ecology and environment, inc.
Global Environmental Specialists
720 Third Avenue, Suite 1700
Seattle, Washington 98104
Tel: (206) 624-9537, Fax: (206) 621-9832

May 22, 2013

Jeffrey Fowlow, On-Scene Coordinator
United States Environmental Protection Agency, Region 10
1200 Sixth Avenue, ECL-116
Seattle, Washington 98102

Re: Site-Specific Sampling Plan for the Stubblefield Salvage Soil Removal, Walla Walla, Washington
Contract Number EP-S7-06-02, Technical Direction Document Number 13-03-0009

Dear Mr. Fowlow:

Enclosed please find the final Site-Specific Sampling Plan for the Stubblefield Salvage Soil Removal Action. If you have any questions regarding this submittal, please call Jake Moersen at (206) 624-9537 or me at (206) 920-1739.

Sincerely,

ECOLOGY AND ENVIRONMENT, INC.

Steven G. Hall
START-3 Project Leader

cc: Kathy Parker, EPA, Region 10 ERU QA Coordinator, Seattle, Washington
Jake Moersen, START-3 Project Manager, Seattle, Washington

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UNITED STATES ENVIRONMENTAL PROTECTION AGENCY
REGION 10 Emergency Response Unit
1200 Sixth Avenue, Suite 900
Seattle, Washington 98101-3140

Site Specific Sampling Plan

Project Name: Stubblefield Salvage Soil Removal Site ID: 10HD

Author: Jake Moersen Company: Ecology and Environment, Inc Date Completed: May 2, 2013

This Site Specific Sampling Plan (SSSP) is prepared and used in conjunction with the Quality Assurance Plan (QAP) for the Emergency Response Unit for collecting samples during this Removal Program project. The information contained herein is based on the information available at the time of preparation. As better information becomes available, this SSSP will be adjusted.

When inadequate time is available for preparing the SSSP in advance of the sampling event, a Field Sampling Form may be prepared on-site immediately prior to sampling. This full length version of the SSSP is written after the sampling event and the completed Field Sampling Form attached to it.

1. Approvals

Name, Title	Telephone, Email, Address	Signature
Jeffrey Fowlow **On-Scene Coordinator**	206-553-2751, fowlow.jeffrey@epa.gov USEPA, M/S: ECL-116 1200 Sixth Ave Suite 900 Seattle, WA 98101	Jeffrey Fowlow Digitally signed by Jeffrey Fowlow DN: cn=Jeffrey Fowlow, o=USEPA, ou=ECL/ERU, email=fowlow.jeffrey@epa.gov, c=US Date: 2013.07.24 12:25:38 -07'00'
Kathy Parker **ERU Quality Assurance Coordinator**	206-553-0062, parker.kathy@epa.gov USEPA, M/S: ECL-116 1200 Sixth Ave Suite 900 Seattle, WA 98101	Kathy Parker Kathy Parker cn=Kathy Parker, o=USEPA, ou=OSC, email=parker.kathy@epa.gov, c=US 2013.07.24 12:48:05 -07'00'

I. Project Management and Organization

2. Personnel and Roles involved in the project:

Name	Telephone, Email, Company, Address	Project Role	Data Recipient
Jeffrey Fowlow	206-553-2751, fowlow.jeffrey@epa.gov USEPA, M/S: ECL-116 1200 Sixth Ave Suite 900, Seattle, WA 98101	On Scene Coordinator	Yes
Jake Moersen	206-624-9537, jmoersen@ene.com Ecology and Environment, Inc. (E & E), 720 Third Ave Suite 1700, Seattle, WA 98104	Author of SSSP, Superfund Technical Assessment and Response Team (START) Project Manager	Yes
Kathy Parker	206-553 0062, parker.kathy@epa.gov USEPA, M/S: ECL-116 1200 Sixth Ave Suite 900 Seattle, WA 98101	Emergency Response Unit (ERU) Quality Assurance Coordinator	No
Mark Woodke	206-624-9537, mwoodke@ene.com E & E, 720 Third Ave Suite 1700 Seattle, WA 98104	START Quality Assurance Reviewer	No
TBD	TBD	Laboratory Contact	No

3. Physical Description and Site Contact Information:

Site Name	Stubblefield Salvage Soil Removal	
Site Location	980 NW Offner Road Walla Walla, Washington 99362 Latitude: 46.065044° N Longitude: 118.369051° W (See Figure 1)	
Property Size	11.3 acres (current salvage yard footprint, see Figure 2)	
Site Contact	Adena Hodgins	**Phone Number:** Not Available
Primary Land Uses Surrounding the Site	Municipal (Walla Walla Wastewater Treatment Plant [WWTP]), farmland, residential. Mill Creek and Myra Road are also in the vicinity of the site.	

4. The proposed schedule of project work follows:

Activity	Estimated Start Date	Estimated Completion Date	Comments
SSSP Review/Approval	04/11/2013	05/2/2013	
Mobilize to / Demobilize from Site	05/13/2013	07/30/2013	
Sample Collection	05/14/2013	07/15/2013	
Laboratory Sample Receipt	05/15/2013	07/18/2013	
Laboratory Analysis	05/15/2013	07/25/2013	
Data Validation	05/30/2013	08/25/2013	

5. Historical and Background Information

Describe briefly what you know about the site that is relevant to sampling and analysis for this investigation.

The Stubblefield Salvage Soil Removal site was a salvage/scrap yard for over 60 years until it ceased operation in 2010. Emory Stubblefield was the original owner/operator of the facility until his death in 2008, and the estate is currently represented by Adena Hodgins. The salvage yard was initially 40 acres in size but has been subdivided with parcels sold to the City of Walla Walla, the County of Walla Walla, and Myra Road Properties LLC, a real estate development site. The current property of 11.3 acres is located in the eastern section of the original site.

Myra Road is located to the west of the site, farmland is to the east and mixed-use residential and farmland is to the south. Mill Creek is located directly to the north and downgradient of the site, and flows from east to west (Figure 2).

EPA and START performed seven field sampling events at the site, including three times in 2009 (May, September, and October), and two times in 2010 (March and October), and one time each in June 2011 and April 2012.

The May 2009 site visit was a limited preliminary removal assessment to determine if sufficient contamination, or threat of contamination, existed to justify a removal action. This removal assessment established the presence of incorrectly labeled drums, open steel tanks, and other containers of hazardous substances including target analyte list (TAL) metals, pesticides, polychlorinated biphenyls (PCBs), semivolatile organic compounds (SVOCs) including a subset of carcinogenic compounds known as polycyclic aromatic hydrocarbons (PAHs), total petroleum hydrocarbons (TPHs), and friable chrysotile asbestos-containing material (ACM) including cement asbestos siding (CAB).

In September 2009, EPA conducted a removal assessment to perform soil, bulk, and subsurface sampling. The

removal assessment found contamination in surface and subsurface boreholes throughout the site at concentrations that exceeded the State of Washington Model Toxics Control Act (MTCA) Unrestricted Cleanup Levels and EPA Regional Screening Levels (RSLs) for residential properties. The removal assessment identified a source area that was heavily impacted by hydrocarbons, TAL metals, PCBs and SVOCs.

In October 2009, EPA performed a removal action to address contamination identified during the two previous removal assessments. This removal action resulted in the disposal of a number of 55-gallon drums and their contents, ACM from the side of the shop building and from a pile of debris found at the site, and surface soil with metals contamination. The primary source area identified during the removal assessment was not addressed because ongoing releases of hydraulic fluid were witnessed in the vicinity of the operational bailer machine. EPA determined that additional removal actions would not be conducted until the source area could be characterized in greater detail and the ongoing release of hydraulic fluid could be evaluated.

In March 2010, START submitted to EPA an Alternatives Evaluation report that outlined several potential removal action alternatives. During the course of preparing the Alternatives Evaluation, EPA determined that four monitoring wells should be installed to determine the impact from the source area on groundwater. The subsequent removal assessment included the installation and sampling of four monitoring wells.

In October 2010, EPA mobilized to the site to collect groundwater samples from the four previously installed monitoring wells as part of an on-going removal assessment.

In June 2011, EPA mobilized to the site to further characterize the horizontal and vertical extent of contamination in the main process area using direct push subsurface soil sampling technology. The removal assessment included the collection of groundwater samples from some of the soil sampling locations in addition to groundwater samples from the four monitoring wells.

In April 2012, EPA performed a removal action at the site to dispose of approximately 60 containers of hazardous substances and approximately 13 drums containing purge water and other investigation-derived waste (IDW).

The current mobilization will include the excavation of contaminated soil in the process area. A generalized approach to the excavation will include the removal of grossly contaminated soil, removal of soil with multiple contaminants (i.e. PCBs and TPHs), and removal of contaminated soil in the process area.

6. Conceptual Site Model

Example: Contaminant: Mercury
Transport Mechanism: vapor moving on air currents
Receptors: people living in the house

The site work addressed by this SSSP pertains to the collection of the following:

1. Groundwater samples from monitoring wells and subsequent analyses for PCBs, TAL metals, SVOCs, diesel- and oil-range (TPHs), and volatile organic compounds (VOCs);
2. Surface/subsurface soil samples during excavation of known contamination for PCBs, TAL metals, SVOCs, TPH, and/or toxicity characteristic leaching procedure (TCLP) metals;
3. Surface/subsurface soil samples during the excavation for field analyses for PCBs and/or SVOCs using immunoassay kits and lead and arsenic using a portable x-ray fluorescence (XRF) instrument.

Contaminants:
The site contaminants include metals (including lead and arsenic), PCBs, SVOCs (specifically PAHs including benzo(a)anthracene, benzo(a)pyrene, benzo(b)fluoranthene, benzo(k)fluoranthene, and indeno(1,2,3-cd)pyrene), VOCs, and diesel- and oil-range TPHs.

Transport Mechanisms:
Contaminants on-site may be transported by vertical migration through soil to groundwater, and by surface runoff to surface water (Mill Creek). Contaminated soil and vapors can be a direct contact hazard both on site and off site.

Receptors:
Potential receptors include recreational users or trespassers, residential users living near the site, site workers, terrestrial ecological receptors on site, and aquatic ecological receptors in Mill Creek.

Note: Particulates are not site contaminants; however, air monitoring for health and safety purposes will be conducted using DataRAM Particulate Air Monitors placed downwind and around the perimeter of the excavation

areas. Viper remote monitoring software will collect the DataRAM information. Sample numbers will not be assigned to the DataRAM results.

7. Decision Statement

Examples: 1) Determine whether surface contamination exceeds the established action level;
2) Determine appropriate disposal options for contaminated materials.

The decision(s) to be made from this investigation is/are to:

1. Conduct an excavation of contaminated soil in the area known as the Process Area that exceeds the established cleanup criteria.
2. Collect samples of surface and subsurface soil media for investigative and confirmation purposes and compare contaminant concentrations to established cleanup criteria.
3. Collect samples of groundwater media from the four previously-installed monitoring wells and/or groundwater exposed during the excavation of contaminated soil, and compare concentrations to state and/or federal screening criteria.
4. Determine the seasonal depth and direction of groundwater flow.
5. Determine if particulate air results exceed action levels (for health and safety purposes).

8. Action Level

State the analyte, concentration, and units for each selected action level. Describe the rationale for choosing each action level and its source (i.e. MTCA, PRG, ATSDR, etc.) Example: The action level for total mercury in soil is 6.7 mg/kg (from Regional Screening Level residential).

1. Soil (TPH): State of Washington MTCA Cleanup Levels for Unrestricted and Industrial Properties.
2. Soil (PCBs, TAL Metals, SVOCs): EPA Residential and/or Industrial RSLs.
3. Groundwater (TPH): State of Washington MTCA Cleanup Levels for Groundwater, Federal Maximum Contaminant Level (MCL) Drinking Water Standards.
4. Groundwater (PCBs, TAL Metals, SVOCs, VOCs): EPA Tapwater RSLs, Federal MCL Drinking Water Standards.
5. Particulate Air: 2.5 milligrams per cubic meter (for health and safety purposes).

Specific screening levels are presented in the attached data tables. If the reporting limit exceeds the screening level, the reporting limit will be used.

II. Data Acquisition and Measurement Objectives

9. Site Diagram and Sampling Areas

A Sampling Area is an area within in which a specific action will be performed.
Examples : 1) Each drum on the site is a Sampling Area;
2) Each section of sidewalk in front of the residence is a Sampling Area;
3) Each sampling grid section is a Sampling Area.

1. Each soil sample location is a decision area. The process area has been divided into the southern process area (SPA) and northern process area (NPA) for the purpose of this removal action (Figure 3).
2. Each groundwater sample location is a decision point. There are four previously-installed monitoring wells on the property.
3. Each DataRAM location is a decision area for health and safety purposes. These locations will move during the excavation activities depending on wind direction.

10. The Decision Rules

These can be written as logical If…, Then.. statements. Describe how the decisions will be made and how to address results falling within the error range of the action level. Examples: 1) In the Old Furnace Sampling Area, the soil in the area around the furnace structure will be excavated until sample analysis with XRF shows no mercury concentrations in surface soil above the lower limit of the error associated with the action level, 18.4 mg/kg. 2) If the concentrations of contaminants in a SA are less than the lower limit of the error associated with the action level, then the area may be characterized as not posing an unacceptable risk to human health or the environment and may be dismissed from additional RP activities. The area may be referred to other Federal, State or Local government agencies.

The following statement(s) describe the decision rules to apply to this investigation.

1. Contaminated soil in the SPA will generally be excavated to 8 feet below ground surface (bgs) or to the depth of groundwater, at the EPA task monitors discretion. The structural integrity of the shop building and/or retaining wall may affect the depth of excavation along the shop building and/or retaining wall, respectively. Soil samples will be collected along the north, west, and east sidewalls of the excavation. The extent of excavation will be determined by visual observation of stained soil, results of in-situ XRF analysis, immunoassay screening for PAHs and PCBs, screening with a photoionization detector

(PID)/flame ionization detector (FID) instrument, petroleum sheen testing, and/or analytical data from a commercial laboratory for PCBs, metals, TPHs, and/or SVOCs.

2. Contaminated soil in the NPA may be sampled and analyzed by field screening and/or laboratory methods. The removal will focus on areas with multiple contaminants (i.e. PCBs and TPHs) in the process area. The extent of excavation will be determined by visual observation of stained soil, results of in-situ XRF analysis, screening with a PID/FID, and/or analytical data from a commercial laboratory for PCBs, metals, TPHs, and/or SVOCs.
3. If groundwater has contaminants of concern for PCBs, metals, SVOCs, TPHs, and/or VOCs that exceed the established action levels, then the conceptual site model (CSM) will be revised accordingly.
4. If the direction of groundwater flow has changed, then the groundwater flow direction map will be revised accordingly.
5. Evaluate health and safety measures when particulate air levels exceed 2.5 milligrams per cubic meter.

11. Information Needed for the Decision Rule

What information needs to be collected to make the decisions – this includes non-sampling info as well: action levels, climate history, direction of water flow, etc. Examples: Current and future on-site and off-site land use; wind direction, humidity and ambient temperature; contaminant concentrations in surface soil.

The following inputs to the decision are necessary to interpret the analytical results:

1. Screening data (immunoassay and/or XRF, visual observations by technical personnel, readings from a PID/FID, petroleum sheen testing, etc.) will be collected to assist in determining which soil samples will be submitted for laboratory analysis.
2. Depth to groundwater and other geological and hydrogeological observations will be used to determine groundwater flow, direction, and other characteristics, which will be used to complete the groundwater CSM and provide context for evaluation of groundwater analytical results.
3. Future on- and off-site land use.
4. Contaminant concentrations.
5. Wind direction.

12. Sampling and Analysis

For each SA, describe:

1. sampling pattern (random, targeted, scheme for composite)
2. number of samples, how many to be collected from where, and why
3. sample type (grab, composite)
4. matrix (air, water, soil)
5. analytes and analytical methods
6. name and locations of off-site laboratories, if applicable.

Groundwater:

An estimated four targeted grab groundwater samples will be collected from the four previously installed monitoring wells and analyzed for SVOCs (EPA Method 8270), TAL Metals (EPA 6000/7000 series methods), PCBs (EPA Method 8082), TPHs (Ecology Method NWTPH-Dx), and VOCs (EPA Method 8260) at a commercial laboratory, to be determined.

Soil

An estimated 200 grab soil samples may be collected for PCBs and/or SVOCs using immunoassay technologies (EPA Methods 4020 and 4035) and metals including arsenic and lead (EPA Method 6200) for XRF field analysis; an estimated 64 targeted grab soil samples will be collected and analyzed for SVOCs (EPA Method 8270), TAL Metals (EPA 6000/7000 series methods), PCBs (EPA Method 8082), TPHs (Ecology Method NWTPH-Dx), and/or TCLP Metals (EPA Methods 1311 and 6000/7000 series) at a commercial laboratory, to be determined.

Particulate Air

The DataRAM will continuously collect targeted grab particulate air samples from several locations for health and safety purposes during the excavation activities following the manufacturer's instructions and the Quick Start Guide.

13. Applicability of Data (place an X in front of the data categories needed, explain with comments)

Do the decisions to be made from the data require that the analytical data be:
1) definitive data, 2) screening data (with definitive confirmation) or 3) screening data (without definitive confirmation)?

X A) Definitive data** is analytical data of sufficient quality for final decision-making. To produce definitive data on-site or off-site, the field or lab analysis will have passed full Quality Control (QC) requirements (continuing calibration checks, Method Detection Limit (MDL) study, field duplicate samples, field blank, matrix spikes, lab duplicate samples, and other method-specific QC such as surrogates) AND the analyst will have passed a Precision and Recovery (PAR) study AND the instrument will have a valid Performance Evaluation sample on file. This category of data is suitable for: **1) enforcement purposes, 2) determination of extent of contamination, 3) disposal, 4) RP verification or 5) cleanup confirmation.

Comments: All samples submitted to the off-site analytical laboratory will produce definitive data.

*X **B) Screening data with definitive confirmation** is analytical data that may be used **to support preliminary or intermediate decision-making** until confirmed by definitive data. However, even after confirmation, this data is often not as precise as definitive data. To produce this category of data, the analyst will have passed a PAR study to determine analytical error AND 10% of the samples are split and analyzed by a method that produced definitive data with a minimum of three samples above the action level and three samples below it.*

Comments: Screening data obtained with field instruments (metals by XRF and PCBs and SVOCs by immunoassay) will be collected to assist with determination of contaminated areas and determination of which samples to submit for fixed laboratory analysis.

***C) Screening data** is analytical data which has not been confirmed by definitive data. The QC requirements are limited to an MDL study and continuing calibration checks. This data can be used for making decisions: **1) in emergencies, 2) for health and safety screening, 3) to supplement other analytical data, 4) to determine where to collect samples, 5) for waste profiling, and 6) for preliminary identification of pollutants**. This data is not of sufficient quality for final decision-making.*

Comments: DataRAM particulate air samples will be collected for health and safety purposes and data obtained using the PID/FID instrument will be considered screening data.

14. Special Sampling or Analysis Directions

Describe any special directions for the planned sampling and analysis such as additional quality controls or sample preparation issues. Examples: 1) XRF and Lumex for sediment will be calibrated before each day of use and checked with a second source standard. 2) A field blank will be analyzed with each calibration to confirm the concentration of non-detection. 3) A Method Detection Limit determination will be performed prior to the start of analysis so that the lower quantitation limit can be determined. 4) If particle size is too large for accurate analyses, the samples will be ground prior to analysis. If the sample contains too much moisture for accurate analyses, the sample will be decanted and air dried prior to analysis.

1. The XRF instrument will require a method detection limit (MDL) study prior to use, and will also require daily MDL and precision and recovery (PAR) studies prior to use as a field screening tool.
2. The START operations guide for the XRF will be followed to ensure effective operation of the instrument.
3. Matrix Spike (MS)/MS Duplicate (MSD) samples for the groundwater matrix will require the collection of additional sample containers.
4. Samples collected from the excavation may be analyzed using an expedited turnaround time.
5. Monitoring wells will be sampled in accordance with the E & E groundwater sampling standard operating procedure (SOP) including the use of low flow sampling pumps and screening of water quality parameters (temperature, pH, salinity and conductivity).
6. Soil samples will be collected in accordance with the E & E subsurface soil sampling SOP.

15. Method Requirements

[Describe the restrictions to be considered in choosing an analytical method due to the need to meet specific regulations, policies, ARARs, and other analytical needs. Examples: 1) Methods must meet USEPA Drinking Water Program requirements. 2) Methods must achieve lower quantitation limits of less than 1/10 the action levels.3) Methods must be performed exactly as written without modification by the analytical laboratory.]

Fixed laboratory methods must achieve quantitation limits equal to or lower than the action levels.

16. Sample Collection Information

[Describe any activities that will be performed related to sample collection]

The applicable sample collection Standard Operating Procedures (SOPs) or methods and other guidelines will be followed and include:

1. Field Activity Logbook SOP.
2. Sample Packaging and Shipping SOP.
3. Sampling Equipment Decontamination SOP.
4. XRF Instrument SOP
5. Groundwater Sampling SOP.
6. Subsurface Soil Sampling SOP.
7. SW-846 Method 6200 (Field Portable XRF).
8. SW-846 Methods 4020 and 4035 (Immunoassay).
9. DataRAM Quick Start Guide.
10. Sheen Test. The procedure for conducting the petroleum sheen test will consist of collecting approximately 50 grams of representative soil at the selected locations within a container and applying water until the soil is saturated and water collects around it. Visual classification of the representative soils will be recorded according to the magnitude of oil sheen observed, as described below:
    1) None (no sheen visually detected);
    2) Sheen (oil film present, but does not display rainbow); and
    3) Rainbow (definite oil sheen, film, or product that displays rainbow).

    A passing test will be defined as soil that does not exhibit a rainbow sheen.

17. Optimization of Sampling Plan (Maximizing Data Quality While Minimizing Time and Cost)

[Describe what choices were made to reduce cost of sampling while meeting the needed level of data quality. Example: The XRF will be used in situ whenever possible to achieve accurate results. Reproducibility and accuracy of in situ XRF analyses will be checked by collecting, air drying, analyzing and comparing five in situ samples at the start of sampling. Where interferences are suspected, steps will be taken to eliminate the interferences by mechanisms such as drying, grinding or sieving the samples or analyzing them using the Lumex with soil attachment.]

The XRF and immunoassay methods will be used in the field to provide guidance and assist in determining which soil samples will be submitted to the commercial laboratory.

The format for sample number identification is summarized in Table 1. Sample collection and analysis information is summarized in Table 2.

Table 1
SAMPLE CODING

Project Name Stubblefield Salvage Soil Removal **Site ID:**10HD

SAMPLE NUMBER [(1)]		
Digits	**Description**	**Code (Example)**
1,2,3,4	Year and Month Code	YYMM (1305)
5,6,7,8	Consecutive Sample Number (grouped by SA as appropriate)	0001 – First sample of SA

SAMPLE NAME / LOCATION ID [(2)] ***(Optional)***		
1,2	Sampling Area	MW – Monitoring Well NP – Northern Processing Area SP – Southern Processing Area
3,4	Consecutive Sample Number	01 – First sample of Sampling Area
5,6	Matrix Code	GW – Groundwater RB – Rinsate Blank SB – Subsurface Soil SS – Surface Soil TB – Trip Blank
7,8,9,10	Depth (Optional)	00_04 (Subsurface interval, in feet)

Notes:
(1) The Sample Number is a unique, 8-digit number assigned to each sample.
(2) The Sample Name or Location ID is an optional identifier that can be used to further describe each sample or sample location.

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Table 2. Sampling and Analysis

Data Quality	Sampling Area	Matrix	Sampling Pattern	Sample Type	Data Quality	Estimated Number of Field Samples	Analyte or Parameter	EPA Method Number	Action Level	Method Quant. Limit	#/type of Sample Containers per Sample	Preservative	Hold Time	Field QC
Field Screening (XRF)	Source Area	Soil	Targeted	Grab	Screening	200	TAL Metals	6200	See Tables	10 – 100 mg/kg	1x8-ounce ziplocking bag	None	6 months (28 days for mercury)	10 Field Duplicates
Field Screening (DataRam)	Source Area	Air	Targeted	Grab	Screening	Continuous	Total Particulates	N/A	2.5 mg/cm^3	0.0001 ug/m^3	N/A	None	N/A	N/A
Field Screening (Immunoassay)	Source Area	Soil	Targeted	Grab	Screening	200	PCBs SVOCs	4020 4035	See Tables	0.5 mg/kg 1 mg/kg	1x2-ounce glass jar	None	14 days/40 days	10 Field Duplicates
Lab Analysis	Source Area	Soil	Targeted	Grab	Definitive	49	TAL Metals	6000/ 7000 Series	See Tables	0.1 – 500 mg/kg	1x8 ounce glass jar	None	6 months (28 days for mercury)	3 Field Duplicates
Lab Analysis	Source Area	Soil	Targeted	Grab	Definitive	64	PCBs	8082	See Tables	20 ug/kg	1x8 ounce glass jar	None	14 days/40 days	4 Field Duplicates
Lab Analysis	Source Area	Soil	Targeted	Grab	Definitive	64	SVOCs	8270	See Tables	50 – 200 ug/kg	1x8 ounce glass jar	None	14 days/40 days	4 Field Duplicates
Lab Analysis	Source Area	Soil	Targeted	Grab	Definitive	64	TPHs	NWTPH-Dx	See Tables	25 - 100 mg/kg	1x8 ounce glass jar	None	14 days/40 days	4 Field Duplicates
Lab Analysis	Source Area	Soil	Targeted	Grab	Definitive	18	TCLP Metals	1311, 6000/ 7000 Series	See Tables	0.2 – 100 mg/L	1x8 ounce glass jar	None	6 months (28 days for mercury)	1 Field Duplicate
Lab Analysis	Monitoring Wells	Water	Targeted	Grab	Definitive	13	TAL Metals	6000 and 7000 Series	See Tables	0.2 – 5,000 ug/L	1x1-liter polyethylene	$HN0_3$ to pH < 2	6 months (28 days for mercury)	1 Field Duplicate 3 Rinsate Blanks
Lab Analysis	Monitoring Wells	Water	Targeted	Grab	Definitive	13	VOCs	8260	See Tables	5 – 10 ug/L	3x40-mL VOA vials	HCl to pH < 2	14 days	1 Field Duplicate 3 Rinsate Blanks
Lab Analysis	Monitoring Wells	Water	Targeted	Grab	Definitive	13	TPHs	NWTPH-Dx	See Tables	250 ug/L	2x1-liter amber glass	None	7 days/40 days	1 Field Duplicate 3 Rinsate Blanks
Lab Analysis	Monitoring Wells	Water	Targeted	Grab	Definitive	13	PCBs	8082	See Tables	1 ug/L	2x1-liter amber glass	None	7 days/40 days	1 Field Duplicate 3 Rinsate Blanks
Lab Analysis	Monitoring Wells	Water	Targeted	Grab	Definitive	13	SVOCs	8270	See Tables	5 ug/L	2x1-liter amber glass	None	7 days/40 days	1 Field Duplicate 3 Rinsate Blanks

Note:
For matrix spike and/or duplicate samples, no extra volume is required for air (unless co-located samples are collected), oil, product, or soil samples except soil VOC or NWTPH-Gx samples (triple volume). Triple volume is also required for organic water samples (double volume for inorganic).

Table 3. Common Sample Handling Information

Analysis Type	Sub Analysis	Matrix	Analytical Method	Container Type	Minimum Volume	Preservative	Temperature/ Storage	Hold Time	Source
Metals	Metals Not including Mercury or Hexachrome. Includes TAL, PP, RCRA lists)	Solid	EPA 6000 / 7000 Series	Glass Jar	200 g	n/a	None	6 months	SW-846 ch. 3
		Aqueous	EPA 6000 / 7000 Series	PTFE or HDPE	600 mL	HNO_3 to pH < 2	Not listed	6 months	SW-846 ch. 3
	Mercury	Solid	EPA 7471B	Glass Jar	200 g	n/a	≤ 6° C	28 days	SW-846 ch. 3
		Aqueous	EPA 7470A	PTFE or HDPE	400 mL	HNO_3 to pH < 2	Not listed	28 days	SW-846 ch. 3
	Hexavalent Chromium, (Hexachrome, Cr+6)	Solid	Lab X method, EPA 7196A	Glass Jar	100 g	n/a	≤ 6° C	28 days to extraction	SW-846 ch. 3
		Aqueous	EPA 218.6 (Drinking Water)	PTFE or HDPE	400 mL	n/a	≤ 6° C	24 hours	SW-846 ch. 3
	XRF	Solid (in situ; on the ground surface)	6200	none	n/a	none	none	Analyze Immediately	n/a
		Solid (ex situ)	6200	plastic bag	200 g	none	none	6 months	n/a
VOCs	VOCs / BTEX	Solid	EPA 5035 / 8260B	*	*	*	*	2 days to lab / 14 days	SW-846 ch. 4
		Aqueous	EPA 8260B	Amber Vial with Septa Lid	2 x 40 mL	HCl to pH< 2	≤ 6° C (headspace free)	14 days	SW-846 ch. 4
SVOCs	SVOCs / PAHs	Solid	EPA 8270D	Glass Jar	8 ounces	n/a	≤ 6° C	14 days	SW-846 ch. 4
		Aqueous	EPA 8270D	Amber Glass	2 x 1 L	n/a	≤ 6° C	7 days	SW-846 ch. 4
PCBs and Dioxins/Furans	PCBs	Solid	EPA 8082	Glass Jar	8 ounces	n/a	≤ 6° C	none	SW-846 ch. 4
		Aqueous	EPA 8082	Amber Glass	2 x 1 L	n/a	≤ 6° C	none	SW-846 ch. 4
	Dioxins/Furans	Solid	EPA 8280 or 8290	Glass Jar	8 ounces	n/a	≤ 6° C	none	SW-846 ch. 4
		Aqueous	EPA 8280 or 8290	Amber Glass	2 x 1 L	n/a	≤ 6° C	none	SW-846 ch. 4
Pesticides and Herbicides	Chlorinated Pesticides	Solid	EPA 8081	Glass Jar	8 ounces	n/a	≤ 6° C	14 days	SW-846 ch. 4
		Aqueous	EPA 8081	Amber Glass	2 x 1 L	n/a	≤ 6° C	7 days	SW-846 ch. 4
	Chlorinated Herbicides	Solid	EPA 8151	Glass Jar	8 ounces	n/a	≤ 6° C	14 days	SW-846 ch. 4
		Aqueous	EPA 8151	Amber Glass	2 x 1 L	n/a	≤ 6° C	7 days	SW-846 ch. 4
NWTPH	Gasoline-Range Organics	Solid	TPHs/NWTPH-Gx	Amber Glass Jar with Septa Lid	4 ounces	n/a	≤ 6° C (headspace free)	14 days	Method
		Aqueous	TPHs/NWTPH-Gx	Amber Vial with Septa Lid	2 x 40 mL	pH < 2 with HCl	≤ 6° C (headspace free)	7 days unpreserved 14 days preserved	Method
	Diesel-Range Organics	Solid	3510, 3540/3550, 8000	Glass Jar	8 ounces	n/a	≤ 6° C	14 days	Method
		Aqueous	3510, 3540/3550, 8000	Glass Amber	2 x 1 L	pH < 2 with HCl	≤ 6° C	7 days unpreserved 14 days preserved	Method
Geotechnical	Particle Size Analysis	Solid	ASTM D-422	Glass Jar or Plastic Bag	2 x 8 ounce	none	n/a	n/a	Method
Miscellaneous	pH	Solid	EPA 9045	Glass Jar	8 ounces	n/a	n/a	Analyze Immediately	SW-846 ch. 3
		Aqueous	EPA 9040	PTFE	25 mL	n/a	n/a	Analyze Immediately	SW-846 ch. 3

Analysis Type	Sub Analysis	Matrix	Analytical Method	Container Type	Minimum Volume	Preservative	Temperature/ Storage	Hold Time	Source
		Aqueous	EPA 9040	PTFE	25 mL	n/a	n/a	Analyze Immediately	SW-846 ch. 3
	Total Organic Carbon (TOC)	Solid	SW-846 9060	Glass Jar	100 mL	n/a	≤ 6° C	28 days	SW-846
		Aqueous	EPA 415.1	PTFE or HDPE	200 mL	store in dark HCL or H_2SO_4 to pH <2	≤ 6° C	7 days unpreserved 28 days preserved	Method
	Cyanide	Solid	SW-846 9013	Glass Jar	5 g	n/a	≤ 6° C	14 days	SW-846 ch. 3
		Aqueous	SW-846 9010C	PTFE or HDPE	500 mL	NaOH to pH > 12	≤ 6° C	14 days	SW-846 ch. 3
	Conductivity	Aqueous	EPA 120.1	PTFE or HDPE	100 mL	n/a	n/a	Analyze Immediately	Method
	Hardness	Aqueous	EPA 130.1	PTFE or HDPE	1 x 1 L	HNO3 to pH<2	≤ 6° C	28 days	Method
	Total Suspended Solids	Aqueous	EPA 160.2	PTFE or HDPE	100 mL	n/a	≤ 6° C	7 days	Method
	Total Dissolved Solids	Aqueous	EPA 160.1	PTFE or HDPE	100 mL	n/a	≤ 6° C	7 days	Method
	Nitrate/nitrite	Aqueous	EPA 353.2	PTFE or HDPE	1 x 250 mL	H_2SO_4 to pH <2	≤ 6° C	28 days	Method
	Nitrate	Aqueous	SW-846 9210A	PTFE or HDPE	1,000 mL	n/a	≤ 6° C	28 days	SW-846 ch. 3
	Nitrite	Aqueous	SW-846 9216	PTFE or HDPE	25 mL	n/a	≤ 6° C	48 hours	SW-846 ch. 3, Method
	Fluoride	Aqueous	SW-846 9214	PTFE or HDPE	300 mL	n/a	≤ 6° C	28 days	SW-846 ch. 3
	Chloride	Aqueous	SW-846 9250	PTFE or HDPE	50 mL	n/a	≤ 6° C	28 days	SW-846 ch. 3
	Sulfate	Aqueous	SW-846 9035	PTFE or HDPE	50 mL	n/a	≤ 6° C	28 days	SW-846 ch. 3
	Sulfide	Solid	SW-846 9215	Glass Jar	1 x 4 ounces	Fill sample surface with 2N zinc acetate until moistened.	≤ 6° C (headspace free)	7 days	SW-846 ch. 3
		Aqueous	SW-846 9031	PTFE or HDPE	100 mL	4 drops 2N zinc acetate/100 mL sample; NaOH to pH>9.	≤ 6° C (headspace free)	7 days	SW-846 ch. 3

Key:

*	= See individual methods. We typically collect 3xEnCore-type samplers and 1x40 mL VOA vial per sample, keep at ≤ 6°C with no chemical preservative, and they must be at the lab within 48 hours of collection.				
C	= Celsius	HNO_3	= nitric acid	SVOCs	= semivolatile organic compounds
Cr	= chromium	L	= liter	SW-846	= EPA Test Methods for Evaluating Solid Waste, Physical/Chemical Methods
EPA	= Environmental Protection Agency	mL	= milliliter	TAL	= Target Analyte List
g	=grams	n/a	= not applicable	TPH	= total petroleum hydrocarbons
H2SO4	= sulfuric acid	NaOH	= sodium hydroxide	VOA	= Volatile Organic Analysis
HCL	= hydrochloric acid	PCBs	= polychlorinated biphenyls	VOCs	= Volatile Organic Compounds
HDPE	= high-density polyethylene	PTFE	= polytetrafluoroethylene		
Hg	= mercury	RCRA	= Resource Conservation and Recovery Act		

III Assessment and Response

A Sample Plan Alteration Form (SPAF) will be used to describe project discrepancies (if any) that occur between planned project activities listed in the final SSSP and actual project work. The completed SPAF will be approved by the On-Scene Coordinator (OSC) and Quality Assurance Coordinator (QAC) and appended to the original SSSP.

A Field Sampling Form (FSF) may be used to capture the sampling and analysis scheme for emergency responses in the field and then the FSF pages inserted into the appropriate areas of the final SSSP.

Corrective actions will be assessed by the sampling team and others involved in the sampling and a corrective action report describing the problem, solution and recommendations will be forwarded to the OSC and the ERU QAC.

IV Data Validation and Usability

The sample collection data will be entered into Scribe and Scribe will be used to print lab Chains of Custody. Results of field and lab analyses will be entered into Scribe as they are received and uploaded to Scibe.net when the sampling and analysis has been completed.

18. Data Validation or Verification will be performed by:

ERU's general recommendation on validation is that a minimum of CLP-equivalent stage IIA verification and validation be performed for every SSSP involving laboratory analyses. However, stage IIB is preferred if the lab can provide it. Dioxins should be validated at CLP-equivalent stage 4.

	Data Verification and Validation Stages						
Performed by:	I	IIA	IIB	III	IV	Verification	Other:
E and E QA Reviewer			100%		10%	100% (field data)	
TechLaw QA Reviewer							
EPA Region 10 QA Office							
MEL staff							
Other:							

R:\3 START 3\Stubblefield\October 2009\Figures\Stubblefield Salvage Yard Site Fig1.mxd 1/14/2009

Site Location

Legend

Site Boundary

0 0.125 0.25 0.5 0.75 1 Miles

Figure 1: Site Location

Stubblefield Salvage Yard Site

Walla Walla, Washington

April 2012 Site Visit

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Seattle, Washington

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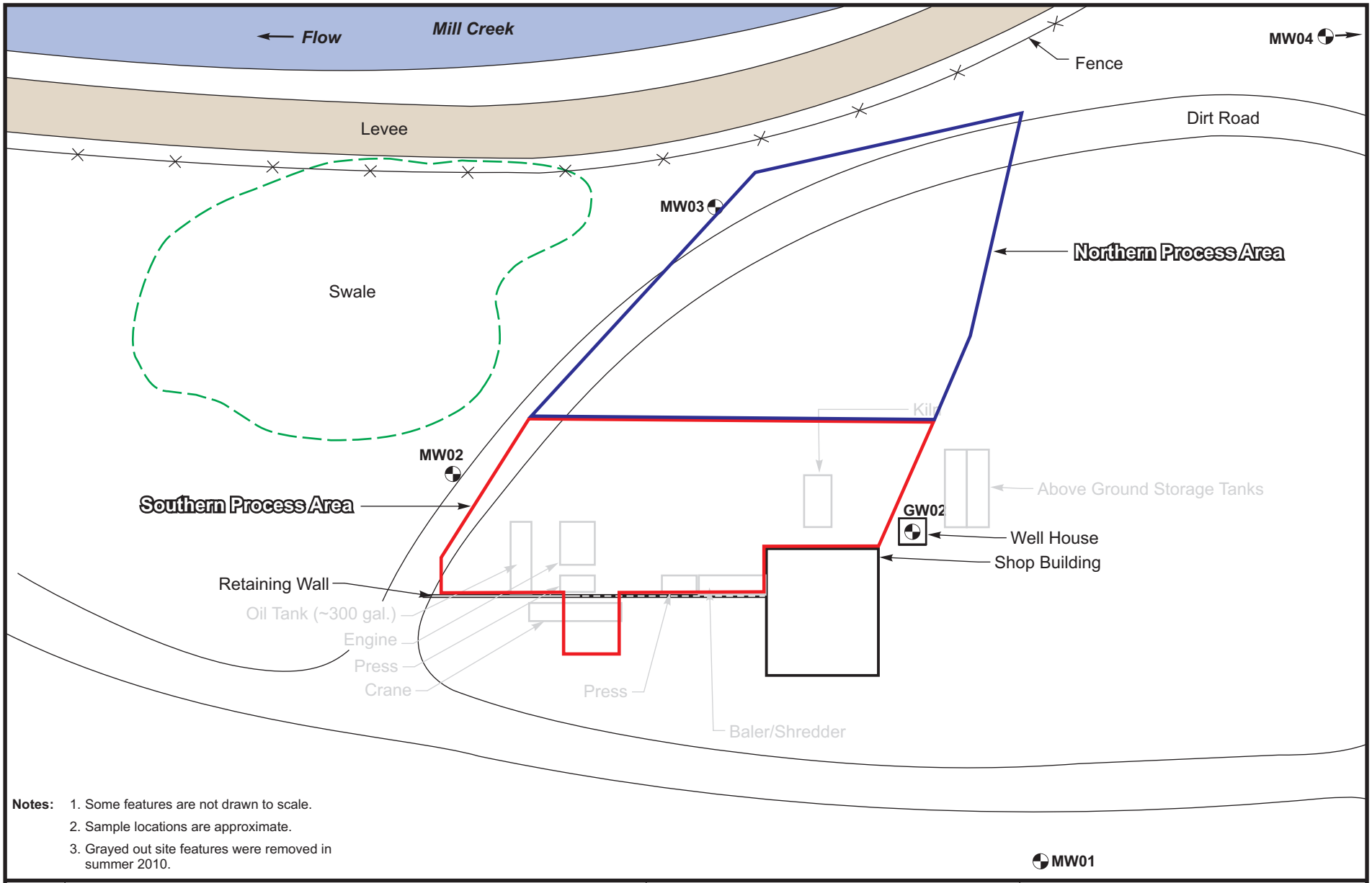
0 200 400
Approximate Scale in Feet

Figure 2: Site Location Map
Stubblefield Salvage Yard Site
Walla Walla, Washington

ecology and environment, inc.
Global Specialists in the Environment
Seattle, Washington



Notes:
1. Some features are not drawn to scale.
2. Sample locations are approximate.
3. Grayed out site features were removed in summer 2010.

Legend

Monitoring Well or Domestic Well

0 36 72

Approximate Scale in Feet

Figure 3: Proposed Excavation Areas
Stubblefield Salvage Yard Site
Walla Walla, Washington

ecology and environment, inc.
Global Specialists in the Environment
Seattle, Washington

Analyte Group	Analyte Name	CAS Number	PRG	Published Date	Matrix	Units	Tapwater	Secondary MCL	Primary MCL
Metals	Aluminum	7429-90-5	EPA Regional Screening Levels - Groundwater	11/1/2012	GW	ug/L	16000	50	
Metals	Antimony (metallic)	7440-36-0	EPA Regional Screening Levels - Groundwater	11/1/2012	GW	ug/L	6		6
Metals	Arsenic, Inorganic	7440-38-2	EPA Regional Screening Levels - Groundwater	11/1/2012	GW	ug/L	0.045		10
Metals	Barium	7440-39-3	EPA Regional Screening Levels - Groundwater	11/1/2012	GW	ug/L	2900		2000
Metals	Beryllium and compounds	7440-41-7	EPA Regional Screening Levels - Groundwater	11/1/2012	GW	ug/L	16		4
Metals	Cadmium (Water)	7440-43-9	EPA Regional Screening Levels - Groundwater	11/1/2012	GW	ug/L	6.9		5
Metals	Chromium, Total (as Cr+3)	7440-47-3	EPA Regional Screening Levels - Groundwater	11/1/2012	GW	ug/L	16000		100
Metals	Cobalt	7440-48-4	EPA Regional Screening Levels - Groundwater	11/1/2012	GW	ug/L	4.7		
Metals	Copper	7440-50-8	EPA Regional Screening Levels - Groundwater	11/1/2012	GW	ug/L	620	1000	1300
Metals	Iron	7439-89-6	EPA Regional Screening Levels - Groundwater	11/1/2012	GW	ug/L	11000	300	
Metals	Lead and Compounds	7439-92-1	EPA Regional Screening Levels - Groundwater	11/1/2012	GW	ug/L			15
Metals	Manganese (Water)	7439-96-5	EPA Regional Screening Levels - Groundwater	11/1/2012	GW	ug/L	320	50	
Metals	Mercury (elemental)	7439-97-6	EPA Regional Screening Levels - Groundwater	11/1/2012	GW	ug/L	0.63		2
Metals	Nickel Soluble Salts	7440-02-0	EPA Regional Screening Levels - Groundwater	11/1/2012	GW	ug/L	300		
Metals	Selenium	7782-49-2	EPA Regional Screening Levels - Groundwater	11/1/2012	GW	ug/L	78		50
Metals	Silver	7440-22-4	EPA Regional Screening Levels - Groundwater	11/1/2012	GW	ug/L	71	100	
Metals	Thallium (Soluble Salts)	7440-28-0	EPA Regional Screening Levels - Groundwater	11/1/2012	GW	ug/L	0.16		2
Metals	Vanadium, Metallic	7440-62-2	EPA Regional Screening Levels - Groundwater	11/1/2012	GW	ug/L	78		
Metals	Zinc and Compounds	7440-66-6	EPA Regional Screening Levels - Groundwater	11/1/2012	GW	ug/L	4700	5000	
Metals	Chromium(III), Insoluble Salts	16065-83-1	EPA Regional Screening Levels - Groundwater	11/1/2012	GW	ug/L	16000		100
Metals	Mercuric Chloride (and other Mercury salts)	7487-94-7	EPA Regional Screening Levels - Groundwater	11/1/2012	GW	ug/L	4.3		2
PCBs	Aroclor 1016	12674-11-2	EPA Regional Screening Levels - Groundwater	11/1/2012	GW	ug/L	0.96		
PCBs	Aroclor 1221	11104-28-2	EPA Regional Screening Levels - Groundwater	11/1/2012	GW	ug/L	0.04		
PCBs	Aroclor 1232	11141-16-5	EPA Regional Screening Levels - Groundwater	11/1/2012	GW	ug/L	0.04		
PCBs	Aroclor 1242	53469-21-9	EPA Regional Screening Levels - Groundwater	11/1/2012	GW	ug/L	0.034		
PCBs	Aroclor 1248	12672-29-6	EPA Regional Screening Levels - Groundwater	11/1/2012	GW	ug/L	0.034		
PCBs	Aroclor 1254	11097-69-1	EPA Regional Screening Levels - Groundwater	11/1/2012	GW	ug/L	0.034		
PCBs	Aroclor 1260	11096-82-5	EPA Regional Screening Levels - Groundwater	11/1/2012	GW	ug/L	0.034		
SVOCs	Acetophenone	98-86-2	EPA Regional Screening Levels - Groundwater	11/1/2012	GW	ug/L	1500		
SVOCs	Aniline	62-53-3	EPA Regional Screening Levels - Groundwater	11/1/2012	GW	ug/L	12		
SVOCs	Atrazine	1912-24-9	EPA Regional Screening Levels - Groundwater	11/1/2012	GW	ug/L	0.26		3
SVOCs	Azobenzene	103-33-3	EPA Regional Screening Levels - Groundwater	11/1/2012	GW	ug/L	0.1		
SVOCs	Benzaldehyde	100-52-7	EPA Regional Screening Levels - Groundwater	11/1/2012	GW	ug/L	1500		
SVOCs	Benzidine	92-87-5	EPA Regional Screening Levels - Groundwater	11/1/2012	GW	ug/L	0.000092		
SVOCs	Benzoic Acid	65-85-0	EPA Regional Screening Levels - Groundwater	11/1/2012	GW	ug/L	58000		
SVOCs	Benzyl Alcohol	100-51-6	EPA Regional Screening Levels - Groundwater	11/1/2012	GW	ug/L	1500		
SVOCs	Bis(2-chloro-1-methylethyl) ether	108-60-1	EPA Regional Screening Levels - Groundwater	11/1/2012	GW	ug/L	0.31		
SVOCs	Bis(2-chloroethoxy)methane	111-91-1	EPA Regional Screening Levels - Groundwater	11/1/2012	GW	ug/L	46		
SVOCs	Bis(2-chloroethyl)ether	111-44-4	EPA Regional Screening Levels - Groundwater	11/1/2012	GW	ug/L	0.012		
SVOCs	Bis(2-ethylhexyl)phthalate	117-81-7	EPA Regional Screening Levels - Groundwater	11/1/2012	GW	ug/L	4.8		6
SVOCs	Bis(chloromethyl)ether	542-88-1	EPA Regional Screening Levels - Groundwater	11/1/2012	GW	ug/L	0.000062		
SVOCs	Bisphenol A	80-05-7	EPA Regional Screening Levels - Groundwater	11/1/2012	GW	ug/L	580		
SVOCs	Butyl Benzyl Phthlate	85-68-7	EPA Regional Screening Levels - Groundwater	11/1/2012	GW	ug/L	14		
SVOCs	Caprolactam	105-60-2	EPA Regional Screening Levels - Groundwater	11/1/2012	GW	ug/L	7700		
SVOCs	Chloroaniline, p-	106-47-8	EPA Regional Screening Levels - Groundwater	11/1/2012	GW	ug/L	0.32		
SVOCs	Chloronaphthalene, Beta-	91-58-7	EPA Regional Screening Levels - Groundwater	11/1/2012	GW	ug/L	550		
SVOCs	Chlorophenol, 2-	95-57-8	EPA Regional Screening Levels - Groundwater	11/1/2012	GW	ug/L	71		
SVOCs	Cresol, m-	108-39-4	EPA Regional Screening Levels - Groundwater	11/1/2012	GW	ug/L	720		
SVOCs	Cresol, o-	95-48-7	EPA Regional Screening Levels - Groundwater	11/1/2012	GW	ug/L	720		
SVOCs	Cresol, p-	106-44-5	EPA Regional Screening Levels - Groundwater	11/1/2012	GW	ug/L	1400		
SVOCs	Cresol, p-chloro-m-	59-50-7	EPA Regional Screening Levels - Groundwater	11/1/2012	GW	ug/L	1100		
SVOCs	Cresols	1319-77-3	EPA Regional Screening Levels - Groundwater	11/1/2012	GW	ug/L	1400		
SVOCs	Dibutyl Phthalate	84-74-2	EPA Regional Screening Levels - Groundwater	11/1/2012	GW	ug/L	670		
SVOCs	Dichlorobenzidine, 3,3'-	91-94-1	EPA Regional Screening Levels - Groundwater	11/1/2012	GW	ug/L	0.11		
SVOCs	Dichlorophenol, 2,4-	120-83-2	EPA Regional Screening Levels - Groundwater	11/1/2012	GW	ug/L	35		
SVOCs	Diethyl Phthalate	84-66-2	EPA Regional Screening Levels - Groundwater	11/1/2012	GW	ug/L	11000		
SVOCs	Dimethylphenol, 2,4-	105-67-9	EPA Regional Screening Levels - Groundwater	11/1/2012	GW	ug/L	270		
SVOCs	Dinitro-o-cresol, 4,6-	534-52-1	EPA Regional Screening Levels - Groundwater	11/1/2012	GW	ug/L	17		
SVOCs	Dinitrobenzene, 1,2-	528-29-0	EPA Regional Screening Levels - Groundwater	11/1/2012	GW	ug/L	1.5		
SVOCs	Dinitrobenzene, 1,3-	99-65-0	EPA Regional Screening Levels - Groundwater	11/1/2012	GW	ug/L	1.5		
SVOCs	Dinitrobenzene, 1,4-	100-25-4	EPA Regional Screening Levels - Groundwater	11/1/2012	GW	ug/L	1.5		
SVOCs	Dinitrophenol, 2,4-	51-28-5	EPA Regional Screening Levels - Groundwater	11/1/2012	GW	ug/L	30		
SVOCs	Dinitrotoluene Mixture, 2,4/2,6-	25321-14-6	EPA Regional Screening Levels - Groundwater	11/1/2012	GW	ug/L	0.092		
SVOCs	Dinitrotoluene, 2,4-	121-14-2	EPA Regional Screening Levels - Groundwater	11/1/2012	GW	ug/L	0.2		
SVOCs	Dinitrotoluene, 2,6-	606-20-2	EPA Regional Screening Levels - Groundwater	11/1/2012	GW	ug/L	15		
SVOCs	Dioxane, 1,4-	123-91-1	EPA Regional Screening Levels - Groundwater	11/1/2012	GW	ug/L	0.67		
SVOCs	Diphenylhydrazine, 1,2-	122-66-7	EPA Regional Screening Levels - Groundwater	11/1/2012	GW	ug/L	0.067		
SVOCs	Dibenzofuran	132-64-9	EPA Regional Screening Levels - Groundwater	11/1/2012	GW	ug/L	5.8		
SVOCs	Hexachlorobenzene	118-74-1	EPA Regional Screening Levels - Groundwater	11/1/2012	GW	ug/L	0.042		1
SVOCs	Hexachlorocyclopentadiene	77-47-4	EPA Regional Screening Levels - Groundwater	11/1/2012	GW	ug/L	22		50
SVOCs	Hexachloroethane	67-72-1	EPA Regional Screening Levels - Groundwater	11/1/2012	GW	ug/L	0.79		
SVOCs	Isophorone	78-59-1	EPA Regional Screening Levels - Groundwater	11/1/2012	GW	ug/L	67		
SVOCs	Nitroaniline, 2-	88-74-4	EPA Regional Screening Levels - Groundwater	11/1/2012	GW	ug/L	150		
SVOCs	Nitroaniline, 4-	100-01-6	EPA Regional Screening Levels - Groundwater	11/1/2012	GW	ug/L	3.3		
SVOCs	Nitrobenzene	98-95-3	EPA Regional Screening Levels - Groundwater	11/1/2012	GW	ug/L	0.12		
SVOCs	Nitroso-di-N-propylamine, N-	621-64-7	EPA Regional Screening Levels - Groundwater	11/1/2012	GW	ug/L	0.0093		
SVOCs	Nitrosodiphenylamine, N-	86-30-6	EPA Regional Screening Levels - Groundwater	11/1/2012	GW	ug/L	10		
SVOCs	Pentachlorobenzene	608-93-5	EPA Regional Screening Levels - Groundwater	11/1/2012	GW	ug/L	2.3		
SVOCs	Pentachlorophenol	87-86-5	EPA Regional Screening Levels - Groundwater	11/1/2012	GW	ug/L	0.035		1
SVOCs	Phenol	108-95-2	EPA Regional Screening Levels - Groundwater	11/1/2012	GW	ug/L	4500		
SVOCs	Acenaphthene	83-32-9	EPA Regional Screening Levels - Groundwater	11/1/2012	GW	ug/L	400		
SVOCs	Anthracene	120-12-7	EPA Regional Screening Levels - Groundwater	11/1/2012	GW	ug/L	1300		
SVOCs	Benz[a]anthracene	56-55-3	EPA Regional Screening Levels - Groundwater	11/1/2012	GW	ug/L	0.029		
SVOCs	Benzo(j)fluoranthene	205-82-3	EPA Regional Screening Levels - Groundwater	11/1/2012	GW	ug/L	0.056		
SVOCs	Benzo[a]pyrene	50-32-8	EPA Regional Screening Levels - Groundwater	11/1/2012	GW	ug/L	0.0029		0.2
SVOCs	Benzo[b]fluoranthene	205-99-2	EPA Regional Screening Levels - Groundwater	11/1/2012	GW	ug/L	0.029		
SVOCs	Benzo[k]fluoranthene	207-08-9	EPA Regional Screening Levels - Groundwater	11/1/2012	GW	ug/L	0.29		
SVOCs	Chrysene	218-01-9	EPA Regional Screening Levels - Groundwater	11/1/2012	GW	ug/L	2.9		
SVOCs	Dibenz[a,h]anthracene	53-70-3	EPA Regional Screening Levels - Groundwater	11/1/2012	GW	ug/L	0.0029		
SVOCs	Dibenzo(a,e)pyrene	192-65-4	EPA Regional Screening Levels - Groundwater	11/1/2012	GW	ug/L	0.0056		
SVOCs	Fluoranthene	206-44-0	EPA Regional Screening Levels - Groundwater	11/1/2012	GW	ug/L	630		
SVOCs	Fluorene	86-73-7	EPA Regional Screening Levels - Groundwater	11/1/2012	GW	ug/L	220		
SVOCs	Indeno[1,2,3-cd]pyrene	193-39-5	EPA Regional Screening Levels - Groundwater	11/1/2012	GW	ug/L	0.029		
SVOCs	Methylnaphthalene, 1-	90-12-0	EPA Regional Screening Levels - Groundwater	11/1/2012	GW	ug/L	0.97		
SVOCs	Methylnaphthalene, 2-	91-57-6	EPA Regional Screening Levels - Groundwater	11/1/2012	GW	ug/L	27		
SVOCs	Naphthalene	91-20-3	EPA Regional Screening Levels - Groundwater	11/1/2012	GW	ug/L	0.14		
SVOCs	Pyrene	129-00-0	EPA Regional Screening Levels - Groundwater	11/1/2012	GW	ug/L	87		
SVOCs	Pyridine	110-86-1	EPA Regional Screening Levels - Groundwater	11/1/2012	GW	ug/L	15		
SVOCs	Trichlorophenol, 2,4,5-	95-95-4	EPA Regional Screening Levels - Groundwater	11/1/2012	GW	ug/L	890		
SVOCs	Trichlorophenol, 2,4,6-	88-06-2	EPA Regional Screening Levels - Groundwater	11/1/2012	GW	ug/L	3.5		
VOCs	Acetone	67-64-1	EPA Regional Screening Levels - Groundwater	11/1/2012	GW	ug/L	12000		
VOCs	Acetonitrile	75-05-8	EPA Regional Screening Levels - Groundwater	11/1/2012	GW	ug/L	130		
VOCs	Acrolein	107-02-8	EPA Regional Screening Levels - Groundwater	11/1/2012	GW	ug/L	0.041		
VOCs	Acrylonitrile	107-13-1	EPA Regional Screening Levels - Groundwater	11/1/2012	GW	ug/L	0.045		
VOCs	Benzene	71-43-2	EPA Regional Screening Levels - Groundwater	11/1/2012	GW	ug/L	0.39		5
VOCs	Bromobenzene	108-86-1	EPA Regional Screening Levels - Groundwater	11/1/2012	GW	ug/L	54		
VOCs	Bromodichloromethane	75-27-4	EPA Regional Screening Levels - Groundwater	11/1/2012	GW	ug/L	7.9		80
VOCs	Bromoform	75-25-2	EPA Regional Screening Levels - Groundwater	11/1/2012	GW	ug/L	0.12		80
VOCs	Bromomethane	74-83-9	EPA Regional Screening Levels - Groundwater	11/1/2012	GW	ug/L	7		
VOCs	Carbon Disulfide	75-15-0	EPA Regional Screening Levels - Groundwater	11/1/2012	GW	ug/L	720		
VOCs	Carbon Tetrachloride	56-23-5	EPA Regional Screening Levels - Groundwater	11/1/2012	GW	ug/L	0.39		5
VOCs	Chlorobenzene	108-90-7	EPA Regional Screening Levels - Groundwater	11/1/2012	GW	ug/L	72		100
VOCs	Chloroform	67-66-3	EPA Regional Screening Levels - Groundwater	11/1/2012	GW	ug/L	0.19		80
VOCs	Chloromethane	74-87-3	EPA Regional Screening Levels - Groundwater	11/1/2012	GW	ug/L	190		
VOCs	Chlorotoluene, o-	95-49-8	EPA Regional Screening Levels - Groundwater	11/1/2012	GW	ug/L	180		
VOCs	Chlorotoluene, p-	106-43-4	EPA Regional Screening Levels - Groundwater	11/1/2012	GW	ug/L	190		
VOCs	Cumene	98-82-8	EPA Regional Screening Levels - Groundwater	11/1/2012	GW	ug/L	390		
VOCs	Cyclohexane	110-82-7	EPA Regional Screening Levels - Groundwater	11/1/2012	GW	ug/L	13000		
VOCs	Dibromo-3-chloropropane, 1,2-	96-12-8	EPA Regional Screening Levels - Groundwater	11/1/2012	GW	ug/L	0.00032		0.2
VOCs	Dibromochloromethane	124-48-1	EPA Regional Screening Levels - Groundwater	11/1/2012	GW	ug/L	0.15		80
VOCs	Dibromoethane, 1,2-	106-93-4	EPA Regional Screening Levels - Groundwater	11/1/2012	GW	ug/L	0.0065		0.05
VOCs	Dibromomethane (Methylene Bromide)	74-95-3	EPA Regional Screening Levels - Groundwater	11/1/2012	GW	ug/L	7.9		

VOCs	Dichlorobenzene, 1,2-	95-50-1	EPA Regional Screening Levels - Groundwater	11/1/2012	GW	ug/L	280	600
VOCs	Dichlorobenzene, 1,4-	106-46-7	EPA Regional Screening Levels - Groundwater	11/1/2012	GW	ug/L	0.42	75
VOCs	Dichlorodifluoromethane	75-71-8	EPA Regional Screening Levels - Groundwater	11/1/2012	GW	ug/L	190	
VOCs	Dichloroethane, 1,1-	75-34-3	EPA Regional Screening Levels - Groundwater	11/1/2012	GW	ug/L	2.4	
VOCs	Dichloroethane, 1,2-	107-06-2	EPA Regional Screening Levels - Groundwater	11/1/2012	GW	ug/L	0.15	5
VOCs	Dichloroethylene, 1,1-	75-35-4	EPA Regional Screening Levels - Groundwater	11/1/2012	GW	ug/L	260	7
VOcs	Dichloroethylene, 1,2- (Mixed Isomers)	540-59-0	EPA Regional Screening Levels - Groundwater	11/1/2012	GW	ug/L	130	
VOCs	Dichloroethylene, 1,2-cis-	156-59-2	EPA Regional Screening Levels - Groundwater	11/1/2012	GW	ug/L	28	70
VOCs	Dichloroethylene, 1,2-trans-	156-60-5	EPA Regional Screening Levels - Groundwater	11/1/2012	GW	ug/L	86	100
VOCs	Dichloropropane, 1,2-	78-87-5	EPA Regional Screening Levels - Groundwater	11/1/2012	GW	ug/L	0.38	5
VOcs	Dichloropropane, 1,3-	142-28-9	EPA Regional Screening Levels - Groundwater	11/1/2012	GW	ug/L	290	
VOCs	Dichloropropanol, 2,3-	616-23-9	EPA Regional Screening Levels - Groundwater	11/1/2012	GW	ug/L	46	
VOCs	Dichloropropene, 1,3-	542-75-6	EPA Regional Screening Levels - Groundwater	11/1/2012	GW	ug/L	0.41	
VOCs	Diisopropyl Ether	108-20-3	EPA Regional Screening Levels - Groundwater	11/1/2012	GW	ug/L	1500	
VOCs	Ethyl Acetate	141-78-6	EPA Regional Screening Levels - Groundwater	11/1/2012	GW	ug/L	14000	
VOCs	Ethyl Chloride	75-00-3	EPA Regional Screening Levels - Groundwater	11/1/2012	GW	ug/L	21000	
VOCs	Ethyl Ether	60-29-7	EPA Regional Screening Levels - Groundwater	11/1/2012	GW	ug/L	3100	
VOCs	Ethyl Methacrylate	97-63-2	EPA Regional Screening Levels - Groundwater	11/1/2012	GW	ug/L	420	
VOCs	Ethylbenzene	100-41-4	EPA Regional Screening Levels - Groundwater	11/1/2012	GW	ug/L	1.3	700
VOCs	Ethylene Oxide	75-21-8	EPA Regional Screening Levels - Groundwater	11/1/2012	GW	ug/L	0.044	
VOCs	Hexachlorobutadiene	87-68-3	EPA Regional Screening Levels - Groundwater	11/1/2012	GW	ug/L	0.26	
VOCs	Hexanone, 2-	591-78-6	EPA Regional Screening Levels - Groundwater	11/1/2012	GW	ug/L	34	
VOCs	Isobutyl Alcohol	78-83-1	EPA Regional Screening Levels - Groundwater	11/1/2012	GW	ug/L	4600	
VOCs	Methacrylonitrile	126-98-7	EPA Regional Screening Levels - Groundwater	11/1/2012	GW	ug/L	1.5	
VOCs	Methyl Acetate	79-20-9	EPA Regional Screening Levels - Groundwater	11/1/2012	GW	ug/L	16000	
VOCs	Methyl Ethyl Ketone (2-Butanone)	78-93-3	EPA Regional Screening Levels - Groundwater	11/1/2012	GW	ug/L	4900	
VOCs	Methyl Isobutyl Ketone (4-methyl-2-pentanone)	108-10-1	EPA Regional Screening Levels - Groundwater	11/1/2012	GW	ug/L	1000	
VOCs	Methyl Methacrylate	80-62-6	EPA Regional Screening Levels - Groundwater	11/1/2012	GW	ug/L	1400	
VOCs	Methyl tert-Butyl Ether (MTBE)	1634-04-4	EPA Regional Screening Levels - Groundwater	11/1/2012	GW	ug/L	12	
VOCs	Methylene Chloride	75-09-2	EPA Regional Screening Levels - Groundwater	11/1/2012	GW	ug/L	9.9	5
VOCs	Propyl benzene	103-65-1	EPA Regional Screening Levels - Groundwater	11/1/2012	GW	ug/L	530	
VOCs	Propylene Oxide	75-56-9	EPA Regional Screening Levels - Groundwater	11/1/2012	GW	ug/L	0.23	
VOCs	Styrene	100-42-5	EPA Regional Screening Levels - Groundwater	11/1/2012	GW	ug/L	1100	100
VOCs	Tetrachloroethane, 1,1,1,2-	630-20-6	EPA Regional Screening Levels - Groundwater	11/1/2012	GW	ug/L	0.5	
VOCs	Tetrachloroethane, 1,1,2,2-	79-34-5	EPA Regional Screening Levels - Groundwater	11/1/2012	GW	ug/L	0.066	
VOCs	Tetrachloroethylene	127-18-4	EPA Regional Screening Levels - Groundwater	11/1/2012	GW	ug/L	9.7	5
VOCs	Toluene	108-88-3	EPA Regional Screening Levels - Groundwater	11/1/2012	GW	ug/L	860	1000
VOCs	Trichlorobenzene, 1,2,3-	87-61-6	EPA Regional Screening Levels - Groundwater	11/1/2012	GW	ug/L	5.2	
VOCs	Trichlorobenzene, 1,2,4-	120-82-1	EPA Regional Screening Levels - Groundwater	11/1/2012	GW	ug/L	0.99	70
VOCs	Trichloroethane, 1,1,1-	71-55-6	EPA Regional Screening Levels - Groundwater	11/1/2012	GW	ug/L	7500	200
VOCs	Trichloroethane, 1,1,2-	79-00-5	EPA Regional Screening Levels - Groundwater	11/1/2012	GW	ug/L	0.24	5
VOCs	Trichloroethylene	79-01-6	EPA Regional Screening Levels - Groundwater	11/1/2012	GW	ug/L	0.44	5
VOCs	Trichlorofluoromethane	75-69-4	EPA Regional Screening Levels - Groundwater	11/1/2012	GW	ug/L	1100	
VOCs	Trichloropropane, 1,2,3-	96-18-4	EPA Regional Screening Levels - Groundwater	11/1/2012	GW	ug/L	0.00065	
VOCs	Trimethylbenzene, 1,2,4-	95-63-6	EPA Regional Screening Levels - Groundwater	11/1/2012	GW	ug/L	15	
VOCs	Trimethylbenzene, 1,3,5-	108-67-8	EPA Regional Screening Levels - Groundwater	11/1/2012	GW	ug/L	87	
VOCs	Urethane	51-79-6	EPA Regional Screening Levels - Groundwater	11/1/2012	GW	ug/L	0.021	
VOCs	Vinyl Acetate	108-05-4	EPA Regional Screening Levels - Groundwater	11/1/2012	GW	ug/L	410	
VOCs	Vinyl Bromide	593-60-2	EPA Regional Screening Levels - Groundwater	11/1/2012	GW	ug/L	0.15	
VOCs	Vinyl Chloride	75-01-4	EPA Regional Screening Levels - Groundwater	11/1/2012	GW	ug/L	0.015	2
VOCs	Warfarin	81-81-2	EPA Regional Screening Levels - Groundwater	11/1/2012	GW	ug/L	4.4	
VOCs	Xylenes	1330-20-7	EPA Regional Screening Levels - Groundwater	11/1/2012	GW	ug/L	190	10000
VOCs	Xylene, P-	106-42-3	EPA Regional Screening Levels - Groundwater	11/1/2012	GW	ug/L	190	
VOCs	Xylene, m-	108-38-3	EPA Regional Screening Levels - Groundwater	11/1/2012	GW	ug/L	190	
VOCs	Xylene, o-	95-47-6	EPA Regional Screening Levels - Groundwater	11/1/2012	GW	ug/L	190	

Analyte Group	Analyte Name	CAS Number	PRG	Published Date	Matrix	Units	Residential Soil	Industrial Soil
Metals	Aluminum	7429-90-5	EPA Regional Screening Levels - Soil	11/1/2012	Soil	mg/kg	77000	990000
Metals	Antimony (metallic)	7440-36-0	EPA Regional Screening Levels - Soil	11/1/2012	Soil	mg/kg	31	410
Metals	Arsenic, Inorganic	7440-38-2	EPA Regional Screening Levels - Soil	11/1/2012	Soil	mg/kg	0.39	1.6
Metals	Barium	7440-39-3	EPA Regional Screening Levels - Soil	11/1/2012	Soil	mg/kg	15000	190000
Metals	Beryllium and compounds	7440-41-7	EPA Regional Screening Levels - Soil	11/1/2012	Soil	mg/kg	160	2000
Metals	Cadmium (Diet)	7440-43-9	EPA Regional Screening Levels - Soil	11/1/2012	Soil	mg/kg	70	800
Metals	Chromium(III), Insoluble Salts	16065-83-1	EPA Regional Screening Levels - Soil	11/1/2012	Soil	mg/kg	120000	1500000
Metals	Cobalt	7440-48-4	EPA Regional Screening Levels - Soil	11/1/2012	Soil	mg/kg	23	300
Metals	Copper	7440-50-8	EPA Regional Screening Levels - Soil	11/1/2012	Soil	mg/kg	3100	41000
Metals	Iron	7439-89-6	EPA Regional Screening Levels - Soil	11/1/2012	Soil	mg/kg	55000	720000
Metals	Lead and Compounds	7439-92-1	EPA Regional Screening Levels - Soil	11/1/2012	Soil	mg/kg	400	800
Metals	Manganese (Non-Diet)	7439-96-5	EPA Regional Screening Levels - Soil	11/1/2012	Soil	mg/kg	1800	23000
Metals	Mercury (elemental)	7439-97-6	EPA Regional Screening Levels - Soil	11/1/2012	Soil	mg/kg	10	43
Metals	Nickel Soluble Salts	7440-02-0	EPA Regional Screening Levels - Soil	11/1/2012	Soil	mg/kg	1500	20000
Metals	Selenium	7782-49-2	EPA Regional Screening Levels - Soil	11/1/2012	Soil	mg/kg	390	5100
Metals	Silver	7440-22-4	EPA Regional Screening Levels - Soil	11/1/2012	Soil	mg/kg	390	5100
Metals	Thallium (Soluble Salts)	7440-28-0	EPA Regional Screening Levels - Soil	11/1/2012	Soil	mg/kg	0.78	10
Metals	Vanadium, Metallic	7440-62-2	EPA Regional Screening Levels - Soil	11/1/2012	Soil	mg/kg	390	5200
Metals	Zinc and Compounds	7440-66-6	EPA Regional Screening Levels - Soil	11/1/2012	Soil	mg/kg	23000	310000
Metals	Chromium, Total (as Cr +3)	7440-47-3	EPA Regional Screening Levels - Soil	11/1/2012	Soil	mg/kg	120000	1500000
Metals	Mercuric Chloride (and other Mercury salts)	7487-94-7	EPA Regional Screening Levels - Soil	11/1/2012	Soil	mg/kg	23	310
PCBs	Aroclor 1016	12674-11-2	EPA Regional Screening Levels - Soil	11/1/2012	Soil	mg/kg	3.9	21
PCBs	Aroclor 1221	11104-28-2	EPA Regional Screening Levels - Soil	11/1/2012	Soil	mg/kg	0.14	0.54
PCBs	Aroclor 1232	11141-16-5	EPA Regional Screening Levels - Soil	11/1/2012	Soil	mg/kg	0.14	0.54
PCBs	Aroclor 1242	53469-21-9	EPA Regional Screening Levels - Soil	11/1/2012	Soil	mg/kg	0.22	0.74
PCBs	Aroclor 1248	12672-29-6	EPA Regional Screening Levels - Soil	11/1/2012	Soil	mg/kg	0.22	0.74
PCBs	Aroclor 1254	11097-69-1	EPA Regional Screening Levels - Soil	11/1/2012	Soil	mg/kg	0.22	0.74
PCBs	Aroclor 1260	11096-82-5	EPA Regional Screening Levels - Soil	11/1/2012	Soil	mg/kg	0.22	0.74
SVOCs	Acetophenone	98-86-2	EPA Regional Screening Levels - Soil	11/1/2012	Soil	mg/kg	7800	100000
SVOCs	Atrazine	1912-24-9	EPA Regional Screening Levels - Soil	11/1/2012	Soil	mg/kg	2.1	7.5
SVOCs	Azobenzene	103-33-3	EPA Regional Screening Levels - Soil	11/1/2012	Soil	mg/kg	5.1	23
SVOCs	Benzaldehyde	100-52-7	EPA Regional Screening Levels - Soil	11/1/2012	Soil	mg/kg	7800	100000
SVOCs	Benzidine	92-87-5	EPA Regional Screening Levels - Soil	11/1/2012	Soil	mg/kg	0.0005	0.0075
SVOCs	Benzoic Acid	65-85-0	EPA Regional Screening Levels - Soil	11/1/2012	Soil	mg/kg	240000	2500000
SVOCs	Benzyl Alcohol	100-51-6	EPA Regional Screening Levels - Soil	11/1/2012	Soil	mg/kg	6100	62000
SVOCs	Bis(2-chloro-1-methylethyl) ether	108-60-1	EPA Regional Screening Levels - Soil	11/1/2012	Soil	mg/kg	4.6	22
SVOCs	Bis(2-chloroethoxy)methane	111-91-1	EPA Regional Screening Levels - Soil	11/1/2012	Soil	mg/kg	180	1800
SVOCs	Bis(2-chloroethyl)ether	111-44-4	EPA Regional Screening Levels - Soil	11/1/2012	Soil	mg/kg	0.21	1
SVOCs	Bis(2-ethylhexyl)phthalate	117-81-7	EPA Regional Screening Levels - Soil	11/1/2012	Soil	mg/kg	35	120
SVOCs	Bis(chloromethyl)ether	542-88-1	EPA Regional Screening Levels - Soil	11/1/2012	Soil	mg/kg	0.000077	0.00039
SVOCs	Bisphenol A	80-05-7	EPA Regional Screening Levels - Soil	11/1/2012	Soil	mg/kg	3100	31000
SVOCs	Butyl Benzyl Phthlate	85-68-7	EPA Regional Screening Levels - Soil	11/1/2012	Soil	mg/kg	260	910
SVOCs	Caprolactam	105-60-2	EPA Regional Screening Levels - Soil	11/1/2012	Soil	mg/kg	31000	310000
SVOCs	Chloroaniline, p-	106-47-8	EPA Regional Screening Levels - Soil	11/1/2012	Soil	mg/kg	2.4	8.6
SVOCs	Chloronaphthalene, Beta-	91-58-7	EPA Regional Screening Levels - Soil	11/1/2012	Soil	mg/kg	6300	82000
SVOCs	Chlorophenol, 2-	95-57-8	EPA Regional Screening Levels - Soil	11/1/2012	Soil	mg/kg	390	5100
SVOCs	Cresol, m-	108-39-4	EPA Regional Screening Levels - Soil	11/1/2012	Soil	mg/kg	3100	31000
SVOCs	Cresol, o-	95-48-7	EPA Regional Screening Levels - Soil	11/1/2012	Soil	mg/kg	3100	31000
SVOCs	Cresol, p-	106-44-5	EPA Regional Screening Levels - Soil	11/1/2012	Soil	mg/kg	6100	62000
SVOCs	Cresol, p-chloro-m-	59-50-7	EPA Regional Screening Levels - Soil	11/1/2012	Soil	mg/kg	6100	62000
SVOCs	Cresols	1319-77-3	EPA Regional Screening Levels - Soil	11/1/2012	Soil	mg/kg	6100	62000
SVOCs	Dibutyl Phthalate	84-74-2	EPA Regional Screening Levels - Soil	11/1/2012	Soil	mg/kg	6100	62000
SVOCs	Dichlorobenzidine, 3,3'-	91-94-1	EPA Regional Screening Levels - Soil	11/1/2012	Soil	mg/kg	1.1	3.8
SVOCs	Dichlorophenol, 2,4-	120-83-2	EPA Regional Screening Levels - Soil	11/1/2012	Soil	mg/kg	180	1800
SVOCs	Diethyl Phthalate	84-66-2	EPA Regional Screening Levels - Soil	11/1/2012	Soil	mg/kg	49000	490000
SVOCs	Dimethylphenol, 2,4-	105-67-9	EPA Regional Screening Levels - Soil	11/1/2012	Soil	mg/kg	1200	12000
SVOCs	Dinitro-o-cresol, 4,6-	534-52-1	EPA Regional Screening Levels - Soil	11/1/2012	Soil	mg/kg	4.9	49
SVOCs	Dinitrobenzene, 1,2-	528-29-0	EPA Regional Screening Levels - Soil	11/1/2012	Soil	mg/kg	6.1	62
SVOCs	Dinitrobenzene, 1,3-	99-65-0	EPA Regional Screening Levels - Soil	11/1/2012	Soil	mg/kg	6.1	62
SVOCs	Dinitrobenzene, 1,4-	100-25-4	EPA Regional Screening Levels - Soil	11/1/2012	Soil	mg/kg	6.1	62
SVOCs	Dinitrophenol, 2,4-	51-28-5	EPA Regional Screening Levels - Soil	11/1/2012	Soil	mg/kg	120	1200
SVOCs	Dinitrotoluene Mixture, 2,4/2,6-	25321-14-6	EPA Regional Screening Levels - Soil	11/1/2012	Soil	mg/kg	0.72	2.5
SVOCs	Dinitrotoluene, 2,4-	121-14-2	EPA Regional Screening Levels - Soil	11/1/2012	Soil	mg/kg	1.6	5.5
SVOCs	Dinitrotoluene, 2,6-	606-20-2	EPA Regional Screening Levels - Soil	11/1/2012	Soil	mg/kg	61	620
SVOCs	Dioxane, 1,4-	123-91-1	EPA Regional Screening Levels - Soil	11/1/2012	Soil	mg/kg	4.9	17
SVOCs	Diphenylhydrazine, 1,2-	122-66-7	EPA Regional Screening Levels - Soil	11/1/2012	Soil	mg/kg	0.61	2.2
SVOCs	Dibenzofuran	132-64-9	EPA Regional Screening Levels - Soil	11/1/2012	Soil	mg/kg	78	1000
SVOCs	Hexachlorobenzene	118-74-1	EPA Regional Screening Levels - Soil	11/1/2012	Soil	mg/kg	0.3	1.1
SVOCs	Hexachlorocyclopentadiene	77-47-4	EPA Regional Screening Levels - Soil	11/1/2012	Soil	mg/kg	370	3700
SVOCs	Hexachloroethane	67-72-1	EPA Regional Screening Levels - Soil	11/1/2012	Soil	mg/kg	12	43
SVOCs	Isophorone	78-59-1	EPA Regional Screening Levels - Soil	11/1/2012	Soil	mg/kg	510	1800
SVOCs	Nitroaniline, 2-	88-74-4	EPA Regional Screening Levels - Soil	11/1/2012	Soil	mg/kg	610	6000
SVOCs	Nitroaniline, 4-	100-01-6	EPA Regional Screening Levels - Soil	11/1/2012	Soil	mg/kg	24	86
SVOCs	Nitrobenzene	98-95-3	EPA Regional Screening Levels - Soil	11/1/2012	Soil	mg/kg	4.8	24
SVOCs	Nitroso-di-N-propylamine, N-	621-64-7	EPA Regional Screening Levels - Soil	11/1/2012	Soil	mg/kg	0.069	0.25
SVOCs	Nitrosodiphenylamine, N-	86-30-6	EPA Regional Screening Levels - Soil	11/1/2012	Soil	mg/kg	99	350
SVOCs	Pentachlorobenzene	608-93-5	EPA Regional Screening Levels - Soil	11/1/2012	Soil	mg/kg	49	490
SVOCs	Pentachlorophenol	87-86-5	EPA Regional Screening Levels - Soil	11/1/2012	Soil	mg/kg	0.89	2.7
SVOCs	Phenol	108-95-2	EPA Regional Screening Levels - Soil	11/1/2012	Soil	mg/kg	18000	180000
SVOCs	Acenaphthene	83-32-9	EPA Regional Screening Levels - Soil	11/1/2012	Soil	mg/kg	3400	33000
SVOCs	Anthracene	120-12-7	EPA Regional Screening Levels - Soil	11/1/2012	Soil	mg/kg	17000	170000
SVOCs	Benz[a]anthracene	56-55-3	EPA Regional Screening Levels - Soil	11/1/2012	Soil	mg/kg	0.15	2.1
SVOCs	Benzo(j)fluoranthene	205-82-3	EPA Regional Screening Levels - Soil	11/1/2012	Soil	mg/kg	0.38	1.3
SVOCs	Benzo[a]pyrene	50-32-8	EPA Regional Screening Levels - Soil	11/1/2012	Soil	mg/kg	0.015	0.21
SVOCs	Benzo[b]fluoranthene	205-99-2	EPA Regional Screening Levels - Soil	11/1/2012	Soil	mg/kg	0.15	2.1
SVOCs	Benzo[k]fluoranthene	207-08-9	EPA Regional Screening Levels - Soil	11/1/2012	Soil	mg/kg	1.5	21
SVOCs	Chrysene	218-01-9	EPA Regional Screening Levels - Soil	11/1/2012	Soil	mg/kg	15	210
SVOCs	Dibenz[a,h]anthracene	53-70-3	EPA Regional Screening Levels - Soil	11/1/2012	Soil	mg/kg	0.015	0.21
SVOCs	Dibenzo(a,e)pyrene	192-65-4	EPA Regional Screening Levels - Soil	11/1/2012	Soil	mg/kg	0.038	0.13
SVOCs	Fluoranthene	206-44-0	EPA Regional Screening Levels - Soil	11/1/2012	Soil	mg/kg	2300	22000
SVOCs	Fluorene	86-73-7	EPA Regional Screening Levels - Soil	11/1/2012	Soil	mg/kg	2300	22000
SVOCs	Indeno[1,2,3-cd]pyrene	193-39-5	EPA Regional Screening Levels - Soil	11/1/2012	Soil	mg/kg	0.15	2.1
SVOCs	Methylnaphthalene, 1-	90-12-0	EPA Regional Screening Levels - Soil	11/1/2012	Soil	mg/kg	16	52
SVOCs	Methylnaphthalene, 2-	91-57-6	EPA Regional Screening Levels - Soil	11/1/2012	Soil	mg/kg	230	2200
SVOCs	Naphthalene	91-20-3	EPA Regional Screening Levels - Soil	11/1/2012	Soil	mg/kg	3.6	18
SVOCs	Pyrene	129-00-0	EPA Regional Screening Levels - Soil	11/1/2012	Soil	mg/kg	1700	17000
SVOCs	Pyridine	110-86-1	EPA Regional Screening Levels - Soil	11/1/2012	Soil	mg/kg	78	1000
SVOCs	Safrole	94-59-7	EPA Regional Screening Levels - Soil	11/1/2012	Soil	mg/kg	0.52	7.8
SVOCs	Trichlorophenol, 2,4,5-	95-95-4	EPA Regional Screening Levels - Soil	11/1/2012	Soil	mg/kg	6100	62000
SVOCs	Trichlorophenol, 2,4,6-	88-06-2	EPA Regional Screening Levels - Soil	11/1/2012	Soil	mg/kg	44	160
VOCs	Acetone	67-64-1	EPA Regional Screening Levels - Soil	11/1/2012	Soil	mg/kg	61000	630000
VOCs	Acetonitrile	75-05-8	EPA Regional Screening Levels - Soil	11/1/2012	Soil	mg/kg	870	3700
VOCs	Acrolein	107-02-8	EPA Regional Screening Levels - Soil	11/1/2012	Soil	mg/kg	0.15	0.65
VOCs	Acrylonitrile	107-13-1	EPA Regional Screening Levels - Soil	11/1/2012	Soil	mg/kg	0.24	1.2
VOCs	Benzene	71-43-2	EPA Regional Screening Levels - Soil	11/1/2012	Soil	mg/kg	1.1	5.4
VOCs	Bromobenzene	108-86-1	EPA Regional Screening Levels - Soil	11/1/2012	Soil	mg/kg	300	1800
VOCs	Bromodichloromethane	75-27-4	EPA Regional Screening Levels - Soil	11/1/2012	Soil	mg/kg	0.27	1.4
VOCs	Bromoform	75-25-2	EPA Regional Screening Levels - Soil	11/1/2012	Soil	mg/kg	62	220
VOCs	Bromomethane	74-83-9	EPA Regional Screening Levels - Soil	11/1/2012	Soil	mg/kg	7.3	32
VOCs	Carbon Disulfide	75-15-0	EPA Regional Screening Levels - Soil	11/1/2012	Soil	mg/kg	820	3700
VOCs	Carbon Tetrachloride	56-23-5	EPA Regional Screening Levels - Soil	11/1/2012	Soil	mg/kg	0.61	3
VOCs	Chlorobenzene	108-90-7	EPA Regional Screening Levels - Soil	11/1/2012	Soil	mg/kg	290	1400
VOCs	Chloroform	67-66-3	EPA Regional Screening Levels - Soil	11/1/2012	Soil	mg/kg	0.29	1.5
VOCs	Chloromethane	74-87-3	EPA Regional Screening Levels - Soil	11/1/2012	Soil	mg/kg	120	500
VOCs	Chlorotoluene, o-	95-49-8	EPA Regional Screening Levels - Soil	11/1/2012	Soil	mg/kg	1600	20000
VOCs	Chlorotoluene, p-	106-43-4	EPA Regional Screening Levels - Soil	11/1/2012	Soil	mg/kg	1600	20000
VOCs	Cumene	98-82-8	EPA Regional Screening Levels - Soil	11/1/2012	Soil	mg/kg	2100	11000
VOCs	Cyclohexane	110-82-7	EPA Regional Screening Levels - Soil	11/1/2012	Soil	mg/kg	7000	29000
VOCs	Dibromo-3-chloropropane, 1,2-	96-12-8	EPA Regional Screening Levels - Soil	11/1/2012	Soil	mg/kg	0.0054	0.069
VOCs	Dibromochloromethane	124-48-1	EPA Regional Screening Levels - Soil	11/1/2012	Soil	mg/kg	0.68	3.3
VOCs	Dibromoethane, 1,2-	106-93-4	EPA Regional Screening Levels - Soil	11/1/2012	Soil	mg/kg	0.034	0.17
VOCs	Dibromomethane (Methylene Bromide)	74-95-3	EPA Regional Screening Levels - Soil	11/1/2012	Soil	mg/kg	25	110

VOCs	Dichlorobenzene, 1,2-	95-50-1	EPA Regional Screening Levels - Soil	11/1/2012	Soil	mg/kg	1900	9800
VOCs	Dichlorobenzene, 1,4-	106-46-7	EPA Regional Screening Levels - Soil	11/1/2012	Soil	mg/kg	2.4	12
VOCs	Dichlorodifluoromethane	75-71-8	EPA Regional Screening Levels - Soil	11/1/2012	Soil	mg/kg	94	400
VOCs	Dichloroethane, 1,1-	75-34-3	EPA Regional Screening Levels - Soil	11/1/2012	Soil	mg/kg	3.3	17
VOCs	Dichloroethane, 1,2-	107-06-2	EPA Regional Screening Levels - Soil	11/1/2012	Soil	mg/kg	0.43	2.2
VOCs	Dichloroethylene, 1,1-	75-35-4	EPA Regional Screening Levels - Soil	11/1/2012	Soil	mg/kg	240	1100
VOcs	Dichloroethylene, 1,2- (Mixed Isomers)	540-59-0	EPA Regional Screening Levels - Soil	11/1/2012	Soil	mg/kg	700	9200
VOCs	Dichloroethylene, 1,2-cis-	156-59-2	EPA Regional Screening Levels - Soil	11/1/2012	Soil	mg/kg	160	2000
VOCs	Dichloroethylene, 1,2-trans-	156-60-5	EPA Regional Screening Levels - Soil	11/1/2012	Soil	mg/kg	150	690
VOCs	Dichloropropane, 1,2-	78-87-5	EPA Regional Screening Levels - Soil	11/1/2012	Soil	mg/kg	0.94	4.7
VOcs	Dichloropropane, 1,3-	142-28-9	EPA Regional Screening Levels - Soil	11/1/2012	Soil	mg/kg	1600	20000
VOCs	Dichloropropanol, 2,3-	616-23-9	EPA Regional Screening Levels - Soil	11/1/2012	Soil	mg/kg	180	1800
VOCs	Dichloropropene, 1,3-	542-75-6	EPA Regional Screening Levels - Soil	11/1/2012	Soil	mg/kg	1.7	8.3
VOCs	Diisopropyl Ether	108-20-3	EPA Regional Screening Levels - Soil	11/1/2012	Soil	mg/kg	2400	10000
VOCs	Ethyl Acetate	141-78-6	EPA Regional Screening Levels - Soil	11/1/2012	Soil	mg/kg	70000	920000
VOCs	Ethyl Chloride	75-00-3	EPA Regional Screening Levels - Soil	11/1/2012	Soil	mg/kg	15000	61000
VOCs	Ethyl Ether	60-29-7	EPA Regional Screening Levels - Soil	11/1/2012	Soil	mg/kg	16000	200000
VOCs	Ethyl Methacrylate	97-63-2	EPA Regional Screening Levels - Soil	11/1/2012	Soil	mg/kg	1500	7500
VOCs	Ethylbenzene	100-41-4	EPA Regional Screening Levels - Soil	11/1/2012	Soil	mg/kg	5.4	27
VOCs	Ethylene Oxide	75-21-8	EPA Regional Screening Levels - Soil	11/1/2012	Soil	mg/kg	0.17	0.83
VOCs	Hexachlorobutadiene	87-68-3	EPA Regional Screening Levels - Soil	11/1/2012	Soil	mg/kg	6.2	22
VOCs	Hexanone, 2-	591-78-6	EPA Regional Screening Levels - Soil	11/1/2012	Soil	mg/kg	210	1400
VOCs	Isobutyl Alcohol	78-83-1	EPA Regional Screening Levels - Soil	11/1/2012	Soil	mg/kg	18000	180000
VOCs	Methacrylonitrile	126-98-7	EPA Regional Screening Levels - Soil	11/1/2012	Soil	mg/kg	7.6	92
VOCs	Methyl Acetate	79-20-9	EPA Regional Screening Levels - Soil	11/1/2012	Soil	mg/kg	78000	1000000
VOCs	Methyl Ethyl Ketone (2-Butanone)	78-93-3	EPA Regional Screening Levels - Soil	11/1/2012	Soil	mg/kg	28000	200000
VOCs	Methyl Isobutyl Ketone (4-methyl-2-pentanone)	108-10-1	EPA Regional Screening Levels - Soil	11/1/2012	Soil	mg/kg	5300	53000
VOCs	Methyl Methacrylate	80-62-6	EPA Regional Screening Levels - Soil	11/1/2012	Soil	mg/kg	4800	21000
VOCs	Methyl tert-Butyl Ether (MTBE)	1634-04-4	EPA Regional Screening Levels - Soil	11/1/2012	Soil	mg/kg	43	220
VOCs	Methylene Chloride	75-09-2	EPA Regional Screening Levels - Soil	11/1/2012	Soil	mg/kg	56	960
VOCs	Propyl benzene	103-65-1	EPA Regional Screening Levels - Soil	11/1/2012	Soil	mg/kg	3400	21000
VOCs	Propylene Oxide	75-56-9	EPA Regional Screening Levels - Soil	11/1/2012	Soil	mg/kg	2	9
VOCs	Styrene	100-42-5	EPA Regional Screening Levels - Soil	11/1/2012	Soil	mg/kg	6300	36000
VOCs	Tetrachloroethane, 1,1,1,2-	630-20-6	EPA Regional Screening Levels - Soil	11/1/2012	Soil	mg/kg	1.9	9.3
VOCs	Tetrachloroethane, 1,1,2,2-	79-34-5	EPA Regional Screening Levels - Soil	11/1/2012	Soil	mg/kg	0.56	2.8
VOCs	Tetrachloroethylene	127-18-4	EPA Regional Screening Levels - Soil	11/1/2012	Soil	mg/kg	0.55	2.6
VOCs	Toluene	108-88-3	EPA Regional Screening Levels - Soil	11/1/2012	Soil	mg/kg	5000	45000
VOCs	Trichlorobenzene, 1,2,3-	87-61-6	EPA Regional Screening Levels - Soil	11/1/2012	Soil	mg/kg	49	490
VOCs	Trichlorobenzene, 1,2,4-	120-82-1	EPA Regional Screening Levels - Soil	11/1/2012	Soil	mg/kg	22	99
VOCs	Trichloroethane, 1,1,1-	71-55-6	EPA Regional Screening Levels - Soil	11/1/2012	Soil	mg/kg	8700	38000
VOCs	Trichloroethane, 1,1,2-	79-00-5	EPA Regional Screening Levels - Soil	11/1/2012	Soil	mg/kg	1.1	5.3
VOCs	Trichloroethylene	79-01-6	EPA Regional Screening Levels - Soil	11/1/2012	Soil	mg/kg	0.91	6.4
VOCs	Trichlorofluoromethane	75-69-4	EPA Regional Screening Levels - Soil	11/1/2012	Soil	mg/kg	790	3400
VOCs	Trichloropropane, 1,2,3-	96-18-4	EPA Regional Screening Levels - Soil	11/1/2012	Soil	mg/kg	0.005	0.095
VOCs	Trimethylbenzene, 1,2,4-	95-63-6	EPA Regional Screening Levels - Soil	11/1/2012	Soil	mg/kg	62	260
VOCs	Trimethylbenzene, 1,3,5-	108-67-8	EPA Regional Screening Levels - Soil	11/1/2012	Soil	mg/kg	780	10000
VOCs	Urethane	51-79-6	EPA Regional Screening Levels - Soil	11/1/2012	Soil	mg/kg	0.12	2.9
VOCs	Vinyl Acetate	108-05-4	EPA Regional Screening Levels - Soil	11/1/2012	Soil	mg/kg	970	4100
VOCs	Vinyl Bromide	593-60-2	EPA Regional Screening Levels - Soil	11/1/2012	Soil	mg/kg	0.11	0.56
VOCs	Vinyl Chloride	75-01-4	EPA Regional Screening Levels - Soil	11/1/2012	Soil	mg/kg	0.06	1.7
VOCs	Warfarin	81-81-2	EPA Regional Screening Levels - Soil	11/1/2012	Soil	mg/kg	18	180
VOCs	Xylenes	1330-20-7	EPA Regional Screening Levels - Soil	11/1/2012	Soil	mg/kg	630	2700
VOCs	Xylene, P-	106-42-3	EPA Regional Screening Levels - Soil	11/1/2012	Soil	mg/kg	600	2600
VOCs	Xylene, m-	108-38-3	EPA Regional Screening Levels - Soil	11/1/2012	Soil	mg/kg	590	2500
VOCs	Xylene, o-	95-47-6	EPA Regional Screening Levels - Soil	11/1/2012	Soil	mg/kg	690	3000

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STANDARD OPERATING PROCEDURE

FIELD ACTIVITY LOGBOOKS

SOP NUMBER: DOC 2.1

REVISION DATE: 8/23/2012 **SCHEDULED REVIEW DATE: 5/24/2017**

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1 Scope and Application

Proper documentation of field activities is a critical component of any field effort. This Standard Operating Procedure (SOP) establishes procedures for initiating, entering information/data into, reviewing, and maintaining/storing hard copy field logbooks for E & E field activities. Field activities may range from simple reconnaissance to complex sampling programs and may include: visual or other observations, in situ or ex situ field measurements (monitoring), or sample collection, and can include meetings with E & E clients, sub-contractors or other stakeholders.

Field logbook documentation may be supplemented by other records (e.g., site safety forms, data collection forms, electronic data, or geotechnical logbooks). Information and data to be recorded on such forms or logbooks are addressed in the applicable SOPs.

Field observations, measurements, and samples have value to data users only to the degree that the observation, measurement, or sample is representative of a specified environment, setting, or process. Field logbooks address representativeness by documenting the following:

- Identification of the subject of the observation, measurement, or sampling;
- Selection of an observation, measurement, or sampling location and time that represents that subject;
- Compliance with or deviation from the work plan, sampling and analysis plan, quality assurance project plan, or other project or program plans; and

Sufficient documentation of how the observation, measurement, or sample represents the same subject as other observations, measurements, or samples from the vicinity. Complete and accurate logbook entries are important for several reasons: to ensure that data collection associated with field activities is sufficient to support the successful completion of the project; to provide sufficient information so that someone not associated with the project can independently reconstruct the field activities at a later date; to maintain quality control throughout the project; to document changes to or deviations from the work plan; to fulfill administrative needs of the project; and to support potential legal proceedings associated with a specific project. This Field Activity Logbook SOP is intended for use by personnel who have knowledge, training and experience in the field activities being conducted.

2 Definitions and Acronyms

Field	Locations (sites) outside the controlled environment of an office or laboratory.
Field Observation	The qualitative and/or quantitative remarks/statements regarding sensory inputs noted in the field.
Field Measurement	The quantitative determination of physical, chemical, biological, geological or radiological properties of a matrix by measurements made in the field.
Field Sampling	The process of obtaining a representative portion of an environmental matrix suitable for laboratory or field measurement or analysis.
E & E	Ecology and Environment, Inc.
EPA	Environmental Protection Agency

ID	Identification
IDW –	Investigation-derived waste
QA	Quality assurance
QC	Quality control
SOP	Standard operating procedure

3 Procedure Summary

Prior to field activity, the program/project manager identifies field personnel; designates a field team leader; and team members responsible for documenting field activities. Since there may be multiple activities with unique logbooks, there may be multiple team members responsible for documenting field activities.

Prior to entering the field, the individual responsible for documenting field activities or other designated author should briefly summarize the field activities that will be conducted in the logbook.

Visual or other observations, in situ or ex situ field measurements (including instrument/equipment calibrations), or sample collection information should be recorded in real-time as field work is conducted. Meetings, including electronic communications, with E & E, clients, sub-contractors or regulatory personnel should be recorded. Compliance with or deviation from the work plan, sampling and analysis plan, quality assurance project plan, or other project or program plans should be highlighted together with authorization for such deviations.

The field team leader should review log book entries on a daily basis or more frequently, if appropriate. The project/program manager should review the logbooks at the close of fieldwork or more frequently for long-term field events. Logbooks may be audited by quality assurance personnel from E & E or a client.

The program/project manager is responsible for storing/archiving applicable logbooks in the project file.

4 Cautions

Logbook entry must be a priority and not left to “later.” Contemporaneous documentation is critical to accurate and precise reporting.

Field logbooks become part of the permanent record for projects/programs and, thus, should include factual material, not opinions. Language used in logbooks should be objective and factual. Pertinent personal observations may be included, but must be clearly identified as such.

If multiple logbooks are used, a project logbook should be used to maintain control of all other logbooks.

Do not leave blank line(s) between logbook entries. Cross out blank spaces with a single line, initial and date the cross out.

Initials should not be used in place of signatures unless specifically allowed by client requirements. Logbooks are considered evidentiary files and full signatures are required under

judicial review guidelines (See EPA NEIC Policy 1991). If initials are used, a table of signatures and initials for all project personnel should be added in the front of the logbook.

5 Equipment and Supplies

Logbooks must be bound with consecutively numbered pages.

Entries should be made using indelible ink (preferably black).

6 Procedure

The following guidelines are used for completing Field Activity Logbooks:

- Logbooks will be assigned by the program/project manager to the field team leader. Additional logbooks may be assigned to other personnel (e.g., health and safety monitors). The program/project manager is responsible for tracking field event logbooks.
- A separate field logbook must be maintained for each project.
- Logbooks must be bound and contain consecutively numbered pages.
- The first entry for each day will be made on a new, previously blank page.
- No pages may be removed for any reason, even if mutilated or illegible. If a page or portion of a page is accidentally skipped during fieldwork, it should be crossed out, signed, and dated.
- Entries should be made in chronological order. Observations that cannot be recorded during field activities should be recorded as soon as possible. If logbook entries are made after field activities, the time of the activity/observation and the time that it is recorded should be noted.
- The time of each entry should be noted. It is customary to record time using a 24-hour clock.
- If corrections are necessary, they must be made by drawing a **single line** through the original entry in such a manner that it can still be read. Do not erase or render an incorrect notation illegible. The corrected entry should be written beside the incorrect entry, and the correction **initialed** and **dated**. Corrected errors may require a footnote explaining the correction.
- Logbooks should be signed at the end of each day (if more than one person makes entries into the logbook, each person should sign and date next to his or her entries). Signatures should be written along a single diagonal line drawn across the blank portion of the page following the last entry of the day.
- If multiple personnel are making entries in a logbook, then a table of personnel, signatures and initials should be added to the front of the logbook.
- At the completion of the field activity, the logbook must be returned to the project manager to include with the project files.

6.1 Format

The following guidelines provide a general format and required information for all routine field activities using the Field Activity Logbooks:

- Title Page
  - The logbook title page should contain the following items:
  - Site name;
  - Site identification (ID) number; if applicable;
  - Location;
  - Project Number;
  - Start/finish date; and
  - Book of . (may be completed at the end of the project)
- First Page
  - The following items should appear on the first page of the logbook prior to daily field activity entries:
  - Project Number;
  - Date;
  - Summary of proposed work (reference work plan and contract documents, as appropriate);
  - Weather conditions;
  - Team members and duties;
  - Health and safety discussion, topics, and attendees;
  - Time work began and time of arrival (using 24-hour clock notation); and
  - Arrival/departure times of each field team member and other personnel if different from overall work times.
- Successive Pages
  - In addition to specific activity entries and observations, the following items should appear on every logbook page:
  - Date at the top of each page,
  - Project Number and site name,
  - Weather conditions if changed from the first entry of the data,
  - Signature and date at the bottom of each page (if more than one person makes entries into the logbook, each person should sign and date next to his or her entries); and
  - Strikethroughs of any unused lines. If more than one person makes entries into the logbook, each person should sign and date next to his or her entry.

- Last Page
  - The last page of the logbook may contain a brief paragraph that summarizes the work that was completed in the field and recorded in this logbook.
  - The last page should indicate if work is continuing in subsequent logbooks or if the project is complete.

6.2 Logbook Information

Field logbook entries will contain a variety of information based on the field activities being conducted (e.g., observing, monitoring, or sampling). The specific type of information recorded in the logbook will depend on the project requirements. In general, information recorded on field forms or electronic data do not need to be recorded in the logbook.

- If not field sampling map is available then a site sketch should be included and updated as necessary identifying the site layout, features and points of interest. A north arrow and rough scale should be included,
- A sketch of individual sampling locations if GPS coordinates are not collected,
- GPS locations, as applicable, for site features,
- Physical description of the site as observed during sample collection,
- Weather conditions, updated as necessary,
- Record of phone calls and/or other contacts with individuals at the site; including names and affiliations,
- Daily brief summary of the site safety meeting if not recorded on separate form,
- Daily brief outline of field activities to be performed that day,
- Pertinent field observations and any unique method to gather observations,
- Documentation of photographs, including:
  - Make and model of the camera(s),
  - Description of the photograph including the date and time,
  - Photograph number,
  - Direction or view angle of the photograph,
  - Name of the photographer(s),
- Brief description of monitoring procedures,
- Model and serial numbers of monitoring equipment,
  - Equipment preparation/calibration procedures, date and time, and results if not recorded on separate form,
  - Field maintenance and/or repairs,
- Sample collection procedures and reference to applicable work plan section or SOP,

- Sample collection activities, including:
  - Pre-sampling activities (e.g., well purging and the number of volumes purged before sample collection),
  - Data associated with pre-sampling activities (e.g., well purging pH, conductivity, temperature data),
  - Equipment decontamination procedure,
- Sample information and observations
  - Sample number, station location ID, programmatic ID , and/or location, including relationship to permanent reference point(s),
  - Name(s) of sampler(s),
  - Sample description, sample depth interval, sample time, sample date, and any field screening results,
  - Sample matrix and number of aliquots if the sample is a composite,
  - Container and preservatives used, recipient laboratory including contact information, and requested analyses, and
  - Any preservative added in the field including preservative type, lot number and expiration date.
- Quality assurance (QA)/quality control(QC) samples,
  - For trip blanks indicate the source of the blanks,
  - For equipment rinsate samples, the equipment from which the rinsate sample is collected should be noted and source of the DI water, and
  - Field duplicates or replicates and a description of how the duplicate was sub-sampled.
- Shipping paper (airbill) numbers, chain-of-custody form numbers.

6.3 Work Plan Changes/Deviation

Compliance with or deviation from the work plan, sampling and analysis plan, quality assurance project plan, or other project or program plans should be highlighted together with authorization for any deviations. Deviations (who, what, where, when, why, and how) from the plans and the circumstances necessitating such changes should be recorded.

6.4 Investigation-Derived Waste

Disposition of non-hazardous versus potentially hazardous IDW should be delineated in the field planning documents. The following information should be included in the logbook:

- Nature and disposition of non-hazardous wastes;
- The type and number of containers of potentially hazardous IDW generated (each “drum” should be numbered and its contents noted);
- Information relevant to characterizing IDW;
- Disposition of IDW (left on site or removed from site); and

- IDW sample information should be recorded the same as other samples.
- The type of paperwork that accompanied the waste/sample shipment (e.g., manifests).

6.5 Data Collection Forms

Certain phases of fieldwork may require the use of separate project-specific data collection forms, such as sample collection, equipment calibration or daily summary forms. Use of such forms and the types of information recorded should be noted in logbook. Information recorded on data entry forms does not need to be repeated in the logbook.

7 Quality Assurance/Quality Control

Compliance with or deviation from work plan, sampling and analysis plan, quality assurance project plan, or other project or program plans should be highlighted together with authorization for any deviations.

Prior to field activity, among other responsibilities, the program/project manager should identify knowledgeable, trained, and experienced field personnel; designate a field team leader; and an individual responsible for documenting field activities. Since there may be multiple activities with unique logbooks, there may be multiple individuals responsible for documenting field activities.

Prior to entering the field, the individual responsible for documenting field activities or other designated author should briefly summarize the field activities being conducted in the logbook.

The field team leader should review log book entries on a daily basis or more frequently if appropriate. The project/program manager should review the logbooks at the close of fieldwork or more frequently for long-term field events. Logbooks may be audited by quality assurance personnel from E & E or a client.

The project/program manager is responsible for storing/archiving applicable logbooks in the project file.

8 Special Project Requirements

Project or program-specific requirements that modify this procedure should be entered in this section and included with the project planning documents.

9 References

The following list sources of technical information on field logbooks.

United States Environmental Protection Agency (EPA). 1988. *Guidance for Conducting Remedial Investigations and Feasibility Studies Under CERCLA Interim Final*, U.S. EPA, EPA/540/G-89/004, October 1988

_____. 1991. *Guidance for Performing Preliminary Assessments Under CERCLA*, U.S. EPA, EPA/540/G-91/013, September 1991

_____. 1991. *NEIC Policies and Procedures Manual*, U.S. EPA, EPA 33019-78-001-R, August 1991

_____. 1992. *Guidance for Performing Site Inspections Under CERCLA, Interim Final*, U.S. EPA, EPA/540/R-92-021, September 1992

Minor Revision Date	Revision Notes
8/1/2012	Added minor clarifications on signatures to address field audit findings.

END OF SOP

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STANDARD OPERATING PROCEDURE

GROUNDWATER WELL SAMPLING

SOP NUMBER: ENV 3.07

REVISION DATE: 4/7/2013 **SCHEDULED REVIEW DATE: 6/14/2017**

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1 Scope and Application

This Standard Operating Procedure (SOP) describes the procedures for the sampling of groundwater wells and is primarily concerned with the collection of water samples from the saturated zone of the subsurface. Every effort must be made to ensure that the sample is representative of the particular zone of water being sampled. There are numerous state and federal standards and guidelines on groundwater sampling that should be relative to project requirements and site conditions. This SOP can be followed for all routine sample collection activities which may include: field measurements (monitoring) or sample collection for chemical, radiological or physical analysis. Site-specific sampling procedures vary depending on the data quality objectives (DQOs) identified in program/project planning documents.

Analysis of groundwater samples may determine pollutant concentrations and its risk to public health, welfare, or the environment; extent of contaminants; and confirmation of remedial standards. Sampling methods should be determined based on regulatory standards needed to report acceptable analytical results. The project planning documents should clearly indicate the type of sampling to be completed.

Procedures for sample handling are defined in E & E Environmental Sample Handling, Packaging and Shipping SOP ENV 3.16. Site-specific sample handling procedures are dependent on the project DQOs.

Procedures for equipment decontamination are defined in E & E Sampling Equipment Decontamination SOP ENV 3.15. Site-specific equipment decontamination procedures are dependent on the project DQOs.

This groundwater sampling SOP is intended for use by personnel who have knowledge, training and experience in the field sampling activities being conducted.

2 Definitions and Acronyms

DQO	Data Quality Objectives
DO	dissolved oxygen
E & E	Ecology and Environment, Inc.
mL/min	milliliters per minute
mV	millivolt
NTU	Nephelometric Turbidity Units
ORP	oxygen reduction potential
SOP	Standard Operating Procedure
SSSP	Site-Specific Safety Plan
SVOC	Semi-volatile organic compound
µm	Micrometer
VOA	Volatile organic analysis
VOC	Volatile organic compound

3 Procedure Summary

This procedure covers routine groundwater sampling. Federal and state regulatory agencies also have standards and guidance for groundwater sampling that supersedes this SOP if required for the project. Before sampling a well, the well must be purged. This may be done with a number of portable devices, including bailers, submersible pumps, bladder pumps, gas-driven pumps, gas-lift pumps, suction-lift pumps, and inertial-lift pumps. Refer to E & E's Guidance 3.06, Groundwater Sampling Devices, for information on different groundwater purging and sampling devices. Domestic drinking water or irrigation wells may have a downhole well pump already installed that could be used for purging and sample collection.

For routine sampling, typically a minimum of three well volumes should be removed during well purging to ensure that groundwater samples collected are representative of aquifer conditions. For low flow sampling, water quality parameters are measured and well purging is complete when the parameters and water depth has stabilized. After purging (routine sampling method or low flow sampling method) is complete and the properly prepared sample containers have been selected, sample collection may proceed. Numerous types of sampling devices are available for the purging and collection of the groundwater sample, but care should be taken when selecting the sampling device, as some will affect the integrity of the sample.

Sampling should occur in a progression beginning with the well(s) suspected to be least contaminated and finishing with those suspected to be most contaminated. Ideally, a dedicated sampling device should be used for each well. However, dedicated sampling devices may not be practical if there are a large number of groundwater samples to be collected. In this case, sampling devices should be cleaned prior to and between sampling locations and events using the decontamination procedures outlined in E & E's SOP for *Sampling Equipment Decontamination* (ENV 3.15).

Domestic well sampling may be conducted to establish base level concentrations of chemicals, metals, bacteria or other potential contaminants prior to work in an area; to assess the impact of nearby activities; or for other reasons. Unique considerations apply to domestic wells due to construction, frequency of use, access, or other factors.

4 Cautions and Considerations

The following general health and safety concerns should be considered during groundwater well sampling:

- Use of tools to open/close the well or during sampling (e.g., sharp knives)
- Use of gas powered equipment when sampling
- Fuel equipment only when equipment is cool to the touch
- Fuel storage (do not use/store near open sampling bottles)
- Use of preserved sample containers(use nitrile gloves when handling containers)
- Use care when handling filled sample containers (use nitrile gloves when handling containers)

4.1 Purging/Stagnant Water

In a nonpumping well, there will be little or no vertical mixing of the water, and stratification will occur. The well water in the screened interval will mix with the groundwater due to normal flow patterns, but the water above the screened interval will remain isolated and become stagnant. Sampling team members should realize that stagnant water will not be representative of aquifer

conditions and may contain foreign material inadvertently or deliberately introduced from the ground surface or from well construction. To safeguard against collecting non-representative stagnant water during sampling, the following guidelines and techniques should be adhered to:

- As a general rule, all wells should be pumped or bailed prior to sample collection (unless otherwise stated in the project planning documents). Typically, evacuation of a minimum of one volume of water in the well casing, and preferably three to five volumes, is recommended for a representative sample. In a high-yielding groundwater formation and where there is no stagnant water in the well above the screened section, evacuation prior to sample collection is not as critical. However, in all cases where the monitoring data are to be used for enforcement actions, evacuation is recommended.
- For wells that can be pumped or bailed dry, the well should be evacuated and allowed to recover prior to sample collection. If the recovery rate is fairly rapid and time allows, evacuation of more than one volume of water is preferred.
- A non-representative sample can also result from excessive pumping of the well. Stratification of the leachate concentrations in the groundwater formation may occur or compounds that are heavier than water may sink to the lower portions of the aquifer. Excessive pumping can dilute or increase the contaminant concentrations from what is representative of the sampling point of interest.

Stagnant water may be a relatively minor issue in domestic drinking water wells that are used on a regular basis; however, such wells should also be purged prior to sample collection. Opening the casing in a domestic well may not be possible or may be impractical and construction information may be unavailable, making well volume calculations difficult or impossible. Treatment systems, filters, pressure tanks, storage tanks, or other apparatus' may be present in a domestic well system. When sampling to assess groundwater supply conditions it is important to collect samples upstream of all such features.

4.2 Materials

The material used to construct groundwater purging and sampling devices can have a significant impact on the analytical results. If practical, equipment that contacts the groundwater should be constructed from stainless steel, Teflon, or glass. The use of plastic should be avoided when analyzing for organics such as SVOCs. In general, the project planning documents should be reviewed to determine which materials are appropriate for a specific site prior to using groundwater purging and sampling devices.

5 Equipment and Supplies

The equipment and supplies required for well sampling depends on the program/project DQOs. The following is a general list of equipment and supplies. A detailed list of equipment and supplies should be prepared based on the project planning documents. In general, the use of dedicated or disposal equipment is preferred but equipment may be re-used after thorough decontamination between sample locations (refer to E & E Sampling Equipment Decontamination SOP ENV 3.15).

- Water level indicator (e.g., electric sounder, steel tape, transducer, reflection sounder, airline, etc.) or oil/water interface indicator (if necessary) selection per project planning documents;
- Appropriate keys for well cap locks;

- Organic vapor meter;
- Timepiece (preferably a stopwatch);
- Field data sheets;
- 5-gallon pails (graduated);
- Plastic sheeting; and
- Tool box (pipe wrenches, wire strippers, electrical tape, hose connectors, Teflon tape, sharp knife, etc., as needed depending on the application).

5.1 Groundwater Sampling Devices

See E & E Guidance 3.06, Groundwater Sampling Devices for detailed information.

General supplies needed for the groundwater sampling devices listed below include:

- Tubing of appropriate size, length, and construction if needed (enough to dedicate to each well);
- Gasoline, generator, or battery and appropriate power cable(s);
- Charger(s) for any battery-operated equipment;
- Winch, pulley, or electric reel (if desired); and
- Appropriately-sized hose barbs, connectors, nipples, and various pipe connectors.

Bailers

- Clean, decontaminated bailers (or disposable bailers) of appropriate size and construction material;
- Nylon or polypropylene line (enough to dedicate to each well); and
- Aluminum foil (to wrap clean bailers if not using disposable bailers).

Submersible Pumps

- Flow controller (if needed);
- Safety cable (i.e., heavy-grade nylon or polypropylene line); and
- Flow meter with gate valve.

Bladder Pumps

- Non-gas contact bladder pump;
- Spare bladder(s); and
- Compressor or compressed nitrogen gas.

Suction Pump (also called Peristaltic Pump)

- Soft, flexible tubing of appropriate size and length for use in peristaltic pump; and
- Flow meter with gate valve.

For low flow sampling, meters for measuring water quality parameters are required. Typically in-line flow-thru cell meters are used to measure water temperature, pH, electrical conductance, conductivity, dissolved oxygen, and oxygen reduction potential. A separate meter is used to

measure turbidity. Refer to the site specific project planning documents to determine which parameters are required prior to sampling. Supporting equipment and supplies also may be required to for sampling using the following:

- Field logbooks and supplies (Refer to project planning documents and the E & E Field Activity Logbooks SOP DOC 2.1 for details)
- Decontamination equipment and supplies (Refer to project planning documents and E & E Sampling Equipment Decontamination SOP ENV 3.15 for details)
- Sample containers, preservatives, and shipping equipment and supplies (Refer to project planning documents and the E & E Environmental Sample Handling, Packaging and Shipping SOP ENV 3.16 for details)
- Waste handling supplies (Refer to project planning documents and E & E Handling Investigation-Derived Wastes SOP ENV 3.26 for details).

6 Procedure

An overview of groundwater sampling procedures is provided in Figure 1. The primary goal of purging is to provide groundwater quality data that are representative of actual aquifer conditions with minimal waste generation caused by variable sampling techniques.

The general methods for groundwater sampling field activities can be reviewed using the American Society for Testing and Materials (ASTM) Standard Guide for Sampling Groundwater Monitoring Wells, D4448-01 (ASTM 2007), or the Florida Department of Environmental Protection SOP, FS 2200 Groundwater Sampling (FDEP 2008). The ASTM Standard Guide for Purging Methods for Wells Used for Groundwater Quality Investigations, D6452-99, can also be reviewed (ASTM 2005).

The methods for the low-flow procedure are included in the appropriate federal or state standards. If no standards are available, follow the United States Environmental Protection Agency (EPA) Region II Guidance document titled Groundwater Sampling Procedure, Low Stress (Low Flow) Purging and Sampling (EPA 1998).

All groundwater sampling is recorded on standard groundwater sampling forms. E & E's has a standard form developed for low-flow sampling, but standard forms are also available in many state and federal standard guidance. The appropriate sampling form should be selected and included with the project planning documents.

6.1 Sampling Preparation

- Start at the least-contaminated well, if known;
- Remove the locking well cap. Note the location of the well, time of day, and date in the field logbook or sample log;
- Remove the well cap covering the well riser;
- If possible, listen for indications of pressure or vacuum when opening the well riser cap;
- If required by the project planning documents, test the well for the presence of organic vapors using appropriate meter(s). Record all readings in the field logbook;
- Allow sufficient time for the water level to equilibrate in the well, to ensure that measurement of groundwater elevation is accurately representative;

- Measure depth to water and total depth of the well in accordance with E & E Measuring Water Level and Well Depth SOP 4.15, and record the measurements in the field logbook;
- Measure the diameter of the well, and calculate the volume of water in the well by multiplying the height of the water column by the appropriate volume per foot conversion factor (see Section 6.7);
- Determine the required volume of groundwater to be removed from the well (e.g., three well volumes or as indicated in the project planning documents);
- Place plastic sheeting on the ground around the well to minimize the likelihood of contamination of sampling equipment from soil adjacent to the well; and
- Prepare the purging and sampling equipment.

Special considerations for domestic well sampling:

- Visually assess the well system, from the well to the tap. Identify the most appropriate tap or spigot from which to sample. Do not touch the open sample bottle to the tap or spigot. Attempt to sample from as close to the well head as possible (before well treatment or softening, etc.). Avoid sampling the well directly (this may disturb the well, loosen rust, etc.). Avoid leaky faucets, sanitary or janitorial tubs, faucets near or below ground level, or other features that may compromise the water sample; and
- Remove any filters, aerators, screens, washers, or hoses from the faucet prior to sampling.

6.2 Purging

The amount of purging that a well receives prior to sample collection depends on the intent of the monitoring program, as well as the hydrogeologic conditions and how much pumping a well undergoes on a routine basis. Programs in which overall quality determinations of water resources are involved may require long pumping periods to obtain a sample that is representative of the groundwater. Refer to site specific project planning documents prior to purging to determine the amount of purging required and the water parameters that need to be measured.

Traditionally, a number of well casing volumes are removed (from three to five) and water stabilization parameters are monitored during removal of the casing volumes. For deeper wells, purging a well in this manner can generate a large volume of contaminated groundwater, which requires proper handling and disposal. In addition, the amount of time purging multiple casing volumes can often be excessive for sites with many wells. For a well that can be purged or bailed dry with the sampling equipment being used, the well should be evacuated and allowed to recover prior to sampling. When recovery is rapid, evacuation of more than one volume of water is recommended.

Low flow purging focuses on pumping a well from the well screen at a flow rate below the recharge capacity of the formation. The specific rate of pumping is generally aquifer dependent (typically less than 500 ml/min). By purging at low flow rates, only ground water that enters through the well screen is purged from the well. Because stagnant water located above the pump intake in the well casing is not drawn into the pump, the casing volume would not have to be purged from the well prior to sampling. The low flow purging approach can effectively reduce the volume of contaminated water generated during purging and the time spent performing the task. For a well that can be purged or bailed dry with the sampling equipment being used, the

well should be evacuated and allowed to recover prior to sampling. When recovery is rapid, evacuation of more than one volume of water is recommended.

Monitoring for defining a contaminant plume requires a representative sample of a small volume of the aquifer. These circumstances require that the well be pumped enough to remove the stagnant water, but not enough to induce flow from other areas.

During purging, water level measurements should be taken at regular intervals and recorded in the field logbook. The data may be used to compute water table or aquifer transmissivity and other hydraulic characteristics.

Information on the most commonly used groundwater purging and sampling devices can be found in E & E's Guidance 3.06, Groundwater Sampling Devices.

6.2.1 Bailers

In order to purge a well using a bailer the following equipment is needed: a clean decontaminated bailer (or disposable bailer); nylon or polypropylene line; a sharp knife; 5-gallon bucket to store the bailed water; and plastic sheeting. Place the plastic sheeting around the well to prevent contact of the bailer or line with the ground. Attach the line to the bailer, and then lower the bailer slowly (trying not to disturb the water) until it is completely submerged. Pull the bailer out of the well; ensuring that the line falls onto the plastic sheeting. Empty the bailer into a 5-gallon bucket. Repeat the procedure until the required purge volume has been removed (per the project planning documents).

6.2.2 Submersible Pumps

- Assemble the pump, hose, and safety cable;
- Lower the pump and assembly into the well to a point a few feet below the water level;
- Attach to a power source and commence purging operations;
- Using a flow meter or bucket and a stopwatch, determine the flow rate and calculate the time required to remove the required volume of water from the well;
- Place the purge water in 5-gallon bucket(s) or as indicated in the project planning documents; and
- Lower the pump by stages until it is in groundwater, and continue to purge until the required volume of water has been removed from the well. In cases where the well will not yield water at a sufficient recharge rate, pump the well dry and allow it to recover.

6.2.3 Non-Gas Contact Bladder Pumps

- Assemble tubing, pump, and compressor/control box;
- For control boxes using external power, connect power source;
- Procedures for purging with a bladder pump are the same as for a submersible pump (Section 6.2.2); and
- Be sure to adjust the flow rate to allow smooth intake and discharge cycles.

6.2.4 Suction Pumps

- Assemble the pump, tubing, and power source; and

- Procedures for purging with a suction pump are the same as for a submersible pump. (Section 6.2.2).

6.2.5 Domestic Wells

- For domestic wells that are consistently used (i.e., daily), extensive purging prior to sampling may not be needed. If the well has been used for normal domestic purposes within the previous 24 hours, this will perform most of the required purging. However, you will need to run water by opening the faucet or spigot at 1-2 gpm for approximately 10-15 minutes prior to taking a sample. This will clear the plumbing system and allow for a fresh water sample to be taken.

- Adjust the flow rate to minimize spikes or dips in flow pressure. Purging and sample collection should be from the cold water supply if given a choice between hot and cold water. Do not sample the hot water line. Do not sample after an in-line filter or after a treatment system, unless you want to determine how well the filter or treatment system is working. Do not touch the sample bottle(s) to the faucet or spigot.
- Monitor the water quality parameters (per the project planning documents). Purging is considered complete and sampling may begin when the water quality parameters have stabilized (see Section 6.2.6 Low Flow Purging for USEPA guidelines).

6.2.6 Low Flow Purging

- Turn on pump and collect the initial water discharged.
- Measure the initial water level.
- Begin purging the well and record the water parameters per the project planning documents on the groundwater sampling form.
- Purge the well using an initial flow rate of 100 to 500 mL/min; however, the flow rate should be adjusted to minimize drawdown to no more than 0.3 foot during purging and sampling. The water level should be monitored with a water level indicator at determined intervals.
- If 0.3-foot drawdown is exceeded and cannot be re-established, establishment of zero drawdown (i.e., water elevation stabilization at a constant or increasing level during purging) will be attempted. The decrease in water level greater than 0.3 foot is allowable as long as the water elevation stabilizes and remains stable or increases during the remainder of purging and sampling.
- Record the water quality parameters per the project planning documents at determined intervals or one quarter of a well volume until stabilization of all parameters is achieved. The purging will be considered complete after the field parameters have stabilized for three successive readings.
- The readings are considered stable when three successive readings are within the following USEPA guidelines or guidelines specific to the project:
    - +/-10 mV for ORP.
    - +/-0.1 for pH.
    - +/- 3% for specific conductivity and temperature.
    - +/- 10% for turbidity and DO.

- Once stabilized and turbidity is 50 NTUs or less, the groundwater sample will be collected.
- If turbidity is unstable (i.e., > 10%), but less than 50 NTUs, the groundwater sample will still be collected and the final turbidity will be recorded,
- If sample turbidity is greater than 50 NTUs, a second sample will be collected by attaching a disposable in-line filter to the end of the tubing and the filtered sample along with an unfiltered sample will be submitted to the laboratory for both dissolved and total (respectively) metals analyses. Refer to the project planning documents for site specific information on sampling.

6.3 Sampling

Groundwater samples can be obtained through the use of a number of groundwater sampling devices. Each groundwater sampling device has its advantages (and disadvantages) over other devices. Ideally, groundwater sampling devices should be completely inert, economical to manufacturer, easily cleaned for reuse, able to operate at remote sites in the absence of power sources, and capable of delivering variable rates for both well purging and sample collection. There are several other factors to consider when choosing a groundwater sampling device and care should be taken when selecting the device. Refer to E & E's Guidance 3.06, Groundwater Sampling Devices for additional information.

6.3.1 Bailers

- Make sure that clean plastic sheeting has been placed around the well;
- Attach a line to the bailer. If a bailer was used for purging, the same bailer and line may be used for sampling;
- Lower the bailer slowly and gently into the well, taking care not to shake the well casing or splash the bailer into the water. Lower the bailer to different points adjacent to the well screen to ensure that a representative water sample is collected;
- Slowly and gently retrieve the bailer from the well, minimizing contact with the well riser;
- Remove the cap from a sample container and place the cap on plastic sheeting or in a location where it will not be contaminated. Refer to Section 6.6 for special considerations for volatile organic analysis (VOA) samples;
- Slowly pour the water into the container;
- Filter and preserve samples as required by the project planning documents.
- Replace sample container cap;
- Prepare the necessary quality assurance samples as outlined in the planning documents;
- Record sample information in the field logbook or on field data sheets, and complete the chain-of-custody form;
- Package samples in accordance with the project planning documents; and
- Repeat this process until all groundwater samples have been collected.

6.3.2 Submersible Pumps

- Attach a gate valve to the discharge hose, and reduce the flow rate to one appropriate for sample collection;
- The VOC aliquot of the sample will be collected first followed any remaining aliquots. Pumping will be performed at a very slow rate to minimize volatilization and turbidity.
- Prepare the sample containers;
- If no gate valve is available, discharge the sample into a clean jar and fill the sample containers from the jar;
- Nonfiltered groundwater samples should be collected directly from the outlet tubing into the sample containers (refer to the project planning documents);
- Filtered groundwater samples should be obtained by connecting the pump outlet tubing directly to the filter unit (refer to the project planning documents);
- Complete the sampling and documentation procedures as outlined in Section 5.1; and
- Upon completion, remove the pump and assembly and properly decontaminate the pump prior to use in the next well. Do not reuse the discharge tubing in a separate well. If dedicated to a particular well, tubing may be left in place for future sampling events.

6.3.3 Bladder Pump

- Prepare the sample containers;
- Turn the pump on. Increase the cycle time and reduce the pressure to the minimum that will allow groundwater to come to the surface;
- Complete the sampling and documentation procedures as outlined in Section 5.1;
- Upon completion, remove the tubing from the well and either replace the Teflon tubing and bladder with new dedicated tubing and bladder, or properly decontaminate the existing material;
- Nonfiltered groundwater samples should be collected directly from the outlet tubing into the sample containers (refer to the project planning documents); and
- Filtered groundwater samples should be obtained by connecting the pump outlet tubing directly to the filter unit (refer to the project planning documents). The pump pressure should be reduced to prevent a pressure buildup on the filter, which could damage the pump bladder.

6.3.4 Suction Pumps

- Attach a gate valve to the discharge line if the suction pump discharge rate cannot be controlled, or discharge the sample into a clean glass jar and fill the sample containers from the jar;
- Complete the sampling and documentation procedures as outlined in Section 5.1; and
- Upon completion, remove the tubing and properly decontaminate the pump prior to use in the next well. Do not reuse the tubing in a separate well. If dedicated to a particular well, tubing may be left in place for future sampling events.

Low Flow

- Prepare the sample containers;
- Turn on pump and collect the initial water discharged;
- Measure the initial water level;
- Begin purging the well and record the water parameters per the project planning documents on the groundwater sampling form;
- Turn the pump on and follow the procedures in Section 6.2.6; and
- Upon completion, remove the tubing and properly decontaminate the pump prior to use in the next well. Do not reuse the tubing in a separate well. If dedicated to a particular well, tubing may be left in place for future sampling events.

6.3.5 Domestic Well Sampling

- Reduce flow rate to a smooth flowing water stream without splashing prior to sample collection. This step is especially important during sample collection for VOC analysis; and
- Complete the sampling and documentation procedures as outlined in Section 5.1.

6.4 Filtering

Samples being analyzed for total dissolved metals and/or other parameters may require filtering per the project planning documents. The most common type of filter is the in-line filter cartridges using a peristaltic pump. The in-line filter cartridges are attached to the end of the tubing prior to the sample entering the sample containers. Barrel filters and vacuum filters may also be used. A barrel filter works with a bicycle pump, which is used to build up positive pressure in the chamber containing the sample. Water is then forced through 0.45-µm filter paper into a jar. The barrel itself is filled manually.

A vacuum filter involves two chambers: the upper chamber contains the sample, and a 0.45-µm filter divides the two chambers. Using a portable vacuum pump, air is withdrawn from the lower chamber, creating a vacuum, which causes the sample to move through the filter into the lower chamber. Repeated pumping may be required to drain all of the sample into the lower chamber. If preservation of the samples is necessary, this should be done after filtering.

6.5 Post Operation

After all samples have been collected and preserved, the sampling equipment should be properly decontaminated to prevent cross-contamination of samples.

- Decontaminate all equipment according to the planning documents;
- Replace sampling equipment in storage containers;
- Prepare groundwater samples for shipment. Check sample documentation and make sure samples are properly packed for shipment; and
- Organize field notes into a report format and transfer logging information to appropriate forms.

6.6 Special Consideration for VOA Sampling

The proper collection of a sample for dissolved VOCs requires minimal disturbance of the sample to limit volatilization and subsequent loss of volatiles from the sample.

Sample retrieval systems suitable for the valid collection of volatile organic samples include: positive-displacement bladder pumps, submersible pumps, and bailers. Field conditions and other constraints will limit the choice of appropriate systems. The principal objective is to provide a valid sample for analysis that has been subjected to the least amount of turbulence possible.

The following procedures should be followed when collecting VOA samples:

- Open the vial and set the cap in a clean place. Determine if the container(s) contains preservative, are pre-preserved or if the proper amount of preservative needs to be added (refer to the project planning documents);
- Fill the vial to the top until a convex meniscus forms on the top of the vial. Do not overfill the vial;
- Check that the cap has not been contaminated, and carefully cap the vial. Place the cap directly over the top and screw down firmly. Do not overtighten and break the cap;
- Invert the vial and tap gently. If an air bubble appears, discard the sample and begin again. It is imperative that no entrapped air remains in the sample vial;
- Place the VOA vial in a cooler; and
- Refer to the project planning documents for the sample holding time. It is recommended that samples be shipped or delivered to the laboratory daily. Ensure that the samples remain at 4 degrees Celsius, but do not allow them to freeze.

6.7 Calculations

Table 1 presents the volume of water in different size casings and holes. To determine the volume of water in a well, the calculations are as follows:

$$V = \pi r^2 h$$

Where:

V = Static well volume

h = Height of water in well

r = Inside radius of well casing

- e.g., If V will be in gallons, h measured in feet, and r measured in inches, then the formula becomes:

$$V = r^2 h\, C$$

C = Constant depends on units of measurement (see Table 1).

Table 1 Volume of Water in Casing or Hole

Diameter of Casing or Hole (in)	Gallons per Foot of Depth	Cubic Feet per Foot of Depth	Liter per Meter of Depth	Cubic Meters per Meter of Depth
1	0.041	0.0055	0.509	0.509×10^{-3}
1.5	0.092	0.0123	1.142	1.142×10^{-3}
2	0.163	0.0218	2.024	2.024×10^{-3}
2.5	0.255	0.0341	3.167	3.167×10^{-3}
3	0.367	0.0491	4.558	4.558×10^{-3}
3.5	0.500	0.0668	6.209	6.209×10^{-3}
4	0.653	0.0873	8.110	8.110×10^{-3}
6	1.469	0.1963	18.240	18.240×10^{-3}
8	2.611	0.3491	32.430	32.430×10^{-3}
12	5.875	0.7854	72.960	72.960×10^{-3}
24	23.500	3.1420	291.850	291.850×10^{-3}
36	52.880	7.0690	656.720	656.720×10^{-3}

1 Gallon = 3.785 liters
1 Meter = 3.281 feet
1 Gallon water weighs 8.33 pounds = 3.785 kilograms
1 Liter water weighs 1 kilogram = 2.205 pounds
1 Gallon per foot of depth = 12.419 liters per foot of depth
1 Gallon per meter of depth = 12.319×10^{3} cubic meters per meter of depth

7 Quality Assurance/Quality Control

Prior to initiating field work, the project planning documents should be reviewed by field personnel to identify sampling procedure(s) that will most likely provide sediment samples that meet project DQOs.

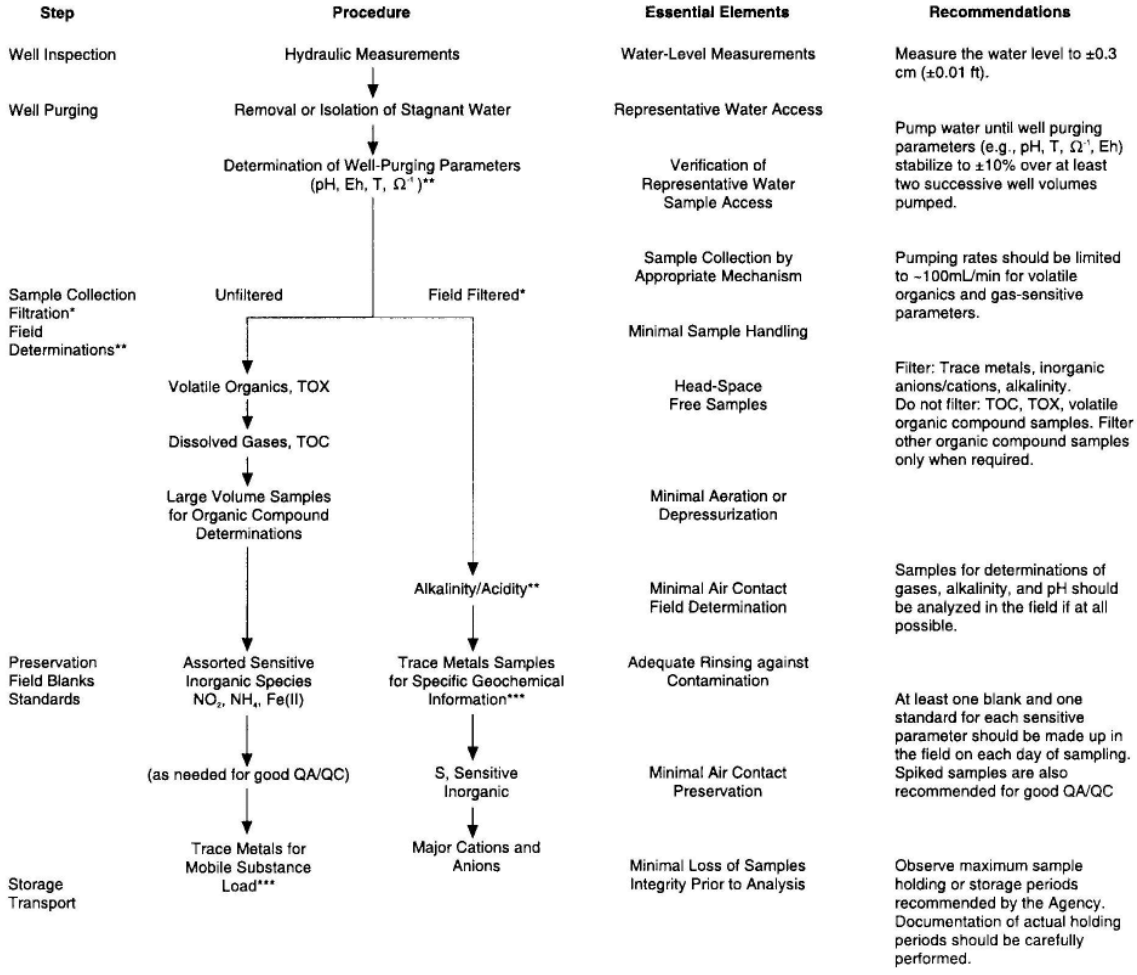
The program/project manager should identify personnel for the field team who have knowledge, training and experience in the groundwater sampling activities being conducted. One member of the field team should be designated as the lead for groundwater sampling and will be responsible, with support from other field personnel, for implementing the procedures in this SOP. The program/project manager should also identify additional personnel, if necessary, to complete ancillary procedures (e.g., field logbook documentation, equipment decontamination, sample shipment, and waste disposal).

The groundwater sampling lead should prepare a detailed equipment checklist before entering the field and verify that sufficient and appropriate equipment and supplies are taken into the field.

Collecting representative groundwater samples is an important quality consideration. The areas should be addressed during implementation of the sampling procedures:

- Log documentation should be reviewed to determine whether the required volume of purge water was removed from the well and that the water quality parameters (per the project planning documents) had been stabilized to ensure that a representative water sample of the aquifer was obtained;
- The purging and sampling devices should be made of materials and utilized in a manner that will not interact with or alter the analysis;
- The results generated by these procedures are reproducible as demonstrated through the use of duplicate samples (should be specified in the project planning documents); and

- The possibility of cross-contamination is reduced by collecting samples from the least contaminated well first. Rinsate blanks should be incorporated where dedicated sampling and purging equipment is not utilized and decontamination of the equipment between sampling events is required.



* Denotes samples that should be filtered to determine dissolved constituents. Filtration should be accomplished preferably with in-line filters and pump pressure or by N_2 pressure methods. Samples for dissolved gases or volatile organics should not be filtered. In instances where well development procedures do not allow for turbidity-free samples and may bias analytical results, split samples should be spiked with standards before filtration. Both spiked samples and regular samples should be analyzed to determine recoveries from both types of handling.

** Denotes analytical determinations that should be made in the field.

Figure 1 Generalized Flow Diagram of Groundwater Sampling Protocol

8 Health and Safety

Prior to entering the field, all field personnel should formally acknowledge that they have read and understand the project specific health and safety plan.

Standard safe operating practices should be followed, such as minimizing contact with potential contaminants in both the vapor phase and liquid matrix through the use of respirators and protective clothing.

9 Special Project Requirements

Project or program-specific requirements that modify this procedure should be entered in this section and included with the project planning documents.

10 References

American Society for Testing and Material (ASTM), 2007, *Standard Guide for Sampling Groundwater Monitoring Wells*, D4448 – 01, ASTM International, West Conshohocken, PA, www.astm.org.

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Florida Department of Environmental Protection (FDEP). 2008. Standard Operating Procedures for Field Activities. DEP-SOP-001/01. December 2008. Online at http://www.dep.state.fl.us/labs/qa/sops.htm. FS 2200 Groundwater Sampling

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END OF SOP

ecology and environment, inc.

STANDARD OPERATING PROCEDURE

ENVIRONMENTAL SAMPLE HANDLING, PACKAGING AND SHIPPING

SOP NUMBER: ENV 3.16

REVISION DATE: 5/24/2012 **SCHEDULED REVIEW DATE: 5/24/2017**

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1 Scope and Application

This Standard Operating Procedure (SOP) describes the packing, marking, labeling, and shipping procedures routinely used by E & E field personnel to transfer environmental samples from the field to off-site laboratories. Unpreserved and/or properly preserved environmental samples include the following matrices:

- Drinking water;
- Groundwater;
- Surface water;
- Soil;
- Sediment;
- Treated municipal and industrial effluent;
- Biological specimens (i.e., non-pathogenic plant and/or animal tissue); or
- Samples not expected to be contaminated with high levels of hazardous substances.

Shipping includes transport by air, rail, or motor vehicle.

Samples containing known or suspected International Air Transport Authority (IATA)-defined dangerous goods and/or United States Department of Transportation (DOT)-defined hazardous materials or which have anesthetic, noxious, or other properties that could inhibit the abilities of transporters do not meet the criteria for shipping as "environmental" samples.

This environmental sample packaging and shipping SOP is intended for use by personnel who have knowledge, training, and experience in the procedures described herein and who have received training on E & E's On-line Hazardous Materials/Dangerous Goods Shipping Guidance Manual. Regional Hazardous Materials Transportation Coordinators (RHTCs) are available to provide technical support for environmental sample shipping.

In the event the sample material meets the established criteria of a DOT hazardous material, consult one of the RHTC personnel and follow guidelines in E & E's Hazardous Materials/Dangerous Goods Shipping Guidance Manual (see http://www.corp.ene.com/departments/health_&_safety/shipping_manual.asp).

2 Definitions and Acronyms

^{o}C	degrees Celsius
COC	Chain-of-Custody
DNAPL	dense non-aqueous phase liquid
DOT	(United States) Department of Transportation
DQO	Data Quality Objective
EPA	United States Environmental Protection Agency
IATA	International Air Transport Authority
LNAPL	light non-aqueous phase liquid
RHTC	Regional Hazardous Materials Transportation Coordinator

SHASP	Site-specific Health and Safety Plan
SOP	Standard Operating Procedure
UN	United Nations
VOA	volatile organic analysis

3 Procedure Summary

Sample packaging, marking, labeling and shipping procedures vary depending on the data quality objectives (DQOs) identified in the program/project planning documents (e.g., work plan, sampling and analysis plan, quality assurance project plan, SOPs, and site-specific health and safety plan [SHASP]). These documents address the types and degrees of contamination anticipated and identify appropriate shipping and handling procedures.

Properly identified, preserved, and sealed individual sample bottles/jars provided by field samplers are sealed in plastic bags and placed in lined shipping containers. Packing material (e.g., bubble wrap) is used reduce the risk of damage to sample bottles/jars and loss of samples during transport. Absorbent material (e.g., highly absorbent small animal bedding material made from recycled paper/wood waste) is added to the shipping container to contain spills from sample bottles/jars during transport. Double-bagged ice is added to the shipping containers as a preservative. Chain-of-custody (COC) documents are prepared and enclosed in the shipping containers. Shipping containers are marked in compliance with DOT/IATA regulations. Shipping papers (e.g., Federal Express shipping documents) are completed and attached to the shipping containers. Shipping containers are custody sealed and taped. Clients, program/project managers, shippers and laboratories already scheduled to receive samples are notified daily of impending shipments.

4 Cautions

Samples collected from sources, such as waste lagoons, drums, tanks, heavily stained soils, and groundwater contaminated with LNAPL or DNAPL, do not qualify as environmental samples.

Known or suspected samples of IATA-defined dangerous goods and/or DOT-defined hazardous materials do not meet the criteria for shipping as “environmental” samples.

Shipping of IATA dangerous goods and/or DOT hazardous materials is not covered by this SOP. Guidance on shipping dangerous goods and hazardous materials is presented in E & E’s Hazardous Materials/Dangerous Goods Shipping Guidance Manual (see http://www.corp.ene.com/departments/health_&_safety/shipping_manual.asp).

Samples preserved in accordance with United States Environmental Protection Agency (EPA) Contract Laboratory Program guidance (most current version) are routinely shipped as environmental samples.

A RHTC should be consulted prior to any biological specimen shipping.

Transboundary/International shipping requirements are presented in program/project planning documents.

Samples preserved with methanol are not shipped as environmental samples. DOT/IATA regulations apply to the shipment of methanol preseverved samples.

Individual sample bottle/jar labels are the responsibility of the field samplers who verify that labels are complete and correct, and match the COC forms prior to shipment to laboratories.

Known or suspected PCB and dioxin samples require additional packaging (i.e., sealing in metal cans) and are not covered by this environmental sample packaging and shipping SOP.

It is E & E's intent to package samples so securely to prevent leakage during shipment. This is to prevent the loss of samples and the expenditure of funds for emergency responses to spills and the efforts necessary to re-obtain the sample. Liquid samples are particularly vulnerable. Because transporters (carriers) are not able to know the difference between a package leaking distilled water and a package leaking a hazardous chemical, they will react to a spill in an emergency fashion, potentially causing enormous expense to E & E for the cleanup of the sample material. Therefore, liquids are to be packed in plastic bags and absorbent/cushioning material to help prevent possibility of leaks from a package.

5 Equipment and Supplies

Coolers, sample bottles/jars, COC forms, and sample labels are typically supplied by the laboratory.

Federal Express or other shippers provide shipping forms.

Packaging material, such as plastic bags, ice, and absorbent material, are purchased locally.

E & E-purchased durable packaging equipment, such as coolers, are labeled with the applicable E & E office (or, in some cases, field office) address.

6 Procedure

6.1 Prior to Field Activity

- Program/project managers or designated personnel utilize the project planning documents to stage the equipment and supplies required to meet project DQOs.
- Labeled temperature blanks, tap water filled 40-mL volatile organic analysis (VOA) vials, are prepared for use in the field.
- The project manager or designee arranges for shipper support and coordinates with the laboratory(ies) necessary to conduct the tests needed to meet project DQOs.

6.2 Field Sampler Support

Field samplers collect samples in accordance with the program/project planning documents and provide properly identified, preserved, and sealed individual sample bottles/jars to the field personnel responsible for sample packaging, marking, labeling, and shipping.

6.3 Environmental Sample Packaging Procedures

Environmental samples are usually shipped in 80-quart solid outer shell plastic or metal coolers (although other size coolers may be used if they meet program/project needs). Disposable, pressed Styrofoam coolers are not used. Before use, shipping cooler drain holes are sealed to prevent leakage. Non-applicable labels are removed from the cooler. Marking, Labeling, and Shipping procedures are presented in Section 6.4 of this SOP.

The following steps are used for routine packaging:

- Verify that the bottle is clean and labeled;
- Verify the caps are secure cap and if necessary use fiber reinforced tape;

- Seal each sample bottle and temperature blank in a sealable plastic bag; and
- Add one temperature blank to each cooler.

When a precut foam block insert is used to prevent sample bottle breakage during shipping:

- Verify cooler has this side up labels/arrows;
- Place at least 1 inch of inert absorbent material in the bottom of the cooler;
- Line the cooler with two double-bagged plastic (e.g., large heavy-duty garbage) bags;
- Place a foam insert (with holes cut to receive the sample bottles) inside the plastic bag;
- Place the bottles upright in the holes in the foam block;
- Fill void spaces with double-bagged ice to the top of the cooler;
- Seal each plastic bag lining the cooler with tape;
- Place a COC form in a waterproof, sealable bag taped to the inside of the cooler lid;
- Place custody seals over top edge of cooler so cooler cannot be opened without braking seals;
- Cover the custody seals with clear tape; and
- Secure the cooler with strapping tape over the hinges and around the entire cooler.

When bubble wrap or similar packing is used to prevent sample bottle breakage during shipping:

- Verify cooler has this side up labels/arrows,
- Place at least 1 inch of inert absorbent material in the bottom of the cooler,
- Line the cooler with two double-bagged plastic (e.g., large heavy-duty garbage) bags,
- Surround each bottle/jar (including the bottom) with bubble wrap, taping the wrap securely around the bottle,
- Place the bottles upright in the inner bag,
- Fill void spaces with double-bagged ice to the top of the cooler,
- Seal each plastic bag lining the cooler with tape,
- Place a COC form in a waterproof, sealable bag taped to the inside of the cooler lid, and
- Place custody seals over top edge of cooler so cooler cannot be opened without breaking seals;
- Cover the custody seals with clear tape; and
- Secure the cooler with strapping tape over the hinges and around the entire cooler.

When only absorbent material is used to prevent sample bottle breakage during shipping:

- Place at least 1 inch of inert absorbent material in the bottom of the cooler;
- Line the cooler with two double-bagged plastic (e.g., large heavy-duty garbage) bags;
- Place at least 1 inch of inert absorbent material in the bottom of the inner bag;

- Place each sample bottle upright inside the inner bag maintaining at least 3 inches between bottles;
- Fill the void spaces around the bottles with absorbent to at least half the height of the largest bottles;
- Fill void spaces with double-bagged ice to the top of the cooler;
- Seal each plastic bag lining the cooler with tape;
- Place a COC form in a waterproof, sealable bag taped to the inside of the cooler lid;
- Place custody seals over top edge of cooler so the cooler cannot be opened without braking the seals;
- Cover the custody seals with clear tape; and
- Secure the cooler with strapping tape over the hinges and around the entire cooler.

6.4 Marking, Labeling and Shipping Procedures

Program/project planning documents provide the information necessary to initiate filling out the COC forms. Additional information is available in the site field logbook(s).

Environmental samples are shipped as nonhazardous cargo.

Outer marking and labeling on each container is compliant with requirements for the carrier that will be used requirements. Coolers have this side up or arrow labels affixed. Extraneous markings are removed.

Markings indicating ownership of the container, destination, and shipping company labels are acceptable and attached as required.

Hazardous materials/dangerous goods airbills are not used when shipping environmental samples.

Environmental sample packages generally shipped overnight by Federal Express or equivalent. Field personnel check with shippers in advance to verify both pick-up and delivery schedules; especially when weekend and/or holiday pick-up and/or delivery may be required.

7 Quality Assurance/Quality Control

Hazardous Materials/Dangerous Goods Shipping training is provided to personnel responsible for shipping environmental samples. RHTCs are available to provide technical support for environmental sample shipping.

COC forms may be completed electronically or by hand. Samples recorded on the COC form are checked against the packaged samples.

Custody seals are attached to shipping containers so the receiving laboratory may verify the temperature of the samples.

Field samplers and shipping personnel verify the samples in the cooler and the samples listed on the COC match.

Site-identifying information is not listed on samples, forms, or other documents and is not provided to the receiving laboratory(ies).

Clients, program/project managers, shippers, and laboratories already scheduled to receive samples are notified daily of impending shipments. E & E personnel verify shipping addresses and confirm the receiving facility's commitment to accept samples based on shipment dates.

Samples shipped on ice require preservation to to 4°C (±2°C). Samples that arrived at the laboratory outside this range could have compromised data quality. Samples should be cooled prior to packaging and sufficient ice used to keep samples cool particularly in warm weather. If samples are being shipped for Saturday or holiday delivery, then the availability of personnel should be verified with the laboratory and the shipping documentation checked to verify the appropriate delivery date is noted. Always confirm delivery of the samples with the shipper.

8 Health and Safety

Prior to entering the field, personnel will formally acknowledge that they have read and understand the project specific health and safety plan (SHASP).

Preserved samples (e.g., samples containing acids, solvents, and formalin) will be handled in accordance with the SHASP.

Good basic lifting and handling procedures will be followed when handling filled coolers.

9 Special Project Requirements

Special project requirements may be found in the program/project planning documents.

10 References

U.S. Environmental Protection Agency (EPA). 2011. Office of Superfund Remediation and Technology Innovation, *Contract Laboratory Program Guidance for Field Samplers*, OSWER 9240.0-47 EPA 540-R-09-03, January 2011. Accessed online at: http://www.epa.gov/superfund/programs/clp/download/sampler/CLPSamp-01-2011.pdf.

END OF SOP

ecology and environment, inc.

STANDARD OPERATING PROCEDURE

SAMPLING EQUIPMENT DECONTAMINATION

SOP NUMBER: ENV 3.15

REVISION DATE: 5/24/2012 **SCHEDULED REVIEW DATE: 5/24/2017**

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1 Scope and Application

This Standard Operating Procedure (SOP) describes the routine procedures utilized by E & E personnel in the field for decontaminating sampling equipment that is not dedicated or disposal and that may have come into contact with site contaminants. It is applicable for equipment that will be re-used in the field and for equipment that will be returned to a warehouse or other storage facility prior to re-use.

Program/project specific data quality objectives (DQOs) dictate the types of sampling equipment requiring decontamination and site-specific sampling procedures should be identified in program/project planning documents. This SOP applies to equipment routinely used for:

- Water quality sampling (e.g., buckets, bailers, Kemmerers, and Niskins);
- Flow/water depth measuring (e.g., velocity meters, stream gauges, and depth sounders);
- Soil and sediment sampling (e.g., corers, augers, Van Veens, direct-push samplers, homogenization buckets, and mixing tools); and
- Miscellaneous tools (e.g., shovels, scoops, tapes/rulers/meter sticks, and cutting tools).

Decontamination is time consummng and expensive, often including analyses of field rinsates and other “blanks” to verify decontamination procedures provide equipment that meet program/project DQOs. The use of clean, dedicated, disposable equipment (e.g., Teflon or plastic bailers for groundwater sampling, aluminum bowls for soil homogenization) is preferred, whenever practicable.

This sampling equipment decontamination SOP is intended for use by personnel who have knowledge, training, and experience in the field sampling activities being conducted and who understand the importance of decontamination in meeting program/project-specific DQOs.

The SOP does not address personnel decontamination. As part of the health and safety plan, a personnel decontamination plan should be developed and set up before any personnel or equipment enters the areas of potential contamination.

2 Definitions and Acronyms

`ASTM American Society for Testing and Materials

De-ionized water Purified water produced by distillation or by filtration through de-ioniizning columns or other means (e.g., reverse osmosis) or some combination of treatments. Program/project DQOs establish the level of purity required (e.g., maximum level of electrical conductivity)

`DQO Data quality objective

Potable water Tap water from a treated drinking water supply

`SHASP Site-specific Health and Safety Plan

`SOP Standard Operating Procedure

`USEPA United States Environmental Protection Agency

3 Procedure Summary

Sampling equipment decontamination procedures vary depending on the DQOs identified in the program/project planning documents . These documents address the types and degrees of contamination anticipated and indentify appropriate decontamination procedures, materials, and wastes handling.

A decontamination line is set up in the contamination reduction zone, outside of the contamination "hot" zone, where personnel follow a multi-step decontamination procedure. If a formal decontamination line is established for the site, then all equipment decontamination must be completed with the "hot" zone.

This procedure can be expanded to include additional or alternate wash/rinse steps designed to remove specific target analytes/compounds, if required by site-specific work plans or as directed by a particular client.

4 Cautions

Decontamination of sampling equipment left in situ for long periods (e.g., groundwater pumps, stack samplers, continuous flow samplers) is addressed in program/project-specific planning docments.

Sites with biohazards are not considered routine operations. Biohazard site sampling equipment decontamination is addressed site-specific program/project planning documents.

Sites with explosive hazards are not considered routine operations. Explosives site sampling equipment decontamination is addressed in site-specific program/project planning documents.

Sites requiring ultra-clean sampling methods (e.g., United States Environmental Protection Agency [USEPA] Method 1669) require ultra-clean sampling equipment decontamination. Ultra-clean sampling equipment decontamination is addressed in site-specific program/project planning documents.

Decontamination of contaminated or potentially contaminated sampling equipment may generate incompatable hazardous wastes. Only compatable waste streams, as defined in the program/project planning documents are combined for disposal.

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. Analyte-free deionized water is can be obtained from the project analytical laboratories if available. Distilled water available from local grocery stores and pharmacies is generally not acceptable for final decontamination rinses. Contaminant-free deionized water is that has been stored on site should not be used without testing. Any new source of water should be tested prior to use if not certified by a vendor or laboratory.

In general, use of solvents is avoided for low level environmental analysis, but may be necessary for more contaminated areas.

5 Equipment and Supplies

Planning documents provide direction on the specific equipment and supplies, and the numbers/volumes required to meet program/project-specific DQOs. The following equipment and supplies are used for routine sampling equipment decontamination:

- Appropriate protective clothing (including safety glasses or splash shield and nitrile gloves);
- Galvanized or similar wash basins;
- Waste collection drums (if required) ;
- Plastic buckets (5-gallon);
- Long-handled brushes;
- Spray/squeeze bottles;
- Non-phosphate detergent (e.g., Liquinox™ or Alconox™);
- Pesticide grade (or equivalent) organic solvents (e.g., methanol, hexane, or other as specified in the planning documents.) if necessary based on the contaminants
- Ten percent, by volume in de-ionized water, nitric acid (ultrapure);
- Tap water;
- Deionized water (usually American Society for Testing and Materials [ASTM] Type II);
- Organic-free water;
- Plastic sheeting for ground cover;
- Paper towels;
- Trash bags;
- Aluminum foil; and
- Waste handling supplies. (Refer to project planning documents and E & E Investigation-Derived Waste SOP for details.)

Note all waters, acids and detergents should be are stored in their original containers or clearly marked clean sealable glass, plastic, or Teflon® bottles in which information from the original label has been transferred. The secondary labeling should include reagent name, source, date opened/transferred, and expiration date as well as any hazardous labels.

6 Procedures

Before entering the field personnel reviews relevant program/project planning documents (e.g., work plan, sampling and analysis plan, quality assurance project plan, health and safety plan);and select the sampling equipment decontamination procedures (e.g., organic solvent[s] to be used) that meet project DQOs.

In the field personnel should follow best practices to minimize contamination of equipment and prevent cross contamination of cleaned equipment.

- Set-up a zone that isolates areas of contamination from clean areas of the site. All equipment should be decontaminated within the contamination area.
- Employing work practices that minimize contact with hazardous or toxic substances (e.g., avoid areas of obvious contamination, avoid touching potentially contaminated materials);
- 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.

6.1 Decontamination Methods for Direct Sample Contact Equipment

Field personnel should set-up a decontamination line that moves contaminated equipment through the decontamination process to a clean zone. At all stations in the decontamination line, contaminated and/or potentially contaminated fluids and/or wastes are collected and containerized.

Routine decontamination steps for equipment that directly contacts samples are described below.

1. Physically remove gross contamination from equipment by abrasive scraping and/or brushing.
2. Wash equipment with non-phosphate detergent (i.e., Alconox™ or Liquinox™) in tap water.
3. Rinse with tap water
4. Rinse with de-ionized water.
5. Rinse with 10% nitric acid, if specified in planning documents. Nitric acid washes are typically used for metals contamination.
6. Rinse with de-ionized water (if the acid rinse is conducted).
7. Rinse with organic solvent(s) to remove high levels of organic contamination, refer to the planning documents for the site/activity-specific solvent choice.

   Use a methanol rinse to dissolve and remove soluble organic contaminants for high concentration samples.

   Use a hexane rinse to dissolve waste lubricating oils, tars, and bunker fuels for high concentration samples.
8. Air drying
9. Rinse with deionized, organic-free water, usually only if alternative solvents are used.
10. Wrap sampling equipment in aluminum foil or plastic ; if it will not be used immediately. Determine the best material to wrap equipment based on site contaminants for example plastic bags should not be used is sampling for volatile and extractable organics.

11. Containerize all solvent rinsing wastes, detergent wastes and other chemical wastes requiring off-site or regulated disposal. Dispose of all wastes in conformance with applicable regulations as defined in the project planning documents.

6.2 Decontamination Methods for Other Equipment and Meters

Several types of sampling equipment such as meters, pumps and tubing that cannot be cleaned directly as described in 6.1. Consult the manufacturers guidelines before decontaminating and equipment.

General decontamination steps are described below.

1. Physically remove visible contamination from equipment by brushing the outside of the equipment or wiping with paper towel.
2. If tubing or other portions of the equipment comes into contact with the sample then pump any decontamination solvents through the equipment.
3. Rinse/or pump with tap water
4. Rinse/or pump with de-ionized water.
5. Air dry
6. Wrap sampling equipment in aluminum foil or plastic ; if it will not be used immediately. Determine the best material to wrap equipment based on site contaminants.

6.3 Decontamination Methods for Heavy Equipment

For heavy equipment, a decontamination pad should be established by the driller or subcontractor. Heavy sampling equipment (e.g., augers) decontamination may include a steam cleaning and/or high-pressure water wash step after gross contamination is removed by detergent and brushing.

7 Quality Assurance/Quality Control

Program/project planning documents define the quality assurance/quality control procedures (e.g., collection and analysis of equipment rinsate and other “blanks”) necessary to meet program/project DQOs. Typically, a field blank (equipment rinsate blank) consists of a sample of analyte-free water passed through/over a decontaminated sampling device to assess possible cross contamination from equipment to sample contamination.

8 Health and Safety

Personnel review and acknowledge that they understand the project planning documents, especially the SHASP prior to entering the field. Material Safety Data Sheetss are taken into the field for hazardous materials used at a site.

Some types of sampling equipment are inherently dangerous pieces of heavy equipment with high pinch or crush potential. Proper handling procedures are followed during decontamination of heavy equipment.

Decontamination procedures may pose hazards, especially when chemical decontamination procedures, high pressure, and/or steam are used. Exposure to hazardous materials or wastes is controlled by the use of appropriate personal protective equipment and proper handling and storage of the materials/wastes, as specified in the project planning documents, especially the SHASP.

Steam cleaning - follow equipment manufacturer operating and safety guidelines.

High-pressure water cleaning - follow equipment manufacturer operating and safety guidelines.

Waste collection and disposal procedures are presented in program/project planning documents and E & E Investigation-Derived Waste SOP.

Avoiding practices that increase tendencies for hand-to-mouth contact including: eating, drinking, smoking, or using chewing tobacco is a basic procedure employed during all field activities.

9 9 Special Project Requirements

Special project requirements are presented in the program/project planning documents. If required, contract or other client-specific, site-specific requirements may be entered in this section.

10 References

The following list sources of technical information on decontamination procedures.

ASTM D 5088 – 02 Standard Practice for Decontamination of Field Equipment Used at Waste Sites, 2008

USEPA Environmental Response Team “Sampling Equipment Decontamination”, SOP #: 2006, REV.#:0.0, 08/11/94

USEPA Environmental Investigations Standard Operating Procedures and Quality Assurance Manual, Region 4, November 2001

USEPA Region IV, Field Equipment Cleaning and Decontamination, SESDPROC-205-R2, December 20, 2011

Navy Environmental Compliance Sampling and Field Testing Procedures Manual, NAVSEA T0300-AZ-PRO-010

END OF SOP

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STANDARD OPERATING PROCEDURE

SURFACE and SHALLOW SUBSURFACE SOIL SAMPLING

SOP NUMBER: ENV 3.13

REVISION DATE: 5/24/2012 **SCHEDULED REVIEW DATE: 5/25/2017**

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1 Scope and Application

This Standard Operating Procedure (SOP) describes the procedures utilized by E & E for collecting surface and shallow subsurface environmental soil samples. The purpose of soil sampling may range from simple reconnaissance to complex sampling programs. This SOP can be followed for all routine sample collection activities which may include: visual or other observations, in situ or ex situ field measurements (monitoring), or sample collection for biological, chemical, geological, radiological or physical analysis. Site-specific sampling procedures vary depending on the data quality objectives (DQOs) identified in program/project planning documents.

E & E routinely utilizes three types of surface and shallow subsurface environmental soil collection procedures, hand scoop, hand coring, and hand auger. Powered hand augers are sometimes used and the procedure is addressed in this SOP. The definition of the depth of a "surface" soil sample is dependent on the program/project specific DQOs); and may be driven by regulatory, risk-based or other considerations. Hand sampling is generally limited to no more than three feet (one meter) below ground surface. The site-specific depth interval of soil collection is identified in the project planning documents.

Procedures for collecting soil samples for volatile organic compound (VOC) analyses are presented in the E & E VOC Soil and Sediment Sampling SOP ENV 25.

Procedures for collecting "deeper" subsurface soil samples (using back hoes, drill rigs and direct push equipment) are presented in the E & E Borehole Installation Methods SOP GEO 4.7.

Procedures for sample handling are defined in E & E Environmental Sample Handling, Packaging and Shipping SOP ENV 3.16. Site-specific sample handling procedures are dependent on the project DQOs.

Procedures for equipment decontamination are defined in E & E Sampling Equipment Decontamination SOP ENV 3.15. Site-specific equipment decontamination procedures are dependent on the project DQOs.

This surface and shallow subsurface soil sampling SOP is intended for use by personnel who have knowledge, training and experience in the field soil sampling activities being conducted.

2 Definitions and Acronyms

cm	centimeter
DQO	Data Quality Objective
E & E	Ecology and Environment, Inc.
SHASP	Site Specific Health and Safety Plan
SOP	Standard Operating Procedure
VOC	Volatile Organic Compound

3 Procedure Summary

Pre-cleaned spoons, trowels, or other types of scoops are used to collect shallow (usually less than 6 inches [15 cm] deep) soil samples using a hand scoop procedure. Shallow subsurface soil is collected manually using scoops from the sides of hand dug excavations. Pre-cleaned hand soil core samplers and/or bucket augers are used for collecting relatively undisturbed shallow (usually no deeper than 3 feet [1 meter]) subsurface soil samples. The corer barrel/bucket auger is advanced into the soil to the pre-determined depth identified in the project planning documents. In some cases, corers may include a liner on the interior of the core barrel. Soil cores may be sectioned to provide vertical profiles of soil characteristics.

Disturbed soil samples are collected directly from the auger when continuous flight (screw) augers are used

Unless otherwise specified, surface soil scoop aliquots are combined, homogenized and then placed in appropriate sample containers. Volatile organic and sulfide samples are collected immediately after sample retrieval, regardless of the sampling procedure used. VOC samples are not homogenized (see E & E VOC Soil and Sediment Sampling SOP ENV 25) If multiple samples are required to provide the sample volume identified in the project planning documents, then samples are thoroughly homogenized prior to collection of aliquots for testing.

4 Cautions

This SOP is applicable to routine E & E surface and shallow subsurface soil sampling and is limited to relatively shallow soil sampling depths Hand augers and corers used in this SOP are generally effective only to a maximum depth of 3 feet (1 meter) below the soil surface. The depth of sample collection will be limited if soil is sandy, clayey or rocky. Grass, roots, or other natural or anthropogenic materials may not be considered part to the soil sample.

Because the sampling devices specified within this SOP provide limited sample volumes, multiple samples may be required to collect sufficient volume for sample analysis. Samples from multiple locations also may be collected and composited to provide a sample representative of a larger area. Sample compositing and homogenization should be addressed in the project planning documents. If a compositing scheme is employed and an area(s) is not visually consistent with other areas, then observations should be noted in the field log and a course of action determined based on the program/project DQOs. Samples for volatile organics, sulfide, or similar analyses are normally collected as discrete aliquots and should be containerized as soon as possible after collection and prior to compositing and homogenization. Field personnel must maintain an awareness of the soil sample volume collected versus the volume required to meet program/project DQOs.

Maintaining sample integrity requires selecting a soil sampling device and procedure that meets project DQOs. Carefully following procedures minimizes the disruption of the soil structure and subsequent changes in physiochemical and biological characteristics.

Continuous flight augers are satisfactory for use when a composite of the soil column is desired.

If a powered auger is used, if possible, position the power unit downwind of the sample location to avoid fumes from fuel used to power the unit.

At sites with known or suspected contamination, based on the data available, samples are collected moving from least to most contaminated soil.

Re-use of equipment may be unavoidable given size and cost. Decontamination matched to DQOs is specified in the project planning documents.

Experience has shown that real-world conditions (e.g., variable soil conditions such as the presence of rocks or trash) may lead to unacceptable soil sample recoveries and multiple attempts to collect soil samples will be required at some locations.

Abandon auger and/or core holes according to applicable regulations. Generally, shallow holes can simply be backfilled with the removed soil material.

Standard measures, such as the use of disposable gloves, that meet project DQOs, are used to avoid cross contamination of samples.

As with all intrusive sampling work, project planning should address the potential for encountering subsurface "utilities" and the measures to be taken to avoid problems in the field.

5 Equipment and Supplies

The equipment and supplies required for field work depend on the program/project DQOs. The following is a general list of equipment and supplies. A detailed list of equipment and supplies should be prepared based on the project planning documents. In general, the use of dedicated or disposal equipment is preferred but equipment may be re-used after thorough decontamination between sample locations (refer to E & E Sampling Equipment Decontamination SOP ENV 3.15).

- Stainless-steel or Teflon™ spoons, trowels, or scoops. Other construction material may be acceptable depending upon the program/project planning documents and DQOs
- Stainless-steel mixing bowls. Other bowl construction material may be acceptable depending upon the program/project planning documents and DQOs
- Hand-driven bucket/continuous flight auger(s), split core sampler(s), and single or multistage core sampler(s)
- Rubber mallet or T-bar to help drive hand augers
- Powered auger(s)
- Spade(s) and/or shovel(s)
- Liners and/or catchers for augers or core samplers as specified in the project planning documents
- Pipe cutter(s), stainless steel knives(s), or power saw to cut liners
- Survey stakes or flags to mark locations
- Ancillary equipment and supplies, e.g., meter stick or tape measure, aluminum foil, plastic sheeting, disposable gloves

Supporting equipment and supplies also may be required to address the following:

- Field logbooks and supplies (Refer to project planning documents and the E & E Field Activity Logbooks SOP DOC 2.1 for details)
- Decontamination equipment and supplies (Refer to project planning documents and E & E Sampling Equipment Decontamination SOP ENV 3.15for details)

- Sample containers, preservatives, and shipping equipment and supplies (Refer to project planning documents and the E & E Environmental Sample Handling, Packaging and Shipping SOP ENV 3.16 for details)
- Waste handling supplies (Refer to project planning documents and E & E Handling Investigation-Derived Wastes SOP ENV 3.26 for details)

6 Procedures

E & E staff will use the following procedures for completing soil sampling:

- Review relevant project planning documents, e.g., work plan, sampling and analysis plan, quality assurance project plan, health and safety plan, etc.
- Select the sampling procedure(s) that meet project DQOs.
- Refer to the E & E Field Activity Logbooks SOP DOC 2.1 for guidance on the types of information that should be recorded for each sample.
- Refer to the E & E Environmental Sample Handling, Packaging and Shipping SOP ENV 3.16 for guidance on how samples should be labeled, packaged, and shipped.

6.1 Hand Scoop Surface and Subsurface Soil Sampling

- Surface and shallow subsurface soil samples may be collected by hand using scoops.
- Pre-cleaned spoons, trowels, or scoops are used to excavate shallow soil.
- Sample collection intervals are identified in the project planning documents.
- Clear the area to be sampled of surface debris (e.g., twigs, rocks, and litter).
- Carefully remove the top layer of soil to the desired sample depth with a precleaned tool.
- When sampling from the sides or bottom of an excavation, use a pre-cleaned, scoop, spoon, or trowel to remove and discard the thin layer of soil from the area that came into contact with the shovel or spade.
- Collect sufficient sample volume to meet the DQOs identified in the project planning documents
- Place aliquots to be analyzed for volatile organic analytes and/or sulfides directly into sample containers (i.e., prior to homogenization). Procedures for collecting soil samples for VOC analyses are presented in the (see E & E VOC Soil and Sediment Sampling SOP ENV 25).
- Empty hand-collected samples into a pre-cleaned stainless steel bowl (or other type as specified in the project planning documents).
- If multiple hand collected samples are necessary to collect adequate sample volume, they should all be combined in the bowl prior to homogenization.
- Homogenize the sample(s) as thoroughly as possible.
- Transfer sample aliquots to appropriate sample containers and preserve as required in the project planning documents.
- Return unused soil to the excavation, level the area, replace grass turf as necessary.

6.2 Subsurface Soil Sampling with a Soil Core Samplers

This system consists of pre-cleaned corer barrels (with liners and liner caps, as appropriate), caps, core tips, and slide hammer. The dimensions of the core barrel define the volume and depth interval of possible sample collection. Core sampling is recommended if accurate resolution of sample depths is a DQO. Hand coring will generally be limited to 2-inch diameter – 3 foot (1 meter) long samples.

There are a variety of manual soil core sampling devices available for collecting undisturbed soil core samples. Split core, single core, and multistage core samplers may be used with or without liners that are used to avoid contact between the soil and the corer.

The following procedures are used for collecting soil samples with the soil core sampler:

- Assemble the soil core sampler based on manufacturer instructions and project DQOs (e.g., using a liner and/or catcher).
- Clear the area to be sampled of surface debris (e.g., twigs, rocks, and litter).
- Using the slide hammer or sledge hammer or pounding sleeve, begin driving the pre-cleaned corer into the soil until the desired upper sampling depth is reached.
- Carefully retrieve the corer from the boring.
- Decontamination or replace the core barrel with a pre-cleaned core barrel and resume coring. See E & E Sampling Equipment Decontamination SOP ENV 3.15 for decontamination procedures.
- Soil cores should be extruded or split as soon as possible following collection.
    - Place core barrel or liner on clean surface
    - Carefully remove end caps and/or catchers
    - Evaluate compaction (core length versus depth of penetration)
    - For transverse sectioning, beginning at the soil surface, measure and mark the sample sections on the outside of the liner
        - Cut the liner with a manual pipe cutter or core liner and core with a decontaminated saw blade into marked sections.
        - Extrude the soil from the cut segments of the liner. If necessary use a plunger cover with aluminum foil to aid in extruding the core.
        - Empty the core segment into a stainless steel bowl (or other type as specified in the project planning documents).
        - Record observations of the soil types.
        - Immediately collect volatile organic analyte and sulfide samples.
    - For longitudinal sectioning, open the split tube or use a knife to cut the liner and expose the upper half of the soil cylinder.
        - Beginning at the soil surface, measure and mark the sample sections using a tape measure set aside the core.
        - Record observations of the soil types.
        - Immediately collect volatile organic analyte and sulfide samples.

    - Scope the core segment into a stainless steel bowl (or other type as specified in the project planning documents).
- If multiple core segments are necessary to collect adequate sample volume, they should all be combined in the bowl prior to homogenization
- Homogenize the sample as thoroughly as possible
- Transfer sample aliquots to appropriate sample containers and preserve as required in the project planning documents.
- Return unused soil to the boring, level the area, replace grass turf as necessary.

6.3 Subsurface Soil Sampling with Bucket Augers

This system consists of pre-cleaned bucket augers, a series of extensions, and a T-handle. The dimensions of the bucket define the volume and depth interval of possible sample collection. The following procedures are used for collecting soil samples with the bucket auger:

- Attach the bucket auger bit to a drill rod extension, and attach T-handle to the drill rod.
- Clear the area to be sampled of surface debris (e.g., twigs, rocks, and litter).
- Begin augering, periodically removing and depositing accumulated soils onto a plastic sheet spread near the hole until the desired upper sampling depth is reached.
- Decontaminate the bucket auger or replace the bucket auger with a pre-cleaned auger bucket and resume augering. After reaching the desired depth (no more than the maximum length of the auger bucket), carefully remove the auger from the boring.
- Empty bucket auger-collected samples into a pre-cleaned stainless steel bowl (or other type as specified in the project planning documents) OR use pre-cleaned scoops and carefully subsample soil from within the bucket that has not come in contact with the auger.
- Immediately collect volatile organic analyte and sulfide samples.
- If multiple bucket auger collected samples are necessary to collect adequate sample volume, they should all be combined in the bowl prior to homogenization.
- Homogenize the sample(s) as thoroughly as possible.
- Transfer sample aliquots to appropriate sample containers and preserve as required in the project planning documents.
- If another sample is to be collected in the sample hole, but at a greater depth, decontaminate or re-attach a pre-cleaned auger bucket, and follow steps above.
- Return unused soil to the excavation, level the area, replace grass turf as necessary

6.4 Subsurface Soil Sampling with Continuous Flight Augers

This system consists of pre-cleaned continuous flight augers, a series of extensions, and a T-handle. The dimensions of the flight define the volume and depth interval of possible sample collection.

When continuous flight augers are used, the sample can be collected directly off the flights. Continuous flight augers are satisfactory for use when a composite of the soil column is desired.

A powered auger may be used at this time. The following procedures are used for collecting soil samples with an auger:

- Attach the continuous flight auger to a drill rod extension, and attach T-handle to the drill rod.
- Clear the area to be sampled of surface debris (e.g., twigs, rocks, and litter).
- Begin augering, periodically removing and depositing accumulated soils onto a plastic sheet spread near the hole until the desired upper sampling depth is reached.
- Decontaminate or replace the auger flight with a pre-cleaned auger flight and resume augering. After reaching the desired depth (no more than the maximum length of the auger flight), carefully remove the auger from the boring.
- Place auger-collected samples into a pre-cleaned stainless steel bowl (or other type as specified in the project planning documents) OR use pre-cleaned scoops and carefully subsample soil from within the auger flights as it comes to the surface.
- Immediately collect volatile organic analyte and sulfide samples.
- If multiple auger flight-collected samples are necessary to collect adequate sample volume, they should all be combined in the bowl prior to homogenization
- Homogenize the sample(s) as thoroughly as possible.
- Transfer sample aliquots to appropriate sample containers and preserve as required in the project planning documents.
- If another sample is to be collected in the sample hole, but at a greater depth, decontaminate or re-attach a pre-cleaned auger flight, and follow steps above.
- Return unused soil to the excavation, level the area, replace grass turf as necessary.

7 Quality Assurance/Quality Control

Prior to initiating field work, the project planning documents (e.g., work plan, sampling and analysis plan, quality assurance project plan, SHASP, *et al*) should be reviewed by field personnel to identify sampling procedure(s) that will most likely provide surface and shallow subsurface soil samples that meet project DQOs.

The program/project manager should identify personnel for the field team who have knowledge, training and experience in the field soil sampling activities being conducted. One member of the field team should be designated as the lead for soil sampling and will be responsible, with support from other field personnel, for implementing the procedures in this SOP. The program/project manager should also identify additional personnel, if necessary, to complete ancillary procedures, e.g., field logbook documentation, equipment decontamination, sample shipment, and waste disposal.

The soil sampling lead should prepare a detailed equipment checklist before entering the field and verify that sufficient and appropriate equipment and supplies are taken into the field.

Quality assurance/quality control samples (e.g., co-located samples) are collected according to the site quality assurance project plan. Field duplicates are collected from one location and treated as separate samples. Field duplicates are typically collected after the samples have been homogenized. Collocated samples are generally collected from nearby locations and are collected as completely separate samples.

In cases where multiple hand-collected scoop, auger or core samples are required to generate an adequate sample volume, homogenization is important. Field personnel should collect sample aliquots only after mixing has produced soil with textural and color homogeneity.

At sites with known or suspected contamination, samples should be collected moving from least to most contaminated areas.

8 Health and Safety

Prior to entering the field, all field personnel formally acknowledge that they have read and understand the project specific health and safety plan.

Augers and soil core sampling apparatus are inherently dangerous pieces of heavy equipment which a high "pinch" potential. Care should be taken at all times when handling such equipment, not just during sample collection.

Prior to any subsurface work, verify that underground utilities have been located and marked.

9 Special Project Requirements

Project or program-specific requirements that modify this procedure should be entered in this section and included with the project planning documents.

10 References

The following list sources of technical information on soil sampling.

Barth, D. S. and B. J. Mason, 1984, *Soil Sampling Quality Assurance User's Guide*, EPA-600/4-84-043.

de Vera, E. R., B. P. Simmons, R. D. Stephen, and D. L. Storm, 1980, *Samplers and Sampling Procedures for Hazardous Waste Streams*, EPA-600/2-80-018.

Navy Environmental Compliance Sampling and Field Testing Procedures Manual, NAVSEA T0300-AZ-PRO-010

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_____, November 2001*, Environmental Investigations Standard Operating Procedures and Quality Assurance Manual*, USEPA Region 4

_____, 18 February 2000, U.S. EPA Environmental Response Team Standard Operating Procedures, Soil Sampling, SOP #2012

END OF SOP

STANDARD OPERATING PROCEDURE FOR THE INNOV-X X-RAY FLUORESCENCE HANDHELD ANALYZER FOR METALS IN SOIL

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Prepared for:
UNITED STATES ENVIRONMENTAL PROTECTION AGENCY
1200 Sixth Avenue, Suite 900
Seattle, Washington 98101

Prepared by:

ECOLOGY AND ENVIRONMENT, INC.
720 Third Avenue, Suite 1700
Seattle, Washington 98104

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List of Abbreviations and Acronyms

EPA	Environmental Protection Agency
MDL	Minimum Detection Limit
MRL	Minimum Reporting Limit
NIST	National Institutes of Science and Technology
QA	Quality Assurance
QC	Quality Control
XRF	X-Ray Fluorescence

1 Method Summary

This method describes how to use the Innov-X hand held XRF analyzer to perform EPA Reference Method 6200 for "Field Portable X-Ray Fluorescence Spectrometry for the Determination of Elemental Concentrations in Soil and Sediment."

The instrument requires daily standardization and calibration of both a reference metal alloy sample, as well as appropriate soil samples for the elements and concentrations of interest. During calibration, a mathematical relationship between the measured element signal and the known quantity of elements introduced to the instrument is generated. An instrument method detection limit study is also performed prior to field use.

Instrument standardization is performed by measuring the signal associated with known quantities of metal elements in a 316L stainless steel alloy to verify that the XRF analyzer is operating and performing within manufacturer specifications. A blank analysis is then performed on a block of pure Teflon to ensure that there is no contamination of the analyzer window. Then, a NIST traceable soil reference sample, with a known quantity of elements, is analyzed. The signal associated with the quantity of the various elements is determined and compared with the known quantity for that reference sample. Concentrations of the elements of interest should be within 20% of the known value, with the exception of chromium (Cr), which can be within 30%. Field samples can then be run to determine concentration of the elements of interest.

Analysis of field samples can either be done in-situ or ex-situ with varying degrees of preparation to achieve higher quality data. Improper sample preparation is the largest source of error associated with XRF field analysis. Proper sampling techniques are discussed in detail in Section 9. Interferences are also a major concern with XRF analysis and can come from: analysis through bagged samples, excess moisture, and soil chemistry. Interferences are discussed in detail in Section 3.

Continuing quality control activities during analysis determine the error associated with the data. These required quality control activities are described in sections 6 and 7.

2 Equipment and Reagents

2.1 Instrument Setup

The Innov-X field portable X-Ray fluorescence spectrometer is assembled in accordance with the appropriate manufacturer's guidance. A summary of these operations is provided:

- Prior to field deployment, the removable lithium ion batteries, as well as the internal battery for the instrument PDA, should be fully charged. See instrument specific manuals and/or quick start guides for information regarding charging, as they vary between analyzers in EPA R10. Once on site, the instrument should be powered on and allowed to warm up in the environment it will be operating in for at least 15 minutes prior to standardization.

- Launch the application software, InnovX, if it does not launch automatically.

- Choose the "Soil" analysis mode. Unless a new selection is made, the application starts on the most recent mode used. Use of the Light Element Analysis Program (LEAP) will provide the lowest possible detection limit for elements lighter than iron, and specifically for Ti, Cr, and Ba. See instrument manual for information on activating this program mode.

- Attach the standardization clip over the analyzer window and run the standardization procedure. A radiation safety notice will appear on the screen; read the notice and acknowledge that you are a certified user by tapping **START**. Upon successful instrument standardization, the XRF analyzer is now ready to analyze soil reference standards and field test samples. Be aware that once standardization is complete, the X-ray tube will be energized for the next 4 hours, or until the instrument/software is powered off. A solid red light on the analyzers metal snout indicates the x-ray beam is shuttered, while a blinking light warns the operator that x-rays are being emitted from the instrument.

2.2 Additional Reagents and Supplies

Additional supplies and reagents are required for operation of the Innov-X field portable XRF analyzer:

2.2.1

National Institute of Science and Technology (NIST) standards for quality control samples. Avoid contamination by keeping these containers sealed. The following standards are assumed available in this SOG:

NIST 2702 Inorganics in Marine Sediment
NIST 2709a San Joaquin Soil

NIST 2710 Montana Soil, Highly Elevated Traces
NIST 2711 Montana Soil, Moderately Elevated Traces
(Information on these standards is available in Appendix A).

2.2.2

Disposable spatulas, mixing bowls, a sieve, and extra sample bags should be packed with the instrument. Before going to a site the operator should check that the quantity of these items is adequate for anticipated tasks.

2.2.3

Gloves and eye protection are not packed with the instrument, but should be readily available in the work area.

These additional required materials and reagents (with the exception of sampling supplies, gloves, and eyewear) are maintained in travel cases ready for shipment or transport.

3 Interferences and Instrument Operation

3.1 Interferences:

Generally, instrument precision is the least significant source of error in field portable XRF analysis. User or application related error is generally more significant and varies with each site and method used. Some sources of interferences can be minimized or controlled by the instrument operator, but others cannot. Common sources of user or application related error are discussed below.

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 analytes concentration will vary depending on how fine particles are distributed within the coarser-grained matrix. One way to reduce such error is to sieve soil and or grind samples to a uniform particle size reducing the sample-to-sample particle size variability. Every effort should be made to thoroughly mix and homogenize soil samples before analysis, as field studies have shown that sample heterogeneity has the largest impact on comparability with confirmatory samples.

Sample moisture has two effects on XRF results:

- It alters the soil chemistry, since water is another chemical compound that compromises the soil matrix.
- Moisture impedes the ability to properly prepare samples.

While the presence of significant moisture does impact soil chemistry, modern XRF analyzers all perform automatic corrections for variations in soil chemistry from site to site. EPA Method 6200 states that “Moisture content above 20% may cause problems, since moisture alters the soil chemistry from which the XRF has been calibrated.” On the Innov-X normalization and correction parameters built into the instrument will automatically correct results for changes in the soil matrix, without having a significant effect on accuracy, except for the dilution effect that can cause discrepancies with laboratory results.

Innov-X states that “The inability to adequately prepare a wet sample is, we believe, the single biggest contributor to errors when testing wet samples.” Wet samples are inherently difficult to properly grind, sieve, and completely homogenize to obtain high quality XRF results. Also, laboratories always dry samples prior to analysis and report percent weight content based on a dry sample basis. Portable XRF results are often obtained with a wet sample in the field, and results are thus reported that include the moisture content. With all factors the same, the laboratory will report higher than the portable XRF by the amount of moisture content in the

sample. For example, laboratory results will be 10% higher compared to XRF results, if the sample contained 10% water when it was tested in the field.

Inconsistent positioning of the 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 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.

Lastly, interference can occur when the spectral peak from one element overlaps either partially or completely with the spectral peak of another. Mathematical corrections in the instrument can correct for most of these events, but there are limits to their effectiveness. The following are interference examples relevant to several RCRA metals of interest:

- Lead interferes with arsenic (not vice versa). The net effect is a elevated detection limit for arsenic, and poorer precision. The XRF handles this correction automatically, but the precision is affected. The loss of precision is reported by the XRF
- Z, Z-1, Z+1 types of interference. These types of interference occur when high levels of an element of atomic number Z are present. This can cause elevated levels of elements with atomic number Z-1 and Z+1. Generally, portable XRFs have good correction methods, so this interference only causes problems with very high levels of the element in question. Example: High concentrations of Fe (Z=26) in excess of 10% may cause elevated levels of Mn or Co (Z=25 and Z=27 respectively).

3.2 Instrument Operation

Blanks, quality control standards, and samples are all analyzed using the same general procedure:

- With the instrument warmed up and standardization with the 316L alloy successfully completed, the analyzer is now ready to perform sample analysis. Be sure that the standardization clip is removed prior to sample analysis.
- Ensure the sampling area covers the entire yellow Kapton window. Do not point the unit at yourself or any other person during operation. Do not test small samples in your hand. Place them on a surface for testing.
- Either pull the trigger or tap **Start**. The *Elapsed Time* screen appears. The test time is reported on the *Test Time* screen under Test Condition. Hold analyzer still while analysis is performed.
- After analysis is complete, tap the **Results** tab and the results screen appears.
- Subsequent tests can be started from either the *Results* or *Analysis* screens.
- All analyzer data can be exported to a spreadsheet program by using a comma delimited (comma separated values = CSV) text file format. It is possible to export a single day, single mode, or to export all data saved on the analyzer
- Note that Microsoft ActiveSync software must be installed to export data.
- To export results, tap **Setup**, then Results Management icon, and the export options screen appears.
- Tap the **Export** radio button.
- Tap either the Results or Spectra radio button.
- Use the Mode down arrow to select: All, Standardization, Analytical, Process Analytical, or Soil Analysis.

- Tap **All** or **Select** in Dates; All exports all stored readings (can take several minutes), Select allows the selection of an individual date. It is recommended that all data be backed up on a daily basis and all data be backed up prior to deleting some or all old data.
- Either tap the Save or Save As and name the file to be exported. Tap the up arrow folder icon to navigate to the desired folder on the PC and tap OK. The export screen appears.
- Connect the Innov-X supplied USB cable to analyzer and the PC. ActiveSync or Mobile Device should automatically start. If it does not, start the program manually.
- Open Windows Explorer on the target PC and tap the icon resembling the PDA. The directory structure on the analyzer appears.
- Navigate the directory and copy the file(s) to the target PC.
- Readings can be deleted on the analyzer by tapping on **Setup** and then on **Results Management** and the export options screen appears.
- Tap the **Delete** radio button and the delete screen appears. Select desired data to be permanently removed (some or all). Be certain that all data is backed up prior to tapping **Delete Results and Spectra** and confirming.

4 Instrument Calibration

4.1 Standardization and Performance Checks

As described previously, the analyzer must be standardized using the 316L stainless steel alloy clip before the analyzing any samples. The software will not permit further analysis of samples until standardization is performed. Standardization of the instrument allows the analyzer to optimize the detector's electrical gain settings, which can drift, especially with temperature fluctuations, and ensures that the instrument hardware and software are performing within the manufacturer tolerances (± 20%). Standardization can be done at any time, but is required by the analyzer if the software is restarted or after 4 hours of continuous use. The recommended standardization intervals are:

- At the start of the work day,
- After lunch,
- And at the close of the work day.

Once a successful standardization of the analyzer is completed, performance checks with NIST traceable soil standards are vital to proper use of the field portable XRF spectrometer before field samples are to be analyzed. While similar to the standardization using 316L stainless steel, the analysis of various elements of interest in the soil matrix with known quantities, allows the operator to gauge how the analyzer will perform with field samples. For data to be considered adequately precise, the RSD for the analytes of interest should not be greater than 20% with the exception of chromium, RSD values for Chromium should not be greater than 30%.

- An acceptable performance check of one or more NIST soil standards must be performed at the beginning of every day in which the instrument is to be used to perform elemental analysis of soil.
- Variations in analyzer response are element specific and can fluctuate. Additional calibrations may be performed if operating conditions change or if quality control materials dictate.

The general procedure for performing a performance check is the same as described previously in Section 3, Instrument Operation. Exporting the analyzer results for performance checks and comparing them to the known elemental concentration allows the operator to calculate percent differences for the elements of interest. A repeated series of performance checks on a standard will allow the operator to determine the method detection limit (MDL), which is described in detail in Section 9.

5 Field Usage & Sample Preparation

Field portable XRF is generally used in three ways to test for analytes in soils (Adapted from Innov-X instrument manual, Appendix B):

In-Situ Soil Testing:

The XRF is placed directly onto the ground for soil testing. Operators remove any plant growth and foreign objects so the analyzer probe is flush to the soil. A thin sheet of plastic may be used to prevent contamination of the analyzer and possible puncturing of the Kapton window (see note below on analysis through plastic). Precision can be improved with in-situ analysis by averaging multiple readings of the same area (ie 4 analyses taken within a 4in by 4in soil surface and averaged). Multiple analysis of the same area can also inform the operator as to the homogeneity of the soil to decide if bagged sampling and mixing is necessary.

Bagged Soil Sample Testing:

A soil sample is collected in a thin plastic bag, mixed for 3-5 minutes, and testing occurs directly through the bag. Collect the sample from a 4 by 4 inch square that is 1 inch deep, which will be enough soil to fill an 8-ounce jar. Except for a few elements – namely Cr, V, and Ba – testing through the thin plastic bag has little effect on the test result. Results for Cr, V, and Ba will be 20-30% lower. Multiple analysis of a sample can also give the operator insight as to how successful homogeneous hand mixing is and if prepared sampling may be necessary to achieve the desired data quality.

Prepared Soil Sample Testing:

Representative sampling is essential for good analytical results. Prepared sample testing assures the operator of the maximum possible accuracy. Prepared sample tests require a sample to be collected, dried if necessary, sieved and ground into a powder. The prepared sample is them placed into a sample baggie or XRF cup for analysis.

Samples with high moisture levels will have a low bias, and will not correlate well with samples sent for offsite laboratory analysis. Therefore samples with free water should be drained prior to analysis. Slurries and obviously wet samples must be dried prior to analysis. Any wet sample result which is between half of an action level and the action level itself, should either be air-dried overnight or dried at 60°C until visibly dry (soil is crumbly) and re-run. Accelerated drying of soils at high temperatures or with high volume air flow will cause loss of mercury from the samples, and should be avoided if mercury is known or thought to be present. If necessary, a

separate sample can be dried rapidly and weighed, and these results can be used to convert results to a dry weight basis.

Data Quality Objectives:

It is important to understand your data quality objectives (DQO) in order to determine the appropriate mix of field screening and prepared sample testing. In-situ testing usually provides only screening-level data. The correlation coefficient (r) for the results should be 0.7 or greater for the field portable XRF data to be considered screening level data. This is because analytical testing always requires a uniform, homogeneous sample matrix. A laboratory achieves this by digesting the sample in hot acid prior to analysis. Testing directly on the ground does not ensure uniformity is met. Preparing a sample provides a uniform sample and likely better analytical data quality, although several minutes of testing time is required. If the r is 0.9 or greater and inferential statistics indicate the field data and confirmatory data are statistically equivalent at a 99% confidence level, the data could potentially meet definitive level data criteria.

XRF operators may use a mixture of in-situ and prepared sample testing. The exact mixture of in-situ and prepared sample testing depends upon the goals of the soil testing. Innov-X provides three examples of to serve as testing guidelines in the Omega Manual on pages 126-128. They can also be contacted directly to discuss specific testing requirements (1-781-938-5005).

6 Analysis

The following is a general schedule that describes analysis activities, beginning with the instrument standardization that has already been described. Once standardization has been successfully performed, analysis can begin.

6.1 Calibration

- An energy calibration (standardization) check is performed at the beginning of day.

6.2 Initial QC

- Calibration blank using Teflon block to check for contamination on analyzer window
- Choose a quality control NIST standard that is at a concentration near the action level or MRL for the element(s) of interest.

6.3 Samples

- Up to 20 samples may be run in a batch
- Refer to the Site Specific Sampling Plan for conformation sampling criteria, as this will vary.

6.4 Continuing and Final QC

- An instrument blank for every 20 environmental samples
- Calibration verification check sample with appropriate NIST standard every 20 samples
- Precision sample performed once per day.

Example of how a log book page may look following these guidelines for a 20 sample batch:

Sample ID	Time	Arsenic (mg/kg)	Chromium (mg/kg)	Comments
Energy Cal	0800			Pass
Instrument Blank				OK
NIST 2709a				As <20% , Cr <30% diff
1				In-situ
2				In-situ
3				Leaves & twigs removed
4				In-situ
5				In-situ
6				In-situ
7				In-situ
8				In-situ
9				In-situ
10				Bag, split sample for lab
11				In-situ
12				In-situ
13 precision -1				Bag
13 precision – 2				Bag
13 precision – 3				Bag
13 precision – 4				Bag
13 precision – 5				Bag
13 precision – 6				Bag
13 precision – 7				RSD: As<20%, Cr<30%
14				In-situ
15				In-situ
16				In-situ
17				Bag, split sample for lab
18				In-situ
19				In-situ
Energy Cal	1200			4 hrs elapsed, Pass
20				In-situ
Instrument Blank				OK
NIST 2709a				As<20%, Cr<30% diff
Lunch taken	1230			XRF off, put on charger

7 Quality Assurance/ Quality Control

Initial and continuing quality control, in the form of the Calibration Blank and the Quality Control Standard, ensure that the instrument is operating at an acceptable limit of accuracy, and the results of these analyses must be compared to the following guidelines.

7.1 Quality Assurance

Quality assurance activities are preventive measures taken to ensure the quality of analytical results. The single most important quality assurance activity undertaken in the field laboratory is rigorous attention to the possibility of errors in sample identification and data entry. Organizing the workspace so that only one sample is on the bench at a particular time is one option. Assume that the analytical environment will be chaotic, and that work processes will be interrupted, and organize the workspace to eliminate identification and recording errors.

7.2 Quality Control

Quality control activities are tests performed during analysis to ensure that the analytical error associated with analysis does not become unacceptably large. These control activities were introduced in Section 6. In this section the criteria for acceptable quality control results are described. Control activities and acceptance criteria are summarized in Table 7-1.

Table 7-1: Quality Control Samples – Frequency and Acceptance Criteria

Quality Control Material	Frequency	Acceptance Criterion
MDL Study	Performed annually	± 20 % of certified Concentration.
Energy Calibration Check	3 times per day, or every 4hrs	Pass or Fail
Instrument Blank	At start and every 20 samples	Between MDL and negative MDL
Calibration Verification	At start and every 20 samples	± 20 % of certified concentration (± 30 % for Cr)
Precision Sample	Once per day	± 20 % of certified concentration (± 30 % for Cr)

7.2.1 Instrument Blank

This value should be between the Minimum Detection Limit (MDL) and the negative of the MDL.

7.2.2 Calibration Verification

This operator is to perform a two minute test on an appropriate NIST reference standard. The result values should be within 20% of the true value of the standard (± 30 % for Cr).

7.2.3 Precision Verification

A minimum of one precision sample should be run per day by conducting from 7 to 10 replicate measurements of the sample. The precision is assessed by calculating the relative standard deviation (RSD) of the replicate measurements ofr the analyte. The RSD values should be within 20% for most analytes, with the exception of Cr, for which th value should be less than 30 percent.

7.4 Corrective Actions

Corrective action must be taken when quality control results are not acceptable. Acceptable corrective actions may include, but are not limited to:

- Repetition of the Calibration Blank or Calibration Verification one time. It is not acceptable to run quality control materials time after time in the hope that a random quality control result will fall in an acceptable range.
- Use of a new sample bag as the blank, changing out of dirty Kapton window, or a new soil standard as the Calibration Verification is acceptable. However, if this fixes the problem, it must be determined what was wrong with the faulty bag or standard before it can be used further.
- Re-calibration of the instrument is an acceptable corrective action, but re-calibration often merely masks other analytical problems.
- If other corrective actions fail, the minimum reporting limit may be raised. This should be done in consultation with subject matter experts.

8 Data Reporting, Qualification and Calculation

8.1 Data Reporting and Qualification

Analytical results above the minimum reporting limit are reported to three significant figures. Analytical results below the minimum reporting limit are reported with two significant figures. Analytical results less than the minimum detection limit are reported as undetectable (U) at the value of the minimum detection limit.

8.2 Calculation

Some calculations must be performed by the operator. These calculations are described below.

8.2.1 Percentage Relative Difference (% RPD)

$$\left[\frac{(x_1 - x_2)}{\frac{(x_1 + x_2)}{2}}\right] \times 100 = \%RPD$$

Where x_1 is a sample result, and x_2 is a separate but comparable sample result.

8.2.2 Percentage True Value of Quality Control Standard

$$\text{Percentage True Value} = \left[\frac{x_1 - x_2}{x_2}\right] \times 100$$

Where x_1 is the Quality Control Standard sample result, and x_2 is the true value of the Quality Control Standard.

8.2.3 Percentage Solids

$$\text{Percentage Solids} = \frac{\text{dry weight}}{\text{wet weight}} \times 100$$

9 Determination of the MDL

The Method Detection Limit (MDL) is determined at least yearly or more frequently if changing instrument conditions cause concern about instrument sensitivity. The determination is performed as follows:

- When the Innov-X XRF spectrometer is ready for analysis, perform seven repetitions of a low level sample using the appropriate NIST standard corresponding to the element(s) of interest.
- Determine the mean and standard deviation of the seven repetitions. Many calculators and spreadsheet programs will perform these calculations, but they are included below as a reference:

$$Mean = \frac{\sum measurements}{7}$$

$$Deviation = \frac{\sqrt{\sum\ (individual - mean)^2}}{6}$$

- 3.14 times the standard deviation yields the calculated method detection limit (The 3.14 value is obtained from the Student's T Test and is based on 7 samples.
- Ten times the standard deviation yields the minimum reporting limit.

10 Health, Safety and Waste Disposal

10.1 Health and Safety in the Field Laboratory

Laboratory operators will always wear gloves and eye protection during operations, and food and beverages will never appear in laboratory areas. A further special hazard posed by handheld XRF analyzers is ionizing radiation emitted from the front of the instrument. The XRF should only be used by a trained operator; never point the analyzer towards others and keep the hands away from the metal snout while analyzing samples. Personal dosimeter badges are required to be worn;if available, ring style finger dosimeters may also be worn.

Drying of samples that contain high levels of water is advised, but samples that potentially contain mercury should only be air dried (with proper ventilation) and not heated to avoid exposure to mercury vapors.

Covering the analyzer window with a clean bag is advised for in-situ sampling, as to avoid contamination, and cross contamination of the Kapton window.

10. 2 Waste Disposal

Excess soil samples should be returned to the site and the containers emptied. The empty containers can be thrown in the trash. Investigational-Derived Waste can be thrown in the trash unless it contains high levels of contamination. The best way to dispose of these objects is to include them in waste destined for analysis, hazard determination, and off-site disposal.

Samples must not be brought from the site back to the warehouse without the express authorization of the EPA OSC.

11 References

Innov-X Omega XRF Analyzer instrument operating manual.

EPA SW-846 Method 6200, “Field Portable X-Ray Fluorescence Spectrometry for the Determination of Elemental Concentrations in Soil and Sediment.”

12 Appendices

Appendix A: NIST Standard Reference Material (SRM) Values

SRM 2702 Inorganics in Marine Sediment

Table 1. Certified Concentrations for Selected Elements

Elements	Mass Fraction mg/kg (unless noted as %)			Elements	Mass Fraction mg/kg (unless noted as %)		
Al [b,c,f,G]	8.41 %	±	0.22 %	Ni [A,a,b,c,d,e]	75.4	±	1.5
As [b,c,d,G]	45.3	±	1.8	P [b,c,f,H]	0.1552 %	±	0.0066 %
Ba [A,b,e]	397.4	±	3.2	Pb [A,a,b,c,e]	132.8	±	1.1
Ce [b,c,e,G]	123.4	±	5.8	Rb [b,e,G]	127.7	±	8.8
Cd [A,a,b,d,I]	0.817	±	0.011	Sb [a,b,e,G]	5.60	±	0.24
Co [b,c,G]	27.76	±	0.58	Sc [b,c,G]	25.9	±	1.1
Cr [a,b,c,e,G]	352	±	22	Sr [A,b,c,e]	119.7	±	3.0
Fe [b,c,f,G]	7.91 %	±	0.24 %	Th [b,c,e,G]	20.51	±	0.96
Hg [A-1,d,H]	0.4474	±	0.0069	Ti [b,c,f,G]	0.884 %	±	0.082 %
K [b,c,f,G]	2.054 %	±	0.072 %	Tl [A,a]	0.8267	±	0.0060
La [b,c,e,G]	73.5	±	4.2	V [b,c,G]	357.6	±	9.2
Mn [b,c,G]	1757	±	58	Zn [a,b,c,e,G]	485.3	±	4.2
Na [b,c,f,G]	0.681 %	±	0.020 %				

Table 2. Reference Values for Concentrations of Selected Elements

Elements	Mass Fraction mg/kg (unless noted as %)			Elements	Mass Fraction mg/kg (unless noted as %)		
Ag [a,d]	0.622	±	0.078	Mg [b,c,f]	0.990 %	±	0.074 %
Ca [b,c,f]	0.343 %	±	0.024 %	Mo [b,c,e]	10.8	±	1.6
Cu [a,b,c,d,e]	117.7	±	5.6	Se [b,c,e]	4.95	±	0.46
Ga [b,c,e]	24.3	±	1.9	Sn [a,c,e]	31.6	±	2.4

Table 3. Information Values for Selected Elements

Elements	Mass Fraction mg/kg (unless noted as %)	Elements	Mass Fraction mg/kg (unless noted as %)
Be [b,c]	3.0	Nb [c,e]	63
C (total) [j]	3.36 %	Nd [G]	56
C (organic) [j]	3.27 %	S [j]	1.5 %
Cs [b,G]	7.1	Sm [G]	10.8
Hf [G]	12.6	U [b,e]	10.4
Li [b,c]	78.2	W [G]	6.2

SRM 2709a San Joaquin Soil – Baseline Trace Element Concentrations:

Table 1. Certified Values[a] (Dry-Mass Basis) for Selected Elements in SRM 2709a

Element	Mass Fraction (%)	Element	Mass Fraction (mg/kg)
Aluminum	7.37 ± 0.16	Antimony	1.55 ± 0.06
Calcium	1.91 ± 0.09	Barium	979 ± 28
Iron	3.36 ± 0.07	Cadmium	0.371 ± 0.002
Magnesium	1.46 ± 0.02	Chromium	130 ± 9
Phosphorus	0.0688 ± 0.0013	Cobalt	12.8 ± 0.2
Potassium	2.11 ± 0.06	Lead	17.3 ± 0.1
Silicon	30.3 ± 0.4	Manganese	529 ± 18
Sodium	1.22 ± 0.03	Strontium	239 ± 6
Titanium	0.336 ± 0.007	Vanadium	110 ± 11
		Zirconium	195 ± 46

Table 2. Reference Values[a] (Dry-Mass Basis) for Selected Elements in SRM 2709a

Element	Mass Fraction (mg/kg)
Arsenic	10.5 ± 0.3
Cerium	42 ± 1
Cesium	5.0 ± 0.1
Copper	33.9 ± 0.5
Europium	0.83 ± 0.02
Gadolinium	3.0 ± 0.1
Lanthanum	21.7 ± 0.4
Mercury[b]	0.9 ± 0.2
Nickel	85 ± 2
Rubidium	99 ± 3
Scandium	11.1 ± 0.1
Thallium	0.58 ± 0.01

Table 3. Information Values[a] (Dry Mass Basis) for Selected Elements in SRM 2709a

Element	Mass Fraction (mg/kg)
Boron	74
Dysprosium	3
Hafnium	4
Lutetium	0.3
Neodymium	17
Samarium	4
Selenium	1.5
Tantalum	0.7
Terbium	0.5
Ytterbium	2

SRM 2710 Montana Soil – Highly Elevated Trace Element Concentrations

Table 1. Certified Values

Element	Mass Fraction (%)			Element	Mass Fraction (mg/kg)		
Aluminum	6.44	±	0.08	Antimony	38.4	±	3
Calcium	1.25	±	0.03	Arsenic	626	±	38
Iron	3.38	±	0.10	Barium	707	±	51
Magnesium	0.853	±	0.042	Cadmium	21.8	±	0.2
Manganese	1.01	±	0.04	Copper	2950	±	130
Phosphorus	0.106	±	0.015	Lead	5532	±	80
Potassium	2.11	±	0.11	Mercury	32.6	±	1.8
Silicon	28.97	±	0.18	Nickel	14.3	±	1.0
Sodium	1.14	±	0.06	Silver	35.3	±	1.5
Sulfur	0.240	±	0.006	Vanadium	76.6	±	2.3
Titanium	0.283	±	0.010	Zinc	6952	±	91

Table 2. Noncertified Values

Element	Mass Fraction (%)	Element	Mass Fraction (mg/kg)
Carbon	3	Bromine	6
		Cerium	57
		Cesium	107
		Chromium	39
		Cobalt	10
		Dysprosium	5.4
		Europium	1
		Gallium	34
		Gold	0.6
		Hafnium	3.2
		Holmium	0.6
		Indium	5.1
		Lanthanum	34
		Molybdenum	19
		Neodymium	23
		Rubidium	120
		Samarium	7.8
		Scandium	8.7

SRM 2711 Montana Soil – Moderately Elevated Trace Element Concentrations:

Table 1. Certified Values

Element	Mass Fraction (%)			Element	Mass Fraction (µg/g)		
Aluminum	6.53	±	0.09	Antimony	19.4	±	1.8
Calcium	2.88	±	0.08	Arsenic	105	±	8
Iron	2.89	±	0.06	Barium	726	±	38
Magnesium	1.05	±	0.03	Cadmium	41.70	±	0.25
Phosphorus	0.086	±	0.007	Copper	114	±	2
Potassium	2.45	±	0.08	Lead	1162	±	31
Silicon	30.44	±	0.19	Manganese	638	±	28
Sodium	1.14	±	0.03	Mercury	6.25	±	0.19
Sulfur	0.042	±	0.001	Nickel	20.6	±	1.1
Titanium	0.306	±	0.023	Selenium	1.52	±	0.14
				Silver	4.63	±	0.39
				Strontium	245.3	±	0.7
				Thallium	2.47	±	0.15
				Vanadium	81.6	±	2.9
				Zinc	350.4	±	4.8

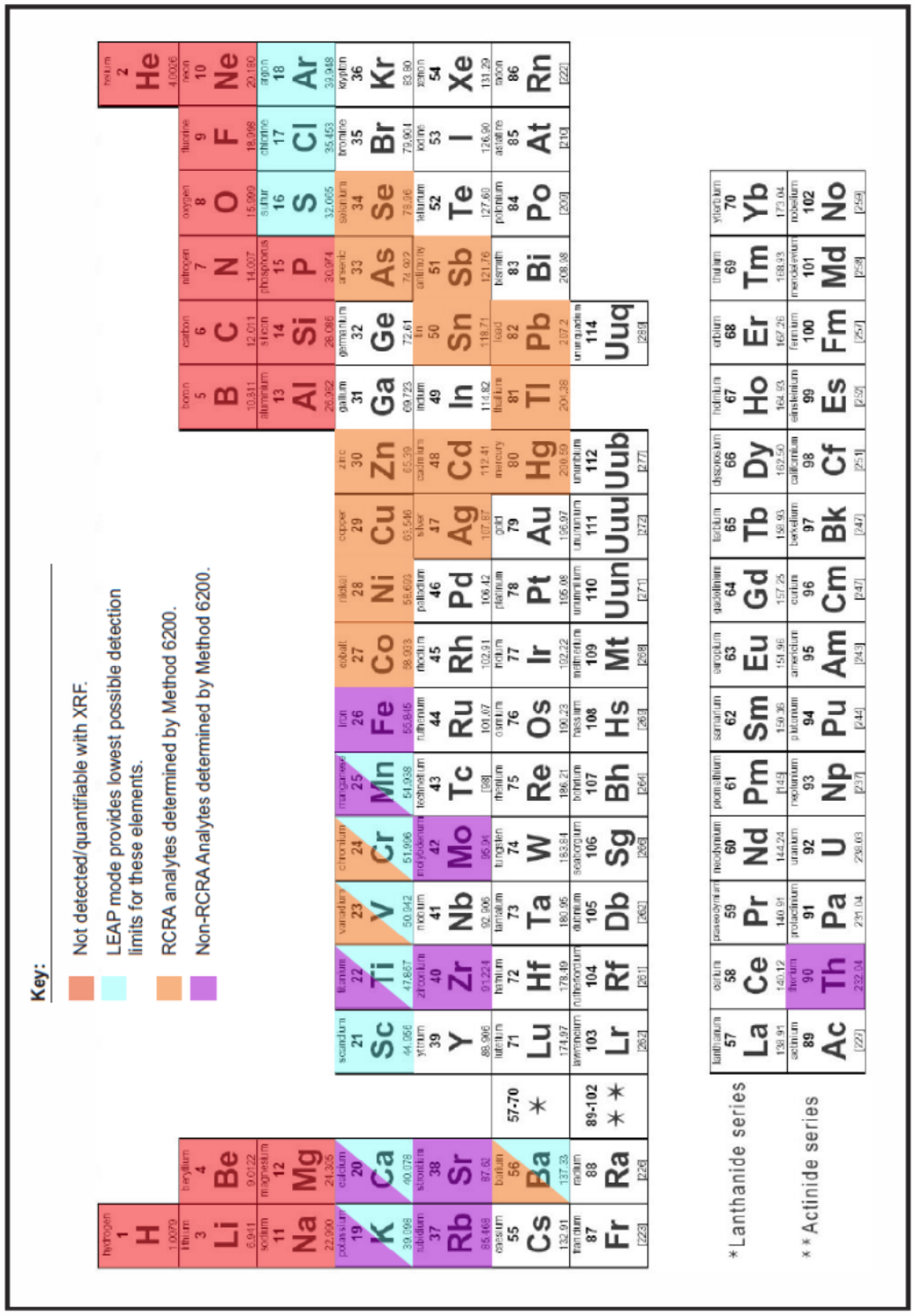
Table 2. Noncertified Values

Element	Mass Fraction (%)	Element	Mass Fraction (µg/g)
Carbon	2	Bromine	5
		Cerium	69
		Cesium	6.1
		Chromium	47
		Cobalt	10
		Dysprosium	5.6
		Europium	1.1
		Gallium	15
		Gold	.03
		Hafnium	7.3
		Holmium	1
		Indium	1.1
		Iodine	3
		Lanthanum	40
		Molybdenum	1.6

Appendix B:
Metals of Interest to ERU for Annual MDL Precision Studies:

Analytes	**Residential Soil RSL (mg/kg)**	**CAS Registry No.**
Arsenic (As)	0.39	7440-38-0
Chromium (Cr)	230	7440-47-3
Copper (Cu)	3100	7440-48-4
Lead (Pb)	400	7439-92-1
Mercury (Hg)	6.7	7439-97-6
Nickel (Ni)	1600	7440-02-0
Silver (Ag)	390	7440-22-4

Appendix C:
Periodic Table of the Elements Highlighting XRF Instrument Capabilities and Method 6200 Determined Analytes



DataRAM 4

GENERAL INFORMATION

Equipment Name:	DataRAM 4
Model:	DR-4000
Manufacturer:	Thermo Corporation
National Manufacturer Contact:	Telephone: 866-282-0430 Website: http://www.thermoscientific.com/ search "DataRAM 4"

NOTE: Guides are to be used by trained personnel only and DO NOT replace the manufacturer's operations or technical manuals. These guides were developed by field personnel for utilization by EPA and their contractors and are helpful in quick start-up and operations. Various limitations have been identified through the experience of the development group. Different makes, models, and updates to this equipment may change the limitations. It is recommended that calibration, maintenance, and use be recorded in a logbook. Additional product information may be found in the accompanying Equipment Operating Guides.

SPECIFICATIONS

Uses:	The DataRAM 4 Model DR-4000 measures the concentration of airborne particulate matter (aerosolized liquid or solid), mean particle size, and air temperature and humidity. The DR-4000 provides direct and continuous readout as well as electronic recording of the monitoring data.
Limitations:	The DR-4000 is not designed to sample highly corrosive aerosols or solvent fumes. The relatively low flow rate of 2.0 liters per minute (LPM) may preclude the instrument from fence line monitoring involving low concentrations of contaminants in soil. The instrument must be protected from precipitation and may fail under extreme temperature. High humidity may cause elevated readings.
Response Range:	Response range: 0.0001 mg/m^3 (0.1 µg/m^3) to 400 mg/m^3 (400,000 µg/m^3)
Alarm Level:	The alarm function can be enabled and the alarm level (trigger threshold) can be set per site-specific requirements. Press any key to momentarily silence an activated alarm.
Product Safety:	Not intrinsically safe.
Battery:	The instrument can be powered by an internal rechargeable sealed lead-acid gel-cell battery with 7.2 Ah, 6V, 20-hour average run time, and 12 hour average recharge period. The instrument can also be powered by alternating current via the universal voltage charger/power supply, 100-250 V, 50 - 60 HZ. *Note: To enable operation with either internal battery or the charger/power supply, the 3-position power selector switch on the back of the unit should indicate* ***INT. BATT.***
Calibration:	The DR-4000 should be annually cleaned and calibrated by the manufacturer. Prior to collecting data in the field the instrument should be automatically zeroed with internal check out; follow prompts within the start-up menu for details. A reading of BACKGROUND HIGH following the zeroing of the instrument is indicative that the internal optics require factory cleaning or servicing.

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Additional Information:	Do not operate the DR-4000 with the pump cap covering inlet in place. Do not operate the DR-4000 without the internal filter in place. Routinely change dust filters and maintain a record of the replacement. The instrument may be equipped to monitor for PM-10 or PM-2.5 size particles only. The instrument may be configured using the DR4-COM software and an RS-232 cable.

QUICK OPERATIONS GUIDE

START UP:	1.	Ensure the rear panel power switch is in the upward **INT. BATT.** position. Remove the sampling inlet protective cap by pulling up on the knurled metal outer piece and lifting it off. Place the sampling inlet protective cap on the inlet storage post located on the bottom left corner of the rear panel of the instrument. Install inlet tubing, omni-directional sampling inlet, and PM 10 or 2.5 separators (if required). Confirm that the internal HEPA filter is installed.
	2.	Press and hold **ON/OFF** until product information appears on screen; the MAIN MENU screen will follow shortly.
	3.	**ZERO/INITIALIZE OPERATION** From the MAIN MENU screen press ▲ or ▼ until the flashing cursor appears next to ZERO/INITIALIZE. Ensure the instrument is located in a background environment and press **ENTER**. The pump will run for 299 seconds to complete the zero/initialize process. Press **EXIT** to return to the MAIN MENU screen. Refer to the operator's manual for troubleshooting if the instrument did not zero/initialize correctly.
	4.	**SELECTING LOGGING PARAMETERS** From the MAIN MENU screen press **NEXT** to display the EDIT MENU screen**.** Press ▲ or ▼ until the flashing cursor appears next to LOGGING PARAMETERS, then press **ENTER**. • Press ▲ or ▼ until the flashing cursor appears on the LOG DATA row. Press **+/–** to toggle between the DISABLED and ENABLED functions. • Press ▼ until the flashing cursor appears on the LOG PERIOD row. Press ◄ or ► to select between hours, minutes, and seconds, then press **+/–** to increase or decrease the values. For example, a 1 minute log period is displayed as 00:01:00. • Press ▼ until the flashing cursor appears on the TAG # row; this value is usually 01. • Press ▼ until the flashing cursor appears on the AUTO START row. Press **+/–** to toggle between the DISABLED and ENABLED functions; for most field applications this setting will be DISABLED. Return to the MAIN MENU screen by pressing **EXIT,** then **NEXT**.
	5.	**SELECTING SET-UP PARAMETERS** From the MAIN MENU screen press **NEXT** to display the EDIT MENU screen**.** Press ▲ or ▼ until the flashing cursor appears next to SETUP PARAMETERS, then press **ENTER** to access the first of five parameter displays. This initial display will indicate DISPLAY AVG, CAL FACTOR, UNITS, and SIZE CORRECT • Press ▲ or ▼ until the flashing cursor appears on the DISPLAY AVG row. Press **+/–** to adjust the display averaging times. Short averaging times provide faster response but noisier (more fluctuating) data, whereas long averaging times decrease response time but provide smoother (less fluctuating) data. • Press ▼ until the flashing cursor appears on the CAL FACTOR row. The calibration factor is a multiplier of the calibration slope programmed at the factory; a factor of 1.00 indicates that the calibration slope is identical with the factory slope. • Press ▼until the flashing cursor appears on the UNITS row. The measurement

		parameters are mass concentration in µg/m^3, scattering coefficient in (Mm)$^{-1}$, or visual range in kilometers. Typically this value is set to µg/m^3. • Press ▼ until the flashing cursor appears on the SIZE CORRECT row. The particle size correction refers to the computation of the mass correction, and is usually set to DISABL. Press **NEXT** to access the second parameter display of RH CORRECTION, TEMPERATURE UNITS, and FLOW RATE. • Press ▲ or ▼ until the flashing cursor appears on the RH CORRECTION row. Press **+/–** to toggle between the DISABLED and ENABLED functions. The relative humidity correction, when enabled, automatically corrects for particle growth due to a high humidity environment. *Note: This correction applies only when mass concentration units have been selected.* • Press ▼ until the flashing cursor appears on the TEMPERATURE UNITS row. Press **+/–** to toggle between degrees Celsius (°C) and degrees Fahrenheit (°F). Temperature data is usually collected in degrees Celsius. • Press ▼ until the flashing cursor appears on the FLOW RATE row. The flow rate can be adjusted over the range of 1.00 to 3.00 liters per minute (LPM); the standard operating flow rate is 2.00 LPM. Press **NEXT** to access the third parameter display of ANLG OUT, SERIAL MODE, and DEVICE #. Refer to the DR-4000 Instruction Manual for specific information related to analog output signal, serial mode digital communication, and instrument identification number. Press **NEXT** to access the fourth parameter display of TIME and DATE. • Press ▲ or ▼ until the flashing cursor appears on the TIME row. Press ◄ or ► to select between hours, minutes, and seconds, then press **+/–** to adjust the values. For example, 3:45 pm is displayed as 15:45:00. • Press ▼ until the flashing cursor appears on the DATE row. Press the ◄ or ► to select between day, month, and year, and press **+/–** to adjust the values. • Press **ENTER** to activate the changes. Press **NEXT** to access the fifth and final parameter display of ALARM, LEVEL, AUTO ZERO, and INTERVAL. Refer to the DR-4000 Instruction Manual for specific information related to alarm function, action levels, auto zero function, and time intervals between consecutive automatic zeroing. Press **EXIT** to return to the EDIT MENU screen, then press **NEXT** to return to the MAIN MENU screen.
	6.	**START RUN OPERATION** From the MAIN MENU screen press ▲ or ▼ until the flashing cursor appears next to START RUN, then press **ENTER** to begin data collection. Press **EXIT** to terminate data collection, then confirm the termination by pressing **ENTER**. The DR-4000 will perform a purge function for approximately 1 minute after termination.
VIEW DATA:	1.	From the MAIN MENU screen press ▲ or ▼ so the flashing cursor appears next to VIEW/ TRANSFER DATA, then press **ENTER**. On the following screen press ▲ or ▼ so the flashing cursor appears next to VIEW LOGGED DATA, then press **ENTER.**
	2.	The first of three data screens will be displayed; press **NEXT** to scroll through the remaining data display screens. After reviewing the data press **EXIT** twice to return to the MAIN MENU**.** *(Note: The VIEW LOGGED DATA display screen conveniently displays Start Time, End Time, Average Concentration, and Average Diameter. Consider recording this data in the site logbook since it is otherwise not readily accessible).*

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TRANSFER DATA TO A PC:	1.	Connect the DR-4000 to the communication port on the PC using an RS-232 cable.
	2.	Open the DR4-COM software to allow the DR-4000 to communicate with the PC; (refer to the DR-4000 Instruction Manual for a copy of the DR4-COM software). From the DR4-COM window on the PC select the DATA TEXT tab.
	3.	From the MAIN MENU screen press ▲ or ▼ so the flashing cursor appears next to VIEW/ TRANSFER DATA, then press **ENTER**. Press the ▲ or ▼ so the flashing cursor appears next to TRANSFER TEXT FILE, then press **ENTER** to transfer the data to the PC. Upon successful completion of the data transfer press **EXIT** to return to the MAIN MENU.
DELETING LOGGED DATA:	1.	From the MAIN MENU screen press ▲ or ▼ so the flashing cursor appears next to VIEW/ TRANSFER DATA, then press **ENTER**. Press ▲ or ▼ so the flashing cursor appears next to DELETE LOGGED DATA, then press **ENTER**.
	2.	Press ▲ or ▼ to select either DELETE TAG DATA or DELETE ALL DATA, then press **ENTER**. Press **ENTER** again to confirm deletion, then **EXIT** to return to the MAIN MENU.
SHUT DOWN:	1.	Press **ON/OFF** one time. Press **ENTER** to confirm shut down or **EXIT** to return to the previous display.
	2.	Remove the inlet tubing, omni-directional sampling inlet, or PM separators (if attached). Replace the sampling inlet protective cap. Properly store the instrument and recharge the batteries.

DataRAM 4

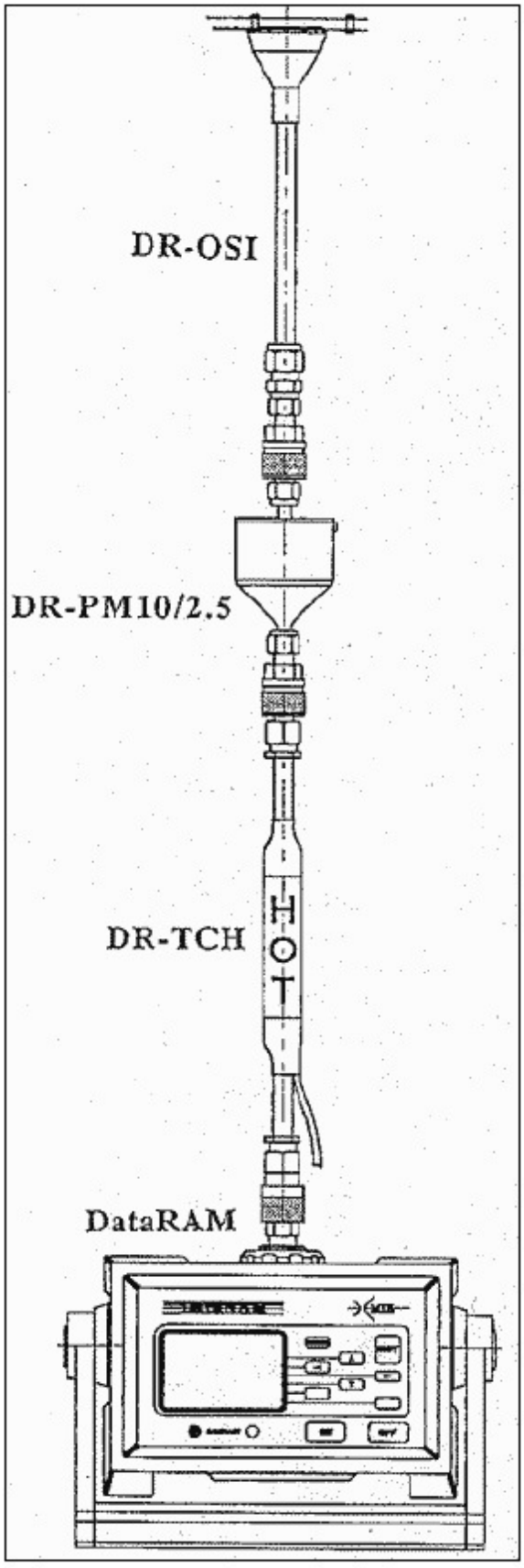

Figure 1

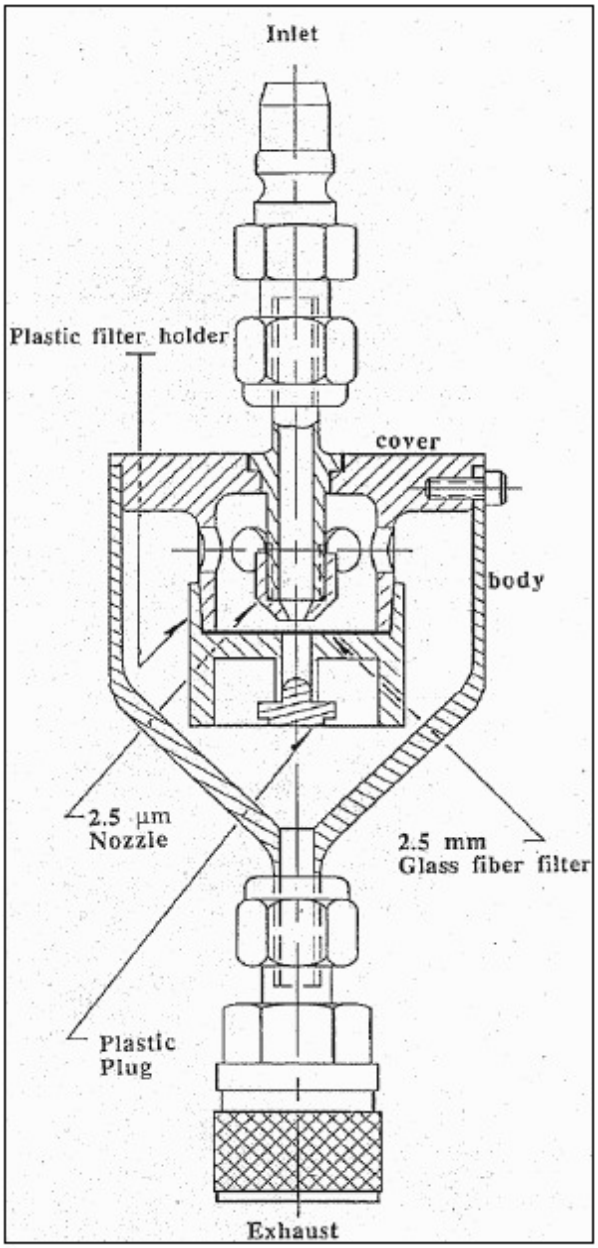

Figure 2

METHOD 4020

SCREENING FOR POLYCHLORINATED BIPHENYLS BY IMMUNOASSAY

1.0 SCOPE AND APPLICATION

1.1 Method 4020 is a procedure for screening soils and non-aqueous waste liquids to determine when total polychlorinated biphenyls (PCBs) are present at concentrations above 5, 10 or 50 mg/kg. Method 4020 provides an estimate for the concentration of PCBs by comparison with a standard.

1.2 Using the test kit from which this method was developed, 95% of soil samples containing 0.625 ppm or less of PCBs will produce a negative result in the 5 ppm test configuration. Using another commercially available test kit, 97% of soil samples containing 0.25 ppm or less of PCBs will produce a negative result in the assay and greater than 99% of the samples containing 1.0 ppm or more will produce a positive result. Tables 2-5, 7, 10, and 11 present false positive and false negative data generated from commercially available test kits. Using a test kit commercially available for screening non-aqueous waste liquids, >95% of samples containing 0.2-0.5 ppm or less of PCB will produce a negative result.

1.3 In cases where the exact concentrations of PCBs are required, quantitative techniques (i.e., Method 8082) should be used.

1.4 This method is restricted to use by or under the supervision of trained analysts. Each analyst must demonstrate the ability to generate acceptable results with this method.

2.0 SUMMARY OF METHOD

2.1 Test kits are commercially available for this method. The manufacturer's directions should be followed.

2.2 In general, the method is performed using a sample extract. Sample and an enzyme conjugate reagent are added to immobilized antibody. The enzyme conjugate "competes" with PCB present in the sample for binding to immobilized anti-PCB antibody.

2.3 The test is interpreted by comparing the response produced by testing a sample to the response produced by testing standard(s) simultaneously.

3.0 INTERFERENCES

Chemically similar compounds and compounds which might be expected to be found in conjunction with PCB contamination were tested to determine the concentration required to produce a positive test result. These data are shown in Tables 1A, 1B, 1C, and 1D.

4.0 APPARATUS AND MATERIALS

4.1 Immunoassay test kit: PCB RISc™ (EnSys, Inc.), EnviroGard™ PCB in Soil (Millipore, Inc.), D TECH ™ PCB test (Strategic Diagnostics Inc.), PCB RISc™ Liquid Waste Test System (EnSys, Inc.), or equivalent.

4.2 Each commercially available test kit will supply or specify the apparatus and materials necessary for successful completion of the test.

5.0 REAGENTS

Each commercially available test kit will supply or specify the reagents necessary for successful completion of the test.

6.0 SAMPLE COLLECTION, PRESERVATION, AND HANDLING

6.1 See the introductory material to this chapter, Organic Analytes, Section 4.1. Also refer to Reference 9 for the collection and handling of non-aqueous waste liquids.

6.2 Samples may be contaminated, and should therefore be considered hazardous and handled accordingly.

7.0 PROCEDURE

7.1 Follow the manufacturer's instructions for the test kit being used.

7.2 Those test kits used must meet or exceed the performance specifications indicated in Tables 2-11.

8.0 QUALITY CONTROL

8.1 Follow the manufacturer's instructions for the test kit being used for quality control procedures specific to the test kit used. Additionally, guidance provided in Method 4000 and Chapter One should be followed.

8.2 Use of replicate analyses, particularly when results indicate concentrations near the action level, is recommended to refine information gathered with the kit.

8.3 Do not use test kits past their expiration date.

8.4 Do not use tubes or reagents designated for use with other test kits.

8.5 Use the test kits within their specified storage temperature and operating temperature limits.

8.6 Method 4020 is intended for field or laboratory use. The appropriate level of quality assurance should accompany the application of this method to document data quality.

9.0 METHOD PERFORMANCE

9.1 A study was conducted with the PCB RISc™ test kit using fourteen standard soils and three soil samples whose PCB concentration had been established by Method 8082. Replicates were performed on seven of the standard soils and on one of the soil samples for a total of 25 separate analyses. Each of two different analysts ran the 25 analyses. Results indicated that "<" assignments are accurate with almost 99% certainty at the 50 ppm level while ">" assignments can be up to about 96% inaccurate as the sample concentration approaches that of the testing level. Corresponding certainties at the 5 ppm level are 92% and 82% respectively. Tables 2 and 3 summarize these results.

9.2 Table 4 presents method precision data generated using the PCB RISc™ test kit, comparing immunoassay test results with results obtained using Method 8082.

9.3 Method precision was determined with the EnviroGard PCB in Soil test kit by assaying 4 different soils (previously determined to contain 5.04, 9.78, 11.8, and 25.1 mg/kg by Method 8082), at three different sites, using three different lots of assay kits, three times a day for 9 days. A total of 81 analyses were performed for each soil. Error attributable to site, lot, date, and operator were determined. Separately, the relative reactivity of Aroclors 1242, 1248, 1254, and 1260 were determined. Based on Aroclor heterogeneity, and method imprecision, concentrations of Aroclor 1248 were selected that would result in greater than 99% confidence for negative interpretation. A study was conducted (Superfund SITE demonstration) on 114 field samples whose PCB concentration were also determined by Method 8082. 32 of the field samples were collected in duplicate (as coded field duplicates) and assayed by standard and immunoassay methods. The results for all 146 samples are summarized in Tables 5 and 6.

9.4 Grab samples were obtained from sites in Pennsylvania, Iowa and Illinois using a stainless steel trowel. Each sample was homogenized by placing approximately six cubic inches in a stainless steel bucket and mixing with the trowel for approximately two minutes. The soils was aliquotted into 2 six ounce glass bottles. The samples were tested on site using the D TECH PCB test kit, and sent to an analytical laboratory for analysis by Method 8082. These data are compared in Table 7.

9.5 Tables 8 and 9 present data on the inter- and intra-assay precision of the PCB RISc™ Liquid Waste Test System. The data were generated using 11 samples, each spiked at 0, 0.2 and 5 ppm, and assayed 4 times.

9.6 Tables 10 and 11 provide data from application of the PCB RISc™ Liquid Waste Test System to a series of liquid waste samples whose PCB concentration had been established by Method 8082.

10.0 REFERENCES

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10. PCB RISc™ Liquid Waste Test System, User's Guide, EnSys Environmental Products, Inc.

TABLE 1A

CROSS REACTIVITY OF DIFFERENT COMPOUNDS[a]

Compound	Soil Equivalent Concentration (ppm) Required to Yield a Positive Result
1-Chloronaphthalene	10,000
1,2,4-Trichlorobenzene	10,000
2,4-Dichlorophenyl-benzenesulfonate	1,000
2,4-Dichloro-1-naphthol	>10,000
Bifenox	500
Diesel fuel	>10,000
Pentachlorobenzene	>10,000
2,5-Dichloroaniline	>10,000
Hexachlorobenzene	>10,000
Gasoline	>10,000
Dichlorofenthion	10,000
Tetradifon	125

(a) PCB RISc™ test kit, Ensys, Inc. publication

TABLE 1B

CROSS REACTIVITY OF DIFFERENT COMPOUNDS[a]

Compound	% Cross Reactivity
Aroclor 1248	100
Aroclor 1242	50
Aroclor 1254	90
Aroclor 1260	50
1,2-, 1,3-, & 1,4-Dichlorobenzene	<0.5
1,2,4-Trichlorobenzene	<0.5
biphenyl	<0.5
2,4-dichlorophenol	<0.5
2,5-dichlorophenol	<0.5
2,4,5-trichlorophenol	<0.5
2,4,6-trichlorophenol	<0.5
Pentachlorophenol	<0.5

[a] EnviroGard PCB Test Kits (Millipore Corporation)

TABLE 1C

CROSS REACTIVITY OF DIFFERENT COMPOUNDS[a]

Compound	MDL[b] (ppm)	IC 50[c] (ppm)	% Cross Reactivity[d]
Aroclor 1016	5.7	83	12
Aroclor 1221	25.5	300	3
Aroclor 1232	9.0	105	10
Aroclor 1242	1.5	31	32
Aroclor 1248	0.8	24	42
Aroclor 1254	0.5	10	100
Aroclor 1260	0.75	10	100
Aroclor 1262	0.5	10	100
Aroclor 1268	3.8	40	25

METHOD: The compounds listed were assayed at various concentrations and compared against an inhibition curve generated using Aroclor 1254. The concentration of the compound required to elicit a positive response at the MDL as well as the concentration required to yield 50% inhibition compared to the standard curve were determined.

[a] D TECH™ PCB test kit

[b] The Minimum Detection Limit (MDL) is defined as the lowest concentration of compound that yields a positive test result.

[C] The IC_{50} is defined as the concentration of compound required to produce a test response equivalent to 50% of the maximum response.

[d] % Cross reactivity is determined by dividing the equivalent Aroclor 1254 concentration by the actual compound concentration at IC_{50}

TABLE 1D

CROSS REACTIVITY OF DIFFERENT COMPOUNDS[a]

Compound	% Cross-Reactivity	Soil Equivalent Concentration (ppm) Required to Yield a Positive Result
1-Chloronaphthalene	0.05%	10,000
1,2,4-Trichlorobenzene	0.05%	10,000
2,4-Dichloro-1-naphthol	<0.20%	>10,000
Bifenox	<0.10%	500
Pentachlorobenzene	<0.05%	>10,000
2,5-Dichloroaniline	<0.05%	>10,000
Hexachlorobenzene	<0.05%	>10,000
Dichlorofenthion	0.05%	10,000
Tetradifon	<0.10%	125

(a) PCB RISc™ Liquid Waste Test System, Ensys, Inc.

TABLE 2

ESTIMATED ERROR RATES FOR 5 PPM DILUTION[a]

True Value (ppm)	0	1	2	3	4	5	6	7	8	9	10	20
Estimated Rate of False Positives (%)	1.3	13.2	39.2	65.2	82.3	.	.	.	.	.	.	.
Estimated Rate of False Negatives (%)	.	.	.	.	.	8.5	4.1	2.0	1.0	0.5	0.3	<0.1

TABLE 3

ESTIMATED ERROR RATES FOR 50 PPM DILUTION[a]

True Value (ppm)	0	5	10	15	20	30	40	50	60	70	80	100
Estimated Rate of False Positives (%)	1.0	7.9	24.5	46.0	65.0	87.3	95.6	.	.	.	.	.
Estimated Rate of False Negatives (%)	.	.	.	.	.	.	.	1.7	0.7	0.3	0.2	<0.1

(a) PCB RISc™ test kit

TABLE 4

Comparison of PCB RISc™ Test Kit with GC

Sample ID	Screening Test Results	GC Results (Method 8082)	Agreement[a] Y, FP, FN
101	<5 ppm	<0.5 ppm	Y
284	<5 ppm	<0.5 ppm	Y
292	<5 ppm	<0.5 ppm	Y
199	<5 ppm	0.5 ppm	Y
264	<5 ppm	1 ppm	Y
257	<5 ppm	1.8 ppm	Y
259	<5 ppm	4 ppm	Y
265	<5 ppm	4.5 ppm	Y
200	<5 ppm	5 ppm	Y
170	5-50	5.8 ppm	Y
198	<5 ppm	2.2-5.8 ppm	Y
172	5-50	6.2 ppm	Y
169	5-50	7.2 ppm	Y
171	5-50	7.2 ppm	Y
202	<5 ppm, 5-50	1.3-7.2 ppm	Y
163	5-50	8.7 ppm	Y
165	5-50	9 ppm	Y
168	5-50	9 ppm	Y
166	5-50	9.3 ppm	Y
164	5-50	11.9 ppm	Y
204	5-50	12.8 ppm	Y
253	5-50	13 ppm	Y
203	5-50	13.5 ppm	Y
258	5-50	15 ppm	Y
106	5-50	15-19 ppm	Y
161	5-50	15.3 ppm	Y
167	5-50	16.2 ppm	Y

TABLE 4 (cont.)

Sample ID	Screening Test Results	GC Results (Method 8082)	Agreement[a] Y, FP, FN
247	5-50	18 ppm	Y
148	>50	18-34 ppm	FP
205	5-50	20 ppm	Y
162	5-50	20.4 ppm	Y
175	5-50	21.2 ppm	Y
176	5-50	21.6 ppm	Y
197	5-50	32 ppm	Y
243	5-50	32 ppm	Y
252	5-50	32 ppm	Y
178	5-50	43.7 ppm	Y
201	5-50	43 ppm	Y
254	5-50, >50	56 ppm	Y
238	>50	46-60 ppm	Y
248	5-50	44-60 ppm	Y
250	>50	68 ppm	Y
242	5-50	30-69 ppm	Y
256	>50	73 ppm	Y
249	>50	96 ppm	Y
245	>50	102 ppm	Y
241	5-50	154 ppm	FN
246	>50	154 ppm	Y
261	>50	204 ppm	Y
240	>50	251 ppm	Y
267	>50	339 ppm	Y
239	>50	460 ppm	Y
104	>50	200-3772 ppm	Y
108	>50	531-1450 ppm	Y

[a] Y=Yes, FN=False Negative, FP=False Positive

TABLE 5

Comparison of EnviroGard™ PCB Kit with GC

Sample Number	Screening Result[c,d]	GC Result[c] [8082]	Agreement[e] Y, FN, FP
001	>10	5.98	FP[g]
002	>10	1.27	FP
003	<10	0.11	Y
004	>10	6.71	FP[g]
005	>10	1.37	FP
006	>10	0.68	FP
007	>10	0.55	FP
008	>10	2.00	FP
009	>10	1.30	FP
010	>10	0.17	FP
011	>10	1.15	FP
012	<10	ND[f]	Y
013	<10	1.13	Y
014	<10	0.18	Y
015	>10	9.13	FP[g]
015	>10	9.84	FP[g]
016	>10	2110	Y
017	>10	2.55	FP
018	>10	45.4	Y
019	>10	6.70	FP[g]
020	<10	0.07	Y
021	<10	0.06	Y
022	<10	0.54	Y
022	<10	0.72	Y
023	>10	20.8	Y
024	<10	0.06	Y

TABLE 5 (cont.)

Sample Number	Screening Result[c,d]	GC Result[c] [8082]	Agreement[e] Y, FN, FP
024D	<10	0.05	Y
025	>10	11.7	Y
026	<10	1.96	Y
027	<10	0.06	Y
028	<10	0.22	Y
028D	<10	0.22	Y
029	<10	0.23	Y
030	<10	1.15	Y
031	<10	0.26	Y
032	>10	47.6	Y
033	>10	6.00	FP[g]
034	>10	34.0	Y
035	<10	ND[f]	Y
035D	<10	ND[f]	Y
036	>10	816	Y
037	<10	0.06	Y
037D	<10	0.04	Y
038	>10	1030	Y
039	<10	0.68	Y
040	>10	4.25	FP
041	<10	ND[f]	Y
042	>10	0.52	FP
042D	>10	0.47	FP
043	>10	1.69	FP
043D	>10	1.74	FP

TABLE 5 (cont.)

Sample Number	Screening Result[c,d]	GC Result[c] [8082]	Agreement[e] Y, FN, FP
044	<10	0.59	Y
045	<10	ND[f]	Y
046	<10	ND[f]	Y
046D	<10	ND[f]	Y
047	<10	0.09	Y
047D	<10	0.10	Y
048	<10	ND[d]	Y
049	<10	ND[d]	Y
050	>10	3.60	FP
050D	>10	4.41	FP
051	<10	ND[f]	Y
052	>10	4.21	FP
053	<10	0.96	Y
054	<10	0.52	Y
055	<10	2.40	Y
056	<10	0.51	Y
057	<10	ND[f]	Y
058	<10	0.69	Y
059	>10	7.86	FP[g]
060	>10	0.62	FP
060D	<10	0.58	Y
061	>10	580	Y
062	>10	2.35	FP
063	<10	0.09	Y
063D	<10	0.15	Y

TABLE 5 (cont.)

Sample Number	Screening Result[c,d]	GC Result[c] [8082]	Agreement[e] Y, FN, FP
064	>10	19.0	Y
065	>10	3.08	FP
066	<10	1.98	Y
067	<10	0.08	Y
068	<10	0.50	Y
069	<10	ND[f]	Y
069D	<10	ND[f]	Y
070	<10	ND[f]	Y
071	<10	0.05	Y
071D	<10	ND[f]	Y
072	<10	0.04	Y
073	>10	15.8	Y
074	>10	13.3	Y
075	>10	23.0	Y
076	>10	46.7	Y
077	<10	ND[f]	Y
078	>10	2.27	FP
079	>10	42.8	Y
080	<10	3.77	Y
081	<10	0.69	Y
081D	<10	0.45	Y
082	<10	ND[f]	Y
082D	<10	0.24	Y
083	<10	0.48	Y
083D	<10	0.41	Y
084	>10	1.16	FP

TABLE 5 (cont.)

Sample Number	Screening Result[c,d]	GC Result[c] [8082]	Agreement[e] Y, FN, FP
084D	>10	1.08	FP
085	>10	428	Y
085D	>10	465	Y
086	<10	1.42	Y
086D	<10	1.25	Y
087	<10	0.08	Y
087D	<10	ND[f]	Y
088	>10	2.70	FP
088D	>10	1.77	FP
089	>10	45.0	Y
090	<10	1.01	Y
090D	<10	1.40	Y
091	>10	1630	Y
091D	>10	1704	Y
092	<10	1.21	Y
092D	<10	ND[f]	Y
093	<10	0.30	Y
094	<10	0.36	Y
095	>10	17.5	Y
095D	>10	31.2	Y
096	<10	0.06	Y
097	<10	1.23	Y
097D	<10	0.29	Y
098	>10	1.17	FP
098D	>10	0.83	FP
099	<10	ND[f]	Y

TABLE 5 (cont.)

Sample Number	Screening Result[c,d]	GC Result[c] [8082]	Agreement[e] Y, FN, FP
100	>10	177	Y
100D	>10	167	Y
101	>10	1.21	FP
102	>10	293	Y
102D	>10	177	Y
103	>10	40.3	Y
104	>10	7.66	FP[g]
105	<10	0.21	Y
106	<10	2.50	Y
107	>10	14.1	Y
108	>10	3.84	FP
109	<10	ND[f]	Y
109D	<10	ND[f]	Y
110	<10	ND[f]	Y
111	<10	ND[f]	Y
112	>10	315	Y
113	>10	14.9	Y
114	>10	66.3	Y

[c] mg/kg (ppm)

[d] Screening Calibrator is 5 mg/kg Aroclor 1248

[e] Y=Yes, FN=False Negative, FP=False Positive

[f] ND = Not Detectable

[g] Expected Result Based on Calibrator Concentration

TABLE 6

EnviroGard™ PCB Kit Field Performance Summary

Specificity:	[1-(Reported Positives/True Negatives)] = [1-(37/109)] = 66%
Note 1:	8 of the 37 reported positive samples had PCB contamination levels between 5 and 10 mg/kg. Soils in this range should test "positive" because the assay calibrator is 5 mg/kg Aroclor 1248. A positive assay bias is necessary to prevent false negative results. Eliminating these samples from the calculations produces a Specificity of: [1-(Reported Positives/True Negatives)] = [1-(29/101)] = 71%
Note 2:	The distribution of false positives is not random ($p < 0.05$), with a clustering at the beginning of the sample set. This observation was included in *Developers Comments* which were added to the final draft of the Technical Evaluation Report. One explanation for the higher frequency of false positive results at the beginning is inexperience of the operator with the method. If the first 20 samples are eliminated from the Specificity analysis, the following result is obtained: [1-(Reported Positives/True Negatives)] = [1-(20/86)] = 77% In the SITE demonstration, the PCB Immunoassay had a 77% positive predictive value.
Sensitivity:	[1-(Reported Negatives/True Positives)] = [1-(0/31)] = 100% In the SITE demonstration, the PCB Immunoassay had a 100% negative predictive value.

TABLE 7

Comparison of D TECH™ PCB Test Kit with GC

Sample	D TECH™ (ppm)	GC (8082) (ppm)	Agreement[a] Y, FN, FP
J1	4.0-15	5.0	Y
J2	>50	147	Y
J3	15-50	54	Y
J5	15-50	160	FN
J6	>50	1200	Y
J7	4.0-15	12	Y
J8	4.0-15	28	FN
J9	>50	463	Y
J10	>50	1760	Y
J11	>50	28	FP
J12	15-50	17	Y
J13	>50	1300	Y
J14	>50	186	Y
J15	15-50	31	Y
J16	15-50	36	Y
J17	>50	31	FP
J18	>50	130	Y
J19	>50	1310	Y
J20	>50	2620	Y
J21	>50	111000	Y
J22	1.0-4.0	0.01	FP
J23	1.0-4.0	0.60	Y
J24	<0.5	0.10	Y

[a] Y=Yes, FN=False Negative, FP=False Positive

TABLE 7 (cont.)

Sample	D TECH™ (ppm)	GC (8082) (ppm)	Agreement[a] Y, FN, FP
J25	0.5-1.0	0.12	FP
J26	<0.5	0.01	Y
J27	1.0-4.0	1.8	Y
J28	<0.5	0.18	Y
J29	0.5-1.0	0.54	Y
J30	>50	21	FP
J31	4.0-15	13	Y
J32	0.5-1.0	0.72	Y
J33	0.5-1.0	0.32	Y
J34	1.0-4.0	0.36	FP
J35	1.0-4.0	0.26	FP
J36	>50	70	Y
J37	<0.5	0.12	Y
J38	0.5-1.0	0.81	Y
J39	0.5-1.0	0.33	Y
J40	<0.5	0.19	Y
J41	<0.5	0.01	Y
J42	1.0-4.0	0.43	FP
J43	1.0-4.0	0.31	FP
J44	15-50	503.4	FN
J45	15-50	5.6	FP
J46	<0.5	0.02	Y
J47	<0.5	0.22	Y

[a] Y=Yes, FN=False Negative, FP=False Positive

TABLE 7(cont.)

Sample	D TECH™ (ppm)	GC (8082) (ppm)	Agreement[a] Y, FN, FP
G1	15-50	18	Y
G2	4.0-15	11	Y
G3	1.0-4.0	3.4	Y
G4	15-50	6.5	FP
G5	<0.5	0.01	Y
G6	1.0-4.0	1.4	Y
G7	1.0-4.0	0.30	FP
G8	15-50	7.5	FP
G9	4.0-15	33	FN
G10	15-50	8	FP
G11	4.0-15	11	Y
G12	4.0-15	24	FN
G13	4.0-15	4.3	Y
G14	0.5-1.0	1.3	Y
G15	<0.5	0.01	Y
G16	1.0-4.0	3.2	Y
G17	4.0-15	18	Y
G18	4.0-15	4.6	Y
G19	1.0-4.0	2.3	Y
G20	>50	37	FP

[a] Y=Yes, FN=False Negative, FP=False Positive

TABLE 7(cont.)

Sample	D TECH™ (ppm)	GC (8082) (ppm)	Agreement[a] Y, FN, FP
W1A	4.0-15	9.1	Y
W2A	4.0-15	11	Y
W3A	1.0-4.0	2.8	Y
W4A	4.0-15	13	Y
W5A	>50	29	FP
W6A	>50	1200	Y
W7A	>50	57	Y
W8A	4.0-15	18	Y
W9A	1.0-4.0	1.3	Y
W10A	0.5-1.0	0.44	Y
W11A	15-50	120	FN
W12A	15-50	48	Y
W13A	15-50	19	Y
W14A	4.0-15	2.7	Y
W15A	1.0-4.0	1.3	Y
W16A	1.0-4.0	0.3	FP
W17A	4.0-15	1.4	FP
W18A	1.0-4.0	2.2	Y
W19A	4.0-15	8.2	Y
W20A	>50	9.3	FP
W21A	>50	110	Y
W22A	1.0-4.0	0.6	Y
W23A	>50	46	Y

[a] Y=Yes, FN=False Negative, FP=False Positive

TABLE 8

Intraassay Precision of the PCB RISc™ Liquid Waste Test System

PCB 1248 Spike Concentration (ppm)	Signal %RSD (OD_{450nm}) N=44 (11 data sets)	Statistical Percentage of False Results Compared to Standards
0	6.4%	<0.02%
0.2	5.9%	4.1%
5	7.9%	1.4%

TABLE 9

Interassay Precision of the PCB RISc™ Liquid Waste Test System

PCB 1248 Spike Concentration (ppm)	Signal %RSD (OD_{450nm}) N=44 (11 data sets)
0	6.4%
0.2	8.3%
5	8.5%

TABLE 10

Comparison of PCB RISc™ Liquid Waste Test with Method 8082

Sample ID	Sample Matrix	GC Results		IA Results	
		Aroclor	Conc. ppm	Test Results	Corr. with GC Results
302	Condensate	ND[b]	ND	<5	yes
303	Condensate	ND	ND	<5	yes
304	Condensate	1242	25	≥5	yes
306	Condensate	1242	5	≥5	yes
307	Condensate	1242	<10	<5	yes
308	Condensate	1242	58	≥5	yes
310	Condensate	1254	25	≥5	yes
311	Condensate	1242	200	≥5	yes
331	Transformer Oil	1260	183	≥5	yes
380	Transformer Oil	PCB[c]	20	≥5	yes
381	Transformer Oil	PCB	38	≥5	yes
382	Transformer Oil	PCB	163	≥5	yes
383	Transformer Oil	PCB	176	≥5	yes
384	Transformer Oil	PCB	336	≥5	yes
385	Transformer Oil	PCB	6400	≥5	yes
387	Coolant	PCB	10	≥5	yes
388	2,4-D Rinse Water	1254	<10	<5	yes
389	Waste Solvent	1242	29	≥5	yes
390	Herbicide	ND	<2	<5	yes
391	Paint/Solvent	1254	9	≥5	yes
394	Waste Solvent	1242/1260	11/17	≥5	yes
395	Waste Solvent	1242/1260	2/2	<5	yes
396	Waste Oil	1260	323	≥5	yes
398	Chlor. Solvent	ND	<5	<5	yes
399	Paint	ND	<50	<5	yes
400	Pump Oil	ND	<50	<5	yes
401	Waste Solvent	ND	<35	<5	yes
402	Herbicide	ND	<50	<5	yes
403	Paint/Solvent	ND	<5	<5	yes
404	Printing Solvent	ND	<5	<5	yes
405	Waste Solvent	ND	<50	<5	yes

TABLE 10 (cont.)

Sample ID	Sample Matrix	GC Results		IA Results	
		Aroclor	Conc. ppm	Test Results	Corr. with GC Results
407	Waste Oil	ND	ND	≥5	FP[d]
408	Waste Oil	ND	ND	<5	yes
409	Waste Oil	ND	ND	<5	yes
410	Waste Oil	ND	ND	<5	yes
411	Waste Oil	ND	ND	<5	yes
412	Waste Oil	ND	ND	<5	yes
413	Waste Oil	ND	ND	<5	yes
414	Waste Oil	ND	ND	<5	yes
415	Waste Oil	ND	ND	<5	yes
416	Waste Oil	PCB	50	>5	yes
417	Waste Oil	ND	ND	<5	yes
418	Waste Oil	ND	ND	<5	yes
419	Waste Oil	ND	ND	<5	yes
420	Waste Oil	ND	ND	<5	yes
421	Waste Oil	ND	ND	<5	yes
422	Waste Oil	ND	ND	<5	yes
423	Waste Oil	ND	ND	<5	yes
424	Waste Oil	ND	ND	<5	yes
425	Waste Oil	ND	ND	<5	yes
Number of False Positive Results				1/32	
Rate				3.1%	
Number of False Negative Results				0/18	
Rate				0.0%	

[a] Trial 1 data

[b] ND = Not Detectable

[c] PCB = Aroclor was not determined

[d] FP = False positive

TABLE 11

Correlation of PCB RISc™ Liquid Waste Test and Method 8082 Results
Using Spiked and Unspiked Liquid Waste Field Samples

ID	Matrix	GC Results Unspiked ppm	Immunoassay Result		Interp.
			Unspiked ppm	Spiked (5 ppm 1248)	
001	Aromatic solvent	<5	<5	≥5	
002	Aviation gas	<5	<5	≥5	
003	Chiller oil	<5	<5	≥5	
004	Compressor oil	<5	<5	≥5	
005	Coolant + water	<5	<5	≥5	
006	Coolant oil	NR[b]	NR	≥5	
007	Coolant oil	NR	<5	≥5	
008	Cutting oil	<5	<5	≥5	
009	Cutting oil	<5	<5	≥5	
010	Degreaser still bottom	<5	<5	≥5	
011	Dope oil	<5	<5	≥5	
012	Draw Lube oil	<5	<5	≥5	
013	Fleet crankcase oil	<5	<5	≥5	
014	Floor sealer	<5	<5	≥5	
015	Fuel oil	<5	<5	≥5	
016	Hi-BTU oil	<5	<5	≥5	
017	Honing oil	<5	<5	≥5	
018	Hydraulic oil	<5	<5	≥5	
019	Hydraulic oil	<5	<5	≥5	
020	Hydraulic oil	<5	<5	≥5	
021	Machine oil	NR	<5	NR	
022	Mineral oil	<5	<5	≥5	
023	Mineral spirits	<5	<5	≥5	
024	Mineral spirits + ink	<5	≥5	≥5	FP
025	Mixed flammables	<5	<5	≥5	
026	Mixed solvents	<5	<5	≥5	
027	Naphtha	<5	<5	≥5	
028	Oil	<5	<5	≥5	
029	Oil	<5	<5	≥5	
030	Oil	<5	<5	≥5	
031	Oil	<5	<5	≥5	

TABLE 11 (cont.)

ID	Matrix	GC Results Unspiked ppm	Immunoassay Result		Interp.
			Unspiked ppm	Spiked (5 ppm 1248)	
032	Oil	<5	<5	≥5	
033	Oil	<5	<5	≥5	
034	Oil + 1,1,1-trichloroethane	<5	<5	≥5	
035	Oil sludge	<5	≥5	≥5	FP
036	Oil + freon	<5	<5	≥5	
037	Oil + mineral spirits	<5	<5	≥5	
038	Oil + scum solution	<5	<5	≥5	
039	Oily water	<5	<5	≥5	
040	Paint thinner	<5	<5	≥5	
041	Paint thinner	<5	<5	≥5	
042	Paint thinner	<5	<5	≥5	
043	Paint waste	<5	<5	≥5	
044	Paint waste + thinner	<5	<5	≥5	
045	Perce + oil	<5	<5	≥5	
046	Petroleum distillates	<5	≥5	≥5	FP
047	Petroleum naphtha	<5	<5	≥5	
048	Pumping oil	<5	<5	≥5	
049	RAC-1 SKOS	<5	<5	≥5	
050	Sk oil	NR	<5	≥5	
051	Sk oil	<5	<5	≥5	
052	Smog Hog	<5	<5	≥5	
053	Toluene + hexane	<5	<5	≥5	
054	Toluene + stain	<5	<5	≥5	
055	1,1,1-Trichloroethane	<5	≥5	≥5	FP
056	1,1,1-Trichloroethane	<5	<5	≥5	
057	1,1,1-Trichloroethane	<5	<5	≥5	
058	1,1,1-Trichloroethane	<5	<5	≥5	
059	1,1,1-TCE + methanol	<5	<5	≥5	
060	Trichloroethylene	<5	<5	≥5	
061	Trichloroethylene	<5	<5	≥5	
062	Trichloroethylene	<5	<5	≥5	
063	Turpentine	<5	<5	≥5	

TABLE 11 (cont.)

ID	Matrix	GC Results Unspiked ppm	Immunoassay Result		Interp.
			Unspiked ppm	Spiked (5 ppm 1248)	
064	Used n-butylacetate	<5	<5	≥5	
065	Used oil + freon	<5	<5	≥5	
066	Used oil + freon	<5	<5	≥5	
067	Used oils	<5	<5	≥5	
068	Used petroleum	<5	<5	≥5	
069	Used petroleum	<5	<5	≥5	
070	Used synthetic oil	<5	<5	≥5	
071	Varnish + stain	<5	<5	≥5	
072	Varsol	<5	<5	≥5	
073	Waste coolant + oil	<5	<5	≥5	
074	Waste ink + solvent	<5	<5	≥5	
075	Waste naphtha	<5	<5	≥5	
076	Waste oil	<5	<5	≥5	
077	Waste oil	<5	<5	≥5	
078	Waste oil	<5	<5	≥5	
079	Waste oil	<5	<5	≥5	
080	Waste oil	<5	<5	≥5	
081	Waste oil	<5	<5	≥5	
082	Waste oil	<5	<5	≥5	
083	Waste oil	<5	<5	≥5	
084	Waste oil	<5	<5	≥5	
085	Waste oil + kerosene	<5	<5	≥5	
086	Waste oil + gas	<5	<5	≥5	
087	Waste paint	<5	<5	≥5	
088	Waste paint	<5	<5	≥5	
089	Waste paint	<5	<5	≥5	
090	Waste paint	<5	<5	≥5	
091	Waste paint	<5	<5	≥5	
092	Waste paint	<5	<5	≥5	FP
093	Waste SC-49 solvent	<5	<5	≥5	
094	Waste solvent	<5	<5	≥5	
095	Waste stoddard	<5	<5	≥5	
096	Waste toner	<5	<5	≥5	

TABLE 11 (cont.)

ID	Matrix	GC Results Unspiked ppm	Immunoassay Result		Interp.
			Unspiked ppm	Spiked (5 ppm 1248)	
097	Waste tramp oil	<5	<5	≥5	
098	Waste transmission fluid	<5	<5	≥5	
099	Xylene	<5	≥5	≥5	FP
100	Not Recorded	<5	<5	NR	
No. of False Positive Results		6/99			
Rate		6.1%			
No. of False Negative Results				0/98	
Rate				0.0%	

[a] Trial 2 data

[b] NR = not run

METHOD 4035

SOIL SCREENING FOR POLYNUCLEAR AROMATIC HYDROCARBONS BY IMMUNOASSAY

1.0 SCOPE AND APPLICATION

1.1 Method 4035 is a procedure for screening soils to determine when total polynuclear aromatic hydrocarbons (PAHs) are present at concentrations above 1 mg/kg. Method 4035 provides an estimate for the concentration of PAHs by comparison with a PAH standard.

1.2 Using the test kit from which this method was developed, $\geq$95% of samples confirmed to have concentrations of PAHs below detection limits will produce a negative result in the 1 ppm test configuration.

1.3 The sensitivity of the test is influenced by the binding of the target analyte to the antibodies used in the kit. The commercial PAH kit used for evaluation of this method is most sensitive to the three (i.e., phenanthrene, anthracene, fluorene) and four (i.e., benzo(a)anthracene, chrysene, fluoranthene, pyrene) ring PAH compounds listed in Method 8310, and also recognizes most of the five and six ring compounds listed.

1.4 The sensitivity of the test is influenced by the nature of the PAH contamination and any degradation processes operating at a site. Although the action level of the test may vary from site to site, the test should produce internally consistent results at any given site.

1.5 In cases where the exact concentration of PAHs are required, quantitative techniques (i.e., Methods 8310, 8270, or 8100) should be used).

1.6 This method is restricted to use by or under the supervision of trained analysts. Each analyst must demonstrate the ability to generate acceptable results with this method.

2.0 SUMMARY OF METHOD

2.1 An accurately weighed sample is first extracted and the extract filtered using a commercially available test kit. The sample extract and an enzyme conjugate reagent are added to immobilized antibody. The enzyme conjugate "competes" with the PAHs present in the sample for binding to the immobilized anti-PAH antibody. The test is interpreted by comparing the response produced by testing a sample to the response produced by testing standard(s) simultaneously.

2.2 A portion of all samples in each analytical batch should be confirmed using quantitative techniques.

3.0 INTERFERENCES

3.1 Chemically similar compounds and compounds which might be expected to be found in conjunction with PAH contamination were tested to determine the concentration required to produce a positive result. These data are shown in Tables 1 and 2.

3.2 The kit was optimized to respond to three and four ring PAHs. The sensitivity of the test to individual PAHs is highly variable. Naphthalene, dibenzo(a,h)anthracene, and

benzo(g,h,i)perylene have 0.5 percent or less than the reactivity of phenanthrene with the enzyme conjugate.

3.3 The alkyl-substituted PAHs, chlorinated aromatic compounds, and other aromatic hydrocarbons, such as dibenzofuran, have been demonstrated to be cross-reactive with the immobilized anti-PAH antibody. The presence of these compounds in the sample may contribute to false positives.

4.0 APPARATUS AND MATERIALS

PAH RISc™ Soil Test (EnSys, Inc.), or equivalent. Each commercially available test kit will supply or specify the apparatus and materials necessary for successful completion of the test.

5.0 REAGENTS

Each commercially available test kit will supply or specify the reagents necessary for successful completion of the test.

6.0 SAMPLE COLLECTION, PRESERVATION, AND HANDLING

6.1 See the introductory material to this chapter, Organic Analytes, Sec. 4.1.

6.2 Soil samples may be contaminated, and should therefore be considered hazardous and handled accordingly.

7.0 PROCEDURE

7.1 Method 4035 is intended for field or laboratory use.

7.2 Follow the manufacturer's instructions for the test being used. Those test kits used must meet or exceed the performance indicated in Tables 3-7.

7.3 The action limit for each application must be within the operating range of the kit used.

8.0 QUALITY CONTROL

8.1 Follow the manufacturer's instructions for the test kit being used for quality control procedures specific to the test kit used. Additionally, guidance provided in Chapter One should be followed.

8.2 Use of replicate analyses, particularly when results indicate concentrations near the action level, is recommended to refine information gathered with the kit.

8.3 Do not use test kits past their expiration date.

8.4 Do not use tubes or reagents designated for use with other kits.

8.5 Use the test kits within the specified storage temperature and operating temperature limits.

9.0 METHOD PERFORMANCE

9.1 The extraction efficiency of a commercially available test kit was tested (PAH RISc™ Test, EnSys Inc.) by spiking phenanthrene, benzo(a)anthracene and benzo(a)pyrene into PAH negative soil matrices (PAH-116 and PAH-141 are field samples). The soils were spiked using detection limits established for each compound (see Table 1), extracted and determined by immunoassay. The results for these 3-, 4- and 5-ring PAHs (Table 4) demonstrated that they were extracted with good recovery and yielded the correct assay interpretation.

9.2 A single laboratory study was conducted with a commercially available test kit (PAH RISc™ Test, EnSys Inc.), using 25 contaminated soil samples. Four replicate determinations were made on each test sample and the data compared with values obtained using HPLC Method 8310. Several analysts performed the immunoassay analyses. The immunoassay data agreed in all cases with the external HPLC data obtained (Table 5).

9.3 An additional single laboratory validation study on 30 randomly selected, PAH-contaminated field samples from multiple sites was run by the USEPA Region X Laboratory. Results are reported in Table 6 on an as found basis, and reported in Table 7 normalized to phenanthrene, based on cross-reactivity data (from Table 1). The false positive rate at the 1 ppm action level was 13% for unnormalized results and 19% for normalized results based on 31 analyses. The false negative rate at 1 ppm was 0 in both cases. At the 10 ppm action level, the false positive rate was 19% unnormalized and 26% normalized. False negative rates at 10 ppm were 6% unnormalized and 3% normalized.

9.4 The probabilities of generating false positive and false negative results at an action level of 1 ppm are listed in Table 3.

10.0 REFERENCES

1. PAH-RISc™ Users Guide, EnSys Inc.

2. P. P. McDonald, R. E. Almond, J. P. Mapes, and S. B. Friedman, "PAH-RISc™ Soil Test - A Rapid, On-Site Screening Test for Polynuclear Aromatic Hydrocarbons in Soil", J. of AOAC International (accepted for publication document #92263)

3. R. P. Swift, J. R. Leavell, and C. W. Brandenburg, "Evaluation of the EnSys PAH-RISc™ Test Kit", Proceedings, USEPA Ninth Annual Waste Testing and Quality Assurance Symposium, 1993.

TABLE 1

Cross-reactivity of Method 8310 PAHs

Compound	Concentration Giving a Positive Result (ppm Soil Equivalent)	Percent Cross-Reactivity
2 Rings		
Naphthalene	200	0.5
3 Rings		
Acenaphthene	8.1	12
Acenaphthylene	7.5	13
Phenanthrene	**1.0**	**100**
Anthracene	0.81	123
Fluorene	1.5	67
4 Rings		
Benzo(a)anthracene	1.6	64
Chrysene	1.2	84
Fluoranthene	1.4	73
Pyrene	3.5	29
5 Rings		
Benzo(b)fluoranthene	4.6	22
Benzo(k)fluoranthene	9.4	11
Benzo(a)pyrene	8.3	12
Dibenzo(a,h)anthracene	>200	<0.5
6 Rings		
Indeno(1,2,3-c,d)pyrene	11	9.4
Benzo(g,h,i)perylene	>200	<0.5

TABLE 2

Cross Reactivity of Other PAHs and Related Compounds

Compound	Concentration Giving a Positive Result (ppm, Soil Equivalent)	Percent Cross-Reactivity
Other PAHs		
1-Methylnaphthylene	54	1.8
2-Methylnaphthylene	58	1.7
1-Chloronaphthylene	59	1.7
Halowax 1013	18	5.7
Halowax 1051	>200	<0.5
Dibenzofuran	14	7.2
Other Compounds		
Benzene	>200	<0.5
Toluene	>200	<0.5
CCA	>200	<0.5
Phenol	>200	<0.5
Creosote	5.4	18.5
2,4,6-Trichlorobenzene	>200	<0.5
2,3,5,6-Tetrachlorobenzene	>200	<0.5
Pentachlorobenzene	>200	<0.5
Pentachlorophenol	>200	<0.5
Bis(2-ethylhexyl) phthalate	>200	<0.5
Aroclor 1254	>200	<0.5
Aroclor 1260	>200	<0.5

TABLE 3

Probability of False Negative and False Positive Results
for PAHs at A 1 ppm Action Level

Spike Concentration Phenanthrene (ppm)	Probability of False Positive (Mean ± SD)	Probability of False Negative (Mean ± SD)
0	0% ± 0%	N/A
0.4	23% ± 17%	N/A
0.8	94% ± 13%	N/A
1.0	N/A	0% ± 0%

Results were obtained from spiking four different validation lots, using 3 operators, 12 matrices for a total of 201 determinations at each concentration of phenanthrene.

N/A = No false positive or negative possible above action limit.

TABLE 4

Spike Recovery of Phenanthrene, Benzo(a)anthracene and Benzo(a)pyrene

Compound	Spike (ppm)	Soil	PAH RISc™ Results
Blank	0	Wake	<1
Blank	0	PAH-116	<1
Phenanthrene	1	Wake	1-10
Phenanthrene	1	PAH-116	1-10
Phenanthrene	1	PAH-141	1-10
Phenanthrene	10	Wake	>10
Phenanthrene	10	PAH-116	>10
Phenanthrene	10	PAH-141	>10
Benzo(a)anthracene	1.6	Wake	1-10
Benzo(a)anthracene	1.6	PAH-116	1-10
Benzo(a)anthracene	16	Wake	>10
Benzo(a)anthracene	16	PAH-116	>10
Benzo(a)pyrene	8.3	Wake	1-10
Benzo(a)pyrene	8.3	PAH-116	1-10
Benzo(a)pyrene	83	PAH-116	>10

TABLE 5

Powerplant Field Samples (Soil) Evaluated by Immunoassay

Field Sample Number	EnSys Method Immunoassay (ppm)	Method 8310 HPLC (ppm)
PAH-137	>10	<21
PAH-141	<1	<21
PAH-118	1-10	<26
PAH-136	>10	26
PAH-139	>10	<28
PAH-126	1-10, >10	<32
PAH-127	>10	<33
PAH-122	>10	<33
PAH-138	>10	33
PAH-131	>10	<34
PAH-128	>10	<35
PAH-132	>10	<43
PAH-112	>10	<48
PAH-140	>10	50
PAH-130	>10	54
PAH-116	<1	<61
PAH-135	>10	71
PAH-133	>10	<91
PAH-119	>10	<100
PAH-120	>10	<161
PAH-124	>10	<167
PAH-134	>10	182
PAH-114	>10	<247
PAH-113	>10	<294
PAH-115	>10	<343

TABLE 6

Total PAH Content of Region X Field Samples Using EnSys
PAH RISc™ Immunoassay Test Kit

Sample ID	1 ppm Test		10 ppm Test		GC/MS Lab Result (ppm)[1]	False +/-	
	<1	>1	<10	>10		Eval @ 1 ppm	Eval @ 10 ppm
PAH-1		*		*	0.2	+	+
PAH-2				*	12.2		
PAH-3				*	16.0		
PAH-4	*				0.00		
PAH-5	*				0.5		
PAH-6		*		*	8.7		+
PAH-7				*	148		
PAH-8				*	182		
PAH-9		*		*	4.4		+
PAH-10		*		*	0.2	+	+
PAH-11	*				0.00		
PAH-12				*	85.4		
PAH-12Dup				*	85.4		
PAH-13				*	28.5		
PAH-14	*		*		0.3		
PAH-15		*			0.6	+	
PAH-16	*		*		0.00		
PAH-17		*		*	1.8		+
PAH-18		*	*		3.4		
PAH-19		*	*		6.7		
PAH-20	*		*		0.9		
PAH-21				*	43.2		

[1] Sum of all PAHs detected.

TABLE 6 (cont.)

Sample ID	1 ppm Test		10 ppm Test		GC/MS Lab Result (ppm)[1]	False +/-	
	<1	>1	<10	>10		Eval @ 1 ppm	Eval @ 10 ppm
PAH-22				*	72.8		
PAH-23		*		*	1.3		+
PAH-24		*	*		0.3	+	
PAH-25	*		*		0.4		
PAH-26			*		27.9		-
PAH-27	*		*		0.00		
PAH-28			*		16.4		-
PAH-29	*		*		0.4		
PAH-30		*	*		9.6		

TABLE 7

Total PAH Content of Region X Field Samples Using EnSys
PAH RISc™ Immunoassay Test Kit Normalized to Cross-reactivity

Sample ID	1 ppm Test		10 ppm Test		GC/MS Lab Result (ppm)[1]	False +/-	
	<1	>1	<10	>10		Eval @ 1 ppm	Eval @ 10 ppm
PAH-1		*		*	0.1	+	+
PAH-2				*	8.1		+
PAH-3				*	9.0		+
PAH-4	*				0.00		
PAH-5	*				0.2		
PAH-6		*		*	5.2		+
PAH-7				*	56.9		
PAH-8				*	73.2		

[1] Sum o f all PAHs detected.

TABLE 7 (cont.)

Sample ID	1 ppm Test		10 ppm Test		GC/MS Lab Result (ppm) [1]	False +/-	
	<1	>1	<10	>10		Eval @ 1 ppm	Eval @ 10 ppm
PAH-9		*		*	0.1	+	+
PAH-10		*		*	0.00	+	+
PAH-11	*				0.00		
PAH-12				*	47.3		
PAH-12Dup				*	47.3		
PAH-13				*	11.5		
PAH-14	*		*		0.2		
PAH-15		*			0.5	+	
PAH-16	*		*		0.00		
PAH-17		*		*	1.2		+
PAH-18		*	*		1.7		
PAH-19		*	*		3.6		
PAH-20	*		*		0.6		
PAH-21				*	27.5		
PAH-22				*	49.2		
PAH-23		*		*	0.8	+	+
PAH-24		*	*		0.1	+	
PAH-25	*		*		0.2		
PAH-26			*		13.5		-
PAH-27	*		*		0.00		
PAH-28			*		6.4		
PAH-29	*		*		0.2		
PAH-30		*	*		2.8		

[1] Sum of all PAHs detected.

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

SW-846 is not intended to be an analytical training manual. Therefore, method procedures are written based on the assumption that they will be performed by analysts who are formally trained in at least the basic principles of chemical analysis and in the use of the subject technology.

In addition, SW-846 methods, with the exception of required method use for the analysis of method-defined parameters, are intended to be guidance methods which contain general information on how to perform an analytical procedure or technique which a laboratory can use as a basic starting point for generating its own detailed Standard Operating Procedure (SOP), either for its own general use or for a specific project application. The performance data included in this method are for guidance purposes only, and are not intended to be and must not be used as absolute QC acceptance criteria for purposes of laboratory accreditation.

1.0 SCOPE AND APPLICATION

1.1 This method is applicable to the in situ and intrusive analysis of the 26 analytes listed below for soil and sediment samples. Some common elements are not listed in this method because they are considered "light" elements that cannot be detected by field portable x-ray fluorescence (FPXRF). These light elements are: lithium, beryllium, sodium, magnesium, aluminum, silicon, and phosphorus. Most of the analytes listed below 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. The following RCRA analytes have been determined by this method:

Analytes	CAS Registry No.
Antimony (Sb)	7440-36-0
Arsenic (As)	7440-38-0
Barium (Ba)	7440-39-3
Cadmium (Cd)	7440-43-9
Chromium (Cr)	7440-47-3
Cobalt (Co)	7440-48-4
Copper (Cu)	7440-50-8
Lead (Pb)	7439-92-1
Mercury (Hg)	7439-97-6
Nickel (Ni)	7440-02-0
Selenium (Se)	7782-49-2
Silver (Ag)	7440-22-4
Thallium (Tl)	7440-28-0
Tin (Sn)	7440-31-5

Analytes	CAS Registry No.
Vanadium (V)	7440-62-2
Zinc (Zn)	7440-66-6

In addition, the following non-RCRA analytes have been determined by this method:

Analytes	CAS Registry No.
Calcium (Ca)	7440-70-2
Iron (Fe)	7439-89-6
Manganese (Mn)	7439-96-5
Molybdenum (Mo)	7439-93-7
Potassium (K)	7440-09-7
Rubidium (Rb)	7440-17-7
Strontium (Sr)	7440-24-6
Thorium (Th)	7440-29-1
Titanium (Ti)	7440-32-6
Zirconium (Zr)	7440-67-7

1.2 This method is a screening method to be used with confirmatory analysis using other techniques (e.g., flame atomic absorption spectrometry (FLAA), graphite furnance atomic absorption spectrometry (GFAA), inductively coupled plasma-atomic emission spectrometry, (ICP-AES), or inductively coupled plasma-mass spectrometry, (ICP-MS)). This method's main strength is that it is a rapid field screening procedure. The method's lower limits of detection are typically above the toxicity characteristic regulatory level for most RCRA analytes. However, when the obtainable values for precision, accuracy, and laboratory-established sensitivity of this method meet project-specific data quality objectives (DQOs), FPXRF is a fast, powerful, cost effective technology for site characterization.

1.3 The method sensitivity or lower limit of detection depends on several factors, including 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. Example lower limits of detection for analytes of interest in environmental applications are shown in Table 1. These limits apply to a clean spiked matrix of quartz sand (silicon dioxide) free of interelement spectral interferences using long (100 -600 second) count times. These sensitivity values are given for guidance only and may not always be achievable, since they will vary depending on the sample matrix, which instrument is used, and operating conditions. A discussion of performance-based sensitivity is presented in Sec. 9.6.

1.4 Analysts should consult the disclaimer statement at the front of the manual and the information in Chapter Two for guidance on the intended flexibility in the choice of methods, apparatus, materials, reagents, and supplies, and on the responsibilities of the analyst for demonstrating that the techniques employed are appropriate for the analytes of interest, in the matrix of interest, and at the levels of concern.

In addition, analysts and data users are advised that, except where explicitly specified in a regulation, the use of SW-846 methods is *not* mandatory in response to Federal testing requirements. The information contained in this method is provided by EPA as guidance to be used by the analyst and the regulated community in making judgments necessary to generate results that meet the data quality objectives for the intended application.

1.5 Use of this method is restricted to use by, or under supervision of, personnel appropriately experienced and trained in the use and operation of an XRF instrument. Each analyst must demonstrate the ability to generate acceptable results with this method.

2.0 SUMMARY OF METHOD

2.1 The FPXRF technologies described in this method use either sealed radioisotope sources or x-ray tubes to irradiate samples with x-rays. When a sample is irradiated with x-rays, the source x-rays may undergo either scattering or absorption by sample atoms. This latter 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 three electron shells include 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), beta (β), or gamma (γ) etc., 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 15.77 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, specifically, 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 standards.

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, Chapter Three, and the manufacturer's instructions for other definitions that may be relevant to this procedure.

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 (i.e., against the cup window), 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 reporting of a "nondetect" or a "less than" value (e.g., <300 ppm) for As, 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 using other techniques (e.g., flame atomic absorption spectrometry (FLAA), graphite furnance atomic absorption spectrometry (GFAA), inductively coupled plasma-

atomic emission spectrometry, (ICP-AES), or inductively coupled plasma-mass spectrometry, (ICP-MS)).

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 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-specific data quality objectives (DQOs).

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 (see Table 8), the confirmatory method used was Method 3050, and the FPXRF data compared very well with regression correlation coefficients (r often exceeding 0.95, except for barium and chromium). The critical factor is that the digestion procedure and analytical reference method used should meet the 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 Sec. 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° F. The operator should follow the manufacturer's recommendations for gain check frequency.

5.0 SAFETY

5.1 This method does not address all safety issues associated with its use. The user is responsible for maintaining a safe work environment and a current awareness file of OSHA regulations regarding the safe handling of the chemicals listed in this method. A reference file of material safety data sheets (MSDSs) should be available to all personnel involved in these analyses.

NOTE: No MSDS applies directly to the radiation-producing instrument because that is covered under the Nuclear Regulatory Commission (NRC) or applicable state regulations.

5.2 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 operator's 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. 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.

Licenses for radioactive materials are of two types, specifically: (1) a general license which is usually initiated by the manufacturer for receiving, acquiring, owning, possessing, using, and transferring radioactive material incorporated in a device or equipment, and (2) a specific license which is issued to named persons for the operation of radioactive instruments as required by local, state, or federal agencies. A copy of the radioactive material license (for specific licenses only) 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. An additional hazard present with x-ray tubes is the danger of electric shock from the high voltage supply, however, if the tube is properly positioned within the instrument, this is only a negligible risk. Any instrument (x-ray tube or radioisotope based) is capable of delivering an electric shock from the basic circuitry when the system is inappropriately opened.

5.3 Radiation monitoring equipment should be used with the handling and operation 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 or badges should be worn in the area of maximum 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.

6.0 EQUIPMENT AND SUPPLIES

The mention of trade names or commercial products in this manual is for illustrative purposes only, and does not constitute an EPA endorsement or exclusive recommendation for

use. The products and instrument settings cited in SW-846 methods represent those products and settings used during method development or subsequently evaluated by the Agency. Glassware, reagents, supplies, equipment, and settings other than those listed in this manual may be employed provided that method performance appropriate for the intended application has been demonstrated and documented.

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 -- FPXRF instruments use either a sealed radioisotope source or an x-ray tube to provide the excitation source. Many 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 (^{55}Fe), cadmium Cd-109 (^{109}Cd), americium Am-241 (^{241}Am), and curium Cm-244 (^{244}Cm). These sources may be contained in a probe along with a window and the detector; the probe may be 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. This is due to the ever increasing time required for the analysis rather than a decrease in instrument performance. 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 necessary 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 both 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 and by the instrument's ability to cool the x-ray tube. 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 FPXRF instruments operated in the intrusive mode, the probe may be rotated so that the window faces either upward or downward. 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 L. 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 amplitudes (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 ppm on a liquid crystal display (LCD) in the instrument. FPXRF instruments can store both spectra and from 3,000 to 5,000 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 or at any other time, data can be downloaded by means of an RS-232 port and cable to a PC.

6.2 Spare battery and battery charger.

6.3 Polyethylene sample cups -- 31 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 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 Reagent grade chemicals must be used in all tests. Unless otherwise indicated, it is intended that all reagents conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

7.2 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 designated 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.

7.3 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.

7.3.1 The SSCS must be representative of the matrix to be analyzed by FPXRF. These samples must be well homogenized. A minimum of 10 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.

7.3.2 Each sample should be oven-dried for 2 to 4 hr at a temperature of less than 150 °C. If mercury is to be analyzed, a separate sample portion should be dried at ambient temperature 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 homogenized (see Sec. 7.3.3) and then a representative portion ground with a mortar and pestle or other mechanical means, prior to passing through a 60-mesh sieve. Only the coarse rock fraction should remain on the screen.

7.3.3 The sample should be homogenized by using a riffle splitter or by placing 150 to 200 g 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 g 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.

7.4 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 established lower limit of detection. These samples are used to monitor for cross-contamination and laboratory-induced contaminants or interferences.

7.5 Standard reference materials -- Standard reference materials (SRMs) 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. When these SRMs are unavailable, alternate standards may be used (e.g., NIST 2702).

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 Follow the manufacturer’s instructions for the quality control procedures specific to use of the testing product. Refer to Chapter One for additional guidance on quality assurance (QA) and quality control (QC) protocols. Any effort involving the collection of analytical data should include development of a structured and systematic planning document, such as a Quality Assurance Project Plan (QAPP) or a Sampling and Analysis Plan (SAP), which translates project objectives and specifications into directions for those that will implement the project and assess the results.

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 Sec. 4.10, this check also serves as a gain check in the event that ambient temperatures are fluctuating greatly (more than 10 °F).

9.2.1 The energy calibration check should be run at a frequency consistent with manufacturer's 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.

9.2.2 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 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, specifically, instrument blanks and method blanks.

9.3.1 An instrument blank is used to verify that no contamination exists in the spectrometer or on the probe window. The instrument blank can be silicon dioxide, a polytetraflurorethylene (PTFE) 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 established lower limit of detection 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. If the method blank does not contain the target analyte at a level that interferes with the project-specific data quality objectives then the method blank would be considered acceptable. In the absence of project-specific data quality objectives, if the blank is less than the lowest level of detection or less than 10% of the lowest sample concentration for the analyte, whichever is greater, then the method blank would be considered acceptable. If the method blank cannot be considered acceptable, 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. If both in situ and intrusive analytical techniques are used during the course of one day, it is recommended that separate precision calculations be performed for each analysis type.

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 sensitivity, but decreases sample throughput.

9.6 The lower limits of detection should be established from actual measured performance based on spike recoveries in the matrix of concern or from acceptable method performance on a certified reference material of the appropriate matrix and within the appropriate calibration range for the application. This is considered the best estimate of the true method sensitivity as opposed to a statistical determination based on the standard deviation of

replicate analyses of a low-concentration sample. While the statistical approach demonstrates the potential data variability for a given sample matrix at one point in time, it does not represent what can be detected or most importantly the lowest concentration that can be calibrated. For this reason the sensitivity should be established as the lowest point of detection based on acceptable target analyte recovery in the desired sample matrix.

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 project-specific 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) for the results should be 0.7 or greater for the FPXRF data to be considered screening level data. If the r 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, namely: 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 necessary, 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 necessary.

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 Sec. 7.3. 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 Sec. 7.3; 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 necessary. 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 necessary to perform an adequate empirical calibration. The exact number of 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 reading. 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 during 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, remove any large or nonrepresentative debris from the soil surface before analysis. This debris includes rocks, pebbles, leaves, vegetation, roots, and concrete. Also, the soil surface must 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 example performance data for this method, this modest amount of sample preparation was found to take less than 5 min 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 the desired method sensitivity. Due to the heterogeneous nature of the soil sample, in situ analysis can provide only “screening” type data.

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 g or 250 cm^3, which is enough soil to fill an 8-ounce jar. However, the exact dimensions and sample depth should take into consideration the heterogeneous deposition of contaminants and will ultimately depend on the desired project-specific data quality objectives. 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 Sec. 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 time necessary for homogenization procedure using the fluorescein dye ranged from 3 to 5 min per sample. As demonstrated in Secs. 13.5 and 13.7, homogenization has the greatest impact on the reduction of sampling variability. It produces little or no contamination. Often, the direct analysis through the plastic bag is possible without the more labor intensive steps of drying, grinding, and sieving given in Secs. 11.5 and 11.6. Of course, to achieve the best data quality possible all four steps should 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 g) is placed in a suitable container for drying. The sample should be dried for 2 to 4 hr in the convection or toaster oven at a temperature not greater than 150 °C. Samples may also be air dried under ambient temperature conditions using a 10- to 20-g portion. Regardless of what drying mechanism is used, the drying process is considered complete when a constant sample weight can be obtained. Care should be taken to avoid sample cross-contamination and these measures can be evaluated by including an appropriate method blank sample along with any sample preparation process.

CAUTION: 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 min 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 established lower limit of detection of the procedure or DQOs of the analysis. If all recommended sample preparation steps are followed, there is a high probability the desired laboratory data quality may be obtained.

12.0 DATA ANALYSIS AND CALCULATIONS

Most FPXRF instruments have software capable of storing all analytical results and spectra. The results are displayed in ppm and can be downloaded to a personal computer, which can be used to provide a hard copy printout. Individual measurements that are smaller than three times their associated SD should not be used for quantitation. See the manufacturer's instructions regarding data analysis and calculations.

13.0 METHOD PERFORMANCE

13.1 Performance data and related information are provided in SW-846 methods only as examples and guidance. The data do not represent required performance criteria for users of the methods. Instead, performance criteria should be developed on a project-specific basis, and the laboratory should establish in-house QC performance criteria for the application of this method. These performance data are not intended to be and must not be used as absolute QC acceptance criteria for purposes of laboratory accreditation.

13.2 The sections to follow discuss three performance evaluation factors; namely, precision, accuracy, and comparability. The example data presented in Tables 4 through 8 were generated from results obtained from six FPXRF instruments (see Sec. 13.3). 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. These data are provided for guidance purposes only.

13.3 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.

13.4 All example data presented in Tables 4 through 8 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.

13.5 Precision measurements -- The example precision data are presented in Table 4. These data are provided for guidance purposes only. 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 4 is an average RSD for the precision samples that had analyte concentrations at 5 to 10 times the lower limit of detection 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 4. Some analytes such as cadmium, nickel, and tin were only detected at concentrations near the lower limit of detection so that an RSD value calculated at 5 to 10 times this limit 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 5 shows these results. These data are provided for guidance purposes only. The additional nine soil samples were comprised of three from each texture and had analyte concentrations ranging from near the lower limit of detection for 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 5 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 5 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 instead of 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 6 presents a summary of this data. With the exception of cadmium, chromium, and nickel, the values presented in Table 6 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 6. 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 6.

Table 7 provides a more detailed summary of accuracy data for one particular FPXRF instrument (TN 9000) for the 9 soil SRMs and 4 sediment SRMs. These data are provided for guidance purposes only. Table 7 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 8. Similar trends in the data were seen for all instruments. These data are provided for guidance purposes only.

Table 8 shows the regression parameters for the whole data set, broken out by soil type, and by preparation method. These data are provided for guidance purposes only. 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--intrusive, sample collected and homogenized; preparation 3--intrusive, with sample in a sample cup but sample still wet and not

ground; and preparation 4–intrusive, with 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 8 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 8 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.

Sec. 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 necessary to dry and grind the sample for small improvements in comparability. Homogenization requires 3 to 5 min. Drying the sample requires one to two hours. Grinding and sieving requires another 10 to 15 min per sample. Lastly, when grinding and sieving is conducted, time has to 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 A. D. Hewitt, "Screening for Metals by X-ray Fluorescence Spectrometry/Response Factor/Compton K_α Peak Normalization Analysis," American Environmental Laboratory, pp 24-32, 1994.

13.8.2 S. Piorek and J. R. Pasmore, "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, Vol 2, pp 1135-1151, 1993.

13.8.3 S. Shefsky, "Sample Handling Strategies for Accurate Lead-in-soil Measurements in the Field and Laboratory," *International Symposium of Field Screening Methods for Hazardous Waste and Toxic Chemicals*, Las Vegas, NV, January 29-31, 1997.

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 St., N.W. Washington, D.C. 20036, http://www.acs.org.

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, "Energy Dispersive X-ray Fluorescence Spectrometry: An Introduction," 1994.

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

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

17.0 TABLES, DIAGRAMS, FLOWCHARTS, AND VALIDATION DATA

The following pages contain the tables referenced by this method. A flow diagram of the procedure follows the tables.

TABLE 1

EXAMPLE INTERFERENCE FREE LOWER LIMITS OF DETECTION

Analyte	Chemical Abstract Series Number	Lower Limit of Detection 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: Refs. 1, 2, and 3
These data are provided for guidance purposes only.

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	432	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: Refs. 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: Ref. 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

EXAMPLE PRECISION VALUES

Analyte	Average Relative Standard Deviation for Each Instrument at 5 to 10 Times the Lower Limit of Detection					
	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

These data are provided for guidance purposes only.
Source: Ref. 4

[a] These values are biased high because the concentration of these analytes in the soil samples was near the lower limit of detection for that particular FPXRF instrument.

NR Not reported.

NA Not applicable; analyte was reported but was below the established lower limit detection.

TABLE 5

EXAMPLES OF 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

These data are provided for guidance purposes only.
Source: Ref. 4
[a] These values may be biased high because the concentration of these analytes in the soil samples was near the lower limit of detection.
ND Not detected.
NR Not reported.

TABLE 6

EXAMPLE ACCURACY VALUES

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: Ref. 4. These data are provided for guidance purposes only.
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 7

EXAMPLE 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: Ref. 4. These data are provided for guidance purposes only.
[a] All concentrations in milligrams per kilogram.
%Rec.: Percent recovery; ND: Not detected; NA: Not applicable.
-- No data.

TABLE 8

EXAMPLE 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

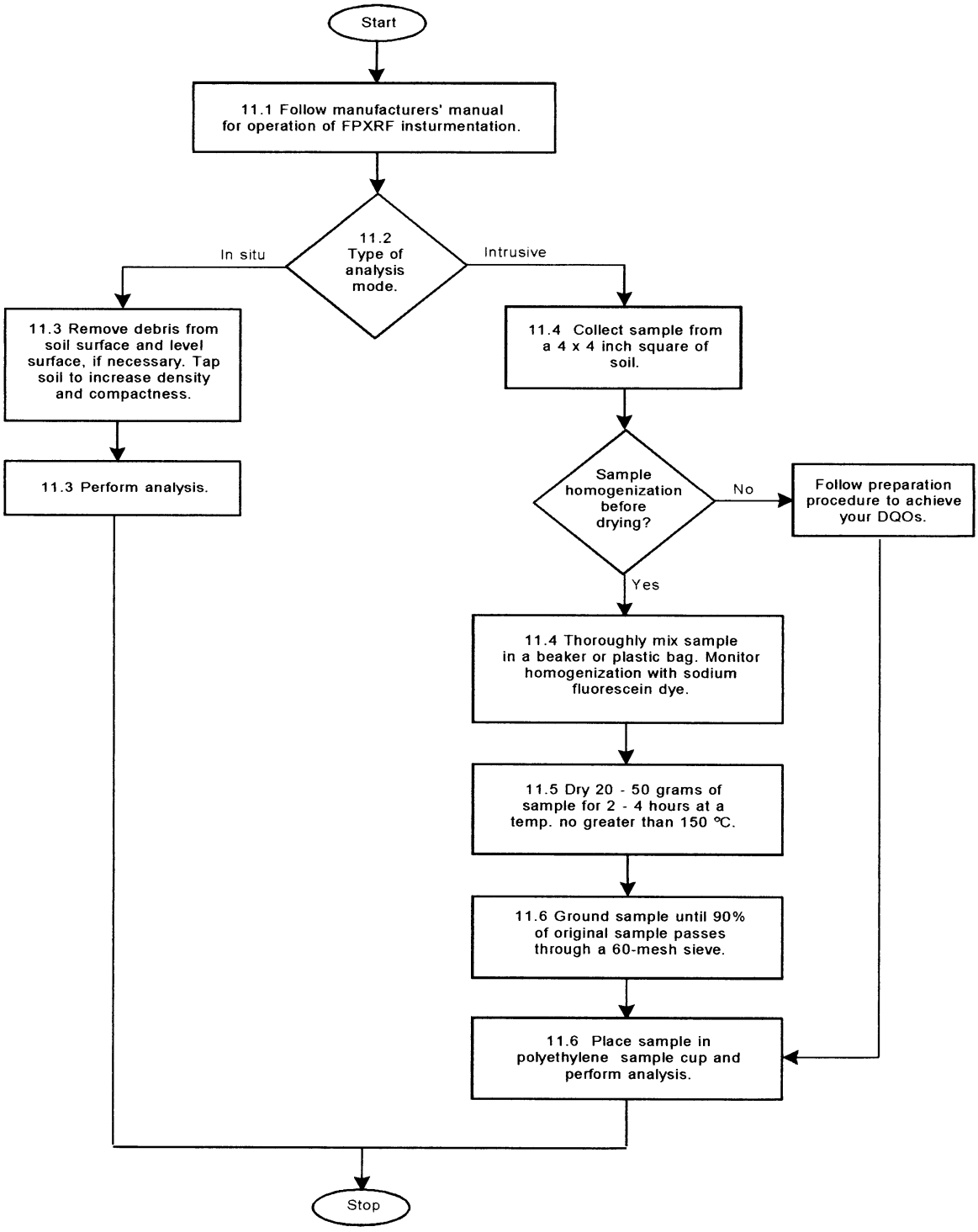
Source: Ref. 4. These data are provided for guidance purposes only.

[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



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