened samples will be submitted to the U.S. EPA Regional laboratory for soil lead analysis using U. S. EPA method 6010C. Sample containers, preservatives, holding times, and estimated number of initial assessment samples, confirmation samples, and Quality Control (QC) samples are summarized in Table 5-1.

To provide analytical quality control for the analytical program, the following measures will be utilized:

- Additional sample volume will be collected for at least 5% of samples for the analytical method, to be utilized for matrix spike/matrix spike duplicate analysis.

Laboratory blind co-located duplicate samples or split duplicate samples will be collected from 10 percent of the sampling locations then submitted for soil analysis. A co-located duplicate sample is a composite sample that is collected and composited separately from its duplicate. A duplicate split sample is a 50/50 split of a sample after collection.

Table 5-1 Assessment Sampling and Analysis Summary

Method	Lead by U. S. EPA 6010C	Lead by XRF Field Analysis
Sample Container	125 or 250 ml glass jars (4 or 8 oz.)	Plastic sample bag
Preservation	4°C	N/A
Analysis Holding Time	6 months	6 months (if transferred to glass jar)
Sampling Location (expected start)	**Number of Samples**	**Estimated Number of Samples**
Unique Samples	(20%) 20	100
Field Co-located Duplicate Samples Duplicates	1	5
Split Duplicate Samples	1	5
Field Analysis Duplicates Detailed Below		
An analysis duplicate run in same batch (same XRF cup, run twice)	N/A	1 per 20 samples (6)
Preparation duplicate run in same batch (2 XRF cups prepared from same sample collection bag)	1	1 per 20 samples (6)
Blank run in same batch	N/A	1 per 10 samples (12)
Control Sample (field analysis only)	1	1 per 10 samples (12)
Total Initial Analyses	**24**	**146**
MS/MSDs	1 per 20 samples (1) Submit one-250 ml glass (8 oz.)	N/A

Source: 2009 Ecology and Environment, Inc.

Note:

A soil duplicate or a preparation duplicate will be prepared once every 10 samples. The type of duplicate, soil, or preparation will be alternated every 10 samples.

6 Field Methods and Procedures

6.1 Field Procedures

The following sections describe field procedures and equipment used during the site activities.

6.1.1 Equipment

The equipment listed below may be utilized to obtain environmental samples from the respective media in accordance with the following sampling SOPs or their equivalent:

- Environmental Response Team SOP #2012 Soil Sampling
- Ecology and Environment Inc. SOP # ENV 3.13: Soil Sampling
- Ecology and Environment Inc. SOP# ENV 3.15: Sampling Equipment Decontamination

The following is a partial list of equipment that is anticipated to come in contact with samples:

- Shovels, hand augers, trowels, scoops
- Stainless steel buckets or glass containers
- Dedicated plastic baggies and disposable trowels

6.1.2 Equipment Maintenance

Field instrumentation for the collection of soil samples will be operated, calibrated, and maintained by the sampling team in accordance with the SOPs listed in Section 6.1.1 or their equivalent. Field instrumentation utilized for health and safety purposes will be operated, calibrated, and maintained by the sampling team according to the manufacturer's instruction. Calibration and field use data will be recorded in the instrument log books.

6.1.3 Inspection/Acceptance Requirements for Supplies and Consumables

There are no project-specific inspection/acceptance criteria for supplies and consumables. It is standard operating procedure that personnel will not use broken or defective materials; items will not be used past their expiration date; supplies and consumables will be checked against order and packing slips to verify the correct items were received; and the supplier will be notified of any missing or damaged items.

6.1.4 Logbooks

Field logbooks will document where, when, how, and from whom any vital project information was obtained. Logbook entries will be complete and accurate enough to permit reconstruction of field activities. A separate logbook will be maintained for each project. Logbooks are bound with consecutively numbered pages. Each page will be dated and the time of entry noted in military time. All entries will be legible, written in ink, and signed by the individual making the entries. Language will be factual, objective, and free of personal opinions. The following information will be recorded, if applicable, during the collection of each sample:

- Sample location and description
- Site sketch showing sample location and measured distances

- Sampler's name(s)
- Date and time of sample collection
- Type of sample (matrix)
- Type of sampling equipment used
- Onsite measurement data (e.g., temperature, pH, conductivity)
- Field observations and details important to analysis or integrity of samples (rain, odors, etc.)
- Type(s) of preservation used
- Field instrument reading (such as Lumex readings for health and safety purposes, etc.)
- Shipping arrangements (air bill numbers)
- Receiving laboratory(ies)

Several START team members will be onsite performing different duties related to sample collection, processing, and analysis. Individual logbooks will be maintained for specific activities at the site, including: Sample collection, sample log-in to the field laboratory, XRF analysis. Each logbook will document the information relevant to the site activity, and at a minimum will include:

- Team members and their responsibilities
- Time of activities
- Deviations from sampling plans, site safety plans, and SAP procedures
- Levels of safety protection
- Calibration information
- Analytical data

6.1.5 Photographs

Photographs will be taken at representative sampling locations and at other areas of interest onsite. They will serve to verify information entered in the field logbook. When a photograph is taken, the following information will be written in the logbook or will be recorded in a separate field photography log:

- Time, date, location, and, if appropriate, weather conditions
- Description of the subject photographed
- Name of person taking the photograph

6.1.6 Electronic Sample Logging

The sampling team may utilize field management software to prepare sample labels and chain-of-custody forms.

The following information should be entered for each sample after collection:

- Sample name
- Sample date and time
- Number of Sample bottles
- Type of Preservation
- Analyses

In addition to these items, the software may also be used to keep track of other information such as sample depth, field measurements (e.g., pH), and split samples.

The field team will generate chain-of-custody forms for each cooler of samples packaged and sent to a laboratory. Each chain-of-custody form will refer to the shipping method and tracking number. Printed chain-of-custody forms will be submitted to the laboratory with the samples.

The use of field management software will require that the field team have access to a computer, a printer, computer paper, and labels while in the field. Field team members will have received specific training in use of the software.

6.1.7 Mapping Equipment

Sample points and site features will be located and documented with a GPS unit. The GPS will be used to assign precise geographic coordinates to sample locations on the site. GPS mapping will be done by personnel trained in the use of the equipment and will be completed in accordance with the manufacturer's instructions. Expected output from the use of GPS mapping will be site maps with sample locations and major site features.

6.2 Surface Soil Sampling Procedures

All sample locations will be recorded in the field logbook as sampling is completed. Each field sampling team will document each individual sampling locations on a field sampling sheet, in which includes: the site address, area sample was collected with a quick representative sketch of the area, GPS coordinates of the sample, photographs taken, date, time, and sampling team members.

6.2.1 Collection Procedure

Surface soil samples will be collected from a depth of 0-6 inches below ground surface (bgs). Surface samples will be collected using a disposable plastic or stainless steel trowel and will be placed in a plastic zip-lock bag for holding and homogenization. A composite sample will be collected from five points within each sampling location. Approximately 2 ounces of soil will be collected from each of the five collection points. A portion of each sample point to be composited will be kept separate for potential future analysis. The soil will be placed into a zip-lock sampling bag.

6.3 Field Analytical Procedures

Soil samples will be field analyzed for total lead using U. S. EPA Method 6200. All total lead analyses using the XRF will be completed in accordance with manufacturer's guidance and the

U. S. EPA SW-846 Method 6200 (Appendix C). Additionally, field duplicate samples, second source control samples, and blanks will be analyzed and evaluated as quality control checks as described in Section 9.1 of this SAP.

Samples will be delivered to the field laboratory in heavy-duty plastic bags. Upon receipt the samples will be logged into the analytical logbook. Twigs, other organic matter and rocks or pebbles will be removed from the samples. Samples will be homogenized while in the sample bag by kneading, crushing, and shaking the sample until mixing of the soil is complete. If the sample is wet, a 30-gram or more aliquot of the sample will be placed in a sample boat or on a coffee filter to air dry. Once the aliquot has dried, it will be placed in a clean bag and homogenized. After the sample is dried, it will be passed through a size #60-mesh sieve to remove large particles. The remaining aliquot will be transferred to a pre-labeled polyethylene cup and covered with Mylar film to be analyzed by XRF.

Sample analysis will be performed in accordance with the manufacturer's guidance and SW-846 Method 6200 (Appendix C). At the beginning of the project and prior to analysis of samples, the START will perform quality control checks including energy calibration, resolution check, background check, and a precision sample analysis. Daily quality control checks to be performed include resolution check, background check, initial calibration verification, method blank, continuing calibration verification, and an instrument blank analysis. Once calibrated and at the end of each set of 10 samples, a second source control standard and sand blank will be analyzed to determine instrument performance. One out of every 10 samples will be selected for a preparation duplicate.

Initial and continuing calibration verifications will be completed using standards at and below the site action level.

One out of every 10 samples will be selected for an analysis duplicate.

After field analysis has been completed, samples for laboratory confirmation analysis will be selected. The START will submit 10 percent of the soil samples analyzed in the field to a laboratory for confirmation analysis of lead. There must be a minimum of ten samples submitted. At least 10 samples representing the dynamic range of non-detect, just below action level, just above action level, and high lead concentrations, as determined by the field analysis, will be submitted to the laboratory for data correlation purposes. The remainder of the confirmatory samples will be selected at the discretion of the START Project Manager but should be somewhat random.

Selected samples will then be transferred from the holding bag to the appropriate sample containers. Samples selected for laboratory analysis will be placed in 4 or 8-ounce jars. Sample containers will be filled to the top, taking care to prevent soil from remaining in the threads prior to being closed to prevent potential contaminant migration to or from the sample. Sample containers will be closed as soon as they are filled, chilled, and processed for shipment to the laboratory. In addition to the jar, the cup that was analyzed using the XRF will be sent to the laboratory for analysis. The 4- or 8-ounce jar will be used to determine percent moisture. The cup and sample jar will be chilled pending shipment to the laboratory. All remaining sample volume will be returned to its point of origin.

6.4 Field Analytical Decontamination Procedures

Decontamination activities will be conducted by the START in accordance with E & E SOP #3.15. All non-dedicated sample handling devices will be decontaminated according to the following procedure:

- Non-phosphate detergent and tap water wash using a brush to scrub solids from the surface
- Tap water rinse
- 10% nitric acid rinse
- Triple deionized/distilled water rinse

The soil sieves, used during preparation of a sample for analysis with the XRF, will be decontaminated by brushing out the excess soil with coarse-hair brushes and wiping out with a paper towel and a small amount of rubbing alcohol. Decontamination procedures for the soil sieves deviate from E & E SOP #3.15 due to the drying time that would be required for the fine mesh sieve.

7 Disposal of Investigation-Derived Waste

In the process of collecting environmental samples at this site, several different types of potentially contaminated investigation derived wastes (IDW) will be generated, including the following:

- Used personal protective equipment (PPE)
- Disposable sampling equipment
- Decontamination fluids

The USEPA's National Contingency Plan required that management of IDW generated during site investigations comply with all relevant or appropriate requirements to the extent practicable. This sampling plan will follow the Office of Emergency and Remedial Response Directive 9345.3-02 (May 1991), which provides the guidance for management of IDW during site investigations. Listed below are the procedures that will be followed for handling IDW. The procedures are flexible enough to allow the site investigation team to use its professional judgment on the proper method for the disposal of each type of IDW generated at each sampling location.

- Used PPE and disposable sampling equipment will be double bagged in plastic trash bags and disposed of in a municipal refuse dumpster. These wastes are not considered hazardous and can be sent to a municipal landfill. Any PPE or dedicated equipment that is to be disposed of that can still be reused will be rendered inoperable before disposal.
- Decontamination fluids will consist of water with residual contaminants and/or non-phosphate detergent. These fluids will be left onsite to evaporate.

8 Sample Identification, Documentation and Shipment

8.1 Sample Nomenclature

A unique, identifiable name will be assigned to each sample. Samples will have a prefix indicating the street block of the site from which they were collected (i.e. Block 1 (B1)). The prefix will be followed by the specific house number in which the samples were collected from. All samples will have a final one letter indicating the area of the yard in which the sample was collected from; front yard (F), back yard (B), or side yard (S).

Field duplicate samples will have the same designations as their originals except the initial block number will be followed by zero (0); thus, the field duplicate of B1-120-F will be B10-120-F. XRF preparation duplicate samples will have the same designations as their originals except the sample number will be followed with a "PD"; thus, the preparatory duplicate of B1-120-F will be B1-120-F PD. A summary of this sample naming system is shown in Table 8-1.

Table 8-1 Sample Numbering System

Site Area	Location	Prefix	Sample ID
ALL	All Sampling Locations	Prefix of pre-determined Block #	B1-address #[1]-yard area[2]
ALL	Field Duplicate	Prefix of pre-determined Block #	B1<plus 0>-address #-yard area
ALL	Preparation Duplicate for U. S. EPA 6200	Prefix of pre-determined Block #	B1-address #-yard area PD

Source: 2009 Ecology and Environment, Inc.

Notes:

[1] Address number is the corresponding house/property number

[2] Yard area is the section of yard in which sample was collected; a one letter indicator (i.e., front yard = F

8.2 Container, Preservation, and Holding Time Requirements

All sample containers will have been delivered to the START in a pre-cleaned condition. Container, preservation, and holding time requirements are summarized in Tables 5-1 and Table 5-2.

8.3 Sample Labeling, Packaging, and Shipping

All samples collected will be labeled in a clear and precise way for proper identification in the field and for tracking in the laboratory. Sample labels will be affixed to the sample containers and will contain the following information:

- Sample number
- Date and time of collection

- Site name
- Analytical parameter and method of preservation

Samples will be stored in a secure location onsite pending onsite analysis and shipment to the laboratory. Sample coolers will be retained in the custody of site personnel at all times or secured so as to deny access to anyone else.

The procedures for shipping soil samples are:

- If ice is used then it will be packed in double zip-lock plastic bags.
- The drain plug of the cooler will be sealed with tape to prevent melting ice from leaking.
- The bottom of the cooler will be lined with bubble wrap to prevent breakage during shipment.
- Screw caps will be checked for tightness.
- Containers will have custody seals affixed so as to prevent opening of the container without breaking the seal.
- All glass sample containers will be wrapped in bubble wrap.
- All containers will be sealed in zip-lock plastic bags.

All samples will be placed in coolers with the appropriate chain-of-custody forms. All forms will be enclosed in plastic bags and affixed to the underside of the cooler lid. If samples require refrigeration during shipment then bags of ice will be placed on top of and around samples. Empty space in the cooler will be filled with bubble wrap or Styrofoam peanuts to prevent movement and breakage during shipment. Each ice chest will be securely taped shut with strapping tape, and custody seals will be affixed to the front, right, and back of each cooler.

Samples will be shipped for immediate delivery to the contracted laboratory. Upon shipping, the laboratory will be notified of:

- Sampling contractor’s name.
- The name of the site.
- Shipment date and expected delivery date.
- Total number of samples, by matrix and the relative level of contamination for each sample (i.e., low, medium, or high).
- Carrier; air bill number(s), method of shipment (e.g., priority).
- Irregularities or anticipated problems associated with the samples.
- Whether additional samples will be sent; whether this is the last shipment.

8.4 Chain-of-Custody Forms and QA/QC Summary Forms

A chain-of-custody form will be maintained for all samples to be submitted for analysis, from the time the sample is collected until its final deposition. Every transfer of custody must be noted

and a signature affixed. Corrections on sample paperwork will be made by drawing a single line through the mistake and initialing and dating the change. The correct information will be entered above, below, or after the mistake. When samples are not under the direct control of the individual responsible for them, they must be stored in a locked container sealed with a custody seal. The chain-of-custody form must include the following:

- Sample identification numbers
- Identification of sample to be used for Matrix Spike/Matrix Spike Duplicate (MS/MSD) purposes
- Site name
- Sample date
- Number and volume of sample containers
- Required analyses
- Signature and name of samplers
- Signature(s) of any individual(s) with control over samples
- Airbill number
- Note(s) indicating special holding times and/or detection limits

The chain-of-custody form will be completed and sent with the samples for each laboratory and each shipment. Each sample cooler should contain a chain-of-custody form for all samples within the sample cooler.

A QA/QC sample summary form will be completed for each method and each matrix of the sampling event. The sample number for all blanks, reference samples, laboratory QC samples (MS/MSDs), and duplicates will be documented on this form. This form is not sent to the laboratory. The original form will be sent to the reviewer who is validating and evaluating the data; a photocopy of the original will be made for the project manager master file.

9 Quality Assurance and Control (QA/QC)

9.1 Field Quality Control Samples

The QA/QC samples described in the following subsections, which are also listed in Tables 5-1, will be collected during this investigation.

9.1.1 Assessment of Field Contamination (Blanks)

9.1.1.1 Equipment Blank Samples

Equipment rinsate blanks will not be collected to evaluate field sampling and decontamination procedures since all sampling equipment will be dedicated.

9.1.1.2 Field Blank

Field blanks will not be collected to evaluate whether contaminants have been introduced into the samples during soil sampling procedures.

9.1.2 Assessment of Sample Variability (Field Duplicate or Co-located Samples

Duplicate soil samples will be collected at selected sample locations. These locations will be chosen in the field based on field observations and will be collected at a rate of 1 for every 10 field samples.

9.1.3 Laboratory Quality Control (QC) Samples

A laboratory QC sample, also referred to as a matrix spike/matrix spike duplicate (MS/MSD), is not an extra sample; rather, it is a sample that requires additional QC analyses and therefore may require a larger sample volume. The chain-of-custody records for these samples will identify them as laboratory QC samples. The location of laboratory QC samples will be selected at random. A minimum of one laboratory QC sample will be submitted per 20 samples (or one per delivery group), per matrix, to be analyzed for each analytical parameter. If the DQIs for analytical parameters are not achieved, further data review will be conducted to assess the impact on data quality. Laboratory QC samples, including laboratory MS/MSD and field duplicate samples, will be selected randomly.

Additional sample volume will be submitted for all mercury samples designated as laboratory QC samples and will be designated as MS/MSD samples on the chain-of-custody to the fixed-base laboratory.

9.1.4 Conformation Samples

The samples submitted to the laboratory for definitive analysis will be used to establish and/or document the comparability and correlation between the definitive and non-definitive data sets. The START will determine correlation of the data sets by linear regression analysis and will determine relative percent differences for each data pair and for the data sets as a whole. These results will be compared to the field screening data and will be used to determine the effectiveness of the field screening technique.

9.1.5 Field Analytical Quality Control (QC) Samples

Field analytical QC samples, also referred to as precision samples, calibration verification samples, and control standards, will be analyzed with field samples to verify and document the precision and accuracy of field analytical methods. QC samples include blanks, preparation duplicates, analysis duplicates, and check standard from two different sources.

9.2 Analytical and Data Package Requirements

It is required that all samples be analyzed in accordance with U.S. EPA Methods listed in Tables 5-1. The laboratory is required to supply documentation to demonstrate that their data meet the requirements specified in the method. A preliminary data summary will be required 15 working days after submission of samples for analysis. A full validation data package will be required five weeks after submission of samples. The laboratory (ies) will also provide all data electronically in a Lotus123-compatible format or delimited text file.

Deliverables for this project must meet the guidelines in *Laboratory Documentation Requirements for Data Evaluation* (EPA Region IX R9/QA/00.4.1, March 2001). The following deliverables are required. Note that the following data requirements are included to specify and emphasize general documentation requirements and are not intended to supersede or change requirements of each method.

- A copy of the chain-of-custody, sample log-in records, and a case narrative describing the analyses and methods used.
- Analytical data (results) for up to three significant figures for all samples, method blanks, MS/MSD, Laboratory Control Samples (LCS), duplicates, Performance Evaluation (PE) samples, and field QC samples.
- QC summary sheets/forms that summarize the following:
  - MS/MSD/LCS recovery summary
  - Method/preparation blank summary
  - Initial and continuing calibration summary (including retention time windows)
  - Sample holding time and analytical sequence (i.e., extraction and analysis)
  - Calibration curves and correlation coefficients
  - Duplicate summary
  - Detection limit information
- Analyst bench records describing dilution, sample weight, percent moisture (solids), sample size, sample extraction and cleanup, final extract volumes, and amount injected.
- Standard preparation logs, including certificates of analysis for stock standards.
- Detailed explanation of the quantitation and identification procedure used for specific analyses, giving examples of calculations from the raw data.
- The final deliverable report will consist of sequentially numbered pages.

9.3 Data Management

Samples will be collected and described in a logbook, as discussed in Section 6.1.2.1. Samples will be kept secure in the custody of the sampler at all times; the sampler will assure that all preservation parameters are being followed. All samples are being submitted to an onsite field laboratory for field analysis. The field analysis laboratory will document sample receipt in an analytical logbook. All samples that are to be sent to the analytical laboratories will be collected and logged on chain-of-custody forms as discussed in Section 8.4. A START member will only submit samples to the analytical laboratory with chain-of-custody documentation. All submitted samples will be in a properly custody-sealed container. Specifics are discussed in Section 8.3. The laboratories will note any evidence of tampering upon receipt.

All data summary reports and complete data packages will be archived by the project manager. The data validation reports and laboratory data summary reports will be included in the final report to be submitted to the EPA. All field data including, XRF, will be managed in SCRIBE.

9.4 Data Validation

Data validation of all data will be performed by the START or their subcontractor in accordance with U.S. EPA Region IX Superfund Data Evaluation/Validation Guidance R9QA/006.1, December 2001.

Standard data quality review requirements, is Tier 2 validation of 100 percent of the data (as defined in *Requirements for Quality Assurance Project Plans*, March 2001), will satisfy the data quality requirements for this project. Upon completion of validation, data will be classified as one of the following: acceptable for use without qualifications, acceptable for use with qualifications, or unacceptable for use.

If during or after the evaluation of the project's analytical data it is found that the data contains excess QA/QC problems or if the data does not meet the DQI goals, then the independent reviewer may determine that additional data evaluation is necessary. Additional evaluation may include U. S. EPA Region IX Superfund Data Evaluation/Validation Guidance R9QA/006.1 for evaluation Tier 3.

To meet evaluation and project requirements, the following criteria will be evaluated during a Tier 2 evaluation:

- Data package completeness
- Laboratory QA/QC summaries
- Holding times
- Blank contamination
- Matrix related recoveries
- Field duplicates
- Random data checks

    - Preservation and holding times

- Initial and continuing calibration
- Blank analyses
- Interference check samples
- Laboratory control samples
- Duplicate sample analysis
- Matrix spike sample analyses
- Sample serial dilution
- Field duplicate/replicate
- Overall assessment of data.

Upon completion of evaluation, an analytical data evaluation Tier 2 review report will be delivered to the project manager, and the data will be classified within the report as one of the following:

- acceptable for use without qualifications
- acceptable for use with qualifications
- unacceptable for use

The data with applicable qualifications will be attached to the report. The analytical data evaluation Tier 1A review report will not compare data to specific project quality objectives, which include target analytes, sensitivity, analytical accuracy, analytical and sampling precision, and analytical completeness.

Unacceptable data may be more thoroughly examined to determine whether corrective action could mitigate data usability.

9.5 Field Variances

As conditions in the field may vary, it may become necessary to implement minor modifications to this plan. When appropriate, the START QA Coordinator will be notified of the modifications and a verbal approval obtained before implementing the modifications. Modifications to the original plan will be recorded in site records and documented in the final report.

9.6 Assessment of Project Activities

9.6.1 Assessment Activities

The following assessment activities will be performed by the START:

- All project deliverables (SAP, Data Summaries, Data Validation Reports, Investigation Report) will be peer reviewed prior to submission to the U.S. EPA. In time critical situations, the peer review may be concurrent with the release of a draft document to the U.S. EPA. Errors discovered in the peer review process will be reported by the reviewer to the originator of the document, who will be responsible for corrective action.
- The QA Coordinator will review project documentation (logbooks, chain-of-custody forms, etc.) to ensure the SAP was followed and that sampling activities were adequately documented. The QA Coordinator will document deficiencies, and the PM will be responsible for corrective actions.

9.6.2 Project Status Reports to Management

It is standard procedure for the START PM to report to the U.S. EPA Task Monitor (TM) any issues, as they occur, that arise during the course of the project which could affect data quality, data use objectives, the project objectives, or project schedules.

As requested, the START will provide XRF results to the U.S. EPA TM daily and unvalidated data will be provided as it is received from the laboratory.

9.6.3 Reconciliation of Data with DQOs

Assessment of data quality is an ongoing activity throughout all phases of a project. The following outlines the methods to be used by the START for evaluating the results obtained from the project.

Review of the DQO outputs and the sampling design will be conducted by the START QA Coordinator prior to sampling activities. The reviewer will submit comments to the START PM for action, comment, or clarification. This process will be iterative.

A preliminary data review will be conducted by the START. The purpose of this review is to look for problems or anomalies in the implementation of the sample collection and analysis procedures and to examine QC data for information to verify assumptions underlying the DQOs and the SAP. When appropriate to sample design, basic statistical quantities will be calculated and the data will be graphically represented. When appropriate to the sample design and if specifically tasked to do so by the U.S. EPA TM, the START will select a statistical hypothesis test and identify assumptions underlying the test.

10 Report References

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U.S. EPA, 2009. *Regional Screening Levels for Chemical Contaminants at Superfund Sites*, April

A Data Quality Objective Process Worksheet

West Oakland
Residential Lead Sampling

Data Quality Objectives (DQO) Process Document
Objective Outputs

Contract: EP-S5-08-01
TDD No.: 09-09-09-0001
Job No.: 002693.2052.01RA

This DQO documentation version reflects the initial project objectives as of October 2009.

1. THE PROBLEM

Background

The South Prescott Residential Neighborhood of West Oakland is the subject of a United States Environmental Protection Agency (U.S. EPA) Region IX Emergency Response Section (ERS) residential soil Lead (Pb) Assessment. The site is located adjacent to the former AMCO Corporation chemical facility (AMCO) National Priorities List (NPL) site and subsequently the DC Metals, Inc. scrap metals site (DC Metals) at 1414 Third Street in Oakland, Alameda County, California. Community concerns arose from a 2007 Remedial Investigation, conducted by the U.S. EPA, in which identified elevated levels of lead in residential soils immediately adjacent to the former AMCO/DC Metals property on Third and Center Streets. This investigation concerns an area of approximately 6 residential blocks located immediately west and northwest of the former AMCO and DC Metals property, known as the West Oakland Residential lead sampling site (site).

The site is defined by residential properties located between Center Street to the east, Third Street to the south, Peralta Street to the west, and the Bay Area Rapid Transit (BART) rail line to the north.

The site is comprised of approximately 130 to 175 residential lots (approximately 23 acres). The site was historically bordered by the former AMCO and DC Metals facilities to the east, which included a railroad spur, above-ground tanks and drums, and scrap metal operations.

Previous investigations have documented that soil at the former AMCO and DC Metals property, as well as soil at residential homes immediately west on Center Street, are contaminated with lead above the U.S. EPA's Regional Screening Levels (RSLs) for Residential Soils, April, 2009.

In 2007, the U.S. EPA conducted a Removal Assessment at residential properties bordering the former AMCO and DC Metals property. This investigation revealed concentrations of lead in bordering residential soils of up to 2700 parts per million (ppm). This prompted the U.S. EPA to

conduct a Remedial Soil Removal at eight (8) residential properties containing lead contaminated soils. According to the available documentation, this investigation is the most recent investigation conducted for lead-contaminated soils within the West Oakland residential neighborhood.

Based on conditions documented by the investigations described above, the U.S. EPA is conducting a Removal Assessment to address data gaps described later in this document. The objectives discussed herein are for the approximate 6 block residential neighborhood area located immediately west and northwest of the formerly assessed/remediated properties at the site.

Conceptual Site Model
Residential Area located west of the former AMCO and DC metals facilities and previously remediated residential properties

- The medium of concern is soil.
- The principal Contaminant of Primary Concern (COPC) is Lead (Pb) above current U.S. EPA RSLs for residential soil.
- The soil medium was potentially contaminated with the COPC due to release of contaminants from adjacent AMCO and DC Metals site operations.

Exposure Scenario
Residential Area soils surrounding the former known hazardous waste operations site

- Concerns based on previous adjacent property conditions include (1) direct exposure of human and/or environmental receptors to contaminants in soil, (2) exposure to contaminated soil that has migrated as particulate matter (dust), and (3) exposure to contaminated water runoff. The residential area is susceptible to direct exposure to human receptors.
- Conditions at the site may pose an additional threat to human health during and/or after potential future construction activities that require grading or excavation in the area. Direct and airborne exposure of human and/or environmental receptors to COPC-contaminated soil and soil-derived particulates are of concern during potential future construction. Previous investigations conducted at the adjacent residential properties identified contaminated surface soils.
- The excess soil generated during potential future site construction may pose a threat to human health during transportation and disposal.
- Development of the properties without investigation and associated remedial action to address potential contamination could expose human and/or environmental receptors to COPCs in soil associated with or derived from this source area.

Available Resources
The current START budget for objective planning and development of a U.S. EPA-approved Sampling and Analysis Plan (SAP) is approximately $6,500.

The available budget for the Removal Assessment currently allocated to the START is $28,200. Other budget constraints on U.S. EPA resources for this project have not been specified. The

primary decision-makers for the project are Federal On-Scene Coordinators (FOSCs) Steve Calanog and Chris Reiner

Planning Team

Mr. Steve Calanog and Mr. Chris Reiner, U.S. EPA FOSCs
Mr. David Neil Ellis, Ecology and Environment, Inc., (E & E) Superfund Technical Assessment and Response Team (START), Project Manager
Mr. Howard Edwards, E & E START, Quality Assurance Officer

Roles and Responsibilities

- The U.S. EPA FOSCs will be the primary decision-makers and will direct the project, specify tasks, and ensure that the project is proceeding on schedule and within budget. Additional duties include coordination of all preliminary and final reporting and communication with the START Project Manager and U.S. EPA Quality Assurance (QA) Office. The U.S. EPA FOSC is also responsible for access to each property to be investigated.

- David Neil Ellis, the START Project Manager, will coordinate with the planning team to develop objectives and complete an approved SAP.

- Howard Edwards, START QA Officer, will oversee development and preparation of the SAP and other START deliverables. Mr. Edwards will provide overall project quality assurance and, if necessary, audit functions.

- START will be responsible for implementation of the SAP, coordination of project tasks, coordination of field sampling, project management, and completion of all preliminary and final reporting.

- The START will use the U.S. EPA Region 9 Laboratory to perform lead sample analysis.

- START or a START contractor will be responsible for data validation if a laboratory other than the U.S. EPA Region 9 Laboratory or a Contract Laboratory Program (CLP) laboratory must be used for any reason.

Other Considerations and Constraints Related to Problem and Resources

- Soil analyses available for assessment are not always useful for determining disposal and remediation costs. Additional waste testing of excavated soil is usually necessary to determine disposal requirements.
- Contamination not found during the soil investigation might be revealed during excavation activities.
- Access and Access agreements to each property.
- Lead concentration on a property may derive for sources that include, but are not limited to: historic use of leaded paint on exterior wall, lead deposition form the us of leaded containing petroleum products, and naturally occurring lead in native soil.

2. THE DECISION

Principal Study Questions

Principal Study Questions : Previous investigations in the vicinity of the *West Oakland Residential site* indicate that surface soils, both at the nearby former hazardous waste operation site and immediately adjacent residential properties, are contaminated with elevated concentrations of lead. What are lead concentrations in exposed residential soils within the study area (*West Oakland Residential site)*? What is lead concentration distribution in individual residential properties within the study area? What is the estimated volume of contaminated soil that is above the action level?

Actions that could result from resolution of the study questions
For Primary Study Questions (regarding the *West Oakland Residential site*):

If it is determined that exposed soils at individual residential properties have lead in soil concentration greater then the action limit, then the information may be used to determine what will need to be excavated/remediated or it may be determined that additional investigation of the property is required.

If it is determined that exposed soils at individual residential properties have lead in soil concentration less then the action limit, then no further action regarding the property will be required.

Decision Statement(s)
For the *Study Area:*
Analytical data will be used to evaluate if soil contains lead concentrations greater than the site-specific action levels in order to determine whether additional investigations is necessary and to assist with determining areas and the quantity of soil that might need to be excavated/ remediated.

3. DECISION INPUTS

New environmental data required to resolve the decision statements
Area surrounding the former hazardous waste operations site within nearby residential soils

- COPC data for soils between 0 and 6 inches below ground surface (bgs) within the residential study area are required.
- Geospatial data for the area and associated sampling locations are needed.

Sources of information to resolve the decision statements

- Visual survey data and global positioning system (GPS) data
- Field analysis data generated by a X-Ray Fluorescence (XRF) Spectrometry from proposed soil sampling
- Analytical data for confirmation analysis from proposed soil sampling
- Risk-based action levels for the COPC

Information Needed To Establish Action Level
Potential action levels for COPCs may come from the following sources:

- April 2009 U.S. EPA RSLs – Residential Soils
- Lawrence Berkeley National Laboratory; Analysis of background distributions of metals in the soil, 2009

Collection methods
Soil samples can be collected using a trowel, hand auger, or shovel.

Measurement methods
Collected soil samples can be analyzed to determine COPC concentrations using the following definitive SW-846 methods:

- U.S. EPA method 6010C for Lead (Pb)

Collected soil samples can be analyzed to determine COPC concentrations using the following less definitive methods:

- U.S. EPA method 6200 using a Portable Field X-Ray Fluorescence (XRF) Spectrometry for analysis of Lead.

Confirm that appropriate analytical methods exist to provide the necessary data:
The definitive and non definitive U.S. EPA methods have sufficient sensitivity, accuracy, precision, and other quality parameters to generate necessary data, provided the data are not needed within a critical timeframe. Field XRF methods can generate time-critical data; however, sensitivity, qualitative selectivity, and quantitative accuracy for these methods will require confirmation by a definitive method.

4. STUDY BOUNDARIES

Specific characteristics that define population being studied

Area Surrounding the former hazardous waste operations site at residential properties

- The COPC concentrations in soil within the specified spatial and temporal boundaries.

- The spatial distribution of COPCs within the specified spatial and temporal boundaries.

Spatial boundaries

The boundary will encompass residential homes located to the west and northwest of the former AMCO and DC Metals hazardous waste operations site located at 1414 Third Street in Oakland, California. The area can initially be described as an approximately 6 blocks of residential properties (approximately 23 acres); bounded by Center Street to the east, Third Street to the south, Peralta Street to the west, and the BART rail line to the north, with depth of 0 to 6 inches bgs.

Temporal boundaries

The decisions will apply to determinations of risk associated with long-term exposure to contaminated surface soil from direct exposure. However, decisions may also apply to short-term (acute) exposure to contaminated soil due to potential development activities.

Lead is environmentally persistent and migrates slowly, so soil concentrations generally do not vary greatly over time. Given the location and human accessibility along with the existing community potential threats are expected to be immediate or imminent.

Thus, the following assessment time-frame has been proposed:

- The SAP will be submitted to U.S. EPA FOSCs by October 13, 2009, and should be reviewed and revised by October 26, 2009, the first day of proposed work.
- Sample collection will take place following SAP approval by the U.S. EPA.
- Preliminary data should be available within 3 weeks of sample delivery to the laboratory.
- Data packages and final data should be reported to project management approximately 5 weeks after sample delivery to the laboratory.
- Laboratory data for lead should be evaluated following U.S. EPA Region 9 Tier 2 guidance. Evaluated data should be reported to project management approximately 4 to 6 weeks after sample delivery to the laboratory.
- Decision statement resolutions are expected to occur approximately 6 weeks after sampling and should take place prior to decisions that may result in exposure of residents or workers to lead contamination soil.

Scale of decision-making

For the *residential area to the west and northwest of the former hazardous waste operations site:* A decision unit will be sampling location (i.e. Such as the back yard at a specific address) The distribution of lead through out the entire study area will also be evaluated. The sampling at residential properties is described in Section 7.

Practical constraints on data collection

Physical constraints

- Sampling refusal for subsurface samples will limit vertical sampling. Repeated attempts to sample at refusal locations (or alternate locations) will proceed within practical time and effort constraints.
- Irrigation/drainage ditch may be filled with water. No water samples will be collected.

Other constraints on data collection

- The turnaround times on data are always estimated and cannot be assured. Sample and system problems may indiscriminately increase data turnaround times.
- Definitive data will undergo a U.S. EPA Region IX Tier 2 validation review prior to final reporting. Problems identified during this review may initiate additional data reviews, which will increase the time needed before data are finalized.
- Specific data may be qualified or rejected based on the results of the data review process.
- Civil constraints such as site access agreements and permit requirements may exist and, if so, will need to be addressed prior to sampling.

5. DECISION RULE

Statistical Parameter

Surrounding the former hazardous waste operations site at residential properties

The geographic distribution of contamination and the range of contaminant concentrations define the statistical population of interest. It will be necessary to consider an individual sampling data point (which is not a statistical parameter) as representing the contaminant concentration within a specific area.

Action Level

Refer to Table 1 for soil action levels.

Decision Rule

1. If the new data indicate that a decision unit is not contaminated above the applicable action-level, then that decision unit will not be considered in need of further investigation or soil removal/ remediation.

2. If the new data indicate that a decision unit is contaminated above an applicable action level, then that decision unit will be considered in need of further investigation or soil removal/ remediation.

3. If the new data for entire study area does not resolve the lead distribution for the study area, then the decision-maker will report data and make recommendations on additional sampling.

4. If the new data for entire study area does resolve the lead distribution for the study area, then the decision-maker will report data and considered in need of further investigation or soil removal/ remediation.

Table 1 – Potential Soil Action Levels
West Oakland Residential Lead Sampling
Oakland, California
All units milligrams per kilogram (mg/kg)

E & E Project No.: 002693.2052.01RA TDD No.: TO2-09-09-09-0001

	RSLs[1] Residential Direct Exposure	CHHSLs[2] Residential Direct Exposure	ESLs[3] Shallow Soils In Residential Land Use	Elevated Range Detected at surrounding area
Lead (Pb)	400	150	200	0 to 2,700

Notes:
[1]The U.S. EPA, Regional Screening Levels, April 2009
[2] The California EPA, California Human Health Screening Levels, January 2005
[3] The California Regional Water Quality Control Board, Environmental Screening Levels, May 2008

Key:
RSLs = Regional Screening Levels
CHHSLs = California Human Health Screening Levels
ESLs = Environmental Screening Levels

6. LIMITS ON DECISION ERRORS

Range of the parameter(s) of interest
For all investigation areas and parameters, the range of interest for COPCs is from ½ the action level to anything above the action levels. Quantitatively precise and accurate determinations of contaminant concentrations that are significantly above (i.e., >100 times) the action level are not necessary.

Based on past investigations, the soil contaminant concentrations are expected to be above action levels.

Baseline Condition *(the Null Hypothesis)*
Residential Area soils surrounding the former known hazardous waste operations site
The contaminant concentrations in soils are greater than or equal to action levels.

Alternative Condition *(the Alternative Hypothesis)*
Residential Area soils surrounding the former known hazardous waste operations site
The contaminant concentrations in soils are less than action levels.

Decision Errors

Table 2 – Decision Error
West Oakland Residential Lead Sampling
Oakland, California

E & E Project No.: 002693.2052.01RA TDD No.: TO2-09-09-09-0001

Decision Error	Deciding that an area or a location within an area is contaminated and requires restriction, mitigation, or additional investigation when the property is not contaminated.	Deciding that an area or a location within an area is not contaminated and requires no restrictions, mitigation, or additional investigation when the property is contaminated.
True Nature of Decision Error	The sample concentrations are either not representative or are biased high.	The sample concentrations are either not representative or are biased low.
The Consequence of Error	Site will undergo additional mitigating activities. These situations would cost additional resources of time, money, and human resources.	The residents in the area could be exposed to existing contaminants.
Which Decision Error Has More Severe Consequences Near the Action Level?	**Less Severe** to human health, but with appreciable economic consequences.	**More Severe** since the contaminated soil may pose risks to human health and/or the environment.
Error Type Based on Consequences	**False Acceptance Decisions** A decision that the area is contaminated when it is not.	**False Rejection Decisions** A decision that the area is not contaminated when it is.

Definitions
A false acceptance decision error occurs when the null hypothesis is not rejected when it is false.
A false rejection decision error occurs when the null hypothesis is rejected when it is true.

Soil Decision Error Limit goals

In order address the study question, decisions will be made on relatively small areas where there is no previous sampling data thus there is no information on the expected sample variance or on the standard deviation. To meet the objectives in Table 3 using a 5 point sample composite the concentration variance must not exceed 65 mg/kg standard deviation.

Table 3 – Decision Error Limit Goals (Soil)
West Oakland Residential Lead Sampling
Oakland, California

E & E Project No.: 002693.2052.01RA TDD No.: TO2-09-09-09-0001

True Average Concentration of Property or Property Portion (% of Action Level)	Decision Error	Typical Decision Error Probability Goals (Based on Professional Judgment)	Type of Decision Error
< 75	A decision that the property or property portion is contaminated when it is not.	< 5%	False Acceptance
75 to < 100	A decision that the property or property portion is contaminated when it is not.	Gray Area[1]	False Acceptance
100 to 150	A decision that the property or property portion is not contaminated when it is.	10%[2]	False Rejection
> 150	A decision that the property or property portion is not contaminated when it is.	less than 1%	False Rejection

The goals in this table are based on professional judgment as relevant to a Phase II Assessment.

[1]Gray Area is where relatively large decision errors are acceptable.

[2]The large probability for the decision error is expected when the true contaminant concentrations are between 100% and 150% of the action level. Decreasing the probability is possible only by significantly increasing sampling number and QA, since sampling and analytical uncertainties and biases can not be eliminated.

7. OPTIMIZED DESIGN FOR OBTAINING DATA

General

All activities and documentation related to the project should proceed under a Quality Management Plan (QMP). All sampling, analytical and quality assurance activities will proceed under a U.S. EPA-approved SAP. A record of sampling activities and deviation from the SAP must be documented in a bound field log book. Prior to sample collection, all project sampling personnel will review relevant sampling procedures and relevant quality assurance and control (QA/QC) requirements for selected analytical methods.

Decision Error Minimization

Average concentrations

To minimize a decision error related to data uncertainty, the decision-maker should consider average concentrations or statistical evaluations.

Contamination hot-spot locations

Data that are above the action level and show contaminant concentration more than 3 times greater than results from adjacent sampling locations should be considered separately to determine whether they represent a contamination hot-spot.

Data from individual sample locations

The decision-maker should consider data uncertainty when making decisions using sampling data and associated estimated values from a single location. An individual data value reported below the action level may be biased low, while a data value reported above the action level may be biased high. The probability of decision error increases when COPC concentrations are around the action level due to both data uncertainty and data bias.

For any reported COPC concentrations near the method detection limit, the uncertainty is relatively large, increasing the probability of decision error. The uncertainty for estimated data (data based on extrapolations and interpolations) is typically greater than for actual data. Therefore the probability of a decision error is greatly increased when extrapolated data are used.

Due to the nature of the contamination, it is unknown whether data from any individual sample locations on a property can represent a larger area. There are insufficient data to determine confidence of any single sampling location. Thus the decision-maker should consider discrete data points as potentially not representative of any greater area.

To minimize decision errors around the action level, all soil data for composite samples that have a reported concentration between 75% and 99% of the action level should be averaged with data from surrounding locations, or the area should be treated as potentially exceeding the action level.

Contamination Distribution Map

Data from sampling locations can be used to create a contaminant distribution map. The mapped COPC concentrations within an area should generally be based on the sample data from that area and the sample data from adjacent locations, particularly if discrete sample data are being used.

The generated map model could be used to estimate the concentration of contamination throughout the property. The decision-maker should consider the data source and statistical sophistication of the distribution map prior to making decisions based on the map.

Design

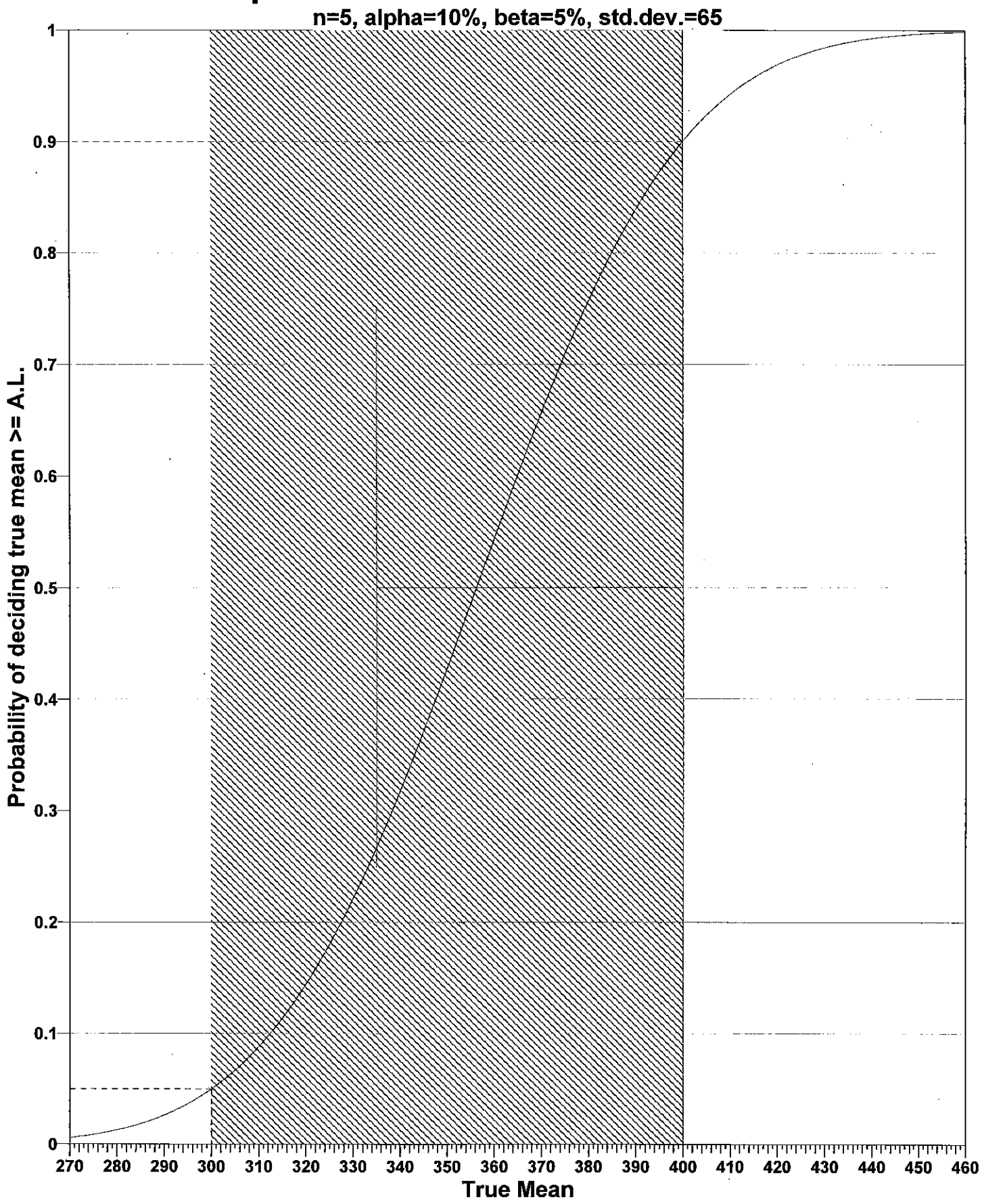
Neither background nor reference soil samples will be collected for this sampling event. Replicates and equipment blanks will be collected. Matrix spike and spike duplicate samples will be collected and are required by method. Data review independent of the laboratory shall be performed on all START-generated analytical data that may be used in decision making. The (GPS) coordinates (latitude and longitude) of each sampling location will be determined and documented during sampling.

Approximately 100 sampling locations identified and sampled. Selected locations will be an entire area such as a back yard, side yard, or front yard. Selected locations will be prioritized based upon physical and legal accessibility and distance from the AMCO NPL site.

Each residential sampling location will be sampled by creating a five point composite sample. The first sampling points will be from centrally located point with the other 4 sampling points approximately at 90 degrees from each other at point half way between the center point the area boundary. Samples will be collected at intervals between 0 and 6 inches bgs using trowels or disposable scoops. X-ray fluorescence (XRF) in situ data may be used to determine if the standard deviation of the mean for measurements within the range indicated in section 6 (e.g <65 mg/kg). If the standard deviation of the mean for measurements is greater than expected the composite number can be increased to meet the situation

The field sampling team will homogenize all soil samples by thoroughly mixing the collected soil for an interval. All samples will be placed in coolers and chilled with ice to 4^{o} Celsius for storage and shipping.

The use of XRF methods in the field immediately following sample collection could immediately provide data to assist the U.S. EPA in determining if contamination above the action level exists and will help reduce the cost of the investigation. Further the relationship of field data to laboratory data will be needed prior to any removal or remedial action where the field data would be used to support decision real-time making.


1-Sample t-Test of True Mean vs. Action Level
n=5, alpha=10%, beta=5%, std.dev.=65
Probability of deciding true mean >= A.L.
1
0.9
0.8
0.7
0.6
0.5
0.4
0.3
0.2
0.1
0
270 280 290 300 310 320 330 340 350 360 370 380 390 400 410 420 430 440 450 460
True Mean

B Site Specific Health and Safety Plan

C Standard Operating Procedures

METHOD 6200

FIELD PORTABLE X-RAY FLUORESCENCE SPECTROMETRY FOR THE DETERMINATION OF ELEMENTAL CONCENTRATIONS IN SOIL AND SEDIMENT

1.0 SCOPE AND APPLICATION

1.1 This method is applicable to the in situ and intrusive analysis of the 26 analytes listed in Table 1 for soil and sediment samples. Some common elements are not listed in Table 1 because they are considered "light" elements that cannot be detected by field portable x-ray fluorescence (FPXRF). They are: lithium, beryllium, sodium, magnesium, aluminum, silicon, and phosphorus. Most of the analytes listed in Table 1 are of environmental concern, while a few others have interference effects or change the elemental composition of the matrix, affecting quantitation of the analytes of interest. Generally elements of atomic number 16 or greater can be detected and quantitated by FPXRF.

1.2 Detection limits depend on several factors, the analyte of interest, the type of detector used, the type of excitation source, the strength of the excitation source, count times used to irradiate the sample, physical matrix effects, chemical matrix effects, and interelement spectral interferences. General instrument detection limits for analytes of interest in environmental applications are shown in Table 1. These detection limits apply to a clean matrix of quartz sand (silicon dioxide) free of interelement spectral interferences using long (600-second) count times. These detection limits are given for guidance only and will vary depending on the sample matrix, which instrument is used, and operating conditions. A discussion of field performance-based detection limits is presented in Section 13.4 of this method. The clean matrix and field performance-based detection limits should be used for general planning purposes, and a third detection limit discussed, based on the standard deviation around single measurements, should be used in assessing data quality. This detection limit is discussed in Sections 9.7 and 11.3.

1.3 Use of this method is restricted to personnel either trained and knowledgeable in the operation of an XRF instrument or under the supervision of a trained and knowledgeable individual. This method is a screening method to be used with confirmatory analysis using EPA-approved methods. This method’s main strength is as a rapid field screening procedure. The method detection limits (MDL) of FPXRF are above the toxicity characteristic regulatory level for most RCRA analytes. If the precision, accuracy, and detection limits of FPXRF meet the data quality objectives (DQOs) of your project, then XRF is a fast, powerful, cost effective technology for site characterization.

2.0 SUMMARY OF METHOD

2.1 The FPXRF technologies described in this method use sealed radioisotope sources to irradiate samples with x-rays. X-ray tubes are used to irradiate samples in the laboratory and are beginning to be incorporated into field portable instruments. When a sample is irradiated with x-rays, the source x-rays may undergo either scattering or absorption by sample atoms. This later process is known as the photoelectric effect. When an atom absorbs the source x-rays, the incident radiation dislodges electrons from the innermost shells of the atom, creating vacancies. The electron vacancies are filled by electrons cascading in from outer electron shells. Electrons in outer shells have higher energy states than inner shell electrons, and the outer shell electrons give off energy as they cascade down into the inner shell vacancies. This rearrangement of electrons

results in emission of x-rays characteristic of the given atom. The emission of x-rays, in this manner, is termed x-ray fluorescence.

Three electron shells are generally involved in emission of x-rays during FPXRF analysis of environmental samples: the K, L, and M shells. A typical emission pattern, also called an emission spectrum, for a given metal has multiple intensity peaks generated from the emission of K, L, or M shell electrons. The most commonly measured x-ray emissions are from the K and L shells; only metals with an atomic number greater than 57 have measurable M shell emissions.

Each characteristic x-ray line is defined with the letter K, L, or M, which signifies which shell had the original vacancy and by a subscript alpha (α) or beta (β), which indicates the higher shell from which electrons fell to fill the vacancy and produce the x-ray. For example, a K_α line is produced by a vacancy in the K shell filled by an L shell electron, whereas a K_β line is produced by a vacancy in the K shell filled by an M shell electron. The K_α transition is on average 6 to 7 times more probable than the K_β transition; therefore, the K_α line is approximately 7 times more intense than the K_β line for a given element, making the K_α line the choice for quantitation purposes.

The K lines for a given element are the most energetic lines and are the preferred lines for analysis. For a given atom, the x-rays emitted from L transitions are always less energetic than those emitted from K transitions. Unlike the K lines, the main L emission lines (L_α and L_β) for an element are of nearly equal intensity. The choice of one or the other depends on what interfering element lines might be present. The L emission lines are useful for analyses involving elements of atomic number (Z) 58 (cerium) through 92 (uranium).

An x-ray source can excite characteristic x-rays from an element only if the source energy is greater than the absorption edge energy for the particular line group of the element, that is, the K absorption edge, L absorption edge, or M absorption edge energy. The absorption edge energy is somewhat greater than the corresponding line energy. Actually, the K absorption edge energy is approximately the sum of the K, L, and M line energies of the particular element, and the L absorption edge energy is approximately the sum of the L and M line energies. FPXRF is more sensitive to an element with an absorption edge energy close to but less than the excitation energy of the source. For example, when using a cadmium-109 source, which has an excitation energy of 22.1 kiloelectron volts (keV), FPXRF would exhibit better sensitivity for zirconium which has a K line energy of 15.7 keV than to chromium, which has a K line energy of 5.41 keV.

2.2 Under this method, inorganic analytes of interest are identified and quantitated using a field portable energy-dispersive x-ray fluorescence spectrometer. Radiation from one or more radioisotope sources or an electrically excited x-ray tube is used to generate characteristic x-ray emissions from elements in a sample. Up to three sources may be used to irradiate a sample. Each source emits a specific set of primary x-rays that excite a corresponding range of elements in a sample. When more than one source can excite the element of interest, the source is selected according to its excitation efficiency for the element of interest.

For measurement, the sample is positioned in front of the probe window. This can be done in two manners using FPXRF instruments: in situ or intrusive. If operated in the in situ mode, the probe window is placed in direct contact with the soil surface to be analyzed. When an FPXRF instrument is operated in the intrusive mode, a soil or sediment sample must be collected, prepared, and placed in a sample cup. The sample cup is then placed on top of the window inside a protective cover for analysis.

Sample analysis is then initiated by exposing the sample to primary radiation from the source. Fluorescent and backscattered x-rays from the sample enter through the detector window and are converted into electric pulses in the detector. The detector in FPXRF instruments is usually either a solid-state detector or a gas-filled proportional counter. Within the detector, energies of the characteristic x-rays are converted into a train of electric pulses, the amplitudes of which are linearly proportional to the energy of the x-rays. An electronic multichannel analyzer (MCA) measures the pulse amplitudes, which is the basis of qualitative x-ray analysis. The number of counts at a given energy per unit of time is representative of the element concentration in a sample and is the basis for quantitative analysis. Most FPXRF instruments are menu-driven from software built into the units or from personal computers (PC).

The measurement time of each source is user-selectable. Shorter source measurement times (30 seconds) are generally used for initial screening and hot spot delineation, and longer measurement times (up to 300 seconds) are typically used to meet higher precision and accuracy requirements.

FPXRF instruments can be calibrated using the following methods: internally using fundamental parameters determined by the manufacturer, empirically based on site-specific calibration standards (SSCS), or based on Compton peak ratios. The Compton peak is produced by backscattering of the source radiation. Some FPXRF instruments can be calibrated using multiple methods.

3.0 DEFINITIONS

3.1 FPXRF: Field portable x-ray fluorescence.

3.2 MCA: Multichannel analyzer for measuring pulse amplitude.

3.3 SSCS: Site specific calibration standard.

3.4 FP: Fundamental parameter.

3.5 ROI: Region of interest.

3.6 SRM: Standard reference material. A standard containing certified amounts of metals in soil or sediment.

3.7 eV: Electron Volt. A unit of energy equivalent to the amount of energy gained by an electron passing through a potential difference of one volt.

3.8 Refer to Chapter One and Chapter Three for additional definitions.

4.0 INTERFERENCES

4.1 The total method error for FPXRF analysis is defined as the square root of the sum of squares of both instrument precision and user- or application-related error. Generally, instrument precision is the least significant source of error in FPXRF analysis. User- or application-related error is generally more significant and varies with each site and method used. Some sources of interference can be minimized or controlled by the instrument operator, but others cannot. Common sources of user- or application-related error are discussed below.

4.2 Physical matrix effects result from variations in the physical character of the sample. These variations may include such parameters as particle size, uniformity, homogeneity, and surface condition. For example, if any analyte exists in the form of very fine particles in a coarser-grained matrix, the analyte's concentration measured by the FPXRF will vary depending on how fine particles are distributed within the coarser-grained matrix. If the fine particles "settle" to the bottom of the sample cup, the analyte concentration measurement will be higher than if the fine particles are not mixed in well and stay on top of the coarser-grained particles in the sample cup. One way to reduce such error is to grind and sieve all soil samples to a uniform particle size thus reducing sample-to-sample particle size variability. Homogeneity is always a concern when dealing with soil samples. Every effort should be made to thoroughly mix and homogenize soil samples before analysis. Field studies have shown heterogeneity of the sample generally has the largest impact on comparability with confirmatory samples.

4.3 Moisture content may affect the accuracy of analysis of soil and sediment sample analyses. When the moisture content is between 5 and 20 percent, the overall error from moisture may be minimal. However, moisture content may be a major source of error when analyzing samples of surface soil or sediment that are saturated with water. This error can be minimized by drying the samples in a convection or toaster oven. Microwave drying is not recommended because field studies have shown that microwave drying can increase variability between FPXRF data and confirmatory analysis and because metal fragments in the sample can cause arcing to occur in a microwave.

4.4 Inconsistent positioning of samples in front of the probe window is a potential source of error because the x-ray signal decreases as the distance from the radioactive source increases. This error is minimized by maintaining the same distance between the window and each sample. For the best results, the window of the probe should be in direct contact with the sample, which means that the sample should be flat and smooth to provide a good contact surface.

4.5 Chemical matrix effects result from differences in the concentrations of interfering elements. These effects occur as either spectral interferences (peak overlaps) or as x-ray absorption and enhancement phenomena. Both effects are common in soils contaminated with heavy metals. As examples of absorption and enhancement effects; iron (Fe) tends to absorb copper (Cu) x-rays, reducing the intensity of the Cu measured by the detector, while chromium (Cr) will be enhanced at the expense of Fe because the absorption edge of Cr is slightly lower in energy than the fluorescent peak of iron. The effects can be corrected mathematically through the use of fundamental parameter (FP) coefficients. The effects also can be compensated for using SSCS, which contain all the elements present on site that can interfere with one another.

4.6 When present in a sample, certain x-ray lines from different elements can be very close in energy and, therefore, can cause interference by producing a severely overlapped spectrum. The degree to which a detector can resolve the two different peaks depends on the energy resolution of the detector. If the energy difference between the two peaks in electron volts is less than the resolution of the detector in electron volts, then the detector will not be able to fully resolve the peaks.

The most common spectrum overlaps involve the K_β line of element Z-1 with the K_α line of element Z. This is called the K_α/K_β interference. Because the K_α:K_β intensity ratio for a given element usually is about 7:1, the interfering element, Z-1, must be present at large concentrations to cause a problem. Two examples of this type of spectral interference involve the presence of large concentrations of vanadium (V) when attempting to measure Cr or the presence of large concentrations of Fe when attempting to measure cobalt (Co). The V K_α and K_β energies are 4.95

and 5.43 keV, respectively, and the Cr K_α energy is 5.41 keV. The Fe K_α and K_β energies are 6.40 and 7.06 keV, respectively, and the Co K_α energy is 6.92 keV. The difference between the V K_β and Cr K_α energies is 20 eV, and the difference between the Fe K_β and the Co K_α energies is 140 eV. The resolution of the highest-resolution detectors in FPXRF instruments is 170 eV. Therefore, large amounts of V and Fe will interfere with quantitation of Cr or Co, respectively. The presence of Fe is a frequent problem because it is often found in soils at tens of thousands of parts per million (ppm).

4.7 Other interferences can arise from K/L, K/M, and L/M line overlaps, although these overlaps are less common. Examples of such overlap involve arsenic (As) K_α/lead (Pb) L_α and sulfur (S) K_α/Pb M_α. In the As/Pb case, Pb can be measured from the Pb L_β line, and As can be measured from either the As K_α or the As $K_ß$ line; in this way the interference can be corrected. If the As K_β line is used, sensitivity will be decreased by a factor of two to five times because it is a less intense line than the As K_α line. If the As K_α line is used in the presence of Pb, mathematical corrections within the instrument software can be used to subtract out the Pb interference. However, because of the limits of mathematical corrections, As concentrations cannot be efficiently calculated for samples with Pb:As ratios of 10:1 or more. This high ratio of Pb to As may result in no As being reported regardless of the actual concentration present.

No instrument can fully compensate for this interference. It is important for an operator to understand this limitation of FPXRF instruments and consult with the manufacturer of the FPXRF instrument to evaluate options to minimize this limitation. The operator's decision will be based on action levels for metals in soil established for the site, matrix effects, capabilities of the instrument, data quality objectives, and the ratio of lead to arsenic known to be present at the site. If a site is encountered that contains lead at concentrations greater than ten times the concentration of arsenic it is advisable that all critical soil samples be sent off site for confirmatory analysis by an EPA-approved method.

4.8 If SSCS are used to calibrate an FPXRF instrument, the samples collected must be representative of the site under investigation. Representative soil sampling ensures that a sample or group of samples accurately reflects the concentrations of the contaminants of concern at a given time and location. Analytical results for representative samples reflect variations in the presence and concentration ranges of contaminants throughout a site. Variables affecting sample representativeness include differences in soil type, contaminant concentration variability, sample collection and preparation variability, and analytical variability, all of which should be minimized as much as possible.

4.9 Soil physical and chemical effects may be corrected using SSCS that have been analyzed by inductively coupled plasma (ICP) or atomic absorption (AA) methods. However, a major source of error can be introduced if these samples are not representative of the site or if the analytical error is large. Another concern is the type of digestion procedure used to prepare the soil samples for the reference analysis. Analytical results for the confirmatory method will vary depending on whether a partial digestion procedure, such as SW-846 Method 3050, or a total digestion procedure, such as Method 3052 is used. It is known that depending on the nature of the soil or sediment, Method 3050 will achieve differing extraction efficiencies for different analytes of interest. The confirmatory method should meet the project data quality objectives.

XRF measures the total concentration of an element; therefore, to achieve the greatest comparability of this method with the reference method (reduced bias), a total digestion procedure should be used for sample preparation. However, in the study used to generate the performance data for this method, the confirmatory method used was Method 3050, and the FPXRF data

compared very well with regression correlation coefficients (r^2 often exceeding 0.95, except for barium and chromium. See Table 9 in Section 17.0). The critical factor is that the digestion procedure and analytical reference method used should meet the data quality objectives (DQOs) of the project and match the method used for confirmation analysis.

4.10 Ambient temperature changes can affect the gain of the amplifiers producing instrument drift. Gain or drift is primarily a function of the electronics (amplifier or preamplifier) and not the detector as most instrument detectors are cooled to a constant temperature. Most FPXRF instruments have a built-in automatic gain control. If the automatic gain control is allowed to make periodic adjustments, the instrument will compensate for the influence of temperature changes on its energy scale. If the FPXRF instrument has an automatic gain control function, the operator will not have to adjust the instrument's gain unless an error message appears. If an error message appears, the operator should follow the manufacturer's procedures for troubleshooting the problem. Often, this involves performing a new energy calibration. The performance of an energy calibration check to assess drift is a quality control measure discussed in Section 9.2.

If the operator is instructed by the manufacturer to manually conduct a gain check because of increasing or decreasing ambient temperature, it is standard to perform a gain check after every 10 to 20 sample measurements or once an hour whichever is more frequent. It is also suggested that a gain check be performed if the temperature fluctuates more than 10 to 20°F. The operator should follow the manufacturer's recommendations for gain check frequency.

5.0 SAFETY

5.1 Proper training for the safe operation of the instrument and radiation training should be completed by the analyst prior to analysis. Radiation safety for each specific instrument can be found in the operators manual. Protective shielding should never be removed by the analyst or any personnel other than the manufacturer. The analyst should be aware of the local state and national regulations that pertain to the use of radiation-producing equipment and radioactive materials with which compliance is required. Licenses for radioactive materials are of two types; (1) general license which is usually provided by the manufacturer for receiving, acquiring, owning, possessing, using, and transferring radioactive material incorporated in a device or equipment, and (2) specific license which is issued to named persons for the operation of radioactive instruments as required by local state agencies. There should be a person appointed within the organization that is solely responsible for properly instructing all personnel, maintaining inspection records, and monitoring x-ray equipment at regular intervals. A copy of the radioactive material licenses and leak tests should be present with the instrument at all times and available to local and national authorities upon request. X-ray tubes do not require radioactive material licenses or leak tests, but do require approvals and licenses which vary from state to state. In addition, fail-safe x-ray warning lights should be illuminated whenever an x-ray tube is energized. Provisions listed above concerning radiation safety regulations, shielding, training, and responsible personnel apply to x-ray tubes just as to radioactive sources. In addition, a log of the times and operating conditions should be kept whenever an x-ray tube is energized. Finally, an additional hazard present with x-ray tubes is the danger of electric shock from the high voltage supply. The danger of electric shock is as substantial as the danger from radiation but is often overlooked because of its familiarity.

5.2 Radiation monitoring equipment should be used with the handling of the instrument. The operator and the surrounding environment should be monitored continually for analyst exposure to radiation. Thermal luminescent detectors (TLD) in the form of badges and rings are used to monitor operator radiation exposure. The TLDs should be worn in the area of most frequent exposure. The maximum permissible whole-body dose from occupational exposure is 5

Roentgen Equivalent Man (REM) per year. Possible exposure pathways for radiation to enter the body are ingestion, inhaling, and absorption. The best precaution to prevent radiation exposure is distance and shielding.

5.3 Refer to Chapter Three for guidance on some proper safety protocols.

6.0 EQUIPMENT AND SUPPLIES

6.1 FPXRF Spectrometer: An FPXRF spectrometer consists of four major components: (1) a source that provides x-rays; (2) a sample presentation device; (3) a detector that converts x-ray-generated photons emitted from the sample into measurable electronic signals; and (4) a data processing unit that contains an emission or fluorescence energy analyzer, such as an MCA, that processes the signals into an x-ray energy spectrum from which elemental concentrations in the sample may be calculated, and a data display and storage system. These components and additional, optional items, are discussed below.

6.1.1 Excitation Sources: Most FPXRF instruments use sealed radioisotope sources to produce x-rays in order to irradiate samples. The FPXRF instrument may contain between one and three radioisotope sources. Common radioisotope sources used for analysis for metals in soils are iron (Fe)-55, cadmium (Cd)-109, americium (Am)-241, and curium (Cm)-244. These sources may be contained in a probe along with a window and the detector; the probe is connected to a data reduction and handling system by means of a flexible cable. Alternatively, the sources, window, and detector may be included in the same unit as the data reduction and handling system.

The relative strength of the radioisotope sources is measured in units of millicuries (mCi). All other components of the FPXRF system being equal, the stronger the source, the greater the sensitivity and precision of a given instrument. Radioisotope sources undergo constant decay. In fact, it is this decay process that emits the primary x-rays used to excite samples for FPXRF analysis. The decay of radioisotopes is measured in "half-lives." The half-life of a radioisotope is defined as the length of time required to reduce the radioisotopes strength or activity by half. Developers of FPXRF technologies recommend source replacement at regular intervals based on the source's half-life. The characteristic x-rays emitted from each of the different sources have energies capable of exciting a certain range of analytes in a sample. Table 2 summarizes the characteristics of four common radioisotope sources.

X-ray tubes have higher radiation output, no intrinsic lifetime limit, produce constant output over their lifetime, and do not have the disposal problems of radioactive sources but are just now appearing in FPXRF instruments An electrically-excited x-ray tube operates by bombarding an anode with electrons accelerated by a high voltage. The electrons gain an energy in electron volts equal to the accelerating voltage and can excite atomic transitions in the anode, which then produces characteristic x-rays. These characteristic x-rays are emitted through a window which contains the vacuum required for the electron acceleration. An important difference between x-ray tubes and radioactive sources is that the electrons which bombard the anode also produce a continuum of x-rays across a broad range of energies in addition to the characteristic x-rays. This continuum is weak compared to the characteristic x-rays but can provide substantial excitation since it covers a broad energy range. It has the undesired property of producing background in the spectrum near the analyte x-ray lines when it is scattered by the sample. For this reason a filter is often used between the x-ray tube and the sample to suppress the continuum radiation while passing the characteristic

x-rays from the anode. This filter is sometimes incorporated into the window of the x-ray tube. The choice of accelerating voltage is governed by the anode material, since the electrons must have sufficient energy to excite the anode, which requires a voltage greater than the absorption edge of the anode material. The anode is most efficiently excited by voltages 2 to 2.5 times the edge energy (most x-rays per unit power to the tube), although voltages as low as 1.5 times the absorption edge energy will work. The characteristic x-rays emitted by the anode are capable of exciting a range of elements in the sample just as with a radioactive source. Table 3 gives the recommended operating voltages and the sample elements excited for some common anodes.

6.1.2 Sample Presentation Device: FPXRF instruments can be operated in two modes: in situ and intrusive. If operated in the in situ mode, the probe window is placed in direct contact with the soil surface to be analyzed. When an FPXRF instrument is operated in the intrusive mode, a soil or sediment sample must be collected, prepared, and placed in a sample cup. For most FPXRF instruments operated in the intrusive mode, the probe is rotated so that the window faces upward. A protective sample cover is placed over the window, and the sample cup is placed on top of the window inside the protective sample cover for analysis.

6.1.3 Detectors: The detectors in the FPXRF instruments can be either solid-state detectors or gas-filled, proportional counter detectors. Common solid-state detectors include mercuric iodide (HgI_2), silicon pin diode and lithium-drifted silicon Si(Li). The HgI_2 detector is operated at a moderately subambient temperature controlled by a low power thermoelectric cooler. The silicon pin diode detector also is cooled via the thermoelectric Peltier effect. The Si(Li) detector must be cooled to at least -90 °C either with liquid nitrogen or by thermoelectric cooling via the Peltier effect. Instruments with a Si(Li) detector have an internal liquid nitrogen dewar with a capacity of 0.5 to 1.0 liter. Proportional counter detectors are rugged and lightweight, which are important features of a field portable detector. However, the resolution of a proportional counter detector is not as good as that of a solid-state detector. The energy resolution of a detector for characteristic x-rays is usually expressed in terms of full width at half-maximum (FWHM) height of the manganese K_α peak at 5.89 keV. The typical resolutions of the above mentioned detectors are as follows: HgI_2-270 eV; silicon pin diode-250 eV; Si(Li)–170 eV; and gas-filled, proportional counter-750 eV.

During operation of a solid-state detector, an x-ray photon strikes a biased, solid-state crystal and loses energy in the crystal by producing electron-hole pairs. The electric charge produced is collected and provides a current pulse that is directly proportional to the energy of the x-ray photon absorbed by the crystal of the detector. A gas-filled, proportional counter detector is an ionization chamber filled with a mixture of noble and other gases. An x-ray photon entering the chamber ionizes the gas atoms. The electric charge produced is collected and provides an electric signal that is directly proportional to the energy of the x-ray photon absorbed by the gas in the detector.

6.1.4 Data Processing Units: The key component in the data processing unit of an FPXRF instrument is the MCA. The MCA receives pulses from the detector and sorts them by their amptitudes (energy level). The MCA counts pulses per second to determine the height of the peak in a spectrum, which is indicative of the target analyte's concentration. The spectrum of element peaks are built on the MCA. The MCAs in FPXRF instruments have from 256 to 2,048 channels. The concentrations of target analytes are usually shown in parts per million on a liquid crystal display (LCD) in the instrument. FPXRF instruments can store both spectra and from 100 to 500 sets of numerical analytical results. Most FPXRF

instruments are menu-driven from software built into the units or from PCs. Once the data–storage memory of an FPXRF unit is full, data can be downloaded by means of an RS-232 port and cable to a PC.

6.2 Spare battery chargers.

6.3 Polyethylene sample cups: 31 millimeters (mm) to 40 mm in diameter with collar, or equivalent (appropriate for FPXRF instrument).

6.4 X-ray window film: Mylar™, Kapton™, Spectrolene™, polypropylene, or equivalent; 2.5 to 6.0 micrometers (µm) thick.

6.5 Mortar and pestle: glass, agate, or aluminum oxide; for grinding soil and sediment samples.

6.6 Containers: glass or plastic to store samples.

6.7 Sieves: 60-mesh (0.25 mm), stainless-steel, Nylon, or equivalent for preparing soil and sediment samples.

6.8 Trowels: for smoothing soil surfaces and collecting soil samples.

6.9 Plastic bags: used for collection and homogenization of soil samples.

6.10 Drying oven: standard convection or toaster oven, for soil and sediment samples that require drying.

7.0 REAGENTS AND STANDARDS

7.1 Pure Element Standards: Each pure, single-element standard is intended to produce strong characteristic x-ray peaks of the element of interest only. Other elements present must not contribute to the fluorescence spectrum. A set of pure element standards for commonly sought analytes is supplied by the instrument manufacturer, if required for the instrument; not all instruments require the pure element standards. The standards are used to set the region of interest (ROI) for each element. They also can be used as energy calibration and resolution check samples.

7.2 Site-specific Calibration Standards: Instruments that employ fundamental parameters (FP) or similar mathematical models in minimizing matrix effects may not require SSCS. If the FP calibration model is to be optimized or if empirical calibration is necessary, then SSCSs must be collected, prepared, and analyzed.

7.2.1 The SSCS must be representative of the matrix to be analyzed by FPXRF. These samples must be well homogenized. A minimum of ten samples spanning the concentration ranges of the analytes of interest and of the interfering elements must be obtained from the site. A sample size of 4 to 8 ounces is recommended, and standard glass sampling jars should be used.

7.2.2 Each sample should be oven-dried for 2 to 4 hours at a temperature of less than 150°C. If mercury is to be analyzed, a separate sample portion must remain undried, as heating may volatilize the mercury. When the sample is dry, all large, organic debris and

nonrepresentative material, such as twigs, leaves, roots, insects, asphalt, and rock should be removed. The sample should be ground with a mortar and pestle and passed through a 60-mesh sieve. Only the coarse rock fraction should remain on the screen.

7.2.3 The sample should be homogenized by using a riffle splitter or by placing 150 to 200 grams of the dried, sieved sample on a piece of kraft or butcher paper about 1.5 by 1.5 feet in size. Each corner of the paper should be lifted alternately, rolling the soil over on itself and toward the opposite corner. The soil should be rolled on itself 20 times. Approximately 5 grams of the sample should then be removed and placed in a sample cup for FPXRF analysis. The rest of the prepared sample should be sent off site for ICP or AA analysis. The method use for confirmatory analysis should meet the data quality objectives of the project.

7.3 Blank Samples: The blank samples should be from a "clean" quartz or silicon dioxide matrix that is free of any analytes at concentrations above the method detection limits. These samples are used to monitor for cross-contamination and laboratory-induced contaminants or interferences.

7.4 Standard Reference Materials: Standard reference materials (SRM) are standards containing certified amounts of metals in soil or sediment. These standards are used for accuracy and performance checks of FPXRF analyses. SRMs can be obtained from the National Institute of Standards and Technology (NIST), the U.S. Geological Survey (USGS), the Canadian National Research Council, and the national bureau of standards in foreign nations. Pertinent NIST SRMs for FPXRF analysis include 2704, Buffalo River Sediment; 2709, San Joaquin Soil; and 2710 and 2711, Montana Soil. These SRMs contain soil or sediment from actual sites that has been analyzed using independent inorganic analytical methods by many different laboratories.

8.0 SAMPLE COLLECTION, PRESERVATION, AND STORAGE

Sample handling and preservation procedures used in FPXRF analyses should follow the guidelines in Chapter Three, Inorganic Analytes.

9.0 QUALITY CONTROL

9.1 Refer to Chapter One for additional guidance on quality assurance protocols. All field data sheets and quality control data should be maintained for reference or inspection.

9.2 Energy Calibration Check: To determine whether an FPXRF instrument is operating within resolution and stability tolerances, an energy calibration check should be run. The energy calibration check determines whether the characteristic x-ray lines are shifting, which would indicate drift within the instrument. As discussed in Section 4.10, this check also serves as a gain check in the event that ambient temperatures are fluctuating greatly (> 10 to 20°F).

The energy calibration check should be run at a frequency consistent with manufacturers recommendations. Generally, this would be at the beginning of each working day, after the batteries are changed or the instrument is shut off, at the end of each working day, and at any other time when the instrument operator believes that drift is occurring during analysis. A pure element such as iron, manganese, copper, or lead is often used for the energy calibration check. A manufacturer-recommended count time per source should be used for the check.

9.2.1 The instrument manufacturer’s manual specifies the channel or kiloelectron volt level at which a pure element peak should appear and the expected intensity of the peak.

The intensity and channel number of the pure element as measured using the radioactive source should be checked and compared to the manufacturer's recommendation. If the energy calibration check does not meet the manufacturer's criteria, then the pure element sample should be repositioned and reanalyzed. If the criteria are still not met, then an energy calibration should be performed as described in the manufacturer's manual. With some FPXRF instruments, once a spectrum is acquired from the energy calibration check, the peak can be optimized and realigned to the manufacturer's specifications using their software.

9.3 Blank Samples: Two types of blank samples should be analyzed for FPXRF analysis: instrument blanks and method blanks. An instrument blank is used to verify that no contamination exists in the spectrometer or on the probe window.

9.3.1 The instrument blank can be silicon dioxide, a Teflon block, a quartz block, "clean" sand, or lithium carbonate. This instrument blank should be analyzed on each working day before and after analyses are conducted and once per every twenty samples. An instrument blank should also be analyzed whenever contamination is suspected by the analyst. The frequency of analysis will vary with the data quality objectives of the project. A manufacturer-recommended count time per source should be used for the blank analysis. No element concentrations above the method detection limits should be found in the instrument blank. If concentrations exceed these limits, then the probe window and the check sample should be checked for contamination. If contamination is not a problem, then the instrument must be "zeroed" by following the manufacturer's instructions.

9.3.2 A method blank is used to monitor for laboratory-induced contaminants or interferences. The method blank can be "clean" silica sand or lithium carbonate that undergoes the same preparation procedure as the samples. A method blank must be analyzed at least daily. The frequency of analysis will depend on the data quality objectives of the project. To be acceptable, a method blank must not contain any analyte at a concentration above its method detection limit. If an analyte's concentration exceeds its method detection limit, the cause of the problem must be identified, and all samples analyzed with the method blank must be reanalyzed.

9.4 Calibration Verification Checks: A calibration verification check sample is used to check the accuracy of the instrument and to assess the stability and consistency of the analysis for the analytes of interest. A check sample should be analyzed at the beginning of each working day, during active sample analyses, and at the end of each working day. The frequency of calibration checks during active analysis will depend on the data quality objectives of the project. The check sample should be a well characterized soil sample from the site that is representative of site samples in terms of particle size and degree of homogeneity and that contains contaminants at concentrations near the action levels. If a site-specific sample is not available, then an NIST or other SRM that contains the analytes of interest can be used to verify the accuracy of the instrument. The measured value for each target analyte should be within ±20 percent (%D) of the true value for the calibration verification check to be acceptable. If a measured value falls outside this range, then the check sample should be reanalyzed. If the value continues to fall outside the acceptance range, the instrument should be recalibrated, and the batch of samples analyzed before the unacceptable calibration verification check must be reanalyzed.

9.5 Precision Measurements: The precision of the method is monitored by analyzing a sample with low, moderate, or high concentrations of target analytes. The frequency of precision measurements will depend on the data quality objectives for the data. A minimum of one precision sample should be run per day. Each precision sample should be analyzed 7 times in replicate. It

is recommended that precision measurements be obtained for samples with varying concentration ranges to assess the effect of concentration on method precision. Determining method precision for analytes at concentrations near the site action levels can be extremely important if the FPXRF results are to be used in an enforcement action; therefore, selection of at least one sample with target analyte concentrations at or near the site action levels or levels of concern is recommended. A precision sample is analyzed by the instrument for the same field analysis time as used for other project samples. The relative standard deviation (RSD) of the sample mean is used to assess method precision. For FPXRF data to be considered adequately precise, the RSD should not be greater than 20 percent with the exception of chromium. RSD values for chromium should not be greater than 30 percent.

The equation for calculating RSD is as follows:

$$RSD = (SD/Mean\ Concentration) \times 100$$

where:

RSD	=	Relative standard deviation for the precision measurement for the analyte
SD	=	Standard deviation of the concentration for the analyte
Mean Concentration	=	Mean concentration for the analyte

The precision or reproducibility of a measurement will improve with increasing count time, however, increasing the count time by a factor of 4 will provide only 2 times better precision, so there is a point of diminishing return. Increasing the count time also improves the detection limit, but decreases sample throughput.

9.6 Detection Limits: Results for replicate analyses of a low-concentration sample, SSCS, or SRM can be used to generate an average site-specific method detection and quantitation limits. In this case, the method detection limit is defined as 3 times the standard deviation of the results for the low-concentration samples and the method quantitation limit is defined as 10 times the standard deviation of the same results. Another means of determining method detection and quantitation limits involves use of counting statistics. In FPXRF analysis, the standard deviation from counting statistics is defined as $SD = (N)^{½}$, where SD is the standard deviation for a target analyte peak and N is the net counts for the peak of the analyte of interest (i.e., gross counts minus background under the peak). Three times this standard deviation would be the method detection limit and 10 times this standard deviation would be the method quantitation limit. If both of the above mentioned approaches are used to calculate method detection limits, the larger of the standard deviations should be used to provide the more conservative detection limits.

This SD based detection limit criteria must be used by the operator to evaluate each measurement for its useability. A measurement above the average calculated or manufacturer's detection limit, but smaller than three times its associated SD, should not be used as a quantitative measurement. Conversely, if the measurement is below the average calculated or manufacturer's detection limit, but greater than three times its associated SD. It should be coded as an estimated value.

9.7 Confirmatory Samples: The comparability of the FPXRF analysis is determined by submitting FPXRF-analyzed samples for analysis at a laboratory. The method of confirmatory analysis must meet the project and XRF measurement data quality objectives. The confirmatory samples must be splits of the well homogenized sample material. In some cases the prepared

sample cups can be submitted. A minimum of 1 sample for each 20 FPXRF-analyzed samples should be submitted for confirmatory analysis. This frequency will depend on data quality objectives. The confirmatory analyses can also be used to verify the quality of the FPXRF data. The confirmatory samples should be selected from the lower, middle, and upper range of concentrations measured by the FPXRF. They should also include samples with analyte concentrations at or near the site action levels. The results of the confirmatory analysis and FPXRF analyses should be evaluated with a least squares linear regression analysis. If the measured concentrations span more than one order of magnitude, the data should be log-transformed to standardize variance which is proportional to the magnitude of measurement. The correlation coefficient (r^2) for the results should be 0.7 or greater for the FPXRF data to be considered screening level data. If the r^2 is 0.9 or greater and inferential statistics indicate the FPXRF data and the confirmatory data are statistically equivalent at a 99 percent confidence level, the data could potentially meet definitive level data criteria.

10.0 CALIBRATION AND STANDARDIZATION

10.1 Instrument Calibration: Instrument calibration procedures vary among FPXRF instruments. Users of this method should follow the calibration procedures outlined in the operator's manual for each specific FPXRF instrument. Generally, however, three types of calibration procedures exist for FPXRF instruments: FP calibration, empirical calibration, and the Compton peak ratio or normalization method. These three types of calibration are discussed below.

10.2 Fundamental Parameters Calibration: FP calibration procedures are extremely variable. An FP calibration provides the analyst with a "standardless" calibration. The advantages of FP calibrations over empirical calibrations include the following:

- No previously collected site-specific samples are required, although site-specific samples with confirmed and validated analytical results for all elements present could be used.

- Cost is reduced because fewer confirmatory laboratory results or calibration standards are required.

However, the analyst should be aware of the limitations imposed on FP calibration by particle size and matrix effects. These limitations can be minimized by adhering to the preparation procedure described in Section 7.2. The two FP calibration processes discussed below are based on an effective energy FP routine and a back scatter with FP (BFP) routine. Each FPXRF FP calibration process is based on a different iterative algorithmic method. The calibration procedure for each routine is explained in detail in the manufacturer's user manual for each FPXRF instrument; in addition, training courses are offered for each instrument.

10.2.1 Effective Energy FP Calibration: The effective energy FP calibration is performed by the manufacturer before an instrument is sent to the analyst. Although SSCS can be used, the calibration relies on pure element standards or SRMs such as those obtained from NIST for the FP calibration. The effective energy routine relies on the spectrometer response to pure elements and FP iterative algorithms to compensate for various matrix effects.

Alpha coefficients are calculated using a variation of the Sherman equation, which calculates theoretical intensities from the measurement of pure element samples. These coefficients indicate the quantitative effect of each matrix element on an analyte's measured

x-ray intensity. Next, the Lachance Traill algorithm is solved as a set of simultaneous equations based on the theoretical intensities. The alpha coefficients are then downloaded into the specific instrument.

The working effective energy FP calibration curve must be verified before sample analysis begins on each working day, after every 20 samples are analyzed, and at the end of sampling. This verification is performed by analyzing either an NIST SRM or an SSCS that is representative of the site-specific samples. This SRM or SSCS serves as a calibration check. A manufacturer-recommended count time per source should be used for the calibration check. The analyst must then adjust the y-intercept and slope of the calibration curve to best fit the known concentrations of target analytes in the SRM or SSCS.

A percent difference (%D) is then calculated for each target analyte. The %D should be within ±20 percent of the certified value for each analyte. If the %D falls outside this acceptance range, then the calibration curve should be adjusted by varying the slope of the line or the y-intercept value for the analyte. The SRM or SSCS is reanalyzed until the %D falls within ±20 percent. The group of 20 samples analyzed before an out-of-control calibration check should be reanalyzed.

The equation to calibrate %D is as follows:

$$\%D = ((C_s - C_k) / C_k) \times 100$$

where:

%D = Percent difference
C_k = Certified concentration of standard sample
C_s = Measured concentration of standard sample

10.2.2 BFP Calibration: BFP calibration relies on the ability of the liquid nitrogen-cooled, Si(Li) solid-state detector to separate the coherent (Compton) and incoherent (Rayleigh) backscatter peaks of primary radiation. These peak intensities are known to be a function of sample composition, and the ratio of the Compton to Rayleigh peak is a function of the mass absorption of the sample. The calibration procedure is explained in detail in the instrument manufacturer's manual. Following is a general description of the BFP calibration procedure.

The concentrations of all detected and quantified elements are entered into the computer software system. Certified element results for an NIST SRM or confirmed and validated results for an SSCS can be used. In addition, the concentrations of oxygen and silicon must be entered; these two concentrations are not found in standard metals analyses. The manufacturer provides silicon and oxygen concentrations for typical soil types. Pure element standards are then analyzed using a manufacturer-recommended count time per source. The results are used to calculate correction factors in order to adjust for spectrum overlap of elements.

The working BFP calibration curve must be verified before sample analysis begins on each working day, after every 20 samples are analyzed, and at the end of the analysis. This verification is performed by analyzing either an NIST SRM or an SSCS that is representative of the site-specific samples. This SRM or SSCS serves as a calibration check. The standard sample is analyzed using a manufacturer-recommended count time per source to check the

calibration curve. The analyst must then adjust the y-intercept and slope of the calibration curve to best fit the known concentrations of target analytes in the SRM or SSCS.

A %D is then calculated for each target analyte. The %D should fall within ±20 percent of the certified value for each analyte. If the %D falls outside this acceptance range, then the calibration curve should be adjusted by varying the slope of the line the y-intercept value for the analyte. The standard sample is reanalyzed until the %D falls within ±20 percent. The group of 20 samples analyzed before an out-of-control calibration check should be reanalyzed.

10.3 Empirical Calibration: An empirical calibration can be performed with SSCS, site-typical standards, or standards prepared from metal oxides. A discussion of SSCS is included in Section 7.2; if no previously characterized samples exist for a specific site, site-typical standards can be used. Site-typical standards may be selected from commercially available characterized soils or from SSCS prepared for another site. The site-typical standards should closely approximate the site's soil matrix with respect to particle size distribution, mineralogy, and contaminant analytes. If neither SSCS nor site-typical standards are available, it is possible to make gravimetric standards by adding metal oxides to a "clean" sand or silicon dioxide matrix that simulates soil. Metal oxides can be purchased from various chemical vendors. If standards are made on site, a balance capable of weighing items to at least two decimal places is required. Concentrated ICP or AA standard solutions can also be used to make standards. These solutions are available in concentrations of 10,000 parts per million, thus only small volumes have to be added to the soil.

An empirical calibration using SSCS involves analysis of SSCS by the FPXRF instrument and by a conventional analytical method such as ICP or AA. A total acid digestion procedure should be used by the laboratory for sample preparation. Generally, a minimum of 10 and a maximum of 30 well characterized SSCS, site-typical standards, or prepared metal oxide standards are required to perform an adequate empirical calibration. The number of required standards depends on the number of analytes of interest and interfering elements. Theoretically, an empirical calibration with SSCS should provide the most accurate data for a site because the calibration compensates for site-specific matrix effects.

The first step in an empirical calibration is to analyze the pure element standards for the elements of interest. This enables the instrument to set channel limits for each element for spectral deconvolution. Next the SSCS, site-typical standards, or prepared metal oxide standards are analyzed using a count time of 200 seconds per source or a count time recommended by the manufacturer. This will produce a spectrum and net intensity of each analyte in each standard. The analyte concentrations for each standard are then entered into the instrument software; these concentrations are those obtained from the laboratory, the certified results, or the gravimetrically determined concentrations of the prepared standards. This gives the instrument analyte values to regress against corresponding intensities during the modeling stage. The regression equation correlates the concentrations of an analyte with its net intensity.

The calibration equation is developed using a least squares fit regression analysis. After the regression terms to be used in the equation are defined, a mathematical equation can be developed to calculate the analyte concentration in an unknown sample. In some FPXRF instruments, the software of the instrument calculates the regression equation. The software uses calculated intercept and slope values to form a multiterm equation. In conjunction with the software in the instrument, the operator can adjust the multiterm equation to minimize interelement interferences and optimize the intensity calibration curve.

It is possible to define up to six linear or nonlinear terms in the regression equation. Terms can be added and deleted to optimize the equation. The goal is to produce an equation with the smallest regression error and the highest correlation coefficient. These values are automatically computed by the software as the regression terms are added, deleted, or modified. It is also possible to delete data points from the regression line if these points are significant outliers or if they are heavily weighing the data. Once the regression equation has been selected for an analyte, the equation can be entered into the software for quantitation of analytes in subsequent samples. For an empirical calibration to be acceptable, the regression equation for a specific analyte should have a correlation coefficient of 0.98 or greater or meet the DQOs of the project.

In an empirical calibration, one must apply the DQOs of the project and ascertain critical or action levels for the analytes of interest. It is within these concentration ranges or around these action levels that the FPXRF instrument should be calibrated most accurately. It may not be possible to develop a good regression equation over several orders of analyte concentration.

10.4 Compton Normalization Method: The Compton normalization method is based on analysis of a single, certified standard and normalization for the Compton peak. The Compton peak is produced from incoherent backscattering of x-ray radiation from the excitation source and is present in the spectrum of every sample. The Compton peak intensity changes with differing matrices. Generally, matrices dominated by lighter elements produce a larger Compton peak, and those dominated by heavier elements produce a smaller Compton peak. Normalizing to the Compton peak can reduce problems with varying matrix effects among samples. Compton normalization is similar to the use of internal standards in organics analysis. The Compton normalization method may not be effective when analyte concentrations exceed a few percent.

The certified standard used for this type of calibration could be an NIST SRM such as 2710 or 2711. The SRM must be a matrix similar to the samples and must contain the analytes of interests at concentrations near those expected in the samples. First, a response factor has to be determined for each analyte. This factor is calculated by dividing the net peak intensity by the analyte concentration. The net peak intensity is gross intensity corrected for baseline interference. Concentrations of analytes in samples are then determined by multiplying the baseline corrected analyte signal intensity by the normalization factor and by the response factor. The normalization factor is the quotient of the baseline corrected Compton K_{α} peak intensity of the SRM divided by that of the samples. Depending on the FPXRF instrument used, these calculations may be done manually or by the instrument software.

11.0 PROCEDURE

11.1 Operation of the various FPXRF instruments will vary according to the manufacturers' protocols. Before operating any FPXRF instrument, one should consult the manufacturer's manual. Most manufacturers recommend that their instruments be allowed to warm up for 15 to 30 minutes before analysis of samples. This will help alleviate drift or energy calibration problems later on in analysis.

11.2 Each FPXRF instrument should be operated according to the manufacturer's recommendations. There are two modes in which FPXRF instruments can be operated: in situ and intrusive. The in situ mode involves analysis of an undisturbed soil sediment or sample. Intrusive analysis involves collection and preparation of a soil or sediment sample before analysis. Some FPXRF instruments can operate in both modes of analysis, while others are designed to operate in only one mode. The two modes of analysis are discussed below.

11.3 For in situ analysis, one requirement is that any large or nonrepresentative debris be removed from the soil surface before analysis. This debris includes rocks, pebbles, leaves, vegetation, roots, and concrete. Another requirement is that the soil surface be as smooth as possible so that the probe window will have good contact with the surface. This may require some leveling of the surface with a stainless-steel trowel. During the study conducted to provide data for this method, this modest amount of sample preparation was found to take less than 5 minutes per sample location. The last requirement is that the soil or sediment not be saturated with water. Manufacturers state that their FPXRF instruments will perform adequately for soils with moisture contents of 5 to 20 percent but will not perform well for saturated soils, especially if ponded water exists on the surface. Another recommended technique for *in situ* analysis is to tamp the soil to increase soil density and compactness for better repeatability and representativeness. This condition is especially important for heavy element analysis, such as barium. Source count times for in situ analysis usually range from 30 to 120 seconds, but source count times will vary among instruments and depending on required detection limits.

11.4 For intrusive analysis of surface or sediment, it is recommended that a sample be collected from a 4- by 4-inch square that is 1 inch deep. This will produce a soil sample of approximately 375 grams or 250 cm^3, which is enough soil to fill an 8-ounce jar. The sample should be homogenized, dried, and ground before analysis. The sample can be homogenized before or after drying. The homogenization technique to be used after drying is discussed in Section 4.2. If the sample is homogenized before drying, it should be thoroughly mixed in a beaker or similar container, or if the sample is moist and has a high clay content, it can be kneaded in a plastic bag. One way to monitor homogenization when the sample is kneaded in a plastic bag is to add sodium fluorescein dye to the sample. After the moist sample has been homogenized, it is examined under an ultraviolet light to assess the distribution of sodium fluorescein throughout the sample. If the fluorescent dye is evenly distributed in the sample, homogenization is considered complete; if the dye is not evenly distributed, mixing should continue until the sample has been thoroughly homogenized. During the study conducted to provide data for this method, the homogenization procedure using the fluorescein dye required 3 to 5 minutes per sample. As demonstrated in Sections 13.5 and 13.7, homogenization has the greatest impact on the reduction of sampling variability. It produces little or no contamination. Often, it can be used without the more labor intensive steps of drying, grinding, and sieving given in Sections 11.5 and 11.6. Of course, to achieve the best data quality possible all four steps must be followed.

11.5 Once the soil or sediment sample has been homogenized, it should be dried. This can be accomplished with a toaster oven or convection oven. A small aliquot of the sample (20 to 50 grams) is placed in a suitable container for drying. The sample should be dried for 2 to 4 hours in the convection or toaster oven at a temperature not greater than 150°C. Microwave drying is not a recommended procedure. Field studies have shown that microwave drying can increase variability between the FPXRF data and confirmatory analysis. High levels of metals in a sample can cause arcing in the microwave oven, and sometimes slag forms in the sample. Microwave oven drying can also melt plastic containers used to hold the sample.

11.6 The homogenized dried sample material should be ground with a mortar and pestle and passed through a 60-mesh sieve to achieve a uniform particle size. Sample grinding should continue until at least 90 percent of the original sample passes through the sieve. The grinding step normally takes an average of 10 minutes per sample. An aliquot of the sieved sample should then be placed in a 31.0-mm polyethylene sample cup (or equivalent) for analysis. The sample cup should be one-half to three-quarters full at a minimum. The sample cup should be covered with a 2.5 µm Mylar (or equivalent) film for analysis. The rest of the soil sample should be placed in a jar, labeled, and archived for possible confirmation analysis. All equipment including the mortar, pestle,

and sieves must be thoroughly cleaned so that any cross-contamination is below the MDLs of the procedure or DQOs of the analysis.

12.0 DATA ANALYSIS AND CALCULATIONS

Most FPXRF instruments have software capable of storing all analytical results and spectra. The results are displayed in parts per million and can be downloaded to a PC, which can provide a hard copy printout. Individual measurements that are smaller than three times their associated SD should not be used for quantitation.

13.0 METHOD PERFORMANCE

13.1 This section discusses four performance factors, field-based method detection limits, precision, accuracy, and comparability to EPA-approved methods. The numbers presented in Tables 4 through 9 were generated from data obtained from six FPXRF instruments. The soil samples analyzed by the six FPXRF instruments were collected from two sites in the United States. The soil samples contained several of the target analytes at concentrations ranging from nondetect to tens of thousands of mg/kg.

13.2 The six FPXRF instruments included the TN 9000 and TN Lead Analyzer manufactured by TN Spectrace; the X-MET 920 with a SiLi detector and X-MET 920 with a gas-filled proportional detector manufactured by Metorex, Inc.; the XL Spectrum Analyzer manufactured by Niton; and the MAP Spectrum Analyzer manufactured by Scitec. The TN 9000 and TN Lead Analyzer both have a HgI_2 detector. The TN 9000 utilized an Fe-55, Cd-109, and Am-241 source. The TN Lead Analyzer had only a Cd-109 source. The X-Met 920 with the SiLi detector had a Cd-109 and Am-241 source. The X-MET 920 with the gas-filled proportional detector had only a Cd-109 source. The XL Spectrum Analyzer utilized a silicon pin-diode detector and a Cd-109 source. The MAP Spectrum Analyzer utilized a solid-state silicon detector and a Cd-109 source.

13.3 All data presented in Tables 4 through 9 were generated using the following calibrations and source count times. The TN 9000 and TN Lead Analyzer were calibrated using fundamental parameters using NIST SRM 2710 as a calibration check sample. The TN 9000 was operated using 100, 60, and 60 second count times for the Cd-109, Fe-55, and Am-241 sources, respectively. The TN Lead analyzer was operated using a 60 second count time for the Cd-109 source. The X-MET 920 with the Si(Li) detector was calibrated using fundamental parameters and one well characterized site-specific soil standard as a calibration check. It used 140 and 100 second count times for the Cd-109 and Am-241 sources, respectively. The X-MET 920 with the gas-filled proportional detector was calibrated empirically using between 10 and 20 well characterized site-specific soil standards. It used 120 second times for the Cd-109 source. The XL Spectrum Analyzer utilized NIST SRM 2710 for calibration and the Compton peak normalization procedure for quantitation based on 60 second count times for the Cd-109 source. The MAP Spectrum Analyzer was internally calibrated by the manufacturer. The calibration was checked using a well-characterized site-specific soil standard. It used 240 second times for the Cd-109 source.

13.4 Field-Based Method Detection Limits: The field-based method detection limits are presented in Table 4. The field-based method detection limits were determined by collecting ten replicate measurements on site-specific soil samples with metals concentrations 2 to 5 times the expected method detection limits. Based on these ten replicate measurements, a standard deviation on the replicate analysis was calculated. The method detection limits presented in Table 4 are defined as 3 times the standard deviation for each analyte.

The field-based method detection limits were generated by using the count times discussed earlier in this section. All the field-based method detection limits were calculated for soil samples that had been dried and ground and placed in a sample cup with the exception of the MAP Spectrum Analyzer. This instrument can only be operated in the in situ mode, meaning the samples were moist and not ground.

Some of the analytes such as cadmium, mercury, silver, selenium, and thorium were not detected or only detected at very low concentrations such that a field-based method detection limit could not be determined. These analytes are not presented in Table 4. Other analytes such as calcium, iron, potassium, and titanium were only found at high concentrations (thousands of mg/kg) so that reasonable method detection limits could not be calculated. These analytes also are not presented in Table 4.

13.5 Precision Measurements: The precision data is presented in Table 5. Each of the six FPXRF instruments performed 10 replicate measurements on 12 soil samples that had analyte concentrations ranging from nondetects to thousands of mg/kg. Each of the 12 soil samples underwent 4 different preparation techniques from in situ (no preparation) to dried and ground in a sample cup. Therefore, there were 48 precision data points for five of the instruments and 24 precision points for the MAP Spectrum Analyzer. The replicate measurements were taken using the source count times discussed at the beginning of this section.

For each detectable analyte in each precision sample a mean concentration, standard deviation, and RSD was calculated for each analyte. The data presented in Table 5 is an average RSD for the precision samples that had analyte concentrations at 5 to 10 times the MDL for that analyte for each instrument. Some analytes such as mercury, selenium, silver, and thorium were not detected in any of the precision samples so these analytes are not listed in Table 5. Some analytes such as cadmium, nickel, and tin were only detected at concentrations near the MDLs so that an RSD value calculated at 5 to 10 times the MDL was not possible.

One FPXRF instrument collected replicate measurements on an additional nine soil samples to provide a better assessment of the effect of sample preparation on precision. Table 6 shows these results. The additional nine soil samples were comprised of three from each texture and had analyte concentrations ranging from near the detection limit of the FPXRF analyzer to thousands of mg/kg. The FPXRF analyzer only collected replicate measurements from three of the preparation methods; no measurements were collected from the *in situ* homogenized samples. The FPXRF analyzer conducted five replicate measurements of the in situ field samples by taking measurements at five different points within the 4-inch by 4-inch sample square. Ten replicate measurements were collected for both the intrusive undried and unground and intrusive dried and ground samples contained in cups. The cups were shaken between each replicate measurement.

Table 6 shows that the precision dramatically improved from the in situ to the intrusive measurements. In general there was a slight improvement in precision when the sample was dried and ground. Two factors caused the precision for the in situ measurements to be poorer. The major factor is soil heterogeneity. By moving the probe within the 4-inch by 4-inch square, measurements of different soil samples were actually taking place within the square. Table 6 illustrates the dominant effect of soil heterogeneity. It overwhelmed instrument precision when the FPXRF analyzer was used in this mode. The second factor that caused the RSD values to be higher for the in situ measurements is the fact that only five versus ten replicates were taken. A lesser number of measurements caused the standard deviation to be larger which in turn elevated the RSD values.

13.6 Accuracy Measurements: Five of the FPXRF instruments (not including the MAP Spectrum Analyzer) analyzed 18 SRMs using the source count times and calibration methods given at the beginning of this section. The 18 SRMs included 9 soil SRMs, 4 stream or river sediment SRMs, 2 sludge SRMs, and 3 ash SRMs. Each of the SRMs contained known concentrations of certain target analytes. A percent recovery was calculated for each analyte in each SRM for each FPXRF instrument. Table 7 presents a summary of this data. With the exception of cadmium, chromium, and nickel, the values presented in Table 7 were generated from the 13 soil and sediment SRMs only. The 2 sludge and 3 ash SRMs were included for cadmium, chromium, and nickel because of the low or nondetectable concentrations of these three analytes in the soil and sediment SRMs.

Only 12 analytes are presented in Table 7. These are the analytes that are of environmental concern and provided a significant number of detections in the SRMs for an accuracy assessment. No data is presented for the X-MET 920 with the gas-filled proportional detector. This FPXRF instrument was calibrated empirically using site-specific soil samples. The percent recovery values from this instrument were very sporadic and the data did not lend itself to presentation in Table 7.

Table 8 provides a more detailed summary of accuracy data for one FPXRF instrument (TN 9000) for the 9 soil SRMs and 4 sediment SRMs. Table 8 shows the certified value, measured value, and percent recovery for five analytes. These analytes were chosen because they are of environmental concern and were most prevalently certified for in the SRM and detected by the FPXRF instrument. The first nine SRMs are soil and the last 4 SRMs are sediment. Percent recoveries for the four NIST SRMs were often between 90 and 110 percent for all analytes.

13.7 Comparability: Comparability refers to the confidence with which one data set can be compared to another. In this case, FPXRF data generated from a large study of six FPXRF instruments was compared to SW-846 Methods 3050 and 6010 which are the standard soil extraction for metals and analysis by inductively coupled plasma. An evaluation of comparability was conducted by using linear regression analysis. Three factors were determined using the linear regression. These factors were the y-intercept, the slope of the line, and the coefficient of determination (r^2).

As part of the comparability assessment, the effects of soil type and preparation methods were studied. Three soil types (textures) and four preparation methods were examined during the study. The preparation methods evaluated the cumulative effect of particle size, moisture, and homogenization on comparability. Due to the large volume of data produced during this study, linear regression data for six analytes from only one FPXRF instrument is presented in Table 9. Similar trends in the data were seen for all instruments.

Table 9 shows the regression parameters for the whole data set, broken out by soil type, and by preparation method. The soil types are as follows: soil 1--sand; soil 2--loam; and soil 3--silty clay. The preparation methods are as follows: preparation 1--in situ in the field; preparation 2--in situ, sample collected and homogenized; preparation 3--intrusive, with sample in a sample cup but sample still wet and not ground; and preparation 4--sample dried, ground, passed through a 40-mesh sieve, and placed in sample cup.

For arsenic, copper, lead, and zinc, the comparability to the confirmatory laboratory was excellent with r^2 values ranging from 0.80 to 0.99 for all six FPXRF instruments. The slopes of the regression lines for arsenic, copper, lead, and zinc, were generally between 0.90 and 1.00 indicating the data would need to be corrected very little or not at all to match the confirmatory laboratory data. The r^2 values and slopes of the regression lines for barium and chromium were

not as good as for the other for analytes, indicating the data would have to be corrected to match the confirmatory laboratory.

Table 9 demonstrates that there was little effect of soil type on the regression parameters for any of the six analytes. The only exceptions were for barium in soil 1 and copper in soil 3. In both of these cases, however, it is actually a concentration effect and not a soil effect causing the poorer comparability. All barium and copper concentrations in soil 1 and 3, respectively, were less than 350 mg/kg.

Table 9 shows there was a preparation effect on the regression parameters for all six analytes. With the exception of chromium, the regression parameters were primarily improved going from preparation 1 to preparation 2. In this step, the sample was removed from the soil surface, all large debris was removed, and the sample was thoroughly homogenized. The additional two preparation methods did little to improve the regression parameters. This data indicates that homogenization is the most critical factor when comparing the results. It is essential that the sample sent to the confirmatory laboratory match the FPXRF sample as closely as possible.

Section 11.0 of this method discusses the time necessary for each of the sample preparation techniques. Based on the data quality objectives for the project, an analyst must decide if it is worth the extra time required to dry and grind the sample for small improvements in comparability. Homogenization requires 3 to 5 minutes. Drying the sample requires one to two hours. Grinding and sieving requires another 10 to 15 minutes per sample. Lastly, when grinding and sieving is conducted, time must be allotted to decontaminate the mortars, pestles, and sieves. Drying and grinding the samples and decontamination procedures will often dictate that an extra person be on site so that the analyst can keep up with the sample collection crew. The cost of requiring an extra person on site to prepare samples must be balanced with the gain in data quality and sample throughput.

13.8 The following documents may provide additional guidance and insight on this method and technique:

13.8.1 Hewitt, A.D. 1994. "Screening for Metals by X-ray Fluorescence Spectrometry/Response Factor/Compton K_{α} Peak Normalization Analysis." *American Environmental Laboratory.* Pages 24-32.

13.8.2 Piorek, S., and J.R. Pasmore. 1993. "Standardless, In Situ Analysis of Metallic Contaminants in the Natural Environment With a PC-Based, High Resolution Portable X-Ray Analyzer." *Third International Symposium on Field Screening Methods for Hazardous Waste and Toxic Chemicals.* Las Vegas, Nevada. February 24-26, 1993. Volume 2, Pages 1135-1151.

14.0 POLLUTION PREVENTION

14.1 Pollution prevention encompasses any technique that reduces or eliminates the quantity and/or toxicity of waste at the point of generation. Numerous opportunities for pollution prevention exist in laboratory operation. The EPA has established a preferred hierarchy of environmental management techniques that places pollution prevention as the management option of first choice. Whenever feasible, laboratory personnel should use pollution prevention techniques to address their waste generation. When wastes cannot be feasibly reduced at the source, the Agency recommends recycling as the next best option.

14.2 For information about pollution prevention that may be applicable to laboratories and research institutions consult *Less is Better: Laboratory Chemical management for Waste Reduction* available from the American Chemical Society's Department of Government Relations and Science Policy, 1155 16th Street N.W., Washington D.C. 20036, (202) 872-4477.

15.0 WASTE MANAGEMENT

The Environmental Protection Agency requires that laboratory waste management practices be conducted consistent with all applicable rules and regulations. The Agency urges laboratories to protect the air, water, and land by minimizing and controlling all releases from hoods and bench operations, complying with the letter and spirit of any sewer discharge permits and regulations, and by complying with all solid and hazardous waste regulations, particularly the hazardous waste identification rules and land disposal restrictions. For further information on waste management, consult *The Waste Management Manual for Laboratory Personnel* available from the American Chemical Society at the address listed in Sec. 14.2.

16.0 REFERENCES

1. Metorex. X-MET 920 User's Manual.

2. Spectrace Instruments. 1994. Energy Dispersive X-ray Fluorescence Spectrometry: An Introduction.

3. TN Spectrace. Spectrace 9000 Field Portable/Benchtop XRF Training and Applications Manual.

4. Unpublished SITE data, recieved from PRC Environment Management, Inc.

17.0 TABLES, DIAGRAMS, FLOWCHARTS, AND VALIDATION DATA

The pages to follow contain Tables 1 through 9 and a method procedure flow diagram.

TABLE 1
INTERFERENCE FREE DETECTION LIMITS

Analyte	Chemical Abstract Series Number	Detection Limit in Quartz Sand (milligrams per kilogram)
Antimony (Sb)	7440-36-0	40
Arsenic (As)	7440-38-0	40
Barium (Ba)	7440-39-3	20
Cadmium (Cd)	7440-43-9	100
Calcium (Ca)	7440-70-2	70
Chromium (Cr)	7440-47-3	150
Cobalt (Co)	7440-48-4	60
Copper (Cu)	7440-50-8	50
Iron (Fe)	7439-89-6	60
Lead (Pb)	7439-92-1	20
Manganese (Mn)	7439-96-5	70
Mercury (Hg)	7439-97-6	30
Molybdenum (Mo)	7439-93-7	10
Nickel (Ni)	7440-02-0	50
Potassium (K)	7440-09-7	200
Rubidium (Rb)	7440-17-7	10
Selenium (Se)	7782-49-2	40
Silver (Ag)	7440-22-4	70
Strontium (Sr)	7440-24-6	10
Thallium (Tl)	7440-28-0	20
Thorium (Th)	7440-29-1	10
Tin (Sn)	7440-31-5	60
Titanium (Ti)	7440-32-6	50
Vanadium (V)	7440-62-2	50
Zinc (Zn)	7440-66-6	50
Zirconium (Zr)	7440-67-7	10

Source: References 1, 2, and 3

TABLE 2
SUMMARY OF RADIOISOTOPE SOURCE CHARACTERISTICS

Source	Activity (mCi)	Half-Life (Years)	Excitation Energy (keV)	Elemental Analysis Range	
Fe-55	20-50	2.7	5.9	Sulfur to Chromium Molybdenum to Barium	K Lines L Lines
Cd-109	5-30	1.3	22.1 and 87.9	Calcium to Rhodium Tantalum to Lead Barium to Uranium	K Lines K Lines L Lines
Am-241	5-30	458	26.4 and 59.6	Copper to Thulium Tungsten to Uranium	K Lines L Lines
Cm-244	60-100	17.8	14.2	Titanium to Selenium Lanthanum to Lead	K Lines L Lines

Source: Reference 1, 2, and 3

TABLE 3
SUMMARY OF X-RAY TUBE SOURCE CHARACTERISTICS

Anode Material	Recommended Voltage Range (kV)	K-alpha Emission (keV)	Elemental Analysis Range	
Cu	18-22	8.04	Potassium to Cobalt Silver to Gadolinium	K Lines L Lines
Mo	40-50	17.4	Cobalt to Yttrium Europium to Radon	K Lines L Lines
Ag	50-65	22.1	Zinc to Technicium Ytterbium to Neptunium	K Lines L Lines

Source: Reference 4

Notes: The sample elements excited are chosen by taking as the lower limit the same ratio of excitation line energy to element absorption edge as in Table 2 (approximately 0.45) and the requirement that the excitation line energy be above the element absorption edge as the upper limit (L2 edges used for L lines). K-beta excitation lines were ignored.

TABLE 4
FIELD-BASED METHOD DETECTION LIMITS (mg/kg)[a]

Analyte	Instrument					
	TN 9000	TN Lead Analyzer	X-MET 920 (SiLi Detector)	X-MET 920 (Gas-Filled Detector)	XL Spectrum Analyzer	MAP Spectrum Analyzer
Antimony	55	NR	NR	NR	NR	NR
Arsenic	60	50	55	50	110	225
Barium	60	NR	30	400	NR	NR
Chromium	200	460	210	110	900	NR
Cobalt	330	NR	NR	NR	NR	NR
Copper	85	115	75	100	125	525
Lead	45	40	45	100	75	165
Manganese	240	340	NR	NR	NR	NR
Molybdenum	25	NR	NR	NR	30	NR
Nickel	100	NR	NA	NA	NA	NR
Rubidium	30	NR	NR	NR	45	NR
Strontium	35	NR	NR	NR	40	NR
Tin	85	NR	NR	NR	NR	NR
Zinc	80	95	70	NA	110	NA
Zirconium	40	NR	NR	NR	25	NR

Source: Reference 4

[a] MDLs are related to the total number of counts taken. See Section 13.3 for count times used to generate this table.

NR Not reported.

NA Not applicable; analyte was reported but was not at high enough concentrations for method detection limit to be determined.

TABLE 5
PRECISION

Analyte	Average Relative Standard Deviation for Each Instrument at 5 to 10 Times the MDL					
	TN 9000	TN Lead Analyzer	X-MET 920 (SiLi Detector)	X-MET 920 (Gas-Filled Detector)	XL Spectrum Analyzer	MAP Spectrum Analyzer
Antimony	6.54	NR	NR	NR	NR	NR
Arsenic	5.33	4.11	3.23	1.91	12.47	6.68
Barium	4.02	NR	3.31	5.91	NR	NR
Cadmium	29.84[a]	NR	24.80[a]	NR	NR	NR
Calcium	2.16	NR	NR	NR	NR	NR
Chromium	22.25	25.78	22.72	3.91	30.25	NR
Cobalt	33.90	NR	NR	NR	NR	NR
Copper	7.03	9.11	8.49	9.12	12.77	14.86
Iron	1.78	1.67	1.55	NR	2.30	NR
Lead	6.45	5.93	5.05	7.56	6.97	12.16
Manganese	27.04	24.75	NR	NR	NR	NR
Molybdenum	6.95	NR	NR	NR	12.60	NR
Nickel	30.85[a]	NR	24.92[a]	20.92[a]	NA	NR
Potassium	3.90	NR	NR	NR	NR	NR
Rubidium	13.06	NR	NR	NR	32.69[a]	NR
Strontium	4.28	NR	NR	NR	8.86	NR
Tin	24.32[a]	NR	NR	NR	NR	NR
Titanium	4.87	NR	NR	NR	NR	NR
Zinc	7.27	7.48	4.26	2.28	10.95	0.83
Zirconium	3.58	NR	NR	NR	6.49	NR

Source: Reference 4

[a] These values are biased high because the concentration of these analytes in the soil samples was near the detection limit for that particular FPXRF instrument.
NR Not reported.
NA Not applicable; analyte was reported but was below the method detection limit.

TABLE 6
PRECISION AS AFFECTED BY SAMPLE PREPARATION

Analyte	Average Relative Standard Deviation for Each Preparation Method		
	In Situ-Field	Intrusive-Undried and Unground	Intrusive-Dried and Ground
Antimony	30.1	15.0	14.4
Arsenic	22.5	5.36	3.76
Barium	17.3	3.38	2.90
Cadmium[a]	41.2	30.8	28.3
Calcium	17.5	1.68	1.24
Chromium	17.6	28.5	21.9
Cobalt	28.4	31.1	28.4
Copper	26.4	10.2	7.90
Iron	10.3	1.67	1.57
Lead	25.1	8.55	6.03
Manganese	40.5	12.3	13.0
Mercury	ND	ND	ND
Molybdenum	21.6	20.1	19.2
Nickel[a]	29.8	20.4	18.2
Potassium	18.6	3.04	2.57
Rubidium	29.8	16.2	18.9
Selenium	ND	20.2	19.5
Silver[a]	31.9	31.0	29.2
Strontium	15.2	3.38	3.98
Thallium	39.0	16.0	19.5
Thorium	NR	NR	NR
Tin	ND	14.1	15.3
Titanium	13.3	4.15	3.74
Vanadium	NR	NR	NR
Zinc	26.6	13.3	11.1
Zirconium	20.2	5.63	5.18

Source: Reference 4

[a] These values may be biased high because the concentration of these analytes in the soil samples was near the detection limit.
ND Not detected.
NR Not reported.

TABLE 7
ACCURACY

Analyte	Instrument															
	TN 9000				TN Lead Analyzer				X-MET 920 (SiLi Detector)				XL Spectrum Analyzer			
	n	Range of % Rec.	Mean % Rec.	SD	n	Range of % Rec.	Mean % Rec.	SD	n	Range of % Rec.	Mean % Rec	SD	n	Range of % Rec.	Mean % Rec.	SD
Sb	2	100-149	124.3	NA	--	--	--	--	--	--	--	--	--	--	--	--
As	5	68-115	92.8	17.3	5	44-105	83.4	23.2	4	9.7-91	47.7	39.7	5	38-535	189.8	206
Ba	9	98-198	135.3	36.9	--	--	--	--	9	18-848	168.2	262	--	--	--	--
Cd	2	99-129	114.3	NA	--	--	--	--	6	81-202	110.5	45.7	--	--	--	--
Cr	2	99-178	138.4	NA	--	--	--	--	7	22-273	143.1	93.8	3	98-625	279.2	300
Cu	8	61-140	95.0	28.8	6	38-107	79.1	27.0	11	10-210	111.8	72.1	8	95-480	203.0	147
Fe	6	78-155	103.7	26.1	6	89-159	102.3	28.6	6	48-94	80.4	16.2	6	26-187	108.6	52.9
Pb	11	66-138	98.9	19.2	11	68-131	97.4	18.4	12	23-94	72.7	20.9	13	80-234	107.3	39.9
Mn	4	81-104	93.1	9.70	3	92-152	113.1	33.8	--	--	--	--	--	--	--	--
Ni	3	99-122	109.8	12.0	--	--	--	--	--	--	--	--	3	57-123	87.5	33.5
Sr	8	110-178	132.6	23.8	--	--	--	--	--	--	--	--	7	86-209	125.1	39.5
Zn	11	41-130	94.3	24.0	10	81-133	100.0	19.7	12	46-181	106.6	34.7	11	31-199	94.6	42.5

Source: Reference 4

n Number of samples that contained a certified value for the analyte and produced a detectable concentration from the FPXRF instrument.
SD Standard deviation.
NA Not applicable; only two data points, therefore, a SD was not calculated.
%Rec. Percent recovery.
-- No data.

TABLE 8
ACCURACY FOR TN 9000[a]

Standard Reference Material	Arsenic			Barium			Copper			Lead			Zinc		
	Cert. Conc.	Meas. Conc.	%Rec.	Cert. Conc.	Meas. Conc.	%Rec.	Cert. Conc.	Meas. Conc.	%Rec.	Cert. Conc.	Meas. Conc.	%Rec.	Cert. Conc.	Meas. Conc.	%Rec.
RTC CRM-021	24.8	ND	NA	586	1135	193.5	4792	2908	60.7	144742	149947	103.6	546	224	40.9
RTC CRM-020	397	429	92.5	22.3	ND	NA	753	583	77.4	5195	3444	66.3	3022	3916	129.6
BCR CRM 143R	--	--	--	--	--	--	131	105	80.5	180	206	114.8	1055	1043	99.0
BCR CRM 141	--	--	--	--	--	--	32.6	ND	NA	29.4	ND	NA	81.3	ND	NA
USGS GXR-2	25.0	ND	NA	2240	2946	131.5	76.0	106	140.2	690	742	107.6	530	596	112.4
USGS GXR-6	330	294	88.9	1300	2581	198.5	66.0	ND	NA	101	80.9	80.1	118	ND	NA
NIST 2711	105	104	99.3	726	801	110.3	114	ND	NA	1162	1172	100.9	350	333	94.9
NIST 2710	626	722	115.4	707	782	110.6	2950	2834	96.1	5532	5420	98.0	6952	6476	93.2
NIST 2709	17.7	ND	NA	968	950	98.1	34.6	ND	NA	18.9	ND	NA	106	98.5	93.0
NIST 2704	23.4	ND	NA	414	443	107.0	98.6	105	106.2	161	167	103.5	438	427	97.4
CNRC PACS-1	211	143	67.7	--	772	NA	452	302	66.9	404	332	82.3	824	611	74.2
SARM-51	--	--	--	335	466	139.1	268	373	139.2	5200	7199	138.4	2200	2676	121.6
SARM-52	--	--	--	410	527	128.5	219	193	88.1	1200	1107	92.2	264	215	81.4

Source: Reference 4

[a] All concentrations in milligrams per kilogram.
%Rec. Percent recovery.
ND Not detected.
NA Not applicable.
-- No data.

TABLE 9
REGRESSION PARAMETERS FOR COMPARABILITY[1]

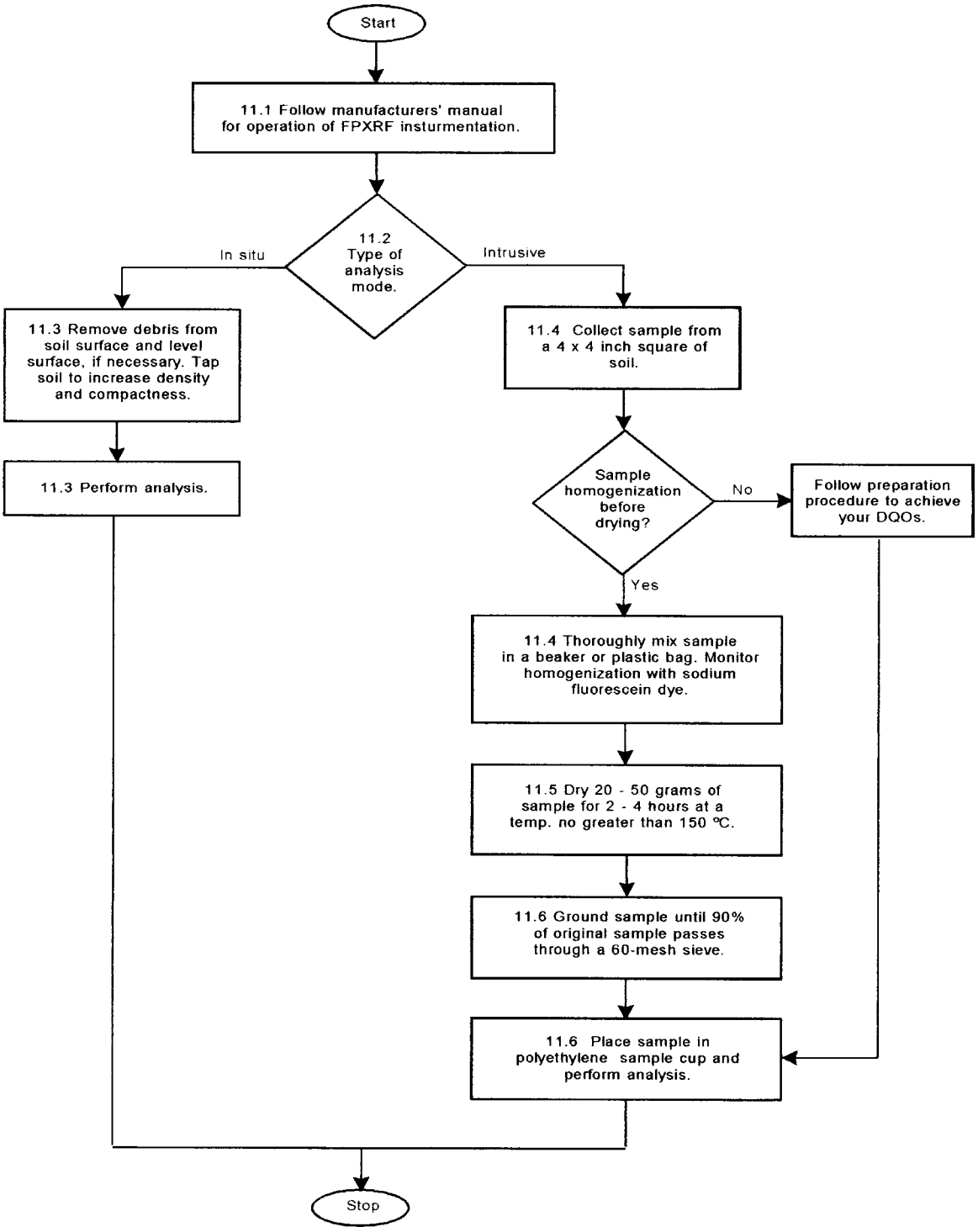
	Arsenic				Barium				Copper			
	n	r^2	Int.	Slope	n	r^2	Int.	Slope	n	r^2	Int.	Slope
All Data	824	0.94	1.62	0.94	1255	0.71	60.3	0.54	984	0.93	2.19	0.93
Soil 1	368	0.96	1.41	0.95	393	0.05	42.6	0.11	385	0.94	1.26	0.99
Soil 2	453	0.94	1.51	0.96	462	0.56	30.2	0.66	463	0.92	2.09	0.95
Soil 3	—	—	—	—	400	0.85	44.7	0.59	136	0.46	16.60	0.57
Prep 1	207	0.87	2.69	0.85	312	0.64	53.7	0.55	256	0.87	3.89	0.87
Prep 2	208	0.97	1.38	0.95	315	0.67	64.6	0.52	246	0.96	2.04	0.93
Prep 3	204	0.96	1.20	0.99	315	0.78	64.6	0.53	236	0.97	1.45	0.99
Prep 4	205	0.96	1.45	0.98	313	0.81	58.9	0.55	246	0.96	1.99	0.96

	Lead				Zinc				Chromium			
	n	r^2	Int.	Slope	n	r^2	Int.	Slope	n	r^2	Int.	Slope
All Data	1205	0.92	1.66	0.95	1103	0.89	1.86	0.95	280	0.70	64.6	0.42
Soil 1	357	0.94	1.41	0.96	329	0.93	1.78	0.93	—	—	—	—
Soil 2	451	0.93	1.62	0.97	423	0.85	2.57	0.90	—	—	—	—
Soil 3	397	0.90	2.40	0.90	351	0.90	1.70	0.98	186	0.66	38.9	0.50
Prep 1	305	0.80	2.88	0.86	286	0.79	3.16	0.87	105	0.80	66.1	0.43
Prep 2	298	0.97	1.41	0.96	272	0.95	1.86	0.93	77	0.51	81.3	0.36
Prep 3	302	0.98	1.26	0.99	274	0.93	1.32	1.00	49	0.73	53.7	0.45
Prep 4	300	0.96	1.38	1.00	271	0.94	1.41	1.01	49	0.75	31.6	0.56

Source: Reference 4

[1] Log-transformed data
n Number of data points
r^2 Coefficient of determination
Int. Y-intercept
— No applicable data

FIELD PORTABLE X-RAY FLUORESCENCE SPECTROMETRY FOR THE DETERMINATION OF ELEMENTAL CONCENTRATIONS IN SOIL AND SEDIMENT



Title:	**SOIL SAMPLING**
Category:	ENV 3.13
Revised:	August 1997

STANDARD OPERATING PROCEDURE

SOIL SAMPLING

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368 Pleasant View Drive / Lancaster, New York 14086 / (716) 684-8060

TITLE:	**SOIL SAMPLING**
CATEGORY:	ENV 3.13
REVISED:	August 1997

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1. Introduction

This document describes the procedures for the collection of representative soil samples. Representative sampling ensures the accurate characterization of site conditions. Analysis of soil samples may determine pollutant concentrations and the accompanying risks to public health, welfare, or the environment.

2. Scope

Included in this discussion are procedures for obtaining representative samples, quality assurance/quality control (QA/QC) measures, proper documentation of sampling activities, and recommendations for personnel safety.

3. Method Summary

Soil samples may be recovered using a variety of methods and equipment. These are dependent on the depth of the desired sample, the type of sample required (disturbed vs. undisturbed), and the soil type.

Samples of near-surface soils may be easily obtained using a spade, stainless-steel spoon, trowel, or scoop. Sampling at greater depths may be performed using a hand auger; a power auger; or, if a test pit is required, a backhoe.

All sampling devices should be cleaned using pesticide-grade acetone (assuming that acetone is not a target compound) or methanol, then wrapped in clean aluminum foil, and custody sealed for identification. The sampling equipment should remain in this wrapping until it is needed. Each sampler should be used for one sample only. However, dedicated tools may be impractical if there is a large number of soil samples required. In this case, samplers should be cleaned in the field using standard decontamination procedures as outlined in E & E's Standard Operating Procedure (SOP) for Sampling Equipment Decontamination (see ENV 3.15).

4. Sample Preservation, Containers, Handling, and Storage

The chemical preservation of solids is not generally recommended. Refrigeration is usually the best approach, supplemented by a minimal holding time.

Soil samples should be handled according to the procedures outlined in E & E's SOP for Sample Packaging (see ENV 3.16).

5. Potential Problems

Potential problems with soil sampling include cross-contamination of samples and improper sample collection. Cross-contamination problems can be eliminated or minimized through the use of dedicated sampling equipment and bottles. If this is not possible or practical, then decontamination of sampling equipment is necessary. Improper sample collection is generally the result of the use of contaminated equipment; the disturbance of the matrix, resulting in compaction of the sample; and inadequate homogenization of the sample where required, resulting in variable, nonrepresentative results. Specific advantages and disadvantages of soil sampling equipment are presented in Table 5-1.

Table 5-1 Soil Sampling Equipment

Equipment	Applicability	Advantages and Disadvantages
Trier	Soft surface soil	Inexpensive; easy to use and decontaminate; difficult to use in stony, dry, or sandy soil.
Scoop, trowel, spoon, or spatula	Soft surface soil	Inexpensive; easy to use and decontaminate; trowels with painted surfaces should be avoided.
Tulip bulb planter	Soft soil, 0 to 6 inches	Easy to use and decontaminate; uniform diameter and sample volume; preserves soil core (suitable for volatile organic analysis (VOA) and undisturbed sample collection); limited depth capability; not useful for hard soils.
Spade or shovel	Medium soil, 0 to 12 inches	Easy to use and decontaminate; inexpensive; can result in sample mixing and loss of volatile organic compounds (VOCs).
Vehimeyer soil outfit	Soil, 0 to 10 feet	Difficult to drive into dense or hard material; can be difficult to pull from ground.
Soil coring device and auger	Soft soil, 0 to 24 inches	Relatively easy to use; preserves soil core (suitable for VOA and undisturbed sample collection); limited depth capability; can be difficult to decontaminate.
Thin-walled tube sampler	Soft soil, 0 to 10 feet	Easy to use; preserves soil core (suitable for VOA and undisturbed sample collection); may be used to help maintain integrity of VOA samples; easy to decontaminate; can be difficult to remove cores from sampler.
Split-spoon sampler	Soil, 0 inches to bedrock	Excellent depth range; preserves soil core (suitable for VOA and undisturbed sample collection); acetate sleeve may be used to help maintain integrity of VOA samples; useful for hard soils; often used in conjunction with drill rig for obtaining deep cores.

Table 5-1 Soil Sampling Equipment

Equipment	Applicability	Advantages and Disadvantages
Shelby tube sampler	Soft soil, 0 inches to bedrock	Excellent depth range; preserves soil core (suitable for VOA and undisturbed sample collection); tube may be used to ship sample to lab undisturbed; may be used in conjunction with drill rig for obtaining deep cores and for permeability testing; not durable in rocky soils.
Laskey sampler	Soil, 0 inches to bedrock	Excellent depth range; preserves soil cores; used in conjunction with drill rig for obtaining deep core; can be difficult to decontaminate.
Bucket auger	Soft soil, 3 inches to 10 feet	Easy to use; good depth range; uniform diameter and sample volume; acetate sleeve may be used to help maintain integrity of VOA samples; may disrupt and mix soil horizons greater than 6 inches in thickness.
Hand-operated power auger	Soil, 6 inches to 15 feet	Good depth range; generally used in conjunction with bucket auger for sample collection; destroys soil core (unsuitable for VOA and undisturbed sample collection); requires two or more equipment operators; can be difficult to decontaminate; requires gasoline-powered engine (potential for cross-contamination).
Continuous-flight auger	Soil, 0 inches to bedrock	Excellent depth range; easy to decontaminate; can be used on all soil samples; results in soil mixing and loss of VOCs.
Dutch auger	Designed specifically for wet, fibrous, or rooted soils (e.g., marshes)	
Eijkelcamp stoney soil auger	Stoney soils and asphalt	
Backhoe	Soil, 0 inches to 10 feet	Good depth range; provides visual indications as to depth of contaminants; allows for recovery of samples at specific depths; can result in loss of VOCs and soil mixing; shoring required at depth.

Note: Samplers may not be suitable for soils with coarse fragments.
Augers are suitable for soils with limited coarse fragments; only the stoney auger will work well in very gravelly soil.

6. Soil Sampling Equipment

Soil Sampling Equipment List

- Stainless-steel spoon
- Trier
- Scoop
- Trowel

- Spatula
- Stainless-steel tulip bulb planter
- Spade or shovel
- Vehimeyer soil sampler outfit
  - tubes
  - points
  - drive head
  - drop hammer
  - fuller jack and grip
- Soil-coring device
- Thin-walled tube sampler
- Split-spoon sampler
- Shelby tube sampler
- Laskey sampler
- Bucket auger
- Hand-operated power auger
- Continuous-flight auger
- Dutch auger
- Eijkelcamp stoney soil auger
- Backhoe
- Hand auger with replaceable sleeves

Sampling Support Equipment and Documentation List

- Sampling plan
- Sample location map
- Safety equipment, as specified in the Health and Safety Plan
- Decontamination supplies and equipment, as described in the Work Plan
- Compass
- Tape measure
- Survey stakes or flags
- Camera
- Stainless-steel buckets or bowls
- Sample containers, precleaned (e.g., I-Chem)
- Logbook
- Chain-of-custody forms
- Plastic sheet
- Soil gas probes
- Infiltrometer
- Pounding sleeve
- Extension rods
- T-handle

Labeling, Packaging, and Shipping Supplies

- Coolers
- Labels for sample containers and coolers (e.g., "fragile")
- Ice
- Plastic bags for sample containers and ice
- ESC paint cans and clamps for polychlorinated biphenyl sampling
- Vermiculite (only if certified asbestos free) or other absorbent
- Duct and strapping tape
- Federal Express airbills and pouches

6.1 Geophysical Equipment

Geophysical techniques can be integrated with field analytical and soil sampling equipment to help define areas of subsurface contamination. For a description of the geophysical techniques and associated applications, refer to E & E's SOP for Surface Geophysical Techniques (see GEO 4.2).

7. Reagents

This procedures does not require the use of reagents except for decontamination of equipment, as required. Refer to E & E's SOP for Sampling Equipment Decontamination (see ENV 3.15) and the Site-Specific Work Plan for proper decontamination procedures and appropriate solvents.

8. Procedures

8.1 Office Preparation

1. The preparation of a Health and Safety Plan is required prior to any sampling. The plan must be approved and signed by the Corporate Health and Safety Officer or his/her designee (i.e., the Regional Safety Coordinator).

2. Prepare a Sampling Plan to meet the data quality objectives of the project in accordance with contract requirements. Review available background information (i.e., topographic maps, soil survey maps, geologic maps, other site reports, etc.) to determine the extent of the sampling effort, the sampling method to be employed, and the type and amounts of equipment and supplies required.

3. Obtain necessary sampling and monitoring equipment (see Section 6), decontaminate or preclean the equipment, and ensure that it is in working order.

4. Contact the delivery service to confirm the ability to ship all equipment and samples. Determine whether shipping restrictions exist.

5. Prepare schedules and coordinate with staff, clients, and regulatory agencies, if appropriate.

8.2 Field Preparation

1. Identify local suppliers of sampling expendables (e.g., ice and plastic bags) and overnight delivery services (e.g., Federal Express).

2. Decontaminate or preclean all equipment before soil sampling, as described in E & E's SOP for Sampling Equipment Decontamination (see ENV 3.15), or as deemed necessary.

3. A general site survey should be performed prior to site entry in accordance with the Health and Safety Plan, followed by a site safety meeting.

4. Identify and stake all sampling locations. If required, the proposed locations may be adjusted based on site access, property boundaries, and surface obstructions. All staked locations will be utility-cleared by the property owner or field team prior to soil sampling.

8.3 Representative Sample Collection

The objective of representative sampling is to ensure that a sample or group of samples adequately reflects site conditions.

8.3.1 Sampling Approaches

It is important to select an appropriate sampling approach for accurate characterization of site conditions. Each approach is defined below. Table 8-1 summarizes the following sampling approaches and ranks them from most to least suitable based on the sampling objective.

8.3.1.1 Judgmental Sampling

Judgmental sampling is based on the subjective selection of sampling locations relative to historical site information, on-site investigation (site walk-over), etc. There is no randomization associated with this sampling approach because samples are collected primarily at areas of suspected highest contaminant concentrations. Therefore, any statistical calculations based on the sampling results would be unfairly biased.

Table 8-1 Representative Sampling Approach Comparison

Sampling Objective	Judgmental	Random	Stratified Random	Systematic Grid	Systematic Random	Search	Transect
Establish Threat	1	4	3	2[a]	3	3	2
Identify Sources	1	4	2	2[a]	3	2	3
Delineate Extent of Contamination	4	3	3	1[b]	1	1	1
Evaluate Treatment and Disposal Options	3	3	1	2	2	4	2
Confirm Cleanup	4	1[c]	3	1[b]	1	1	1[c]

1 Preferred approach.
2 Acceptable approach.
3 Moderately acceptable approach.
4 Least acceptable approach.
a Should be used with field analytical screening.
b Preferred only where known trends are present.
c Allows for statistical support of cleanup verification if sampling over entire site.

8.3.1.2 Random Sampling

Random sampling involves the arbitrary collection of samples within a defined area. Refer to EPA 1984 and EPA 1989 for a random number table and guidelines on selecting sample coordinates. The arbitrary selection of sample locations requires each sample location to be chosen independently so that results in all locations within the area of concern have an equal chance of being selected. To facilitate statistical probabilities of contaminant concentration, the area of concern must be homogeneous with respect to the parameters being monitored. Thus, the higher the degree of heterogeneity, the less the random sampling approach will reflect site conditions (see Figure 8-1).

8.3.1.3 Stratified Random Sampling

Stratified random sampling relies primarily on historical information and prior analytical results to divide the area of concern into smaller sampling areas, or “strata.” Strata can be defined by several factors, including sampling depth, contaminant concentration levels, and contaminant source areas. Sampling locations should be selected within a strata using random selection procedures (see Figure 8-2).

8.3.1.4 Systematic Grid Sampling

Systematic grid sampling involves the division of the area of concern into smaller sampling areas using a square or triangular grid. Samples are then collected from the intersections of the grid lines, or “nodes.” The origin and direction for placement of the grid should be selected by using an initial random point. The distance between nodes is dependent upon the size of the area of concern and the number of samples to be collected (see Figure 8-3).

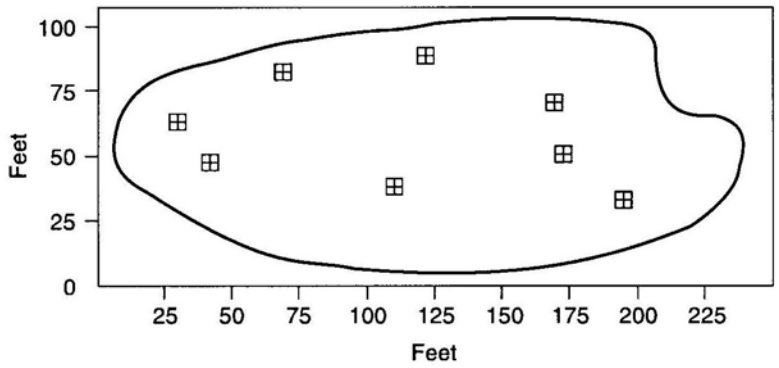


Figure 8-1 Random Sampling**

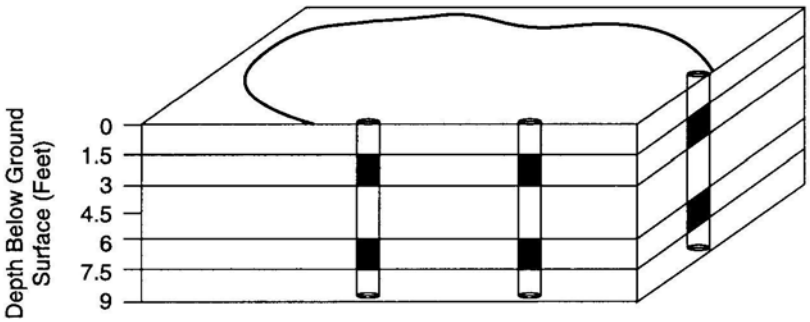


Figure 8-2 Stratified Random Sampling

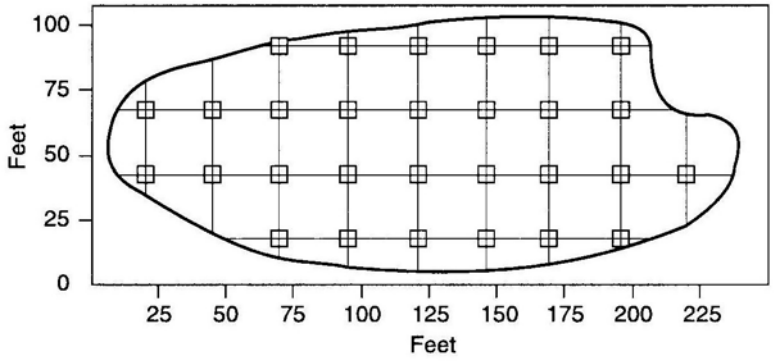
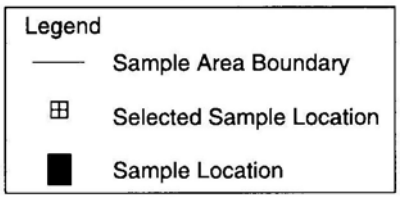


Figure 8-3 Systematic Grid Sampling**

** After EPA, February 1989



8.3.1.5 Systematic Random Sampling

Systematic random sampling involves dividing the area of concern into smaller sampling areas as described in Section 8.3.1.4. Samples are collected within each grid cell using random selection procedures (see Figure 8-4).

8.3.1.6 Biased-Search Sampling

Search sampling utilizes a systematic grid or systematic random sampling approach to define areas where contaminants exceed cleanup standards (i.e., hot spots). The distance between the grid lines and number of samples to be collected are dependent upon the acceptable level of error (i.e., the chance of missing a hot spot). This sampling approach requires that assumptions be made regarding the size, shape, and depth of hot spots (see Figure 8-5).

8.3.1.7 Transect Sampling

Transect sampling involves establishing one or more transect lines, parallel or nonparallel, across the area of concern. If the lines are parallel, this sampling approach is similar to systematic grid sampling. The advantage of transect sampling over systematic grid sampling is the relative ease of establishing and relocating transect lines as opposed to an entire grid. Samples are collected at regular intervals along the transect line at the surface and/or at a specified depth(s). The distance between the sample locations is determined by the length of the line and the number of samples to be collected (see Figure 8-6).

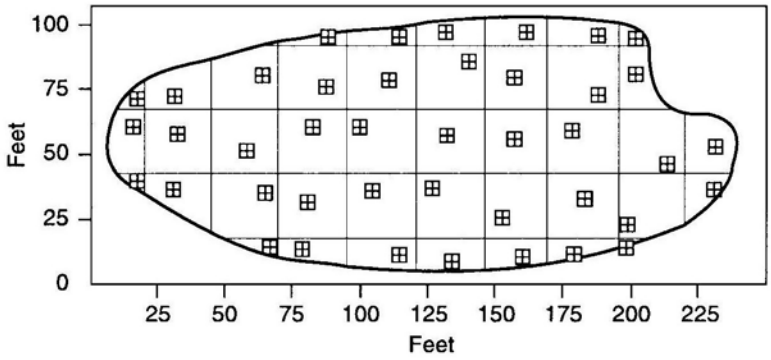


Figure 8-4 Systematic Random Sampling

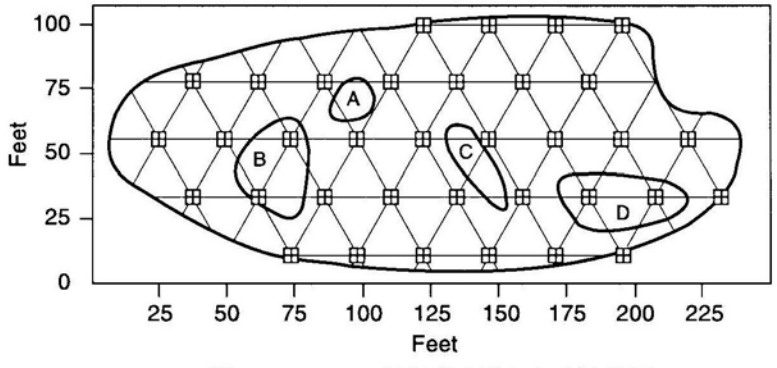


Figure 8-5 Search Sampling

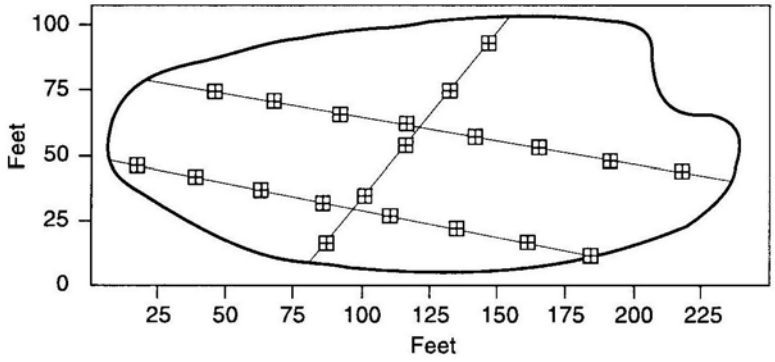
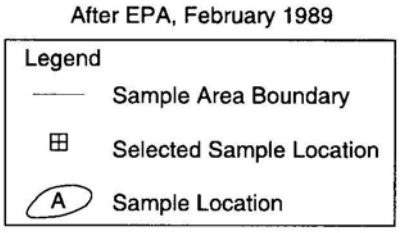


Figure 8-6 Transect Sampling



8.3.2 Surface Soil Samples

Collection of samples from near-surface soil can be accomplished with tools such as spades, spoons, shovels, and scoops. The surface material can be removed to the required depth with this equipment; stainless-steel or plastic scoops can then be used to collect the sample.

This method can be used in most soil types, but is limited to sampling near-surface areas. Accurate, representative samples can be collected with this procedure, depending on the care and precision demonstrated by the sampling technician. The use of a flat, pointed mason trowel to cut a block of the desired soil can be helpful when undisturbed profiles are required (e.g., for volatile organic analyses [VOAs]). A stainless-steel scoop, lab spoon, or plastic spoon will suf-

fice in most other applications. Care should be exercised to avoid the use of devices plated with chrome or other materials, as is common with garden implements such as potting trowels.

Soil samples are collected using the following procedure:

1. Carefully remove the top layer of soil to the desired sample depth with a precleaned spade;

2. Using a precleaned, stainless-steel scoop, spoon, trowel, or plastic spoon, remove and discard the thin layer of soil from the area that came into contact with the shovel;

3. Transfer the sample into an appropriate container using a stainless-steel or plastic lab spoon or equivalent. If composite samples are to be collected, place the soil sample in a stainless-steel or plastic bucket and mix thoroughly to obtain a homogeneous sample representative of the entire sampling interval. Place the soil samples into labeled containers. **(Caution: Never composite VOA samples)**;

4. VOA samples should be collected directly from the bottom of the hole before mixing the sample to minimize volatilization of contaminants;

5. Check to ensure that the VOA vial Teflon liner is present in the cap, if required. Fill the VOA vial fully to the top to reduce headspace. Secure the cap tightly. The chemical preservation of solids is generally not recommended. Refrigeration is usually the best approach, supplemented by a minimal holding time;

6. Ensure that a sufficient sample size has been collected for the desired analysis, as specified in the Sampling Plan;

7. Decontaminate equipment between samples according to E & E's SOP for Sampling Equipment Decontamination (see ENV 3.15); and

8. Fill in the hole and replace grass turf, if necessary.

QA/QC samples should be collected as specified, according to the Work Plan.

8.3.3 Sampling at Depth with Augers and Thin-Walled Tube Samplers

This system consists of an auger, a series of extensions, a T-handle, and a thin-walled tube. The auger is used to bore a hole to a desired sampling depth and is then withdrawn. The auger tip is then replaced with a tube core sampler, lowered down the borehole, and driven into the soil to the completion depth. The core is then withdrawn and the sample is collected.

Several augers are available, including bucket type, continuous-flight (screw), and post-hole augers. Because they provide a large volume of sample in a short time, bucket types are better for direct sample recovery. When continuous-flight augers are used, the sample can be collected directly off the flights, usually at 5-foot intervals. The continuous-flight augers are sat-

isfactory for use when a composite of the complete soil column is desired. Posthole augers have limited utility for sample collection because they are designed to cut through fibrous, rooted, swampy soil.

The following procedures will be used for collecting soil samples with the hand auger:

1. Attach the auger bit to a drill rod extension, and attach the T-handle to the drill rod.

2. Clear the area to be sampled of any surface debris (e.g., twigs, rocks, and litter). It may be advisable to remove the first 3 to 6 inches of surface soil from an area approximately 6 inches in radius around the drilling location.

3. Begin augering, periodically removing and depositing accumulated soils onto a canvas or plastic sheet spread near the hole. This prevents accidental brushing of loose material back down the borehole when removing the auger or adding drill rods. It also facilitates refilling the hole and avoids possible contamination of the surrounding area.

4. After reaching the desired depth, slowly and carefully remove the auger from the boring. When sampling directly from the auger, collect the sample after the auger is removed from the boring and proceed to Step 11.

5. A precleaned stainless-steel auger sleeve can also be used to collect a sample. After reaching the desired sampling depth, remove the auger and place the sleeve inside the auger. Collect the sample with the auger. Remove the auger from the boring. The sample will be collected only from the sleeve. The soil from the auger tip should never be used for the sample.

6. Remove the auger tip from the dill rods and replace with a precleaned thin-walled tube sampler. Install the proper cutting tip.

7. Carefully lower the tube sampler down the borehole. Gradually force the tube sampler into the soil. Care should be taken to avoid scraping the borehole sides. Avoid hammering the drill rods to facilitate coring, because the vibrations may cause the boring walls to collapse.

8. Remove the tube sampler and unscrew the drill rods.

9. Remove the cutting tip and core from the device.

10. Discard the top of the core (approximately 1 inch), because this represents material collected before penetration of the layer in question. Place the remaining core into the sample container.

11. If required, ensure that a Teflon liner is present in the cap. Secure the cap tightly onto the sample container. Place the sample bottle in a plastic bag and put on ice to keep the sample at 4°Celsius.

12. Carefully and clearly label the container with the appropriate sample tag, addressing all the categories or parameters listed in E & E's SOP for Sample Packaging and Shipping (see ENV 3.16).

13. Use the chain-of-custody form to document the types and numbers of soil samples collected and logged. Verify that the chain-of-custody form is correctly and completely filled out.

14. Record the time and date of sample collection, as well as a description of the sample, in the field logbook.

15. If another sample is to be collected in the sample hole, but at a greater depth, re-attach the auger bit to the drill and assembly, and follow Steps 3 through 11, making sure to decontaminate the auger and tube sampler between samples.

16. Abandon the hole according to applicable regulations. Generally, shallow holes can simply be backfilled with the removed soil material.

17. Decontaminate the sampling equipment per E & E's SOP for Sampling Equipment Decontamination (see ENV 3.15).

8.3.4 Sampling at Depth with a Trier

1. Insert the trier into the material to be sampled at a 0° to 45° angle from horizontal. This orientation minimizes the spillage of sample material. Extraction of samples may require tilting of the containers.

2. Rotate the trier once or twice to cut a core of material.

3. Slowly withdraw the trier, making sure that the slot is facing upward.

4. Transfer the sample into a suitable container with the aid of a spatula and brush.

5. If required, ensure that a Teflon liner is present in the cap. Secure the cap tightly onto the sample container. Samples are handled in accordance with E & E's SOP for Sample Packaging and Shipping (see ENV 3.16).

6. Carefully and clearly label the container with the appropriate sample tag, addressing all the categories or parameters listed in E & E's SOP for Sample Packaging and Shipping (see ENV 3.16).

7. Use the chain-of-custody form to document the types and numbers of soil samples collected and logged.

8. Record the time and date of sample collection as well as a description of the sample and any associated air monitoring measurements in the field logbook.

9. Abandon the hole according to applicable regulations. Generally, shallow holes can simply be backfilled with the removed soil material.

10. Decontaminate sampling equipment per E & E's SOP for Sampling Equipment Decontamination (see ENV 3.15).

8.3.5 Sampling at Depth with a Split-Spoon (Barrel) Sampler

The procedure for split-spoon sampling describes the extraction of undisturbed soil cores of 18 or 24 inches in length. A series of consecutive cores may be sampled to give a complete soil column, or an auger may be used to drill down to the desired depth for sampling. The split spoon is then driven to its sampling depth through the bottom of the augured hole and the core extraction.

This sampling device may be used to collect information such as soil density. All work should be performed in accordance with American Society for Testing and Materials (ASTM) D 1586-84, *Penetration Test and Split Barrel Sampling of Soils*.

1. Assemble the sampler by aligning both sides of the barrel and then screwing the bit on the bottom and the heavier head piece on top. Install a retaining cap in the head piece if necessary.

2. Place the sampler in a perpendicular position on the sample material.

3. Using a sledge hammer or well ring, if available, drive the tube. Do not drive past the bottom of the head piece because compression of the sample will result.

4. Record the length of the tube used to penetrate the material being sampled and the number of blows required to obtain this depth.

5. Withdraw the split spoon and open by unscrewing the bit and head. If a split sample is desired, a clean stainless-steel knife should be used to divide the tube contents in half, lengthwise. This sampler is available in 2- and 3.5-inch diameters. The required sample volume may dictate the use of the larger barrel. If needed, stainless-steel or Teflon sleeves can be used inside the split-spoon. If sleeves removed from the split-spoon are capped immediately, volatilization of contaminants can be reduced. When split-spoon sampling is performed to gain geologic information, all work should be performed in accordance with ASTM D 1586-67 (reapproved in 1974).

6. Cap the sample container, place in a double plastic bag, and attach the label and custody seal. Record all pertinent data in the field logbook and complete the sample analysis request form and chain-of-custody record before collecting the next sample.

7. If required, preserve or place the sample on ice.

8. Follow proper decontamination procedures and deliver samples to the laboratory for analysis.

8.3.6 Test Pit/Trench Excavation

These relatively large excavations are used to remove sections of soils when detailed examination of soil characteristics (horizontal, structure, color, etc.) is required. It is the least cost-effective sampling method because of the relatively high cost of backhoe operation.

1. Prior to any excavations with a backhoe, it is important to ensure that all sampling locations are clear of utility lines and poles (subsurface as well as above surface).

2. Using the backhoe, a trench is dug to approximately 3 feet in width and approximately 1 foot below the cleared sampling depth. Place removed or excavated soils on canvas or plastic sheets, if necessary. Trenches greater than 4 feet deep must be sloped or protected by a shoring system, as required by Occupational Safety and Health Administration (OSHA) regulations.

3. A shovel is used to remove a 1- to 2-inch layer of soil from the vertical face of the pit where sampling is to be done.

4. Samples are collected using a trowel, scoop, or coring device at the desired intervals. Be sure to scrape the vertical face at the point of sampling to remove any soil that may have fallen from above, and to expose soil for sampling. Samples are removed and placed in an appropriate container.

5. If required, ensure that a Teflon liner is present in the cap. Secure the cap tightly onto the sample container. Samples are handled in accordance with E & E's SOP for Sample Packaging and Shipping (see ENV 3.16).

6. Carefully and clearly label the container with the appropriate sample tag, addressing all the categories or parameters listed in E & E's SOP for Sample Packaging and Shipping (see ENV 3.16).

7. Use the chain-of-custody form to document the types and numbers of soil samples collected and logged.

8. Record the time and date of sample collection as well as a description of the sample and any associated air monitoring measurements in the field logbook.

9. Abandon the hole according to applicable state regulations. Generally, excavated holes can simply be backfilled with the removed soil material.

10. Decontaminate sampling equipment, including the backhoe bucket, per E & E's SOP for Sampling Equipment Decontamination (see ENV 3.15).

8.4 Sample Preparation

In addition to sampling equipment, representative sample collection includes sample quantity, volume, preservation, and holding time (see Table 8-2). *Sample preparation* refers to all aspects of sample handling after collection. How a sample is prepared can affect its representativeness. For example, homogenizing can result in a loss of volatiles and is therefore inappropriate when volatile contaminants are the concern.

8.4.1 Sample Quantity and Volume

The volume and number of samples necessary for site characterization will vary according to the budget, project schedule, and sampling approach.

8.4.2 Sample Preservation and Holding Time

Sample preservation and holding times are as discussed in Section 4.

8.4.3 Removing Extraneous Material

Discard materials in a sample that are not relevant for site or sample characterization (e.g., glass, rocks, and leaves), because their presence may introduce an error in analytical procedures.

8.4.4 Homogenizing Samples

Homogenizing is the mixing of a sample to provide a uniform distribution of the contaminants. Proper homogenization ensures that the containerized samples are representative of the total soil sample collected. All samples to be composited or split should be homogenized after all aliquots have been combined. Do not homogenize samples for volatile compound analysis.

Table 8-2 Standard Sampling Holding Times, Preservation Methods, and Volume Requirements

Protocol Parameter	Holding Time		Minimum Volume Required		Container Type		Preservation	
	Soil	Water	Soil	Water	Soil	Water	Soil	Water
SW-846								
VOA[e]	14 days from date sampled	14 days from date sampled	15 g	One 40-mL vial; no air space	Two 40-mL vials; no air space	Two 40-mL vials; no air space	Cool to 4°C (ice in cooler)	Add HC1 until pH <2 and cool to 4° (ice in cooler)
Semi-VOA (BNAs)[e]	14 days to extract from date sampled	7 days to extract from date sampled	30 g	1 L	8-oz. glass jar with Teflon-lined cap	½-gallon amber glass bottle	Cool to 4°C (ice in cooler)	Cool to 4°C (ice in cooler)
PCBs[d,e]	14 days to extract from date sampled	7 days to extract from date sampled	30 g	1 L	4-oz. glass jar with Teflon-lined cap	½-gallon amber glass bottle	Cool to 4°C (ice in cooler)	Cool to 4°C (ice in cooler)
Pesticides/PCBs[d,e]	14 days to extract from date sampled	7 days to extract from date sampled	30 g	1 L	8-oz. glass jar with Teflon-lined cap	½-gallon amber glass bottle	Cool to 4°C (ice in cooler)	Cool to 4°C (ice in cooler)
Metals[c]	6 months from date sampled	6 months from date sampled	10 g	300 mL	8-oz. glass jar with Teflon-lined cap	1-L polyethylene bottle with polyethylene-lined cap	Cool to 4°C (ice in cooler)	Add HNO_3 until pH <2 and cool to 4°C (ice in cooler)
Cyanide[c]	14 days from date sampled	14 days from date sampled	10 g	100 mL	8-oz. glass jar with Teflon-lined cap	1-L polyethylene bottle with polyethylene-lined cap	Cool to 4°C (ice in cooler)	Add NaOH until pH >12 and cool to 4°C (ice in cooler)
Hexavalent chromium[a]	24 hours from time sampled	24 hours from time sampled	10 g	50 mL	8-oz. glass jar with Teflon-lined cap	125-mL polyethylene bottle with polyethylene-lined cap	Cool to 4°C (ice in cooler)	Cool to 4°C (ice in cooler)
Total Organic Carbon (TOC)[a]	NA	28 days from date sampled	5 g	10 mL	8-oz. glass jar with Teflon-lined cap	125-mL polyethylene bottle with polyethylene-lined cap	Cool to 4°C (ice in cooler)	Add H_2SO_4 until pH <2 and cool to 4°C (ice in cooler)
Total Organic Halides (TOX)	NA	7 days from date sampled	100 g	200 mL	8-oz. glass jar with Teflon-lined cap	1-L amber glass bottle	Cool to 4°C (ice in cooler)	Add H_2SO_4 until pH <2 and cool to 4°C (ice in cooler)

Table 8-2 Standard Sampling Holding Times, Preservation Methods, and Volume Requirements

Protocol Parameter	Holding Time		Minimum Volume Required		Container Type		Preservation	
	Soil	Water	Soil	Water	Soil	Water	Soil	Water
Total Recoverable Petroleum Hydrocarbons[e]	28 days from date sampled	28 days from date sampled	50 g	1 L	8-oz. glass jar with Teflon-lined cap	1-L amber glass bottle	Cool to 4°C (ice in cooler)	Add H_2SO_4 until pH <2 and cool to 4°C (ice in cooler)
EPA-CLP								
VOA[e]	10 days from date received	10 days from date received	15 g	One 40-mL vial; no air space	Two 40-mL vials; no air space	Two 40-mL vials; no air space	Cool to 4°C (ice in cooler)	Add HC1 until pH <2 and cool to 4°C (ice in cooler)
Semi-VOA (BNAs)[e]	10 days to extract from date received	5 days to extract from date received	30 g	1 L	8-oz. glass jar with Teflon-lined cap	½-gallon amber glass bottle	Cool to 4°C (ice in cooler)	Cool to 4°C (ice in cooler)
PCBs[d,e]	10 days to extract from date received	5 days to extract from date received	30 g	1 L	4-oz. glass jar with Teflon-lined cap	½-gallon amber glass bottle	Cool to 4°C (ice in cooler)	Cool to 4°C (ice in cooler)
Pesticides/PCBs[d,e]	10 days to extract from date received	5 days to extract from date received	30 g	1 L	8-oz. glass jar with Teflon-lined cap	½-gallon amber glass bottle	Cool to 4°C (ice in cooler)	Cool to 4°C (ice in cooler)
Metals[c]	6 months from date sampled	6 months from date sampled	10 g	300 mL	8-oz. glass jar with Teflon-lined cap	1-L polyethylene bottle with polyethylene-lined cap	Cool to 4°C (ice in cooler)	Add HNO_3 to pH <2 and cool to 4°C (ice in cooler)
Cyanide[c]	12 days from date received	12 days from date received	10 g	100 mL	8-oz. glass jar with Teflon-lined cap	1-L polyethylene bottle with polyethylene-lined cap	Cool to 4°C (ice in cooler)	Add NaOH to pH >12 and cool to 4°C (ice in cooler)
NYSDEC-CLP								
VOA[e]	7 days from date received	10 days from date received	15 g	One 40-mL vial; no air space	Two 40-mL vials; no air space	Two 40-mL vials; no air space	Cool to 4°C (ice in cooler)	Add HC1 until pH <2 and cool to 4°C (ice in cooler)
Semi-VOA (BNAs)[e]	5 days to extract from date received	5 days to extract from date received	30 g	1 L	8-oz. glass jar with Teflon-lined cap	½-gallon amber glass bottle	Cool to 4°C (ice in cooler)	Cool to 4°C (ice in cooler)

Table 8-2 Standard Sampling Holding Times, Preservation Methods, and Volume Requirements

Protocol Parameter	Holding Time		Minimum Volume Required		Container Type		Preservation	
	Soil	Water	Soil	Water	Soil	Water	Soil	Water
PCBs[d,e]	5 days to extract from date received	5 days to extract from date received	30 g	1 L	4-oz. glass jar with Teflon-lined cap	½-gallon amber glass bottle	Cool to 4°C (ice in cooler)	Cool to 4°C (ice in cooler)
Pesticides/PCBs[d,e]	5 days to extract from date received	5 days to extract from date received	30 g	1 L	8-oz. glass jar with Teflon-lined cap	½-gallon amber glass bottle	Cool to 4°C (ice in cooler)	Cool to 4°C (ice in cooler)
Metals[c]	6 months from date sampled	6 months from date sampled	10 g	300 mL	8-oz. glass jar with Teflon-lined cap	1-L polyethylene bottle with polyethylene-lined cap	Cool to 4°C (ice in cooler)	Add HNO_3 to pH <2 and cool to 4°C (ice in cooler)
Cyanide[c]	12 days from date received	12 days from date received	10 g	100 mL	8-oz. glass jar with Teflon-lined cap	1-L polyethylene bottle with polyethylene-lined cap	Cool to 4°C (ice in cooler)	Add NaOH to pH >12 and cool to 4°C (ice in cooler)
EPA Water and Waste								
Total Dissolved Solids (TDS)	NA	7 days from date sampled	NA	200 mL	NA	1-L polyethylene bottle with polyethylene-lined cap	NA	Cool to 4°C (ice in cooler)

Note: All sample bottles will be prepared in accordance with EPA bottle-washing procedures. These procedures are incorporated in E & E's Laboratory and Field Personnel Chain-of-Custody Documentation and Quality Assurance/Quality Control Procedures Manual, July 1987.

[a] Technical requirements for sample holding times have been established for water matrices only. However, they are also suggested for use as guidelines in evaluating soil data.
[b] Holding time for GC/MS analysis is 7 days if samples are not preserved.
[c] Maximum holding time for mercury is 28 days from time sampled.
[d] If one container has already been collected for PCB analysis, then only one additional container need be collected for extractable organic, BNA, or pesticides/PCB analysis.
[e] Extra containers required for MS/MSD.

Key:

NA = Not applicable.

8.4.5 Compositing Samples

Compositing is the process of physically combining and homogenizing several individual soil aliquots of the same volume or weight. Compositing samples provides an average concentration of contaminants over a certain number of sampling points. Compositing dilutes high-concentration aliquots; therefore, detection limits should be reduced accordingly. If the composite area is heterogeneous in concentration and its composite value is to be compared to a particular action level, then that action level must be divided by the total number of aliquots making up the composite for accurate determination of the detection limit.

8.4.6 Splitting Samples

Splitting samples (after preparation) is performed when multiple portions of the same samples are required to be analyzed separately. Fill the sample containers simultaneously with alternate spoonfuls of the homogenized sample (see Figure 8-7).

8.5 Post-Operations

8.5.1 Field

Decontaminate all equipment according to E & E's SOP for Sampling Equipment Decontamination (see ENV 3.15).

8.5.2 Office

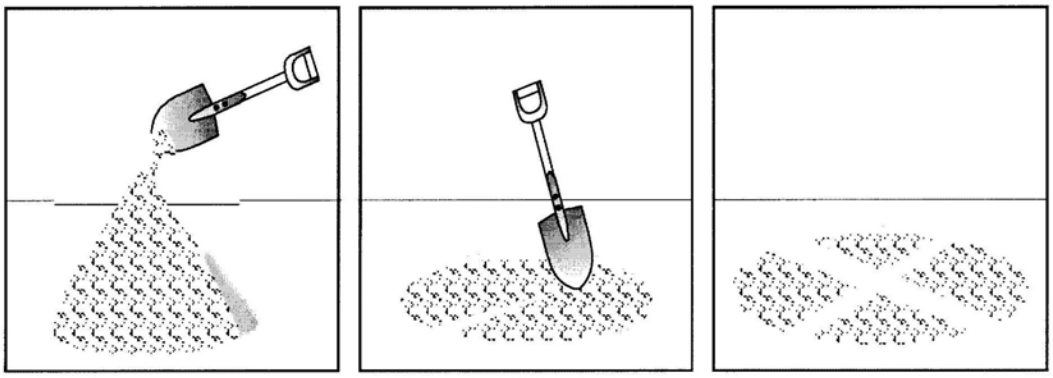
Organize field notes into a report format and transfer logging information to appropriate forms.

9. Calculations

There are no specific calculations required for these procedures.

10. Quality Assurance/Quality Control

The objective of QA/QC is to identify and implement methodologies that limit the introduction of error into sampling and analytical procedures.



Step 1:

- Cone Sample on hard, clean surface
- Mix by forming new cone

Step 2:

- Quarter after flattening cone

Step 3:

- Divide sample into quarters

Step 4:

- Remix opposite quarters
- Reform cone
- Repeat a minimum of 5 times

After: ASTM Standard C702-87

Figure 8-7 Quartering to Homogenized and Split Samples

10.1 Sampling Documentation

10.1.1 Soil Sample Label

All soil samples shall be documented in accordance with E & E's SOP for Sample Packaging and Shipping (see ENV 3.16). The soil sample label is filled out prior to collecting the sample and should contain the following:

1. Site name or identification.
2. Sample location and identifier.
3. Date samples were collected in a day, month, year format (e.g., 03 Jan 88 for January 3, 1988).
4. Time of sample collection, using 24-hour clock in the hours:minutes format.
5. Sample depth interval. Units used for depths should be in feet and tenths of feet.
6. Preservatives used, if any.
7. Analysis required.

8. Sampling personnel.

9. Comments and other relevant observations (e.g., color, odor, sample technique).

10.1.2 Logbook

A bound field notebook will be maintained by field personnel to record daily activities, including sample collection and tracking information. A separate entry will be made for each sample collected. These entries should include information from the sample label and a complete physical description of the soil sample, including texture, color (including notation of soil mottling), consistency, moisture content, cementation, and structure.

10.1.3 Chain of Custody

Use the chain-of-custody form to document the types and numbers of soil samples collected and logged. Refer to E & E's SOP for Sample Packaging and Shipping (see ENV 3.16) for directions on filling out this form.

10.2 Sampling Design

1. Sampling situations vary widely; thus, no universal sampling procedure can be recommended. However, a Sampling Plan should be implemented before any sampling operation is attempted, with attention paid to contaminant type and potential concentration variations.

2. Any of the sampling methods described here should allow a representative soil sample to be obtained, if the Sampling Plan is properly designed.

3. Consideration must also be given to the collection of a sample representative of all horizons present in the soil. Selection of the proper sampler will facilitate this procedure.

4. A stringent QA Project Plan should be outlined before any sampling operation is attempted. This should include, but not be limited to, properly cleaned samplers and sample containers, appropriate sample collection procedures, chain-of-custody procedures, and QA/QC samples.

11. Data Validation

The data generated will be reviewed according to the QA/QC considerations that are identified in Section 10.

11.1 Quality Assurance/Quality Control Samples

QA/QC samples are used to identify error due to sampling and/or analytical methodologies and chain-of-custody procedures.

11.1.1 Field Duplicates (Replicates)

Field duplicates are collected from one location and treated as separate samples throughout the sample handling and analytical processes. These samples are used to assess total error for critical samples with contaminant concentrations near the action level.

11.1.2 Collocated Samples

Collocated samples are generally collected 1.5 to 3.0 feet away from selected field samples to determine both local soil and contaminant variations on site. These samples are used to evaluate site variation within the immediate vicinity of sample collection.

11.1.3 Background Samples

Background or “clean” samples are collected from an area upgradient from the contamination area and representative of the typical conditions. These samples provide a standard for comparison of on-site contaminant concentration levels.

11.1.4 Rinsate (Equipment) Blanks

Rinsate blanks are collected by pouring analyte-free water (i.e., laboratory de-ionized water) on decontaminated sampling equipment to test for residual contamination. These samples are used to assess potential cross contamination due to improper decontamination procedures.

11.1.5 Performance Evaluation Samples

Performance evaluation samples are generally prepared by a third party, using a quantity of analyte(s) known to the preparer but unknown to the laboratory. The percentage of analyte(s) identified in the sample is used to evaluate laboratory procedural error.

11.1.6 Matrix Spike/Matrix Spike Duplicates (MS/MSDs)

MS/MSD samples are spiked in the laboratory with a known quantity of analyte(s) to confirm percent recoveries. They are primarily used to check sample matrix interferences.

11.1.7 Field Blanks

Field blanks are prepared in the field with certified clean sand, soil, or water. These samples are used to evaluate contamination error associated with sampling methodology and laboratory procedures.

11.1.8 Trip Blanks

Trip blanks are prepared prior to going into the field using certified clean sand, soil, or water. These samples are used to assess error associated with sampling methodology and analytical procedures for volatile organics.

12. Health and Safety

12.1 Hazards Associated with On-Site Contaminants

Depending on site-specific contaminants, various protective programs must be implemented prior to soil sampling. The site Health and Safety Plan should be reviewed with specific emphasis placed on a protection program planned for direct-contact tasks. Standard safe operating practices should be followed, including minimization of contact with potential contaminants in both the vapor phase and solid matrix by using both respirators and disposable clothing.

Use appropriate safe work practices for the type of contaminant expected (or determined from previous sampling efforts):

- Particulate or Metals Contaminants
  - Avoid skin contact with, and ingestion of, soils and dusts.
  - Use protective gloves.

- Volatile Organic Contaminants
  - Pre-survey the site with an HNu 101 or OVA 128 prior to collecting soil samples.
  - If monitoring results indicate organic constituents, sampling activities may be conducted in Level C protection. At a minimum, skin protection will be afforded by disposable protective clothing.

13. References

ASTM D 1586-67 (reapproved 1974), ASTM Committee on Standards, Philadelphia, PA.

ASTM D 1586-84, Penetration Test and Split Barrel Sampling of Soils.

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_____, 1984, *Characterization of Hazardous Waste Sites – A Methods Manual: Volume I, Site Investigations*, Section 7: Environmental Monitoring Systems Laboratory, Las Vegas, Nevada, EPA/600/4-84/075.

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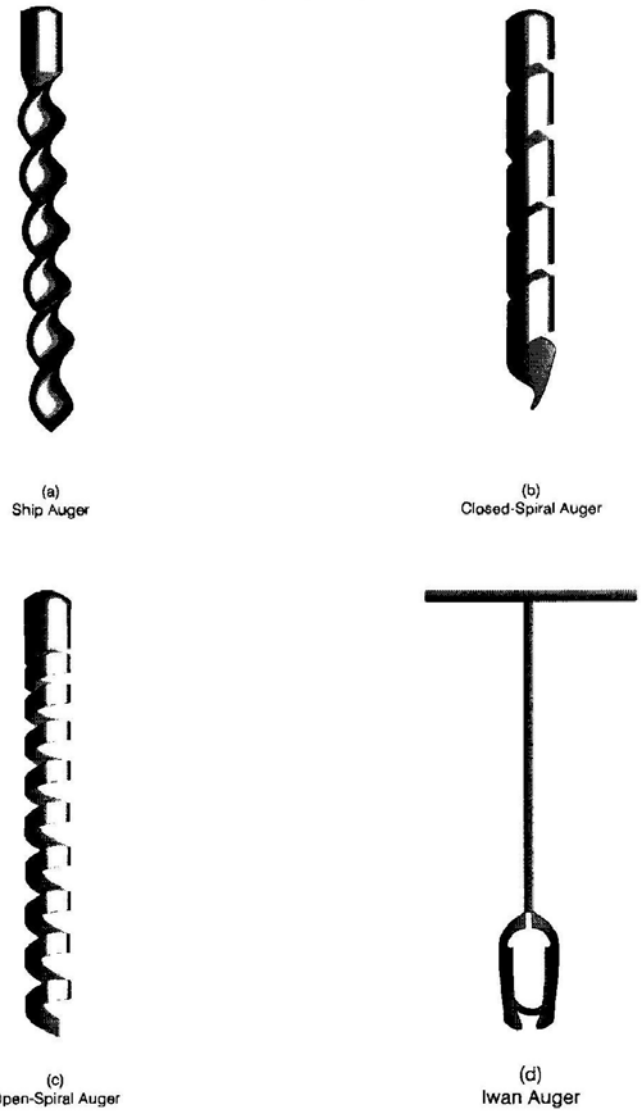
A SAMPLING AUGERS

A. Sampling Augers



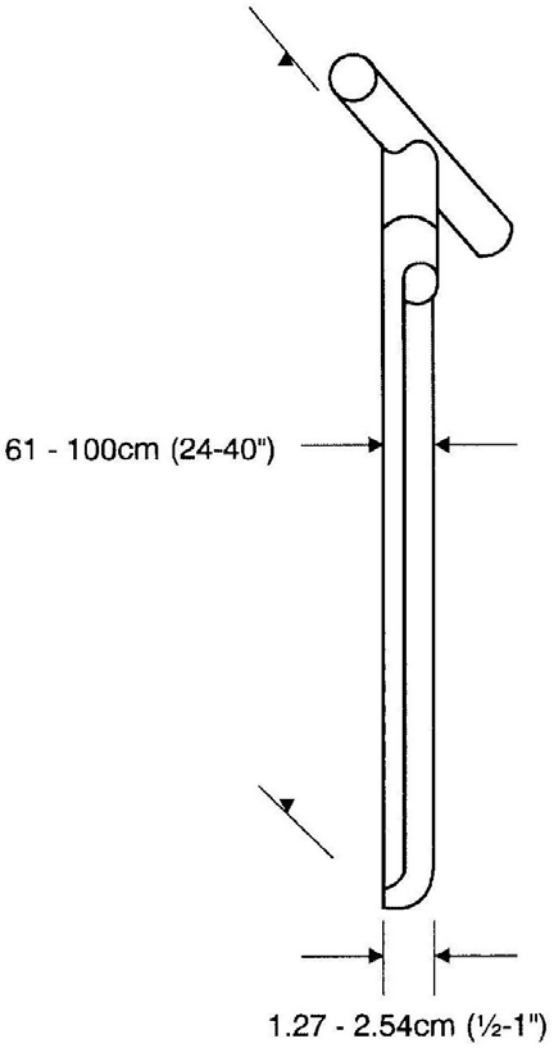
(a) Ship Auger

(b) Closed-Spiral Auger

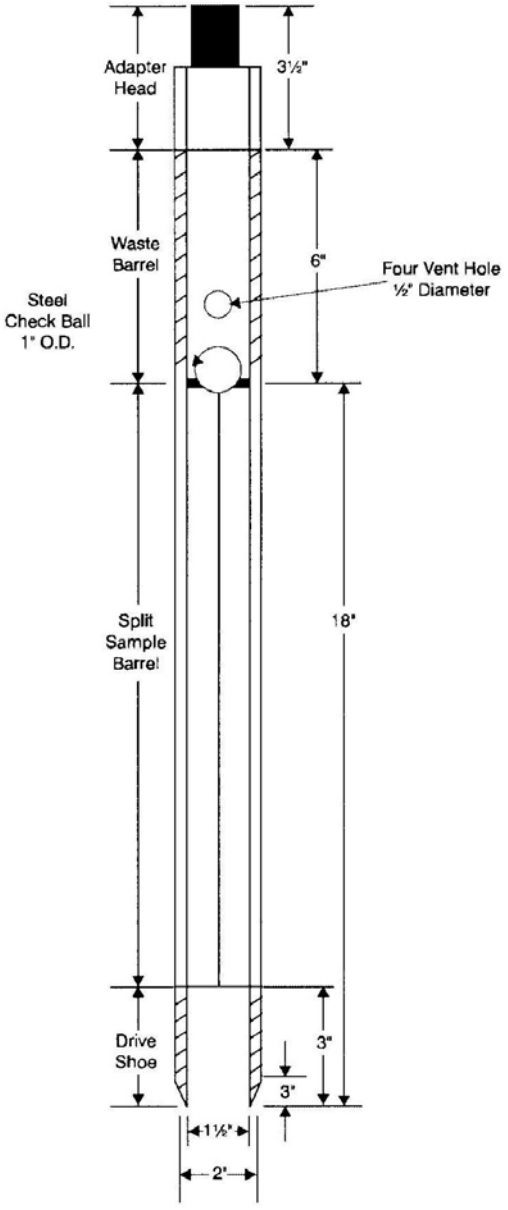
(c) Open-Spiral Auger

(d) Iwan Auger

B SAMPLING TRIER



C SPLIT-SPOON SAMPLER



Title:	**SAMPLING EQUIPMENT DECONTAMINATION**
Category:	ENV 3.15
Revised:	March 1999

STANDARD OPERATING PROCEDURE

SAMPLING EQUIPMENT DECONTAMINATION

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TITLE:	**SAMPLING EQUIPMENT DECONTAMINATION**
CATEGORY:	ENV 3.15
REVISED:	March 1999

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1. Scope and Application

The purpose of this procedure is to provide a description of methods for preventing or reducing cross-contamination and general guidelines for designing and selecting decontamination procedures for use at potential hazardous waste sites. The decontamination procedures chosen will prevent introduction and cross-contamination of suspected contaminants in environmental samples, and will protect the health and safety of site personnel.

2. Method Summary

Removing or neutralizing contaminants that have accumulated on personnel and equipment ensures protection of personnel from permeating substances, reduces/eliminates transfer of contaminants to clean areas, prevents the mixing of incompatible substances, and minimizes the likelihood of sample contamination.

Cross-contamination can be removed by physical decontamination procedures. The abrasive and non-abrasive methods include the use of brushes, high pressure water, air and wet blasting, and high pressure Freon cleaning. These methods should be followed by a wash/rinse process using appropriate cleaning solutions. A general protocol for cleaning with solutions is as follows:

1. Physical removal.
2. Non-phosphate detergent plus tap water.
3. Tap water.
4. 10% nitric acid.
5. Distilled/deionized water rinse.
6. Solvent rinse.
7. Total air dry.
8. Triple rinse with distilled/deionized water.

This procedure can be expanded to include additional or alternate solvent rinses that will remove specified target compounds if required by site-specific work plans (WP) or as directed by a particular client.

3. Interferences

The use of distilled/deionized water commonly available from commercial vendors may be acceptable for decontamination of sampling equipment provided that it has been verified by laboratory analysis to be analyte-free distilled/deionized water. Distilled water available from local grocery stores and pharmacies is generally not acceptable for final decontamination rinses. Contaminant-free deionized water is available from commercial vendors and may be shipped directly to the site or your hotel.

The use of an untreated potable water supply is not an acceptable substitute for tap water. Tap water may be used from any municipal water treatment system.

4. Equipment/Apparatus

The following are standard materials and equipment used as a part of the decontamination process:

- Appropriate protective clothing;
- Air purifying respirator (APR);
- Field log book;
- Non-phosphate detergent;
- Selected high purity, contaminant-free solvents;
- Long-handled brushes;
- Drop cloths (plastic sheeting);
- Trash containers;
- Paper towels;
- Galvanized tubs or equivalent (e.g., baby pools);
- Tap water;
- Contaminant-free distilled/deionized water;
- Metal/plastic container for storage and disposal of contaminated wash solutions;
- Pressurized sprayers, H_2O;
- Pressurized sprayers, solvents;
- Trash bags;
- Aluminum foil;
- Sample containers;

- Safety glasses or splash shield; and
- Emergency eyewash bottle.

5. Reagents

There are no reagents used in this procedure aside from decontamination solutions used for the equipment. The type of decontamination solution to be used shall depend upon the type and degree of contamination present and as specified in the project/site-specific Quality Assurance Project Plan (QAPP).

In general, the following solvents are utilized for decontamination purposes:

- 10% nitric acid wash (reagent grade nitric acid diluted with deionized/distilled water – 1 part acid to 10 parts water)[a];
- Acetone (pesticide grade)[b] ;
- Hexane (pesticide grade)[b];
- Methanol; and
- Methylene chloride[b].

[a] Only if sample is to be analyzed for trace metals.
[b] Only if sample is to be analyzed for organics requiring specific or specialized decontamination procedures. These solvents must be kept away from samples in order to avoid contamination by decon solvents.

6. Procedures

Decontamination is the process of removing or neutralizing contaminants that have accumulated on both personnel and equipment. Specific procedures in each case are designed accordingly and may be identified in either the Health and Safety Plan (HSP), WP, QAPP, or all three.

As part of the HSP, a personnel decontamination plan should be developed and set up before any personnel or equipment enters the areas of potential contamination. Decontamination procedures for equipment will be specified in the WP and the associated QAPP. These plans should include:

- Number and layout of decontamination stations;
- Decontamination equipment needed (see Section 4);

- Appropriate decontamination methods;
- Procedures to prevent contamination of clean areas;
- Methods and procedures to minimize worker contact with contaminants during removal of protective clothing;
- Methods and procedures to prevent cross-contamination of samples and maintain sample integrity and sample custody; and
- Methods for disposal of contaminated clothing, equipment, and solutions.

Revisions to these plans may be necessary for health and safety when the types of protective clothing, site conditions, or on-site hazards are reassessed based on new information.

Prevention of Contamination

Several procedures can be established to minimize contact with waste and the potential for contamination. For example:

- Employing work practices that minimize contact with hazardous substances (e.g., avoid areas of obvious contamination, avoid touching potentially hazardous substances);
- Use of remote sampling, handling, and container-opening techniques;
- Covering monitoring and sampling equipment with plastic or other protective material;
- Use of disposable outer garments and disposable sampling equipment with proper containment of these disposable items;
- Use of disposable towels to clean the outer surfaces of sample bottles before and after sample collection; and
- Encasing the source of contaminants with plastic sheeting or overpacks.

Proper procedures for dressing prior to entrance into contaminated areas will minimize the potential for contaminants to bypass the protective clothing. Generally, all fasteners (zippers, buttons, snaps, etc.) should be used, gloves and boots tucked under or over sleeves and pant legs, and all junctures taped (see the Health and Safety Plan for these procedures).

Decontamination Methods

All personnel, samples, and equipment leaving the contaminated area of a site must be decontaminated to remove any chemicals or infectious organisms that may have adhered to them. Various decontamination methods will either physically remove, inactivate by chemical detoxification/disinfection/sterilization, or remove contaminants by both physical and chemical means.

In many cases, gross contamination can be removed by physical means. The physical decontamination techniques can be grouped into two categories: abrasive methods and non-abrasive methods.

6.1 Abrasive Cleaning Methods

Abrasive cleaning methods work by rubbing and wearing away the top layer of the surface containing the contaminant. The following reviews the available abrasive methods.

Mechanical

Mechanical methods include using brushes with metal, nylon, or natural bristles. The amount and type of contaminants removed will vary with the hardness of bristles, length of time brushing, and degree of brush contact. Material may also be removed by using appropriate tools to scrape, pry, or otherwise remove adhered materials.

Air Blasting

Air blasting equipment uses compressed air to force abrasive material through a nozzle at high velocities. The distance between nozzle and surface cleaned, air pressure, and time of air blasting dictate cleaning efficiency. The method's disadvantages are its inability to control the exact amount of material removed and its large amount of waste generated.

Wet Blasting

Wet blast cleaning involves the use of a suspended fine abrasive. The abrasive/water mixture is delivered by compressed air to the contaminated area. By using very fine abrasives, the amount of materials removed can be carefully controlled.

6.2 Non-abrasive Cleaning Methods

Non-abrasive cleaning methods work by either dissolution or by forcing the contaminant off a surface with pressure. In general, less of the equipment surface is removed using non-abrasive methods.

High-Pressure Water

This method consists of a high-pressure pump, an operator controlled directional nozzle, and high-pressure hose. Operating pressure usually ranges from 340 to 680 psi, which relates to flow rates of 20 to 140 lpm.

Steam Cleaning

This method uses water delivered at high pressure and high temperature in order to remove accumulated solids and/or oils.

Ultra-High-Pressure Water

This system produces a water jet from 1,000 to 4,000 atm. This ultra-high-pressure spray can remove tightly-adhered surface films. The water velocity ranges from 500 m/sec. (1,000 atm) to 900 m/sec. (4,000 atm). Additives can be used to enhance the cleaning action, if approved by the QAPP for the project.

High-Pressure Freon Cleaning

Freon cleaning is a very effective method for cleaning cloth, rubber, plastic, and external/internal metal surfaces. Freon 113 (trichlorotriflorethane) is dense, chemically stable, relatively non-toxic, and leaves no residue. The vapor is easily removed from the air by activated charcoal. A high pressure (1,000 atm) jet of liquid Freon 113 is directed onto the surface to be cleaned. The Freon can be collected in a sump, filtered, and reused.

Physical removal of gross contamination should be followed by a wash/rinse process using cleaning solutions. One or more of the following methods utilize cleaning solutions.

Dissolving

Removal of surface contaminants can be accomplished by chemically dissolving them, although the solvent must be compatible with the equipment and protective clothing. Organic solvents include alcohols, ethers, ketones, aromatics, straight-chain alkanes, and common petroleum products. Halogenated solvents are generally incompatible with protective clothing and are toxic. Table 1 provides a general guide to the solubility of contaminant categories in four types of solvents.

Surfactants

Surfactants reduce adhesion forces between contaminants and the surface being cleaned and prevents reposition of the contaminants. Non-phosphate detergents dissolved in tap water is an acceptable surfactant solution.

Rinsing

Contaminants are removed and rinsing through dilution, physical attraction, and solubilization.

Disinfection/Sterilization

Disinfectants are a practical means of inactivating infectious agents. Unfortunately, standard sterilization methods are impractical for large equipment and personal protective clothing.

6.3 Field Sampling Equipment Cleaning Procedures

The following steps for equipment cleaning should be followed for general field sampling activities.

1. Physical removal (abrasive or non-abrasive methods).
2. Scrub with non-phosphate detergent plus tap water.
3. Tap water rinse.
4. 10% nitric acid (required during sampling for inorganics only).
5. Distilled/deionized water rinse.
6. Solvent rinse (required during sampling for organics only).
7. Total air dry (required during sampling for organics only).
8. Triple rinse with distilled/deionized water.

Table 1 lists solvent rinses which may be required for elimination of particular chemicals. After each solvent rinse, the equipment should be air-dried and triple-rinsed with distilled/deionized water.

Solvent rinses are not necessarily required when organics are not a contaminant of concern. Similarly, an acid rinse is not necessarily required if analysis does not include inorganics.

NOTE: Reference the appropriate analytical procedure for specific decontamination solutions required for adequate removal of the contaminants of concern.

Sampling equipment that requires the use of plastic or teflon tubing should be disassembled, cleaned, and the tubing replaced with clean tubing, if necessary, before commencement of sampling or between sampling locations.

Table 1 Decontamination Solvents

Solvent	Soluble Contaminants
Water	Low-chain compounds Salts Some organic acids and other polar compounds
Dilute Bases For example: ■ detergent ■ soap	Acidic compounds Phenol Thiols Some nitro and sulfonic compounds
Organic Solvents: For example: ■ alcohols (methanol) ■ ethers ■ ketones ■ aromatics ■ straight-chain alkanes (e.g., hexane) ■ common petroleum products (e.g., fuel oil, kerosene)	Nonpolar compounds (e.g., some organic compounds)

WARNING: Some organic solvents can permeate and/or degrade the protective clothing.

7. Quality Assurance/Quality Control

QA/QC samples are intended to provide information concerning possible cross-contamination during collection, handling, preparation, and packing of samples from field locations for subsequent review and interpretation. A field blank (rinsate blank) provides an additional check on possible sources of contamination from ambient air and from sampling instruments used to collect and transfer samples into sample containers.

A field blank (rinsate blank) consists of a sample of analyte-free water passed through/over a precleaned/decontaminated sampling device and placed in a clean area to attempt to simulate a worst-case condition regarding ambient air contributions to sample contamination.

Field blanks should be collected at a rate of one per day per sample matrix even if samples are not shipped that day. The field blanks should return to the lab with the trip blanks originally sent to the field and be packed with their associated matrix.

The field blank places a mechanism of control on equipment decontamination, sample handling, storage, and shipment procedures. It is also indicative of ambient conditions and/or equipment conditions that may affect the quality of the samples.

Holding times for field blanks analyzed by CLP methods begin when the blank is received in the laboratory (as documented on the chain of parameters and associated analytical methods).

Holding times for samples and blanks analyzed by SW-846 or the 600 and 500 series begins at the time of sample collection.

8. Health and Safety

Decontamination can pose hazards under certain circumstances even though performed to protect health and safety. Hazardous substances may be incompatible with decontamination methods (i.e., the method may react with contaminants to produce heat, explosion, or toxic products). Decontamination methods may be incompatible with clothing or equipment (e.g., some solvents can permeate and/or degrade protective clothing). Also, a direct health hazard to workers can be posed from chemical decontamination solutions that may be hazardous if inhaled or may be flammable.

The decontamination solutions must be determined to be compatible before use. Any method that permeates, degrades, or damages personal protective equipment should not be used. If decontamination methods do pose a direct health hazard, measures should be taken to protect personnel or modified to eliminate the hazard.

All site-specific safety procedures should be followed for the cleaning operation. At a minimum, the following precautions should be taken:

1. Safety glasses with splash shields or goggles, neoprene gloves, and laboratory apron should be worn.

2. All solvent rinsing operations should be conducted under a fume hood or in open air.

3. No eating, smoking, drinking, chewing, or any hand-to-mouth contact is permitted.

9. References

Field Sampling Procedures Manual, New Jersey Department of Environmental Protection, 1988.

A Compendium of Superfund Field Operations Methods, EPA 540/p-87/001.

Engineering Support Branch Standard Operating Procedures and Quality Assurance Manual, USEPA Region IV, April 1, 1986.

Occupational Safety and Health Guidance Manual for Hazardous Waste Site Activities, NIOSH/OSHA/USCG/EPA, October 1985.