FINAL
SITEWIDE SAMPLING AND ANALYSIS PLAN
OMAHA LEAD SITE
OMAHA, NEBRASKA

U.S. Environmental Protection Agency Region 7
901 North 5th Street
Kansas City, KS 66101

Revision 2

April 2010

FINAL
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OMAHA LEAD SITE
OMAHA, NEBRASKA

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Superfund Division
USEPA Region 7
Kansas City, Kansas

APPROVED:

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LIST OF ACRONYMS AND ABBREVIATIONS

AA	atomic absorption
AES	Architect and Engineering Services
ASARCO	American Smelting and Refining Company, Inc.
B	bias
bgs	below ground surface
˚C	degrees Celsius
CEC	cation exchange capacity
CERCLA	Comprehensive Environmental Response, Compensation, and Liability Act
CFR	Code of Federal Regulations
CLP	contract laboratory program
COC	chain-of-custody
DCHD	Douglas County Health Department
DQA	data quality assessment
DQCR	daily quality control reports
DQOs	Data Quality Objectives
EPA	U.S. Environmental Protection Agency
EQL	estimated quantitation limit
ERT	Environmental Response Team
ESP	exchangeable sodium percentage
˚F	degrees Fahrenheit
FP	
FPXRF	field portable X-ray fluorescence
FSP	Field Sampling Plan
FTL	field team leader
g	grams
GC	gas chromatography
HGL	HydroGeoLogic, Inc.
HPLC	high performance liquid chromatography
IC	ion chromatographic technique
ICP	inductively coupled plasma
IDW	investigation-derived waste
IEUBK	Integrated Exposure Uptake Biokinetic model

LCS	laboratory control sample/laboratory control sample duplicate
LSW	Lead Sites Workgroup
µg/dl	micrograms per deciliter
µg/m^3	micrograms per cubic meter
µm	micrometer
m^3	cubic meter
m^3/min	cubic meter per minute
MCE	mixed cellulose ester
MDL	method detection limit
mg/kg	milligram per kilogram
mm	millimeter
MQL	method quantitation limit
MS/MSD	matrix spike/matrix spike duplicate
NCP	National Contingency Plan
NELAC	National Environmental Laboratory Accreditation Counsel
NELAP	National Environmental Laboratory Accreditation Program
NOAA	National Oceanic Atmospheric Administration
NPL	National Priorities List
OLS	Omaha Lead Site
OSHA	Occupational Safety and Health Administration
OSWER	Office of Solid Waste and Emergency Response
PAH	polynuclear aromatic hydrocarbons
PCBs	polychlorinated biphenyls
PEL	permissible exposure limit
PPE	personal protective equipment
PR	procurement request
PRGs	preliminary remediation goals
PWS	Performance Work Statement
QA	quality assurance
QA/QC	quality assurance/quality control
QAPP	Quality Assurance Project Plan
QC	quality control
QCC	Quality Control Coordinator
QMP	quality management plan
%R	percent recovery
RAPMA	Remedial Action Plan Monitoring Act
RCRA	Resource Conservation and Recovery Act
RI	Remedial Investigation

ROD	Record of Decision
RPD	relative percent difference
RPM	Remedial Project Manager
RSD	relative standard deviation
SAP	Sampling and Analysis Plan
SOP	standard operating procedure
SOW	statement of work
SRM	standard reference materials
SSCS	site-specific calibration standards
SSHP	Site Safety and Health Plan
STP	standard temperature and pressure
SVOC	semivolatile organic compound
SQL	sample quantitation limit
TCLP	Toxicity Characteristic Leaching Procedure
TO	task order
TSP	total suspended particulate
USDA	United States Department of Agriculture
USGS	United States Geological Survey
VOC	volatile organic compound

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SITEWIDE SAMPLING AND ANALYSIS PLAN
OMAHA LEAD SITE - OMAHA, NEBRASKA

1.0 INTRODUCTION

HydroGeoLogic, Inc. (HGL) has been tasked to prepare a Sitewide Sampling and Analysis Plan (SAP) for the Omaha Lead Site (OLS) to standardize excavation and post-excavation sampling activities. This SAP is being completed under U. S. Environmental Protection Agency (EPA) Region 7 Architect and Engineering Services (AES) Contract Number EP-S7-05-05, Task Order Number 0018.

1.1 SAMPLING AND ANALYSIS PLAN LAYOUT

This SAP consists of a Field Sampling Plan (FSP) and a Quality Assurance Project Plan (QAPP) and is organized as follows:

Section 1 – Introduction
Section 2 – Site Background
Part 1: Field Sampling Plan
Section 3 – Post Excavation Screening
Section 4 – Air Sampling
Section 5 – Clean Fill Sampling
Section 6 – Sod Sampling
Section 7 – Contaminated Stock Pile Sampling
Part 2: Quality Assurance Project Plan
Section 8 – Project Management
Section 9 – Measurement and Data Acquisition
Section 10 – Assessment and Oversight
Section 11 – Data Validation and Usability
Section 12 – References

The SAP organizational structure is illustrated as a flow chart on Figure 1.1.

1.2 OBJECTIVES

The purpose of this SAP is to standardize OLS excavation and post excavation sampling activities. The sample collection and analysis methods described in the FSP and QAPP have been successfully implemented at other lead removal sites (in accordance with the *EPA Superfund Lead-Contaminated Residential Sites Handbook finalized in August 2003*) or are an industry standard. This SAP addresses only soil lead contamination; lead paint and lead in interior dust from impacted residences is beyond the purview of this document.

This Sitewide SAP is intended to serve as a model for EPA's remedial contractors conducting soil excavation activities at the OLS. The site-specific EPA Region 7 QAPP Addendum form covering the specific activities and properties under consideration for each contract should be

prepared based on the procedures and methods detailed in this SAP. A copy of the Addendum form is included as Appendix A.

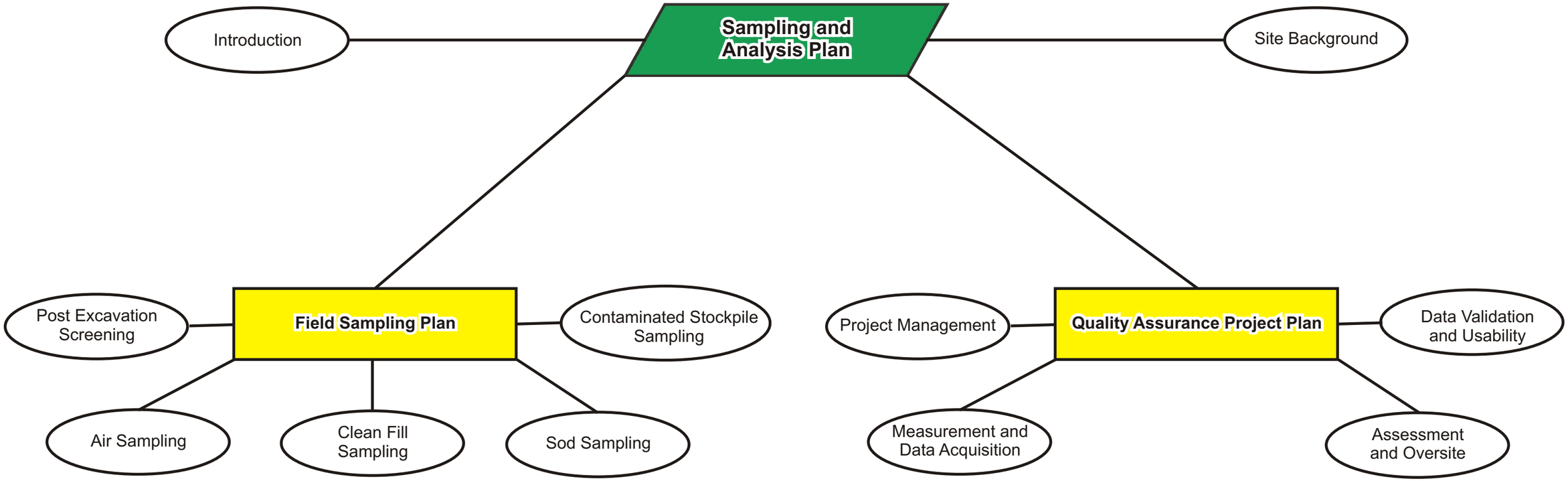
1.3 Distribution List

Copies of the approved sitewide SAP for the OLS will be distributed to the following individuals:

EPA Region 7:	Robert Feild, Project Coordinator and Remedial Project Manager Donald Bahnke, Remedial Project Manager Pauletta France-Issetts, Remedial Project Manager Daniel Garvey, Remedial Project Manager
Contractor:	Program Manager Quality Assurance Officer (Contractor will distribute copies within their organization)

It is assumed that numerous and unknown contractors will abide by this sitewide SAP; thus, they are not listed.

FIGURE



HGL—Sitewide Sampling and Analysis Plan for the Omaha Lead Site—Omaha, NE

Figure 1.1
Sampling and Analysis Organization Chart

U.S. EPA Region 7

Table 9.10
Sampling Requirements, Analytical Methods, and Quantitation Limits for Clean Fill and Sod Samples Analyzed for Polychlorinated Biphenyls (PCBs) in Soil by Gas Chromatography

Analyte	EPA Analytical Method	Bottle Requirements	Preservative	Holding Times	Estimated Quantitation Limits
PCBs in Soil					
PCB-1016	SW8082	250 mL widemouth jar with Teflon™ lined cap	Cool to 4ºC	14 Days	330
PBC-1221					330
PBC-1232					330
PBC-1242					330
PBC-1248					330
PCB-1254					330
PCB-1260					330

Notes:
The estimated quantitation limits are recommended limits listed in micrograms per kilogram. The laboratory derived estimated quantitation limits supersede these values and will be reported in the event a disparity occurs.

ºC degrees Celsius
EPA Environmental Protection Agency
mL milliliter
PBC Polychlorinated Biphenyls

Table 9.11
Sampling Requirements, Analytical Methods, and Quantitation Limits for Clean Fill and Sod Samples Analyzed for Polycyclic Aromatic Hydrocarbons (PAHs)

Analyte	EPA Analytical Method	Bottle Requirements	Preservative	Holding Times	Estimated Quantitation Limits
PAHs in Soil					
Acenaphthene	SW8270D	250 mL widemouth amber jar with TeflonTM lined cap	Cool to 4°C	7 days until extraction. Analyze with 40 days after extraction	330
Acenaphthylene					330
Anthracene					330
Benzo(a)anthracene					330
Benzo(a)pyrene					330
Benzo(b)fluoranthene					330
Benzo(ghi)perylene					330
Benzo(k)fluoranthene					330
Chrysene					330
Dibenzo(a,h)anthracene					330
Fluoranthene					330
Fluorene					330
Indeno(1,2,3-cd)pyrene					330
Naphthalene					330
Phenanthrene					330
Pyrene					330

Notes:
The estimated quantitation limits are recommended limits listed in micrograms per kilogram. The laboratory derived estimated quantitation limits supersede these values and will be reported in the event a disparity occurs.

ºC degrees Celsius
EPA Environmental Protection Agency
mL milliliter
PAH polynuclear aromatic hydrocarbons

2.0 SITE BACKGROUND

2.1 SITE LOCATION

The OLS (CERCLIS ID # NESFN0703481) is located in Omaha, Nebraska, in Douglas County (see Figure 2.1). The site includes properties that have been contaminated with lead as a result of industrial emissions from historical lead smelting/refining operations. The majority of the impacted properties are within an approximately 27 square mile area generally bounded by Read Street on the north, Harrison Street on the south, 56th Street on the west, and the Missouri River to the east (see Figure 2.2). The actual site is defined on a property-by-property basis based on the levels of lead detected in soil, and is not defined by a discrete boundary.

2.2 SITE DESCRIPTION

The OLS includes contaminated surface soils present at residential properties, child-care facilities, and other residential-type properties in Omaha, Nebraska. The site encompasses the eastern portion of the greater metropolitan area in Omaha and is centered on the downtown area where two former lead processing facilities released lead-contaminated particulates to the air. These particulates were transported downwind and deposited on the ground surface. In general, concentrations of lead in soil are greatest near downtown and decrease with increasing distance from downtown. The area includes some of the oldest neighborhoods in the Omaha area. The site area is primarily residential with a population exceeding 125,000, including more than 14,000 children less than 7 years of age (2000 U.S. Census data). The site also includes schools, child care centers, parks and other parcels where residential-level exposure could occur. Approximately 135 acres of wetlands are located within the 4-mile radius of the site (EPA, 2003b).

2.3 SITE HISTORY

The American Smelting and Refining Company Inc., (ASARCO) operated a lead refinery at 500 Douglas Street in Omaha for over 100 years beginning in the 1870s. The 23 acre refinery was located on the west bank of the Missouri River on land originally owned by Union Pacific Railroad. ASARCO bought the property in 1946 and continued refinery operations until 1997. As a routine part of the refinery operation, lead-contaminated particulates were emitted into the atmosphere and were transported downwind in various directions and deposited on the ground surface. After the facility was closed, the property was remediated under the State of Nebraska Remedial Action Plan Monitoring Act (RAPMA) program and transferred to the City of Omaha. Current uses of the property are both commercial and public.

In addition to ASARCO, a secondary lead smelter and lead battery recycling plant was located at 555 Farnam Street in Omaha and operated from the early 1950s until 1963 by the Aaron Ferer and Sons Co. In 1963 it was purchased by Gould Electronics, Inc. (Gould). The blast furnace (smelter) used to smelt the lead at the Gould plant emitted lead particles into the air which were also transported downwind in various directions and deposited on the ground surface. Operations continued at the facility until 1982, when the facility was closed and the property sold

to Douglas County. Douglas County performed a clean up of the site. The property is currently occupied by Heartland of America Park.

The Douglas County Health Department (DCHD) began to monitor the air quality around the ASARCO facility in 1984 and the measurements of ambient lead in the air routinely exceeded the standard. The DCHD also compiled over 25 years of statistics on blood lead screening of children and found that children living within the OLS exceeded the health-based threshold for lead more frequently than children not within the OLS.

In 1998 the Omaha City Council solicited assistance from the EPA in addressing problems with lead contamination in the Omaha area. The EPA initiated a process to investigate the lead contamination under the Comprehensive Environmental Response, Compensation, and Liability Act (CERCLA). After conducting several comprehensive risk and exposure studies between 1998 and 1999, EPA executed an Action Memorandum describing the time-critical removal action that would take place at those properties where soil samples contained lead concentrations equal to or greater than 400 milligrams per kilogram (mg/kg). Several subsequent Action Memorandums and Amendments have been issued since to continue the time-critical removal actions. The EPA response action transitioned from removal authority to remedial authority in 2005. The objective of the remedial action is to eliminate or reduce ingestion exposure caused by the presence of lead in soil.

Lead processing was conducted at the eastern edge of downtown Omaha for more than 100 years. Residential properties located along the directions of prevailing winds were tested to determine the extent of migration. Other properties were later tested to fill in gaps in order to identify a geographic pattern. Surface lead contamination exists above the 400 mg/kg screening level over a wide area of eastern Omaha. In general, concentrations of lead in soil are greatest near downtown, which was the former location of the now closed lead processing industries. Concentrations of lead decrease with increasing distance from downtown. The area is approximately 27 square miles in size and includes some of the oldest neighborhoods in the Omaha area.

Investigations have been conducted at the site since 1999 and several investigations have been performed at the ASARCO facility. Groundwater and soils were characterized and a closure report was developed. Surface soil samples were collected from 38,000 residential and child care properties within the OLS and analyzed for lead between March 1999 and January 2008. Sverdrup/Jacobs Engineering conducted the sampling between March 1999 and July 2000, and since then the sampling has been conducted by Black and Veatch Special Projects Corp. (Black and Veatch). The properties sampled were relatively evenly distributed throughout the site and represent lead concentrations in surface soil in all areas of the site. Previous studies have indicated that the highest lead concentrations were along the direction of the prevailing wind. The RI results appear to support this assertion because most of the homes with soil lead concentrations exceeding 400 mg/kg are clustered along the prevailing wind directions. Past investigations further indicate that lead in soil generally had not migrated beyond the top 2 to 12 inches of soil.

In February 2002, the EPA proposed adding the OLS to the National Priorities List (NPL). It was added to the NPL on April 30, 2003. The Interim Record of Decision (ROD) for the OLS

was issued December 15, 2004 (EPA, 2004a). A Final ROD was issued on May 13, 2009 (EPA 2009).

2.4 ENVIRONMENTAL SETTING

2.4.1 Soils and Geology

Omaha lies in the Dissected Till Plains of the Central Region of the Interior Plains physiographic province. This portion of eastern Nebraska lies above the Nemaha Uplift, which is a deep north-south trending geologic structure that runs from Omaha to central Oklahoma. It is an uplift of Precambrian Granite basement rock and is flanked by the Humboldt fault zone on the eastern edge of the uplift (USGS, 1997).

The Dissected Till Plains is an area that was glaciated, uplifted, and subsequently eroded into a flat-to-rolling terrain that slopes gently toward the Missouri River Valley. Glaciers left behind glacial till consisting of clay, sand, gravel, and boulders intermingled. Over thousands of years, rivers and streams cut into the till to form low, rolling hills and ridges. Much of the Dissected Till Plains is covered by deposits of loess, a wind carried glacial silt. Along the Missouri River, the Peoria loess deposits were piled up by wind action to form steep-sided bluffs that can rise as much as 150 feet above the river surface. The glacial till and loess together constitute unconsolidated materials underlying the OLS. According to the RI, these unconsolidated materials are approximately 90 feet thick in the Missouri River Valley at Council Bluff, Iowa, located immediately to the east of the OLS.

Underlying the loess and glacial till are the Dakota Formation and the Lansing and Kansas City Groups undifferentiated. The Lower Cretaceous-aged Dakota Formation consists of clay, sand, gravel, shale and sandstone. Thickness of the Dakota Formation ranges from 15 to 60 feet (Shiroba, et al, 2001). The Pennsylvanian-aged Lansing and Kansas City Groups consist of interbedded shale and limestone and is greater than 50 feet thick in the Omaha area (Black and Veatch, 2004). Underlying the Kansas City Group is Paleozoic-aged sedimentary rock of unknown thickness.

There are two distinct soil types within the OLS, although both are characterized as Peoria loess or younger (USDA, 1975). The Albaton Haynie Association soils are found along the Missouri River floodplain and cover approximately one-third of the site. These soils are deep, poorly drained to moderately well drained, nearly level clayey and silty soils. The soils on the bluffs to the west of the floodplain are the Monona and Ida Association which are deep, well drained, nearly level to very steep silty soils.

2.4.2 Topography

The eastern portion of Omaha is part of the Missouri River Floodplain and has little topographic relief. The western portion of the site is characteristic of the Dissected Till Plains with terrain that varies from flat to rolling hills and ridges. Elevation varies from approximately 1,030 to 1,200 feet above mean sea level.

2.5 CONTAMINANT OF CONCERN

The contaminant of concern for the OLS is lead. Previous investigations have determined that the lead contamination on the site resulted from air deposition of lead contaminated particulates emitted from the ASARCO and Gould facilities in downtown Omaha. Airborne particulates contaminated with lead traveled downwind and were deposited on the ground surface. Lead concentrations in tested soils indicate that lead contamination is generally limited to the upper 2 to 12 inches of soil and that further migration downward through the soil is not likely based on the characteristics of the soil (Black and Veatch, 2004).

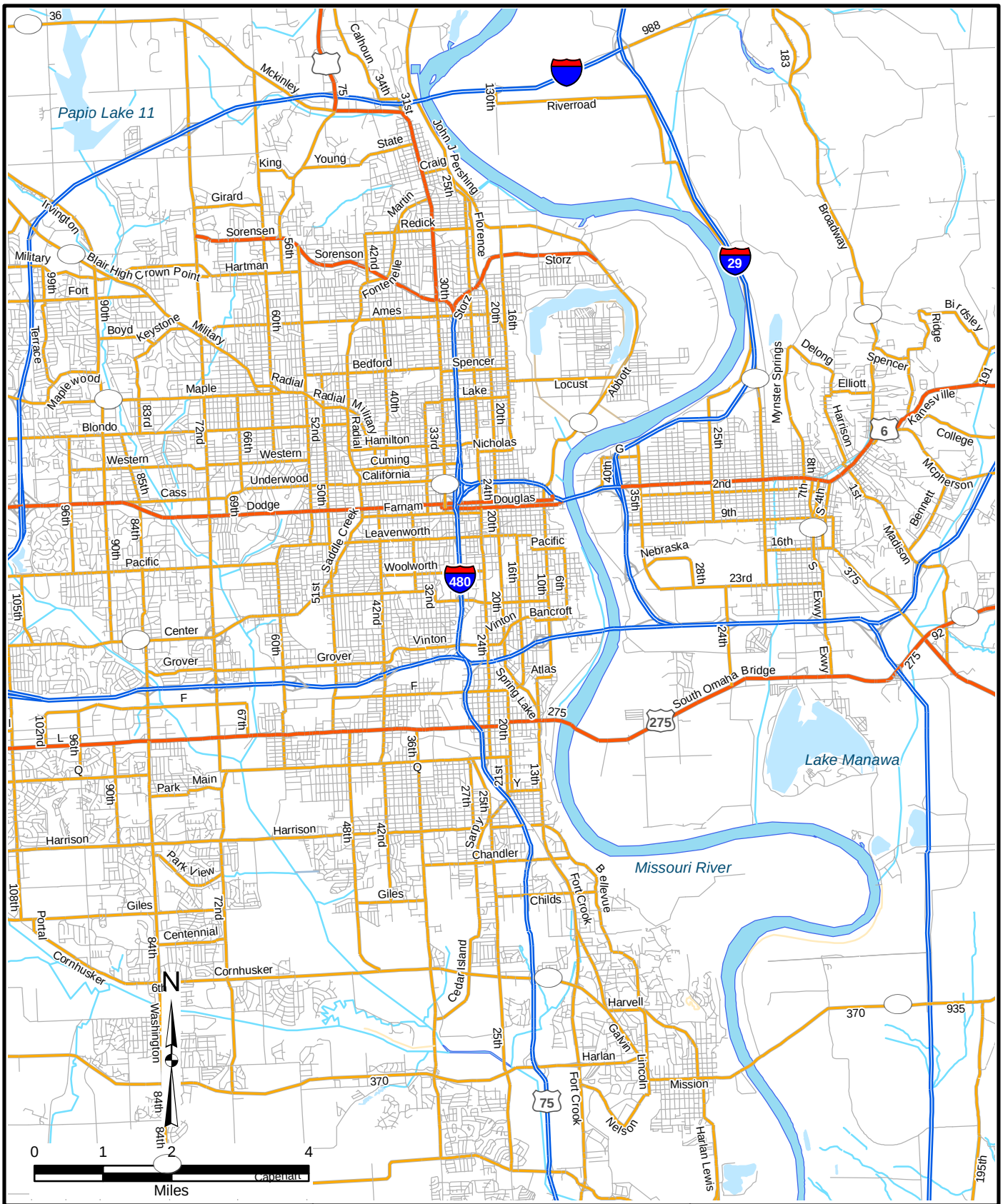
Lead is classified by the EPA as a probable human carcinogen and cumulative toxicant. The Centers for Disease Control and Prevention have set a blood lead standard of 10 micrograms per deciliter (µg/dl) for children less than seven years of age. Lead poisoning can cause learning and behavioral difficulties, loss of hearing, lower IQ and kidney problems.

The Selected Remedy as stated in the Record of Decision (ROD) for the OLS includes:

- Excavation and replacement of soils with lead concentrations exceeding 400 mg/kg at any residential-type property
- Final Management of excavated materials
- Stabilization of loose and flaking exterior lead-based paint
- Response to lead-contaminated interior dust
- Participation in a comprehensive program addressing all potential lead sources

As noted in Section 1.2, this SAP does not address lead paint or lead in interior dust. Lead paint and interior dust are addressed in a separate SAP.

FIGURES



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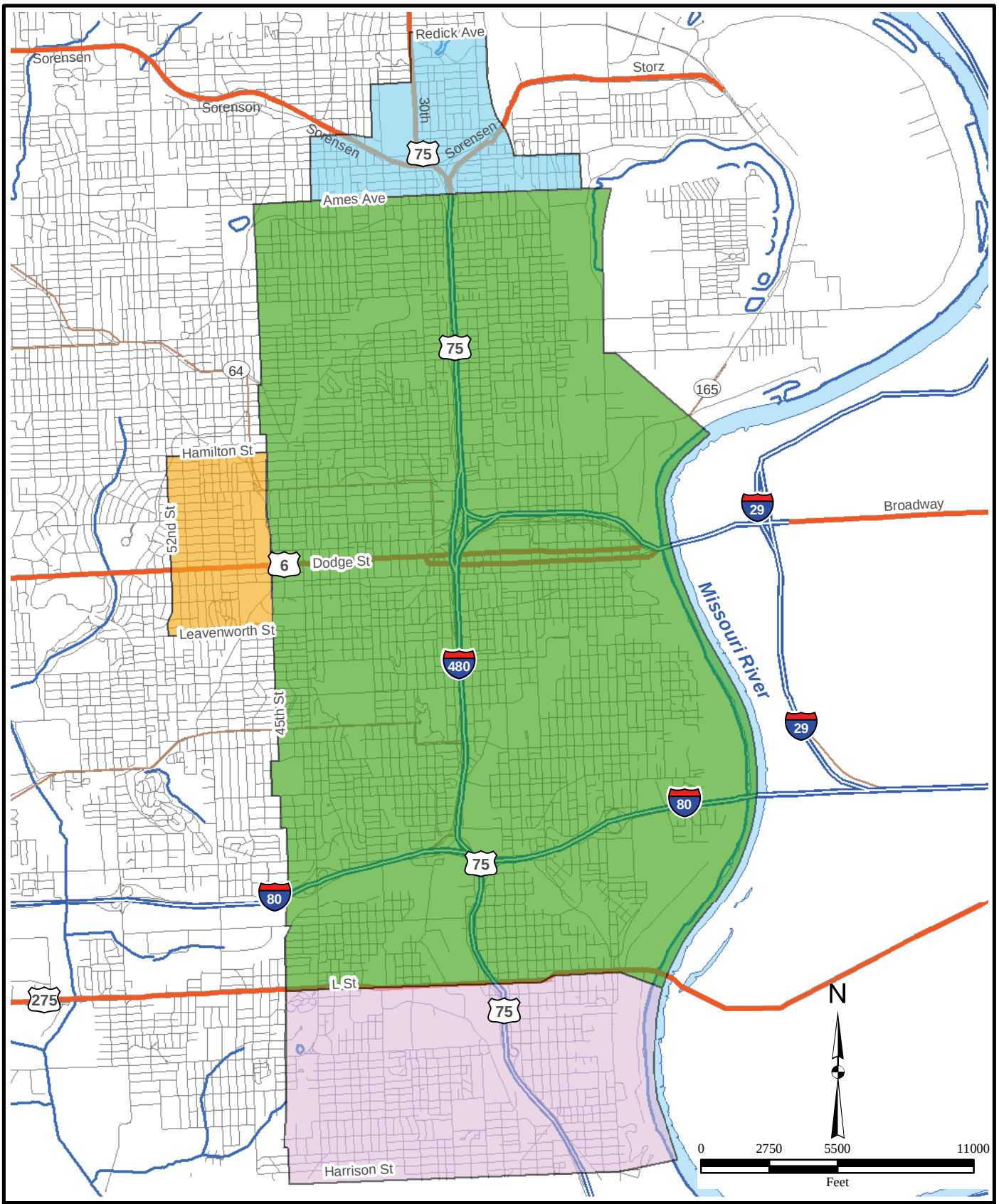
Revised: 09/04/07 CV
Source: ESRI StreetMap USA

United States Environmental Protection Agency

HGL HydroGeoLogic, Inc

Figure 2.1
Omaha Lead Site Location Map



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Project: EPA009-018-03-46
Source: ESRI Streetmap USA

United States Environmental Protection Agency

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Legend

- Ames-L-45
- Ext-North
- Ext-West
- Ext-South

Figure 2.2
Site Layout Map

PART 1: FIELD SAMPLING PLAN

This FSP has been developed to provide the rationale and procedures for EPA-lead field activities related to soil lead contamination that will be conducted at the OLS. These field activities include the following:

- Post excavation screening using x-ray fluorescence (XRF)
- Air sampling at residential properties and the soil stockpile
- Personal air sampling
- Sampling of clean fill
- Sampling of replacement sod
- Sampling of the contaminated stock pile to determine final disposition

The procedures presented in this SAP were primarily obtained from EPA documents, methods, and Standard Operating Procedures (SOPs). Copies of these documents, methods, and SOPs are provided in Appendix B.

3.0 POST EXCAVATION SCREENING

The process of documenting lead concentrations at the excavated properties is described in this section. The sample screening process presented in the following sections has been designed to document that the properties have been remediated in accordance with the guidelines and action levels established by EPA to protect human health and the environment.

3.1 X-RAY FLUORESCENCE INSTRUMENT CALIBRATION AND QUALITY CONTROL

After the excavation has been completed, XRF readings will be taken to confirm that lead levels are below the action level. Readings will be collected with a field portable XRF (FPXRF) unit. The instrument calibration and quality control (QC) measures required to assure accurate operation of the FPXRF are summarized in the following subsections. These procedures were obtained from manufacturers, operation manuals, Code of Federal Regulations (CFR) Title 40 Appendix B to Part 136—Definition and Procedure for the Determination of the Method Detection Limit—Revision 1.11, and draft Method 6200 contained in the EPA SW-846 manual.

3.1.1 Annual and Post-Maintenance Calibration and Quality Control

The calibration and QC measures described in this subsection will be completed annually or after the instrument has undergone maintenance or repair that would affect its calibration.

3.1.1.1 Factory Calibration

The instrument will be calibrated in accordance with the instrument manufacturer's specifications, typically before initial use and after factory servicing. The manufacturer also typically suggests that the calibration be checked annually by a qualified service technician. Most instruments perform a self calibration check when powered on. The energy and field checks will be completed as discussed below and if a discrepancy is noted the manufacturer should be contacted for further clarification.

Instrument calibration procedures vary among manufacturer's; however, three types of calibration techniques are generally implemented by all manufacturers: a fundamental parameters calibration, empirical calibration, and Compton peak ratio calibration. Most FPXRFs perform a self calibration check when powered on. The manufacturer's guidelines will be reviewed and followed to check the unit calibration prior to use.

Fundamental Parameters Calibration
The fundamental parameters (FP) calibration is a "standardless" calibration. Rather than calibrating a unit's calibration curve by measuring its response to standards that contain analytes of known concentrations, fundamental parameters calibration relies on the known physics of the spectrometer's response to pure elements to set the calibration. Built-in mathematical algorithms are used to adjust the calibration for analysis of soil samples and to compensate for the effects of the soil matrix. The FP calibration is performed by the manufacturer, but the analyst can adjust the calibration curves (slope and y-intercept) on the bases of results of analyses of check samples, such as standard reference materials (SRM), which are analyzed in the field.

Empirical Calibration
In performing an empirical calibration, a number of actual samples, such as site-specific calibration standards (SSCS), are used, and the instrument's measurement of the concentrations of known analytes in the samples are measured. Empirical calibration is effective because the samples used closely match the sample matrix. SSCSs are well-prepared samples collected from the site of interest in which the concentrations of analytes have been determined by inductively coupled plasma (ICP), atomic absorption (AA), or other methods approved by the EPA. The standards should contain all the analytes of interest and interfering analytes. Manufacturers recommend that 10 to 20 calibration samples be used to generate a calibration curve.

Compton Normalization
The Compton normalization method incorporates elements of both FP and empirical calibration. A single, well-characterized standard, such as an SRM or a SSCS, is analyzed, and the data are normalized 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 intensity of the Compton peak changes as various matrices affect the way in which source radiation is scattered. For that reason, normalizing to the Compton peak can reduce problems with matrix effects that vary among samples. Compton normalization is similar to the use of internal standards in analysis for organic analytes.

3.1.1.2 Detection Limit Determination

A lead precision-based method detection limit (MDL) and method quantitation limit (MQL) must be calculated for each instrument before it is used to obtain a reading. The detection limits will be calculated annually or after factory maintenance. The detection limits calculated will be documented in the field logbook.

Precision-Based Method Detection Limit
MDL is defined as the minimum concentration of a substance that can be measured and reported with 99 percent confidence that the analyte concentration is greater than zero and is determined

from analysis of a sample in a given matrix containing the analyte. The precision-based MDL will be calculated by following the steps listed below and may be recorded on the attached form (Appendix C).

1. Obtain a calibration check standard that contains lead at a concentration two to five times the expected MDL. The typical FPXRF lead MDL ranges from 50 mg/kg to 100 mg/kg and will not exceed 120 mg/kg. The Scitec MAP Spectrum Analyzer should not be used on this project because its method detection limit is too high to meet the project objectives. If an MDL of 120 mg/kg cannot be achieved, the manufacturer should be consulted to determine whether the instrument is capable of meeting the MDL required by this project. The precision-based MDLs calculated for lead during an EPA technology evaluation project for six common FPXRF instruments are summarized below.

Instrument Method Detection Limit Summary

	Field Portable X-Ray Fluorescence Instruments					
	Innovex TN 9000®	Innovex TN Lead Analyzer®	Metorex X-MET 920-P®	Metorex X-MET 920-MP®	Niton XL Spectrum Analyzer®	Scitec MAP Spectrum Analyzer®
MDL	45	40	45	100	75	165

Notes:
The method detection limits summarized above were obtained from the Environmental Protection Agency *draft method 6200 document*.
The method detection limits are listed in milligrams per kilogram.
The Scitec MAP Spectrum Analyzer should not be used on this project due to its method quantitation limit.
MDL = method detection limit

2. Analyze the calibration check standard 10 times for 30 nominal seconds.
3. Record the instrument readings in the field logbook.
4. Calculate the standard deviation for the 10 results and record in the field logbook.
5. Multiply the calculated standard deviation by 3 to obtain the precision-based MDL and record in the field logbook.

Readings that are greater than the MDL, but less than the MQL are considered to be estimated values.

Method Quantitation Limit

MQL is defined as the lowest amount of analyte in a sample that can be quantitatively determined with stated, acceptable precision and accuracy (bias) under stated analytical conditions. The MQL is calculated by multiplying the standard deviation tabulated for the MDL by 10. The MQLs calculated for lead during an EPA technology evaluation project for six common FPXRF instruments are summarized below. Note: The MQL for the Scitec MAP Spectrum Analyzer® is greater than the MQL allowed at the OLS; thus, it should not be used.

Instrument Method Quantitation Limit Summary

	Field Portable X-Ray Fluorescence Instruments					
	Innovex TN 9000®	Innovex TN Lead Analyzer®	Metorex X-MET 920-P®	Metorex X-MET 920-MP®	Niton XL Spectrum Analyzer®	Scitec MAP Spectrum Analyzer®

MQL	150	133	150	333	250	550

Notes:
The method detection limits summarized above were derived from the Environmental Protection Agency draft method 6200 document.
Agency Field Portable X-ray Fluorescence Analyzer, Niton XL, Technology Verification Report.
The method detection limits are listed in milligrams per kilogram.
The MAP Spectrum Analyzer should not be used on this project due to its method quantitation limit.
MQL = method quantitation limit

3.1.2 Daily Quality Control Checks

This section discusses the QC checks that are required to be completed daily on the FPXRF: energy calibration, instrument blank, calibration verification, and precision measurement. A form has been attached in Appendix D to record results.

3.1.2.1 Energy Calibration Check

The energy calibration check determines whether the characteristic X-ray lines are shifting, which indicates that the instrument is drifting. The check should be run at a frequency consistent with the manufacturer's recommendations to determine whether the FPXRF instrument is operating within resolution and stability tolerances. At a minimum, the check will be performed at the beginning of each working day, after the batteries have been changed or the instrument shut off, at the end of each working day, and at any other time at which the instrument operator believes that drift is occurring during analysis. The resolution listed on the instrument will be recorded in the field logbook and must be within 20 percent of the first reading of the day. Most instruments will not calibrate properly and will list an error if the resolution drifts drastically.

3.1.2.2 Instrument Blank Check

The instrument blank will be analyzed on each working day before and after analyses are conducted and once per every 25 samples. It also will be analyzed if the instrument is suspected to be contaminated. The manufacturer's recommended count time (for example: the Niton® FPXRF lists nominal seconds) should be used for this sample. At a minimum, the count time should allow the instrument to produce a detection limit equal to the MDL. OLS project objectives require that lead will not be detected above the MDL in this sample. If lead is reported above the MDL, the instrument window should be checked to assure the instrument was properly decontaminated. The instrument blank sample should also be checked to determine if it is free of contamination. When residual contamination is not the problem, the instrument will be zeroed in accordance with the manufacturer's guidelines, which may require that it to be returned to the manufacturer for service.

3.1.2.3 Calibration Verification Check

A field calibration check will be performed a minimum of three times a day, generally at the beginning and end of each working day and at the expected mid-point of the day. The field check should follow the manufacturer's instructions for calibration verification. The calibration check sample should contain lead concentrations near the action levels of 400 mg/kg and 1,200 mg/kg. The sample shall be analyzed until the instrument uncertainty is less than $^{+}$/- 10 percent of the measured value. The measured value for the check sample must be within $^{+}$/- 20 percent of the true value of the check sample to be acceptable. If the measured value falls outside the

acceptance range for a mid-day or final verification, the instrument will be recalibrated in accordance with the manufacturer's instructions. Any samples analyzed before the failed calibration check must be reanalyzed.

3.1.2.4 Precision Measurement Check

The method precision must be documented daily before the FPXRF is used. Relative standard deviation (RSD) is used to assess method precision. The RSD must be less than 20 percent for FPXRF readings to meet the project objectives for precision. If the RSD is greater than 20 percent, the method precision should be rechecked and less than 20 percent before used. If the recheck RSD is greater than 20 percent, the manufacturer should be contacted to resolve the issue. The steps listed below will be followed to calculate the method precision.

1. Obtain check samples with varying lead concentrations.
2. Analyze one of the samples seven times. The samples shall be analyzed until the instrument uncertainty is less than $^{+}$/- 10 percent of the measured values. Note: The check samples should be alternated daily to avoid completing a precision calculation using the sample analyzed the previous day.
3. Document the instrument readings in the field logbook.
4. Calculate the standard deviation and mean concentration for the seven readings.
5. Divide the standard deviation by the mean concentration.
6. Multiply value obtained from step 5 by 100 to obtain the RSD.
7. Record all readings and calculations in the field logbook.

3.2 FPXRF SCREENING METHOD

Screening of the soil for lead concentrations will be accomplished using a portable FPXRF instrument. The sample screening process includes collecting, preparing, and analyzing the soil from the bottom of an excavated area to evaluate the concentrations of lead at the bottom of the excavation and document whether the clean-up goals have been achieved.

At the OLS site, several zones may be sampled. The zones can generally be placed in one of two categories: drip zones and non-drip zones. For purposes of soil sampling, a drip zone is defined as that portion of the property that is located within 6 - 30 inches of the exterior building wall. The non-drip zone is defined as that area of the property located away from the exterior wall. These areas typically include zones that are defined as quadrants, play areas, gardens, or easements.

3.2.1 Sample Collection

The steps for collecting a composite soil sample from OLS properties to determine post-excavation lead concentrations are listed below and summarized in the accompanying flow chart. The sample collection steps were derived from the EPA *Superfund Lead-Contaminated Residential Sites Handbook* prepared by the EPA Lead Sites Workgroup (LSW) published in August 2003 (EPA, 2003a) and EPA SOP 4230.19A titled *Soil Sampling at Lead-Contaminated Residential Sites* and dated July 3, 2007 (EPA, 2007b).

1. Acquire a property map that depicts the sample zone boundaries.
2. Determine the extent of the sampling effort, including types and amounts of equipment and supplies. Supplies that may be required to complete the post-excavation FPXRF screening are listed in Table 3.1.
3. Obtain supplies necessary to collect samples.
4. Inspect the supplies to ensure that they are in good condition and are free of contamination (see Section 9.8).
5. Coordinate with staff, client, and regulatory agencies as necessary.
6. Upon arrival at the subject property, complete a general site survey.
7. Don the appropriate health and safety personal protective equipment (PPE). The remaining post-excavation screening sections assume that the appropriate PPE will be worn for all sample collection, preparation and analysis activities. At a minimum, contaminant-free gloves will be worn at all times when handling a sample.
8. In **non-drip zones**, collect five to nine discrete aliquots of near equal amounts from each non-drip zone. In **drip zones**, collect four discrete aliquots from exposed soil surfaces, preferably from each of four sides of the structure. Aliquots will be collected from a depth of 0 to 6 inches below ground surface (bgs) using a sampling device such as a slide hammer. Each aliquot should generally be collected from the midpoint of each side of the house. Collection of additional aliquots should be considered if other factors exist, such as areas where runoff collects from downspouts. Also, if the drip zone near one or more of the exterior walls is covered with concrete, the aliquot(s) will be adjusted and collected from another segment of the drip zone. Examples of aliquot collection locations for typical properties are illustrated on Figure 3.1.
9. Place the aliquots in a clean, disposable pie pan. The benefits of using a pie pan to homogenize and prepare samples for FPXRF analysis are that moisture in the sample is reduced during the preparation phase, debris is easily visible, no decontamination is required, and the potential for cross-contamination is eliminated.

An illustration of the steps listed above is presented on Figure 3.2.

3.2.2 Sample Preparation

Sample preparation will be conducted in accordance with the process described below. The sample preparation process was derived from information obtained from the EPA *Superfund Lead-Contaminated Residential Sites Handbook* (EPA, 2003a)*;* EPA SOP 4230.19A, *Soil Sampling at Lead-Contaminated Residential Sites* (EPA, 2007b), and EPA SW-846 Method 6200 (EPA, 1998b). Figure 3.3 is a flow chart illustrating sample preparation process.

1. Remove visible debris from the aliquots placed in the clean pie pan as it will impact the accuracy of the FPXRF reading. Debris that must be removed includes grass, leaves, concrete, rock, wood chips, roots, and trash, and any large visible paint chips.
2. Homogenize the sample using the clean utensil used to collect the aliquots. Homogenization entails breaking up the sample into small pieces and thoroughly mixing all aliquots to form a composite sample After homogenization, the composite sample should consist primarily of soil pieces less than ¼-inch in size.
3. Continue to mix the sample and inspect for and remove debris. Repeat this step until all debris is removed.

4. Transfer the composite sample to a decontaminated 60-mesh screen sieve. Sieving with a number 10-mesh screen reduces the sample error that may be introduced with varying particle size, lack of homogeneity, and excessive moisture. Other than the debris described in Step 1, the entire composite sample must be sieved.
5. Samples that contain excessive moisture will likely plug the holes in the screen and prevent the sample from being sieved. Excessive moisture can be removed by drying the sample in its pie pan for 2 to 4 hours in a conventional oven at a temperature less than 150 degrees Fahrenheit (º F) or by drying in the open air until it is dry enough to pass through the sieve.
6. The sieved sample will be placed in an analysis cup designed for the FPXRF being used. The cup should be filled to the upper edge with the sieved soil.
7. Cover the sample cup with a lead-free film.

3.2.3 FPXRF Field Analysis

FPXRF field analysis will be conducted in accordance with the guidelines described in this section. Three readings and uncertainty values will be recorded, averaged, and documented for each zone that required excavation. Uncertainty values are expressed as a $^{+}$/- or error value. The three XRF confirmation readings must be $\pm$ 10% of the mean of the three readings if any of the three individual XRF results exceed the soil lead cleanup criteria. If all three XRF readings are below 400 ppm in samples collected from the upper foot, or less than 1,200 ppm in samples collected at depths greater than one foot, agreement of samples within 10% of the mean is not required. This means the average of the three readings will still be considered the sample concentration when the three readings are not within 10% of one another.

Unless otherwise directed by an EPA representative, the parameters listed below must be met before the zone is backfilled.

All Zones (excluding Garden Zone)

- For a zone excavated to a depth of 12 inches or less, the value of the averaged reading must be less than 400 mg/kg.
- For a zone excavated to a depth greater than 12 inches, the total averaged reading must be less than 1,200 mg/kg.
- If a zone is excavated to varying depths, all exposed soil surfaces excavated to a depth of 12 inches or less must be less than 400 mg/kg. All exposed soil surfaces excavated to a depth greater than 12 inches must be less than 1,200 mg/kg.
- If the homeowner will not allow certain items (for example: shrubs and flowers) to be removed from the excavation zone, and the total combined unexcavated area for the property is greater than 4 square feet, the property map must be updated to illustrate the unexcavated area. This map must be maintained in the property file, which will be submitted to EPA at the end of the excavation season.

Garden Zones

- In a garden zone, where soil lead concentrations exceed 400 mg/kg, soil must be excavated until reaching a residual soil lead concentration of less than 400 mg/kg at depths of twenty-four inches or less, or until reaching a residual soil lead concentration of less than 1,200 mg/kg at depths greater than twenty-four inches.

Sample material not analyzed in the field will be added to the contaminated stock pile awaiting disposal or returned to the zone it was collected from. Analyzed sample material will either be submitted to the laboratory, added to the contaminated stock pile awaiting disposal, or returned to the zone it was collected from.

3.3 LABORATORY SAMPLES

Since 1999, EPA has assessed the contamination in residential properties by collecting over 8,000 soil samples that have been screened with an XRF and submitted to a fixed laboratory for confirmation analysis. Correlation with the XRF data and laboratory results has surpassed the quality control limits for screening level data specified by SW-846 Method 6200.

Based on the reliable history of XRF measurements at the Omaha Lead site, EPA will discontinue the collection of confirmation samples in connection with the cleanup activities. Collection of lab confirmation samples for assessing contamination with the residential screening effort (outside the scope of this SAP) will be continued and addressed in a separate SAP. If the correlation between XRF measurements and laboratory data does not meet SW-846 Method 6200 requirements, EPA may reconsider its decision not to collect lab confirmation samples in association with the soil cleanup activities.

3.4 RINSATE SAMPLES

Rinsate samples will be collected at the beginning of each month to assure the sieve is being properly decontaminated. After the sieve has been used to prepare a sample and decontaminated, deionized water will be poured through the sieve screen and collected in an appropriate sample container. The analysis of the rinsate sample is described in Part 2 of this SAP.

3.5 INVESTIGATION-DERIVED WASTE

Investigation-derived waste (IDW) generated by the field screening process will include excess soil sample material, disposable equipment and PPE, and decontamination fluids. The following procedures will be used to dispose of IDW:

Excess soil sample material. Whenever possible, excess soil will be disposed in the general location from where it was collected. If this is not possible, excess soil sample material will be placed with the excavated stockpiled soils for proper disposal.

***Disposable equipment and PPE*.** Non-hazardous and decontaminated disposable equipment and PPE should be double bagged for disposal at a sanitary landfill.

***Decontamination fluids*.** Decontamination fluids from each property will be placed in a watertight container, labeled, sampled, and staged awaiting sample analysis. After sample analysis, non-hazardous fluids will be disposed of in a sanitary sewer. Any IDW that is considered Resource Conservation and Recovery Act (RCRA) hazardous waste will be disposed of at an appropriate licensed hazardous waste disposal facility by a state-licensed waste hauler.

3.6 SAMPLING UNSAMPLED AREAS

Removal contractors can encounter unsampled areas under two different circumstances. Sometimes the sampling contractor will not have sampled part of the property. This is an error by the sampling contractor which should be corrected. The second type of unsampled area is identified when a homeowner requests part of a quadrant remain unexcavated for personal reasons. In this case, the contractor can sample these areas and note the results on the as-built sketch.

3.6.1 Unsampled Areas

In rare instances, the sampling contractor may not have collected a sample from part of a particular residential property. This is the sampling contractor's error and it must be corrected. The excavation contractor should inform the COR of the unsampled area. The COR will make arrangements to have the sampling contractor collect a sample and update the Oracle database with the new data. The sampling contractor will also modify the property sketch (aka field sheet) and upload it into the database. The COR will then provide the revised sketch to the excavation contractor.

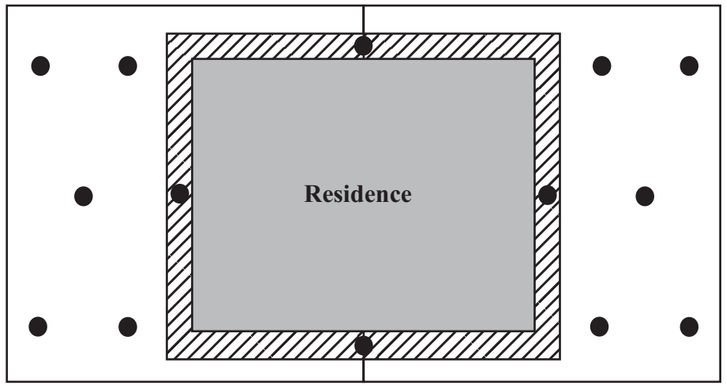
3.6.2 Unexcavated at Owner's Request

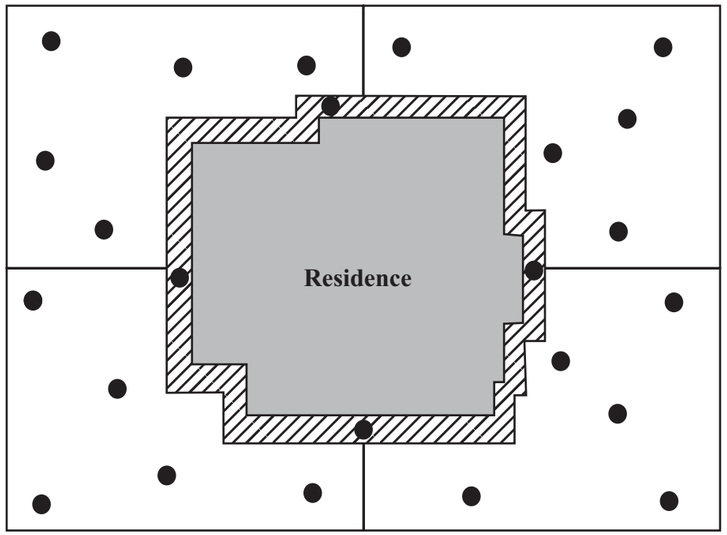
Areas left unexcavated at the request of the homeowner will be located within a quadrant that was previously sampled and found to be 400 ppm or greater. Unexcavated parts of a quadrant need to be sampled by the excavation contractor using the procedures in Section 3.2 FP XRF Screening Method in the Sitewide Sampling and Analysis Plan. The contractor will add the new data to the as-built sketch and the Oracle database.

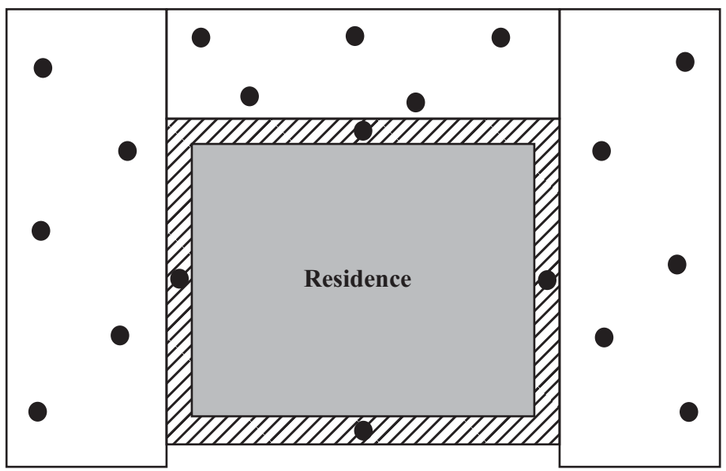
Table 3.1
Field Equipment and Supplies

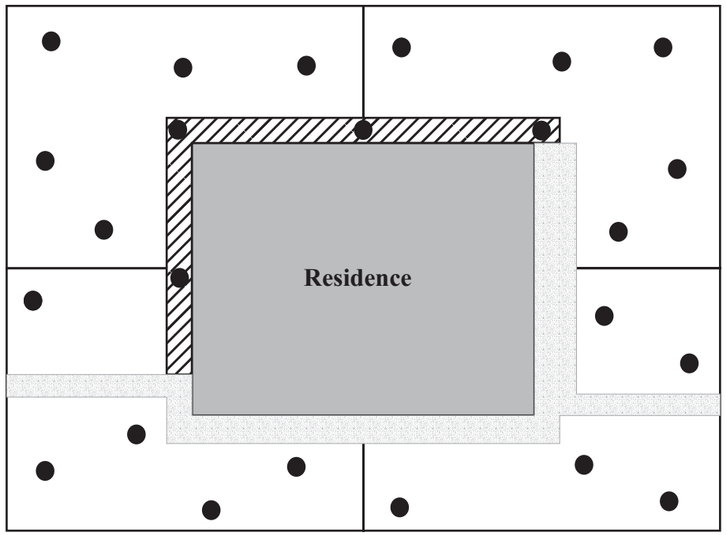
Sampling Supplies	
Appropriate size sample containers	Sampling field forms
Preservatives (nitric acid)	Sample labels
Baggies	Chain-of-Custody forms
Ice	Custody seals
Sample shipping coolers	Vermiculite
Shipping material (packaging tape, bubble wrap)	Labels
Deionized water	Alconox detergent and scrub brushes
Sampling Equipment	
Spatulas	Survey equipment or GPS
Shovel or spade	FPXRF instrument
Scoops	Plastic or stainless steel spoons
60-mesh shaker sieve	Trowels
Disposable pie pans	
Health and Safety	
Latex and nitrile gloves	First aid kit
Eyewash station	Fire extinguisher
Tyvek®	Steel-toed boots
Hard Hat	Safety glasses
High visibility safety Vest	NIOSH N100 dust mask
General Field Operations	
Logbooks	Indelible ink pens
Digital camera or camera and film	Paper towels
Canvas or plastic sheeting	Trash bags
Tape measure	Survey stakes or flags
Maps/plot plan	Plastic bucket of tub for decontamination
Investigation derived waste container	

FIGURES









HGL—Sitewide Sampling and Analysis Plan for the Omaha Lead Site—Omaha, NE

Figure 3.1
Soil Sample Aliquot Location Examples

U.S. EPA Region 7

Legend

- Sample Aliquots
- Residential Structure
- Concrete
- Drip Zone

X:/EPA009/Omaha_Lead SAP/
Soil_Sample_Aliquot_Loc.cdr
Created: 09/04/2007
Project: EPA009-018-03-03

HGL HydroGeoLogic, Inc

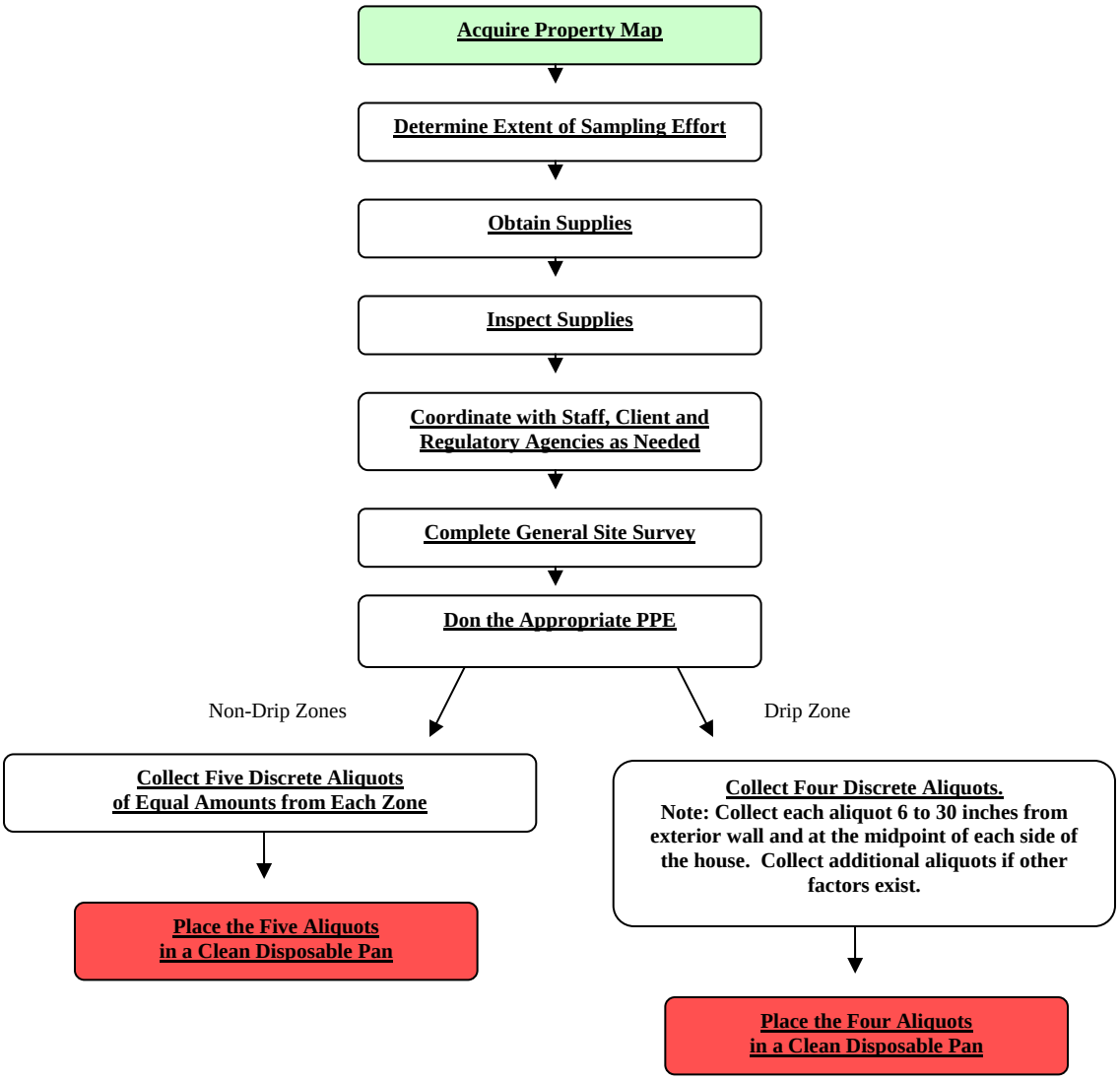


Figure 3.2 Flow chart representing the sample collection process.

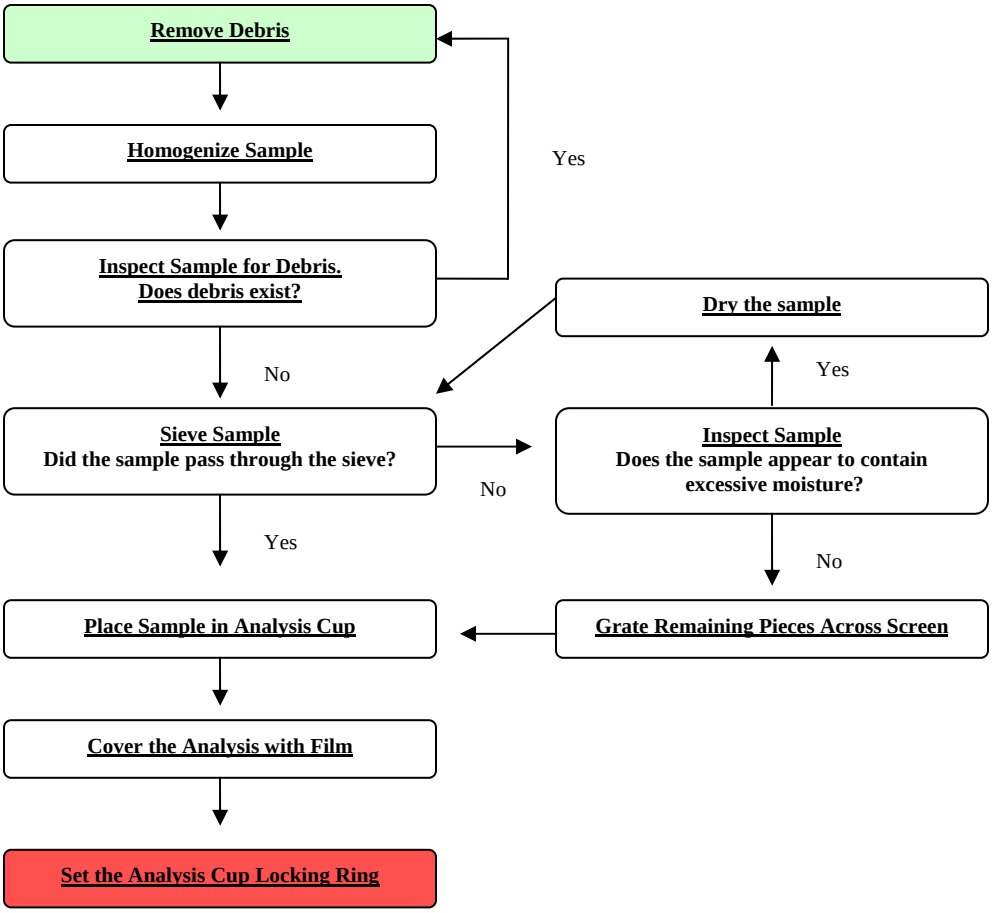


Figure 3.3 Flow chart representing the sample preparation process.

4.0 AIR SAMPLING

This section describes the air sampling procedures that will be implemented at the OLS. Three types of air sampling will be conducted: personal monitoring, sampling at the property being excavated, and monitoring the contaminated stock pile. The air samples will document site worker exposure to potential lead-contaminated particulates, and determine whether contaminant migration is occurring from the excavation sites and stock pile area. The procedures were obtained from EPA SOPs. At a minimum, the air samples will be collected at the beginning of the excavation season and in late July or early August.

4.1 PERSONAL AIR SAMPLING

This section describes the process for collecting personal air samples using a low volume sampling pump. The process follows the guidelines promulgated in the EPA Environmental Response Team (ERT) SOP Number 2119, *Air Sampling for Metals* (EPA, 1994c).

4.1.1 Preparation

The steps for preparing to collect a personal air sample to document worker exposure are listed below.

1. Arrange for sample analysis by a certified laboratory.
2. Determine if the laboratory has any special requirements (for example: lot blanks) and incorporate into sampling protocol.
3. Schedule sampling event with staff.
4. Obtain and organize the necessary sampling equipment. Table 4.1 lists the equipment that may be required to complete personal air monitoring.
5. Don the appropriate PPE. The remaining air sampling sections assumes that the appropriate PPE will be worn for all activities associated with the section. At a minimum, contaminant free gloves will be worn at all times when handling a sample.
6. Decontaminate or pre-clean equipment, and ensure that it is in working order. If possible, precalibrate equipment.
7. Connect the calibration assembly. A typical calibration assembly is illustrated on Figure 4.1. The calibration assembly will consist of a 37 millimeter (mm) 3-stage filter cassette loaded with a 0.8 micrometer mixed cellulose ester (MCE) filter and support pad, tygon tubing, a hose-barb filter adapter, a flow meter, and an adjustable flow sampling pump. The filter cassette is illustrated on Figure 4.2.
8. Turn on the pump and adjust the pump to the desired flow rate.
9. Affix a sticker or tag to the pump that lists the flow rate and sample media.

4.1.2 Personal Air Sampling

The steps for collecting a personal air sample using a low volume sampling pump to document exposure are listed below.

1. Connect the sample assembly. A typical sample assembly is displayed on Figure 4.3.

2. Ensure that all sample connections are tight.
3. Verify the pump flow rate by attaching the meter to running sample pump.
4. Record flow rate on the sample sheet.
5. Connect the filter cassette.
6. Remove the sample cap/plug.
7. Deploy the sample assembly.
8. Record the beginning sample time.
9. Allow the pump to run for 8 hours. Note: Before sampling commences, the laboratory must be contacted to assure that sufficient air volume passes through the filter to attain a detection limit low enough to meet project objectives. The detection limit minimally must be less than the Occupational Safety and Health Administration (OSHA) permissible exposure limit (PEL) of 50 micrograms per cubic meter ($\mu g/m^3$) of lead.
10. Disconnect the cassette.
11. Seal the cassette with plugs or caps.
12. Record the ending sample time.
13. Record the final flow rate.
14. Label and package sample.
15. Calculate the sample volume for the laboratory.

If a personal air sampler indicates the presence of lead at a level that exceeds the 50 $\mu g/m^3$ OSHA PEL, EPA will be contacted to determine whether dust suppression or other measures should be implemented.

4.2 PROPERTY AIR SAMPLING

This section describes the procedure for collecting air samples during property excavation activities using a total suspended particulate (TSP) monitor (also known as hi-vol). The process follows the guidelines listed in EPA SOP 2314.1B titled *Hi-Vol Operation* (EPA, 1985). The SOP was approved on May 13, 1985 and recertified on October 28, 2005. Hi-vol sampling will be conducted twice per year. For each event, four air samples will be collected from two properties at which excavation activities are under way. One sampler will be placed in each cardinal direction surrounding the property and should be inside of or within five feet outside of the property line. Figure 4.4 illustrates example placement of deployed samplers. The sampler maintenance and QC records will be reviewed to ensure that the guidelines established in EPA SOP 2314.1B are strictly followed.

4.2.1 Filter Preparation

The steps for preparing the sample filter are listed below. The filters prepared should not be stored more than one month.

1. Purchase prenumbered 8-inch by 10-inch glass fiber filters. The identification numbers will only exist on one corner of the filter.
2. Using a lighted box, visually inspect the filters for pin holes, weak spots, dirt, or other foreign material embedded in the fiber. Both sides of the filter will be inspected.
3. Document the visual inspection results in a logbook. If flaws are observed, note the flaws and discard the filter.

4. Equilibrate the filters in a conditioning environment (such as a desiccator) for 24 hours before weighing them. The conditioning environment must average between 20 degrees Celsius (ºC) and 25ºC and not vary more than 3ºC. It must also contain a relative humidity of less than 50 percent and the relative humidity should not vary more than 5 percent.
5. Check the scale before weighing the filters.
6. Obtain a standard class-S weight between three and five grams (g). Note that in the S Class weight the knob is a separate piece, which can be unscrewed to reveal a small compartment for adjusting the mass of the weight.
7. Record the weight in the logbook. If the weight is within 0.0005 g of the true value, proceed to the next step. If the value is not within 0.0005 g, the scale must be repaired and recalibrated before it is used.
8. Weigh each flat filter to the thousandth gram place (0.001 g).
9. Record the filter number, date, tared weight, and atmospheric conditions (room temperature and relative humidity) in the logbook.
10. Store the tared filters in the original box or in a manila folder until used.

4.2.2 Hi-Vol Functional Test

The hi-vol functional test requirements are listed below. The functional test includes a check of the following items: elapsed time meter, on-off timer, and an orifice.

1. Check the elapsed time meter against a timepiece of known accuracy. If a gain or loss of more than 2 minutes over 24-hour period is documented, the elapsed time meter must be adjusted or replaced. Document the findings in the logbook.
2. Check the on-off timer. The timer must turn the sampler on or off within 15 minutes of the assigned time. If it does not, the timer will be adjusted or replaced. Document the findings in the logbook.
3. Inspect the sampler for nicks or dents that could impact the collection of the sample.
4. Document the findings in the logbook.

4.2.3 Hi-Vol Calibration

Hi-vol samplers will be calibrated when first purchased, after major maintenance, if the flow rate measure device is changed or repaired, or if any one audit point deviates more than seven percent from the calibration curve. The hi-vol samplers will be calibrated in accordance with the steps listed below.

1. Document the flow recorder number, hi-vol motor number, and the date.
2. Assemble the sampler with the calibration orifice clamped to the filter holder.
3. Attach a water or oil manometer to the calibration orifice with flexible tubing.
4. Attach the flow recorder to the pressure tap of the hi-vol motor housing.
5. Turn the sampler on and allow the motor to warm up for at least 5 minutes at 115 volts. If a step-down transformer is used during normal operation, utilize the transformer during calibration.
6. Turn the motor off.
7. Place a clean chart on the recorder. Zero the pen if necessary.

8. Change the load plate.
9. Turn the motor on and record the manometer and flow recorder readings after they stabilize.
10. Turn the motor off. Repeat steps 8 through 10 for each load plate.
11. Calculate and record the air flow rates from the hi-vol orifice calibration curve for each flow recorder reading.
12. Record the barometric pressure and the ambient temperature.
13. Calculate the sampler calibration curve, slope (m) and intercept (b), by linear regression analysis of the corrected recorder readings (x) versus the standard sample flow rates (y).

Figure 4.5 illustrates the calibration process.

4.2.4 Hi-Vol Sampling

Property air samples will be collected in accordance with the steps listed below. The sampler will be operated for 8 hours at the property.

1. Place the tared filter into the cassette. The filter must centered and the number will face down so that the sample will be collected on the un-numbered side.
2. Place cover over the filter.
3. Record a sample location description, motor number, filter number, and time sample is set to begin collecting in the logbook.
4. Check the on-off device to ensure it is set properly.
5. Record the value listed on the elapsed time meter.
6. Place a recording chart on the flow recorder, checking the zero and pen.
7. Document the current temperature and barometric pressure, which can be obtained from on-site instruments, from the National Oceanic Atmospheric Administration (NOAA) website, or by calling the local National Weather Bureau.
8. Remove the filter cover.
9. At the conclusion of the sampling period, record the final elapsed time meter reading.
10. Before removing the cassette, turn the hi-vol motor on and check the final flow for consistency with the initial flow.
11. Record the date and the sampling time indicated on the elapsed time meter in the logbook.
12. Replace the cover.
13. Remove the filter cassette. Note: when removing the filter cassette grasp it gently at the ends.
14. Remove the flow recording trace.
15. Transport the covered filter cassette to a stable environment.
16. Remove the upper part of the filter cassette holding the filter in place.
17. Carefully remove the glass fiber filter.
18. Inspect the filter and remove any captured insects with tweezers.
19. Record any abnormalities (torn filter, malfunctioning elapsed time meter, flawed recording trace chart, etc.) in the logbook.
20. Fold the filter lengthwise with the sampled side in.
21. Place the filter in an envelope or folder.
22. Record the temperature and average pressure using the sources listed on step 7.

23. If the filters are damp, allow them to equilibrate for 24 hours in a conditioning environment.
24. Check the balance using steps 5 through 7 listed in Section 4.2.1.
25. Record the temperature and average pressure using the sources listed on step 7.
26. Individually, weigh the exposed filters to the nearest milligram
27. Record the weight in a logbook.
28. Place the exposed filter in the laboratory specified container and submit for lead analysis.
29. Calculate the sample air volume using the equation below.

$$v = Q\,t$$

v = air volume sample (cubic meter [m^3])
Q = average sampling rate (m^3/ per minute [m^3/min] at standard temperature and pressure [stp])
t = sampling period (elapsed time in minutes)

The Q will be obtained from the recorder chart. If the flow rate varies less than 0.11 m^3/min during the sampling period, read the flow rate from the chart at 2-hour intervals and average the values from the intervals. For greater flow rate variations, use an hourly average. Then correct the chart reading to standard temperature and pressure (stp), and calculate the average sampling rate using the hi-vol calibration curve.

30. Document the sample volume in the logbook.
31. Calculate the TSP concentration using the equation shown below.

$$TSP = \frac{(W_f - W_i)\,10^6}{V}$$

W_f = Weight of the exposed filter in grams
W_i = Weight of unexposed filter in grams
v = Air volume in m^3

32. Record the TSP concentration in the logbook.

4.3 CONTAMINATED SOIL STOCKPILE AIR SAMPLING

The procedure for collecting air samples at the contaminated soil stockpile is the same as described text for the property sampling procedures. Four air samples will be collected from the stock pile twice a year as described earlier. The samples will also be collected when the pile is active, that is when contaminated soil from the properties is being off-loaded by trucks or the stockpile soil is being loaded into trucks for transport to the landfill. One hi-vol sampler will be placed in each cardinal direction surrounding the pile. The samplers will be placed within five feet of the edge of the pile. The sampler maintenance and QC records will be reviewed to ensure the guidelines established in SOP 2314.1A are strictly followed.

4.3.1 Filter Preparation

The steps to prepare the filter listed in Section 4.2.1 will be followed.

4.3.2 Hi-Vol Sampler Functional Test

The steps to perform the sampler functional test listed in Section 4.2.2 will be followed.

4.3.3 Hi-Vol Calibration

The steps to calibrate the sampler listed in Section 4.2.3 will be followed.

4.3.4 Sampling

The steps to collect the sample listed in Section 4.2.4 will be followed.

Table 4.1
Field Equipment and Supplies for Hi-Vol Air Sampling

Sampling Supplies	
Sample containers	Sampling field forms
Baggies	Sample labels
Sample shipping coolers	Chain-of-Custody forms
Shipping material (packaging tape, bubble wrap)	Custody seals
Trowel/spoon	Paper Towels
Deionized water	Alconox detergent
Low volume sampling pump	High volume sampling pump
8 inch by 10 inch glass fiber filters	
Health and Safety	
Latex and nitrile gloves	First aid kit
Eye wash station	Fire extinguisher
Tyvek®	Hard hat
Safety vest (reflective)	Steel-toed boots
Safety glasses	
General Field Operations	
Logbooks	Indelible ink pens
Digital camera	Paper towels
Trash bags	Scale

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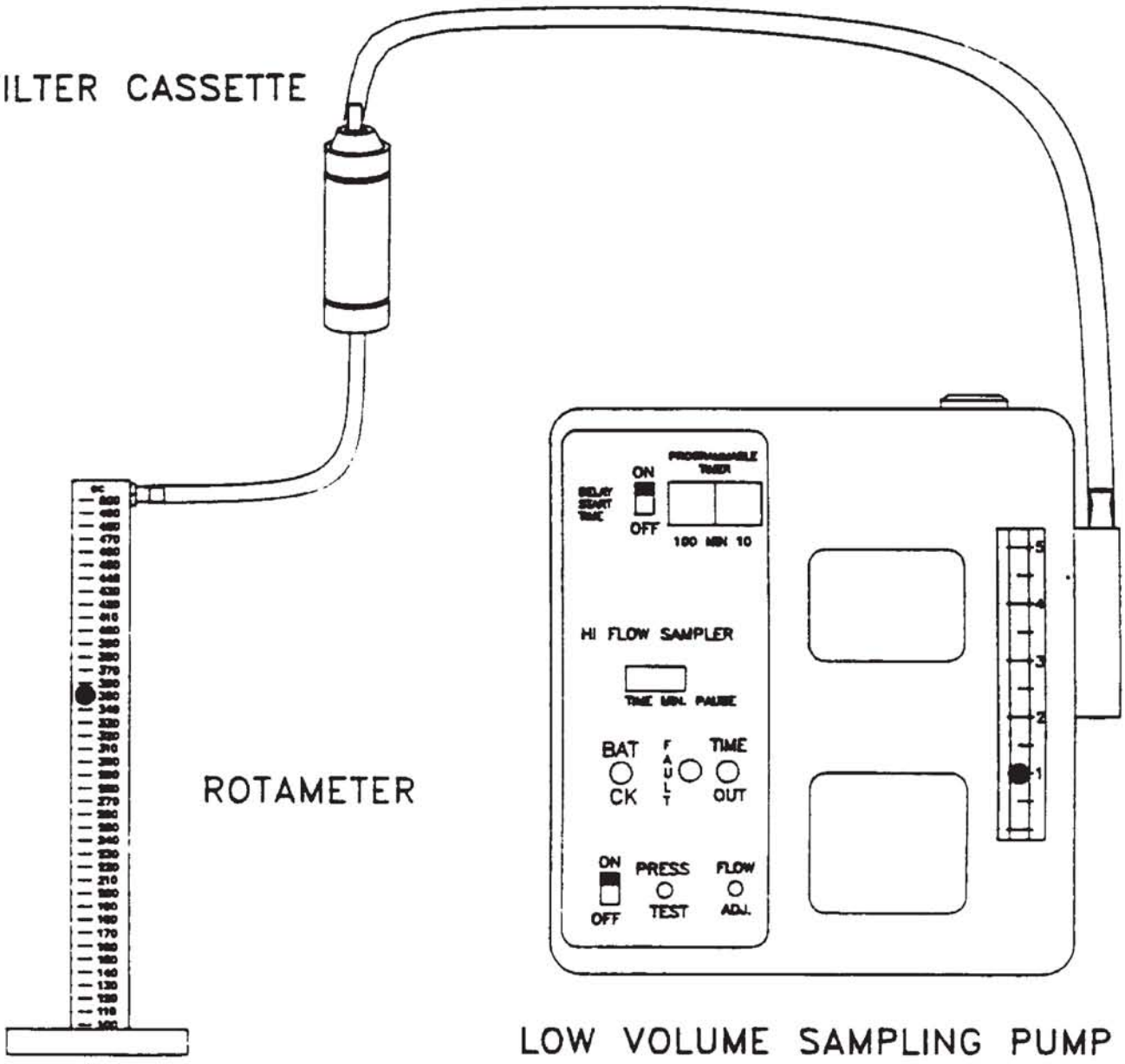
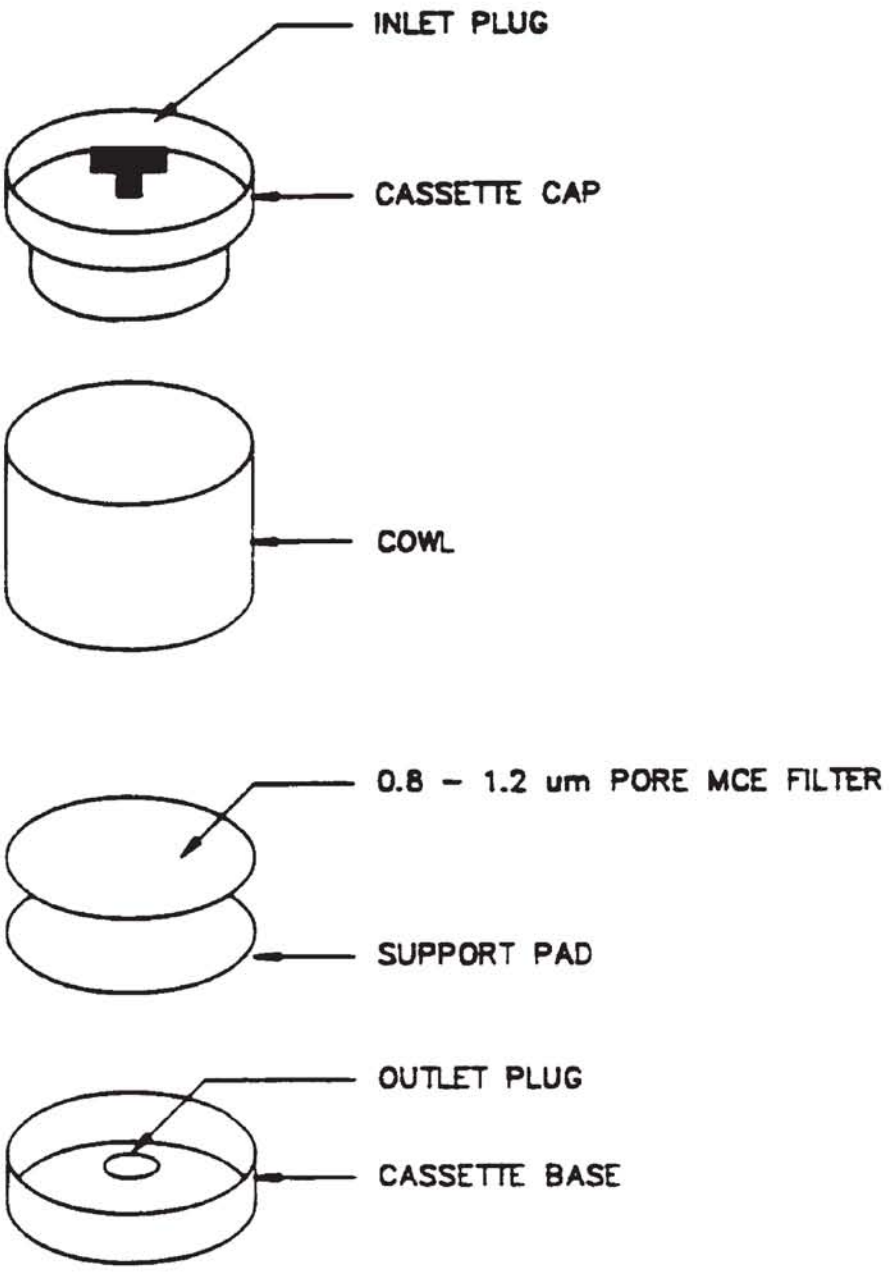
FIGURES



X:/EPA009/Omaha_Lead SAP/
Personal_Air_Monitoring_Calibration.cdr
Created: 09/04/2007
Project: EPA009-018-03-03

Figure 4.1
Personal Air Monitoring
Calibration System



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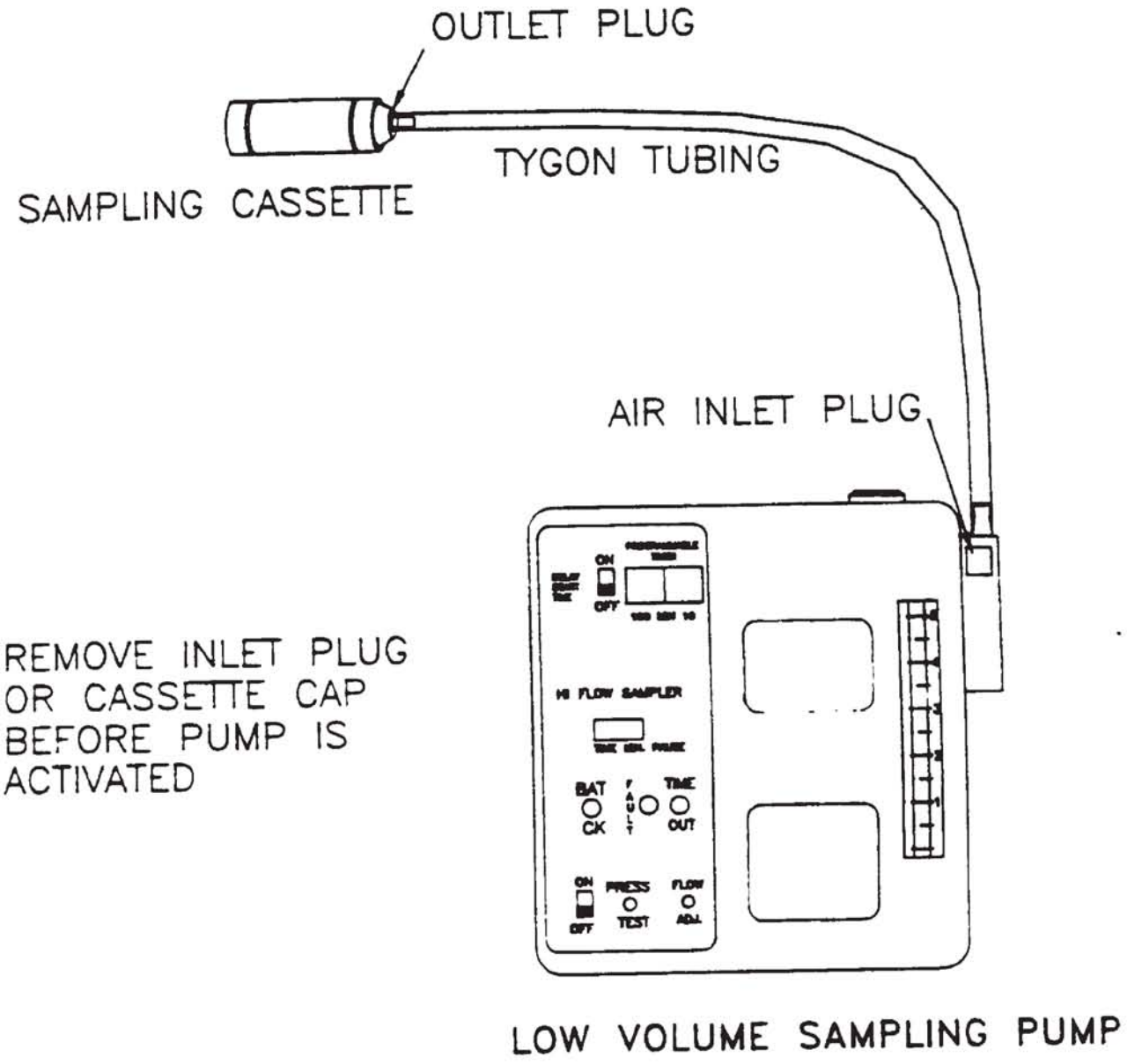
Personal_Air_Monitoring_Filter_Cassette.cdr
Created: 09/04/2007
Project: EPA009-018-03-03

Figure 4.2
Personal Air Monitoring
Filter Cassette



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Personal_Air_Monitoring_Sample_Assembly.cdr
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Project: EPA009-018-03-03

Figure 4.3
Personal Air Monitoring
Sample Assembly

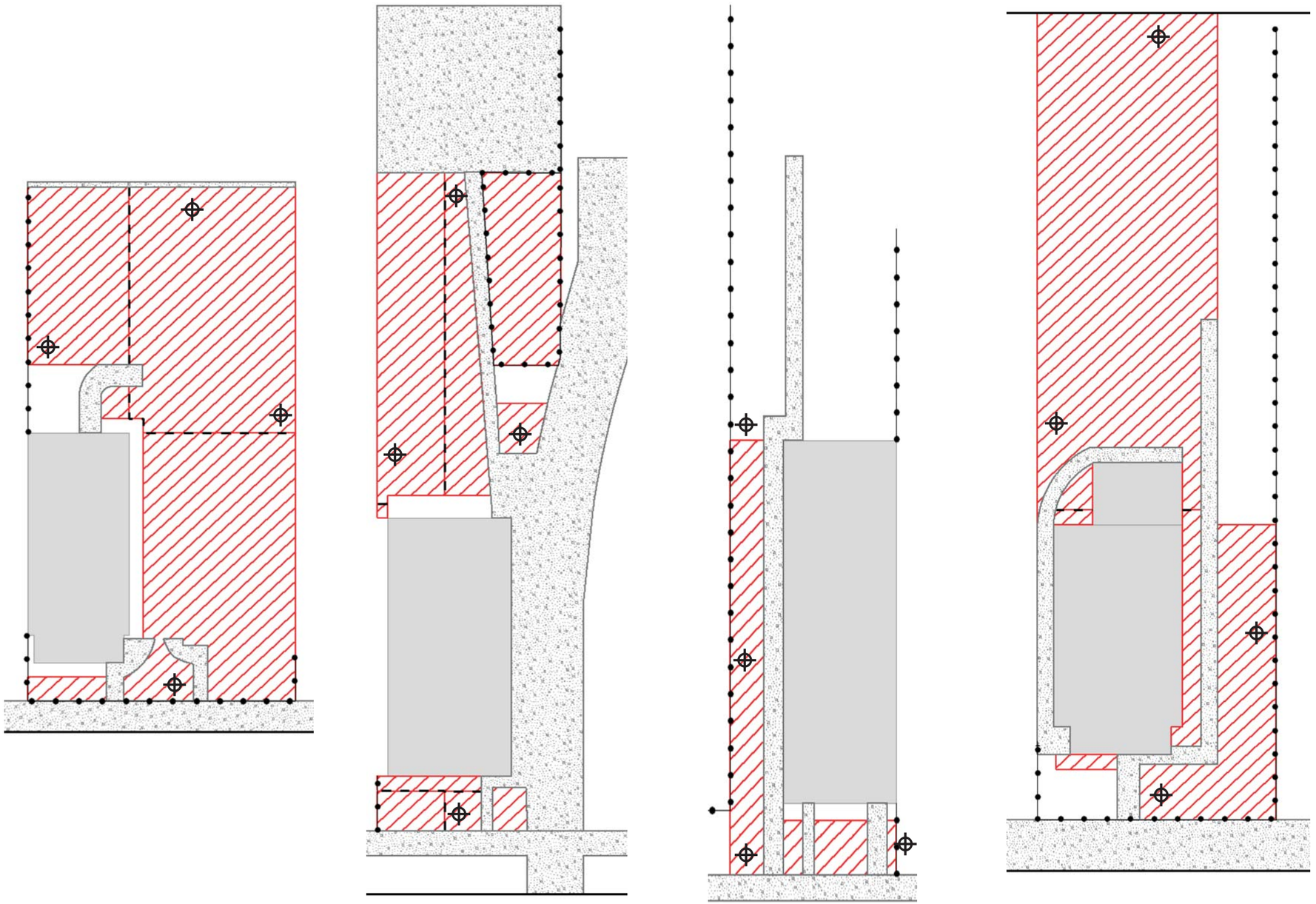

Figure 4.4
Property Air Monitoring Sampler Location Examples

Legend

- Hi-Vol Air Sampler
- Residential Structure
- Area to be Excavated

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Property_Air_Monitoring_Sampler_Loc.cdr
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Project: EPA009-018-03-03

HGL HydroGeoLogic, Inc

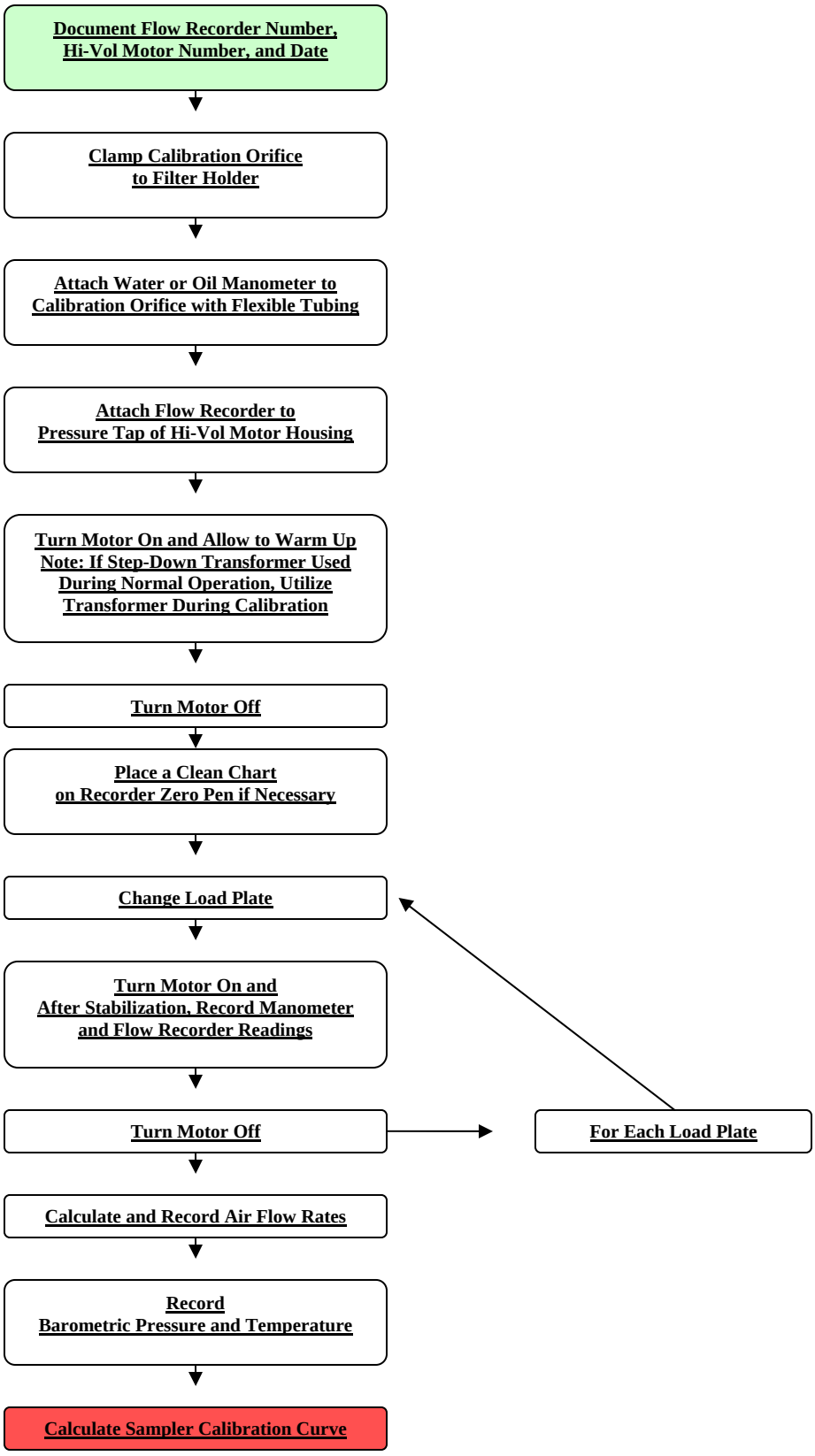

Figure 4.5 Flow chart representing the Hi-Vol calibration proces

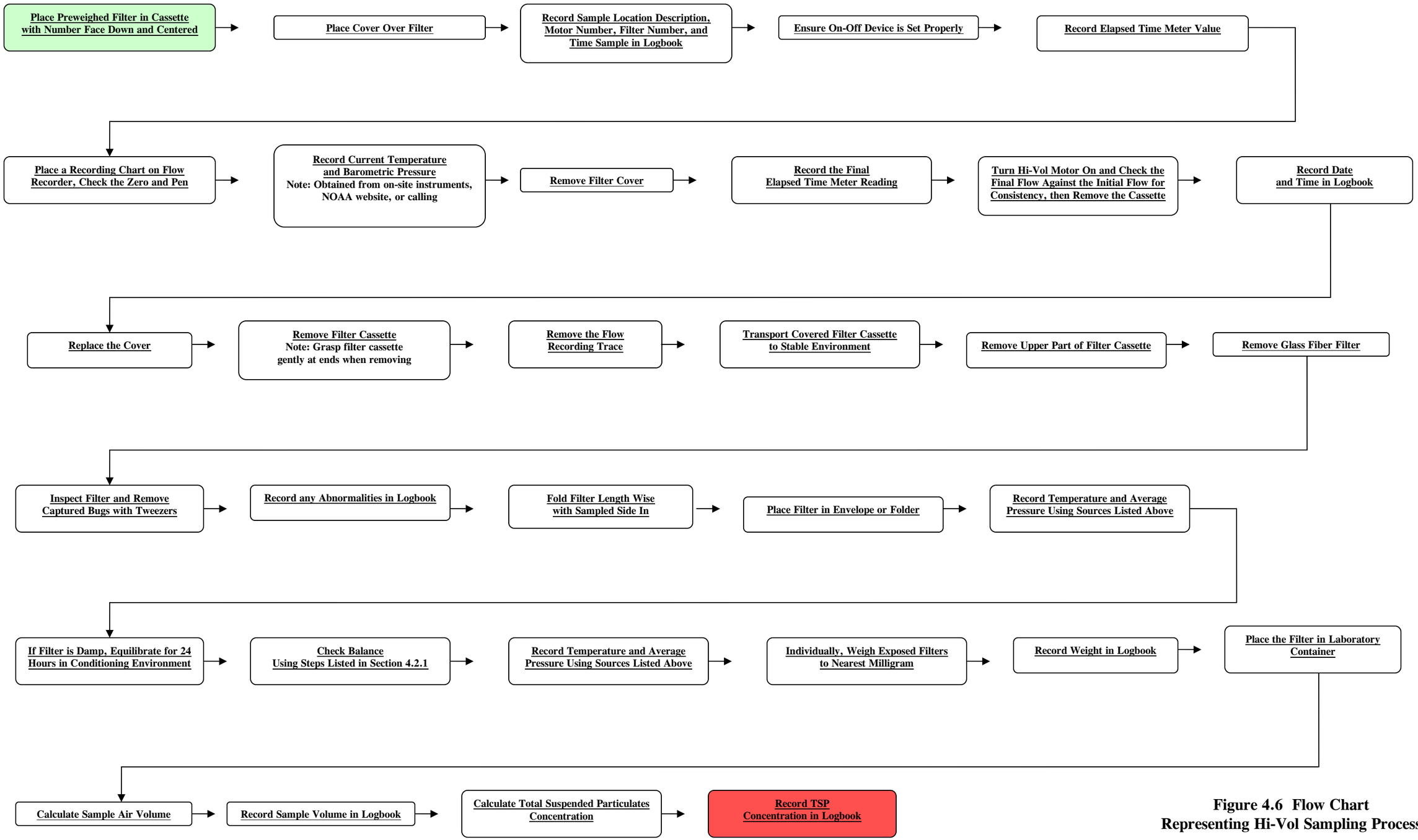

Figure 4.6 Flow Chart Representing Hi-Vol Sampling Process

5.0 CLEAN FILL SAMPLING

The fill material used to backfill excavations will be sampled to assure that it is free of contamination. Clean fill sampling for the OLS will be conducted in accordance with the procedures specified in this section. There are no specific guidance documents for this activity; the steps for collecting the clean fill samples were derived from past EPA site practices at the OLS. The recommended nutrients levels were obtained from the North Central Region for soil nutrient analysis as stated in *Recommended Chemical Soil Test Procedures for the North Central Region*, revised in 1998 (NCR-13, 1998).

Samples must be collected from each fill source location and the source approved by EPA before the fill is placed at a property.

1. Identify origin of clean fill and evaluate current and former land use for potential concerns.
2. Establish a subcontract with a qualified National Environmental Laboratory Accredited Counsel (NELAC) laboratory for the analysis of the parameters listed below.
   - Plant-available nitrogen
     - Nitrate
     - Ammonia
   - Phosphorus
   - Cations
     - Potassium
     - Calcium
     - Magnesium
   - Sulfate-sulfur
   - Micronutrients
     - Boron
     - Zinc
     - Copper
     - Manganese
   - pH
   - Exchangeable Sodium Percentage (ESP)
   - Soluble salts
   - Cation Exchange Capacity (CEC)
   - RCRA metals
   - Low-level volatile organic compounds (VOCs) utilizing SW846 Method 5035 sampling method. Note: The VOC samples must not be homogenized
   - Note: The VOC samples must not be homogenized. They should be collected from the central sampling point or a stained area.
   - Semivolatile organic compounds (SVOCs)
   - Pesticides
   - Polychlorinated biphenyls (PCBs)
   - Polynuclear aromatic hydrocarbons (PAHs)

3. Gather sample collection equipment. Table 5.1 summarizes the equipment that may be used when collecting a clean fill sample.
4. Mobilize to the site.
5. Perform visual analysis of clean fill to ensure there is no obvious staining from spilled or dumped materials.
6. Don PPE
7. Using a clean utensil, collect five large aliquots of soil from the area from which the fill will be removed. The depth and dispersion of the aliquots should vary within the pile or area depending on observed conditions, but generally one aliquot will be collected from the center of the pile or area and four locations approximately equidistant surrounding the center point. However, if any sheen, discolorations, or spots are observed an aliquot of these areas will need to be considered for sampling.
8. Place the five aliquots in a clean disposable pan.
9. Using the utensil used to collect the aliquots, combine the five aliquots and mix soil until a homogenous mixture is created.
10. Using the same utensil, transfer the homogenized composite sample to the laboratory-specified containers.
11. Collect VOC samples following SW-846 method 5035 sample collection guidelines.
12. Label the laboratory-specified container with time, date, and sampling identification. This information also will be added to the logbook and the field sheets.
13. Send samples to specified laboratory in accordance with the packaging and shipping protocols detailed in Section 9.3 of the QAPP.
14. Once analytical results are received from the laboratory, review results against EPA Region 9 preliminary remediation goals (PRGs) for residential soils and nutrient requirements. The PRGs are contained in Appendix E and the nutrient requirements are summarized in Table 5.2. The results must fall between the values listed in this table.
15. Provide EPA with a report summarizing the findings.

Table 5.1
Field Equipment and Supplies for Clean Fill Sampling

Sampling Supplies	
Sample containers	Sampling field forms
Preservatives, i.e., sodium bisulfate	Sample labels
Baggies	Chain-of-Custody forms
Ice	Custody seals
Sample shipping coolers	Small bucket
Shipping material (packaging tape, bubble wrap)	Paper Towels
Trowel/Spoon	Alconox detergent
Deionized water	
Health and Safety	
Latex and nitrile gloves	First aid kit
Eye wash station	Fire extinguisher
Tyvek®	Hard hat
Safety vest (reflective)	Steel-toed boots
Safety glasses	
General Field Operations	
Logbooks	Indelible ink pens
Digital camera	Paper towels
Plastic sheeting	Trash bags

Table 5.2
Clean Fill Nutrient Requirements

Parameter	Low (mg/kg)	High (mg/kg)
CEC	9	41
Nitrate-Nitrogen	<10	20-30
Ammonium-Nitrate	<2	>10
Phosphorous	<10	>40
Potassium	<150	250-800
Calcium	<1,000	>2,000
Magnesium	<60	>180
Sulfate-Sulfur	<2	>10
Boron	<0.5	>2
Zinc	<1.0	NA
Copper	<0.6	NA
Manganese	<1.5	NA
pH	<6.0	>7.5
ESP	NA	>10%
Soluble Salts	<640	>1,280
Organic Content	<5%	

Notes:
> greater than
< less than
% percent
CEC Cation Exchange Capacity
mg/kg milligrams per kilogram
ESP Exchangeable Sodium Percentage

6.0 SOD SAMPLING

The sod used to restore properties to pre-excavation conditions must be sampled before use. Sod sampling for the OLS will be conducted in accordance with the guidelines described in this section. Samples must be collected from each sod source location. These samples must be collected, analyzed for the parameters specified below, the resultant data validated to determine its usability, the data reviewed by the excavation contractor, and the sod source approved by EPA before the sod is emplaced at a property. There is no established guidance for sod sampling; the steps listed below for collecting the sod samples were derived from past practices at the OLS.

1. Identify origin of sod.
2. Establish a subcontract with a qualified laboratory for the analysis parameters listed below.
   - RCRA Metals
   - Low-level VOCs utilizing 5035 sampling method.
   - SVOCs
   - Pesticides
   - PCBs
   - PAHs
3. Gather sample collection equipment. The same supplies used to sample the clean fill may also be used to collect the sod samples (see Table 5.1).
4. Mobilize to the site.
5. Perform visual analysis of sod to ensure that the sod is healthy
6. Don PPE
7. Using a clean utensil, remove five sections of sod from five different pallets or rolls.
8. Shake/scrap soil from each section of sod over one clean disposable pie pan.
9. Mix soil until a homogenous composite sample is created.
10. Using the same utensil, transfer the composite sample to the laboratory-specified containers.
11. Collect VOC samples following SW846 Method 5035 guidelines.
12. Label laboratory-specified container with time, date, and sampling identification. This information will also be recorded in the logbook.
13. Send samples to specified laboratory in accordance with the packaging and shipping protocols detailed in Section 9.3 of the QAPP.
14. Once analytical results are received from the laboratory, review results and compare to EPA Region 9 PRGs for residential soils. The PRGs are provided in Appendix E.
15. Provide EPA with a summary report of the findings.

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7.0 CONTAMINATED SOIL STOCKPILE SAMPLING

7.1 RUN-OFF ASSESSMENT

Contaminated soil removed from individual properties at the OLS is stored and managed at the contaminated soil stockpile awaiting off-site disposal. These stockpiles must have run on and run off controls to prevent potentially contaminated soils from migrating off site; be managed to prevent wind dispersion; and are subjected to inspection, monitoring, and release response requirements. Initial and final assessments of the soils around the pad upon which the stockpile is situated will be conducted to assure that adequate run on and run off practices are being maintained.

7.1.1 Initial and Final Assessments

Before stockpile accumulation begins each year, and after the stockpile has been cleared the final time, the area around the pad will be assessed for the presence of lead. At a minimum the assessment will consist of composite samples collected at each of the four cardinal directions around the pad and in areas of potential surface water run off.

1. Obtain supplies necessary to collect samples. Similar supplies used to sample the clean fill may also be used to collect samples around the pad (see Table 5.1).
2. Don the appropriate PPE.
3. Using a clean utensil, collect five discrete aliquots of near-equal amounts from each cardinal direction around the pad from a depth of 0 to 1 inch bgs. Examples of aliquot collection locations around typical soil stock pile pads are illustrated on Figure 7.1.
4. Place the aliquots in a clean disposable pie pan.
5. Remove visible debris from the aliquots placed in the clean pie pan as it will impact the accuracy of the XRF reading. Debris that must be removed include grass, leaves, concrete, rock, wood chips, roots, and trash.
6. Homogenize the sample using the clean utensil used to collect the aliquots. Homogenization entails breaking up the sample into small pieces and thoroughly mixing all aliquots to form a composite sample After homogenization, the composite sample should consist primarily of soil pieces less than ¼-inch in size.
7. Continue to mix the sample and inspect for and remove debris. Repeat this step until all debris is removed.
8. Transfer the composite soil sample to the laboratory-specified container.
9. Label laboratory-specified container with time, date, and sampling identification. Note: This information will also need to be added to the logbook as well as the field sheets.
10. Send samples to specified laboratory in accordance with the proper shipping procedures (for example: custody seals and COC documentation).
11. Review the data.
12. Provide EPA with the data.

The sample locations will be documented in a logbook along with a site sketch that documents the specific aliquot locations.
If lead levels in excess of 400 mg/kg are identified by the laboratory, EPA will be notified immediately.

7.2 DISPOSAL ASSESSMENT FOR CONTAMINATED SOIL STOCKPILES

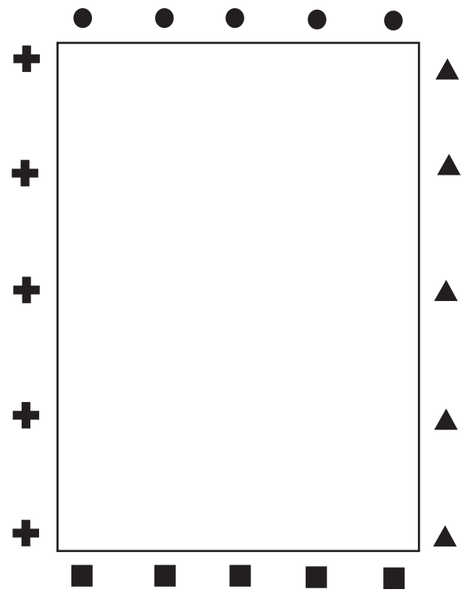
The contaminated soil stockpiles will be sampled before disposal in accordance with the requirements specified by the selected disposal facility. For example, the Loess Hills Landfill in Malvern, Iowa, which has received contaminated soil from the OLS requires one (1) representative sample for every one thousand (1,000) tons to characterize soil for disposal. Different disposal facilities or methods may require different sampling protocols based upon the facilities RCRA permits. All sampling must be done in accordance with the individual disposal facility permit. Representative sampling will be conducted based on a judgmental sampling approach consisting of composite samples. Each composite sample will be composed of at least 20 aliquots. Ten of the aliquots will be collected from a depth equal to one third to two-thirds of the height of the stockpile.

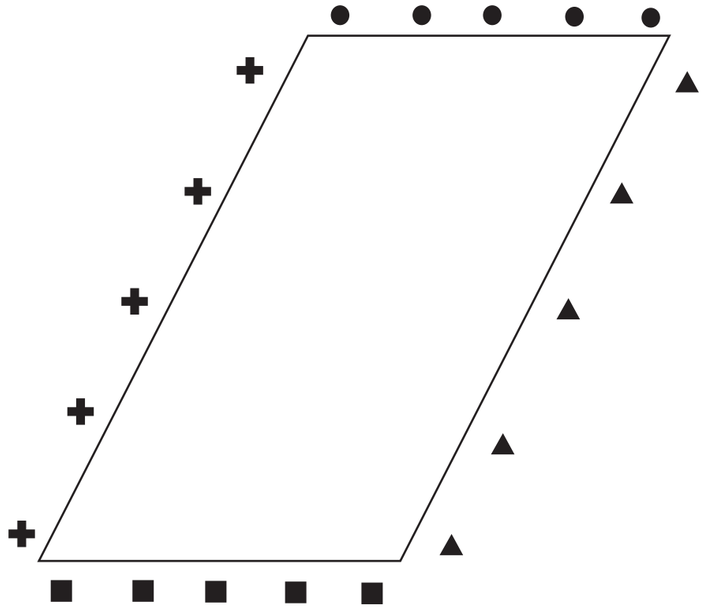
At a minimum the composite soil samples will be analyzed for lead in accordance with EPA Toxicity Characteristic Leaching Procedure (TCLP) method 1311/6010B and total lead. Table 9.4 details sample containers, preservation requirements and holding times for the above sampling method.

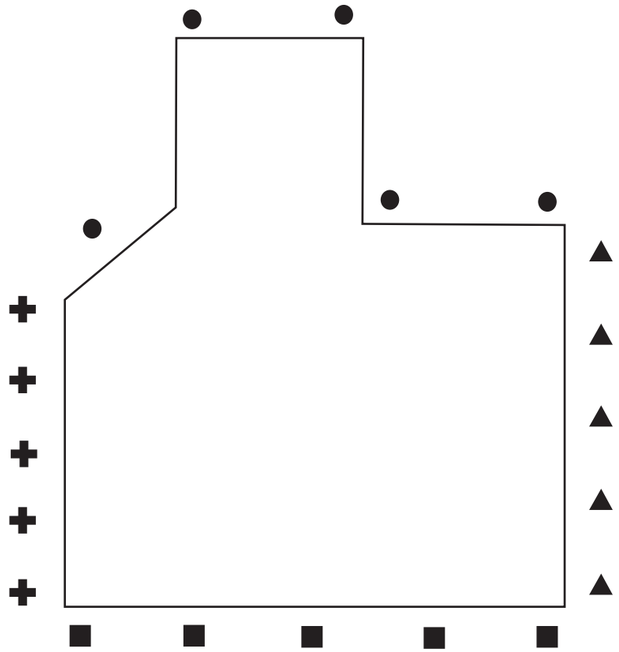
FIGURE

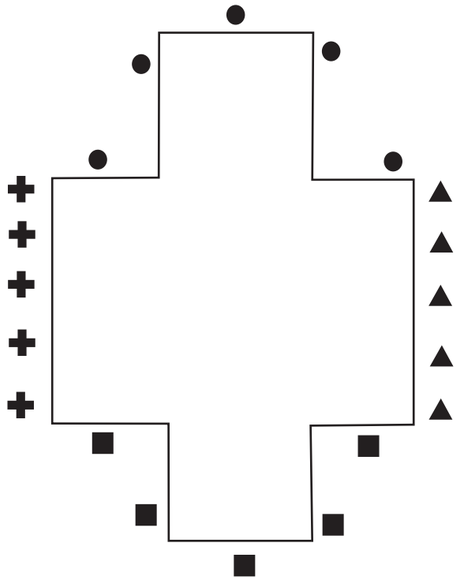
Figure 7.1
Contaminated Stock Pile Run-Off Aliquot Location Examples

U.S. EPA Region 7









Legend

- 1st Sample Group
- 2nd Sample Group
- 3rd Sample Group
- 4th Sample Group
- Concrete/Asphalt Pad

X:/EPA009/Omaha_Lead SAP/
Contamintated_Stock_Pile_Runoff_Aliquot.cdr
Created: 09/04/2007
Project: EPA009-018-03-03

HGL HydroGeoLogic, Inc

PART 2: QUALITY ASSURANCE PROJECT PLAN

This QAPP (Part 2 of the SAP) is organized in accordance with EPA *Requirements for Quality Assurance Project Plans for Environmental Data Operations*, EPA QA, Interim Final, March 2001 (EPA, 2001). Section 8.0 presents overall project management and organization as well as introductory information. Section 9.0 provides guidance for measurement and data acquisition. Section 10.0 details assessment and oversight aspects of the project. Section 11.0 describes data validation and usability criteria. Section 12.0 lists references used in generating this document.

8.0 PROJECT MANAGEMENT

This section covers the basic area of project management, including the project organization, background and purpose, project description, quality objectives and criteria, special training, and documentation and records.

8.1 PROJECT ORGANIZATION

Organization and responsibilities specific to the OLS investigation are discussed in this section. The Remediation Contractor will provide the necessary technical staff, facilities, equipment, materials, and services necessary to perform all the work described in their contract.

8.1.1 EPA Management Organization

The responsibilities of the EPA personnel involved with the Superfund Remedial Program are outlined below.

8.1.1.1 EPA Project Managers

The EPA project managers (for example: Remedial Project Managers [RPMs]) will serve as the project manager for the site activities. The EPA project managers will determine project requirements and ensure that the general scope of work necessary to accomplish the project is provided on the project task order (TO) or procurement request (PR) form and/or otherwise communicated to the contractors. The EPA project managers will help resolve problems and provide details when necessary to help contractor develop and/or select options for the technical approach and methods to be employed for a project and to develop sampling strategies. The EPA project managers will review work plans and cost estimates, and make recommendations to their branch chief for any approval/modifications. The EPA project managers will perform project oversight by conducting document reviews, audits, site visits, or field oversight activities. The EPA project managers will also provide periodic updates to EPA management and/or to EPA Region 7 personnel concerning project status/progress as required.

The EPA project managers will oversee all elements associated with the project and will coordinate field activities and other site-related operations with the contractor's project manager.

The contractor's project manager will be responsible for gathering background information pertaining to the site, obtaining equipment necessary to implement field work, acquiring non-sampling data required to complete the investigation, arranging site access, planning and

implementing all field activities, and preparing reports. The contractor's project manager will ensure that acquisition of non-sampling data and sampling-related information will be thoroughly documented (i.e., in logbooks, telephone conversation records, sample field sheets, etc.), in accordance with EPA Region 7 SOPs and/or contractor SOPs.

8.1.1.2 EPA Regional Quality Assurance Manager

The EPA regional quality assurance manager is required to review and approve this SAP for the OLS and provide general guidance and/or specific instructions to ensure that the SAP is in compliance with EPA guidance documents and policy. Once the SAP is revised to meet the standard requirements, it can be coordinated for approval by the EPA regional quality assurance manager or their designated representatives.

8.1.1.3 EPA Superfund Branch Chief

The EPA Superfund Branch chief will provide overall program management and is the primary decision maker in cooperation with the EPA Region 7 program and/or project managers/coordinators. The branch chief or their designated representative will provide the contractor with the general project scope and objectives and request contractors provide work plans. The branch chief(s) or an appropriate designee will approve recommendations from EPA project managers for project work plans and budgets, direct modifications/revisions if required, and ensure that the proper level of management authorization is obtained (normally by signature as the EPA authorizing official), in order to properly issue a task order (TO) or procurement request (PR) forms to approve the project work plan and associated costs. Copies of TO or PR forms, work plans (including cost proposals and SAPs) will be provided to the appropriate EPA Region 7 personnel upon request.

8.1.2 Contractor Management Organization

The contractor staff responsibilities associated with the OLS are described below.

8.1.2.1 Contractor Quality Assurance Manager

The contractor's Quality Assurance (QA) manager for the contract/site-specific project is responsible for monitoring the quality of technical documents generated by the contractor and its subcontractor(s). They will provide direction and guidance to contractor personnel and, through subcontractor QA/project manager(s), to subcontractor personnel performing activities under the contract. The contractor's QA/project manager will maintain a comprehensive quality program based on this SAP and will issue recommendations about quality to technical staff and management within the contractor's organization. Specific QA/project manager responsibilities include the following:

- Meeting regularly with the contractor's contract administrator to review, discuss, and resolve any quality issues and concerns.
- Reviewing, approving, and/or providing guidance to contractor project managers and/or technical staff for developing plans.
- Interacting with EPA representatives to evaluate the acceptability and qualifications of laboratory and technical subcontractors.

- Conducting field and laboratory audits, identifying nonconformance situations resulting from audits or other quality assurance/quality control (QA/QC) review activities and notifying the appropriate EPA personnel, contractor's project manager(s), the contractor's contract administrator and/or regional office manager, and/or subcontractor personnel.
- Providing recommendations and orders for corrective action for all aspects of work that do not meet program standards.
- Facilitating QA problem identification and resolution at both the project- and contract-levels.
- Managing and overseeing all aspects of laboratory procurement and management, data management, data validation, and document generation and review/revision.

8.1.2.2 Contractor's Contract Administrator

The contractor's contract administrator will serve as the primary EPA point of contact for all activities under the particular EPA Environmental Services contract. The contractor's contract administrator is ultimately responsible for all field data collection and reporting activities performed in accordance with the QAPP and should ensure that contractor's project managers are qualified and provided adequate staff and equipment support to achieve the project requirements. Specific responsibilities of the contractor's contract administrator will include, but may not be limited to the following:

- Receiving, acknowledging, and implementing all TO or PR forms and the resulting approved work plans and other project requirements.
- Designating a project manager for each TO or PR.
- Ensuring work plans (including scheduling of work) are submitted for approval by EPA for each TO or PR and for the proper implementation of those approved work plans.
- Providing overall supervision and administrative support to the projects manager including providing all the support staff, facilities, administrative capabilities, clerical support and all other resources needed to ensure the successful and efficient accomplishment of TOs or PRs issued and/or project assigned under the contract.
- Reporting and correcting all problems encountered in performing work pursuant to TOs or PRs or in the administration of the contract whether noted by the contractor or noted by representatives of the EPA.
- Preparing and submitting all reports, data, or other deliverables required in the TO or PR forms and ensuring that all deliverables are in compliance with the QA/QC requirements described in this SAP.

8.1.2.3 Contractor's Project Manager

The contractor's project manager is responsible for implementing all activities identified in the TOs or PRs issued by EPA. The contractor's project manager have the authority to commit the resources necessary to meet the technical, financial, and scheduling objectives for the project. The contractor's project manager will report directly to the contractor's contract administrator and will serve as or provide access information for the major point of contact(s) and control(s) for project-related activities and/or issues. Specific responsibilities of contractor project manager include the following:

- Preparing project plans.

- Verifying that the contractor and/or subcontractor's project team performs contract work and generate contract-related documents and deliverables that comply with all QA requirements in this SAP and/or the performance work statement (PWS).
- Monitoring and directing field activities and verifying that appropriate field measurement, field testing, and other field procedures are followed and that appropriate QC checks are conducted.
- Working with the contractor's quality assurance manager and the contractor's contract administrator to identify QA problems and to implement effective corrective actions.

On large field investigations the contractor's project manager may be supported by a field team leader (FTL). The FTL is responsible for directing day-to-day field operations and reporting to the contractor's project manager on a daily basis. The FTL will monitor field measurement and sampling procedures to verify the requirements of the work plan documents including this SAP. The FTL will also ensure that proper chain-of-custody procedures for sample handling and shipment are utilized. Other specific responsibilities of the FTL include the following:

- Supervising staffing and mobilization activities for field work.
- Overseeing sample collection and field measurements and maintaining field logbook(s).
- Overseeing the activities of all project personnel in the field, including subcontractor personnel.
- Providing the contractor's project manager with the required planning, cost and schedule control, records documentation, and data management information related to field activities.
- Facilitating project-level QA/QC problem identification and resolution.

8.1.2.4 Contractor's Technical Staff

The contractor's technical staff will conduct field activities, gather and analyze data, and prepare various project reports and support materials. The contractor's technical staff will be required to follow procedures and requirements that are specified in TO or PR and approved work plans, QA/QC documents including this SAP and other guidance and/or instructions provided by appropriate contractor and/or EPA project/contract management personnel. The contractor's contract administrator, with a reasonable amount of assistance from contractor's project manager, is responsible for ensuring that all contractor's technical staff members assigned to a project are experienced professionals, who possess the degree of specialization and technical expertise required to effectively and efficiently perform their duties and responsibilities, necessary to complete the required work/task for all TOs or PRs /projects issued under the contract.

8.1.2.5 Team Subcontractor's Project Managers and Staff

Subcontractors may be assigned responsibility for completing all or part of TOs or PRs issued under a contract. On projects with subcontractors having primary involvement, the subcontractor's project manager(s) are responsible for the planning, scheduling, budgeting, and reporting related to subcontractor activities. On projects where subcontractors play a supporting role, the subcontractor's project manager(s) will coordinate their activities through the prime contractor's project manager. Subcontractor's project managers will provide technical review of all work conducted by their staff. They will also verify that all work is conducted in compliance

with contractor's overall quality requirements and with the quality requirements of any applicable work plans and this SAP.

8.1.2.6 Team Subcontractor's Quality Assurance Manager

For all portions of the project and data collection activities assigned to the subcontractor component of the team, the team subcontractor's quality assurance manager is responsible for ensuring that all technical services provided by the subcontractor comply with overall EPA contract QA/QC requirements and the project-specific QA requirements of any applicable work plans and this SAP. Specific QA/QC responsibilities of the team subcontractor's quality assurance manager include the following:

- Reviewing this SAP or the applicable segments of the SAP under which the subcontractor will provide technical services.
- Monitoring subcontractor performance on the project, including compliance with sample collection, field analysis requirements, sample preparation and analysis methods, sample holding times, required field QC check samples, and data validation as required.
- Maintaining project-specific records of QC data, performance evaluation results, audit comments, and data quality inquiries.
- Applying the subcontractor's QA/QC program to the work done on the project, including reviewing all deliverables before they are submitted to the contractor and verifying that they meet the requirements specified in the project work this SAP.
- Ensuring that corrective action is implemented when required/directed by appropriate representatives of the prime contractor or appropriate EPA project/contract management personnel.
- Assisting the prime contactor in resolving any QA/QC issues related to the applicable analytical and/or field laboratory's work.
- Facilitating project-level QA/QC problem identification and resolution.

8.2 BACKGROUND AND PURPOSE

Site background information for the OLS is provided in Section 2.0 of this SAP. The purpose and objectives of the work assignment are discussed in Section 1.2 of this SAP. The purpose of this QAPP is to ensure that all data collection procedures and measurements are scientifically sound and of known, acceptable, and documented quality. The QAPP is a systematic planning document which documents how environmental data collection operations are planned and implemented and the results are assessed. This QAPP also defines the specific QA and QC activities that will be applied to ensure that the environmental data are the type needed for EPA to make specific decisions concerning the OLS site.

8.3 PROJECT DESCRIPTION

The QAPP addresses field work and sample data collection that will be performed by the contractor. Field and sampling procedures are discussed in Sections 3.0 through 7.0. The contractor will ensure that the QAPP is implemented as prescribed and that the data collected confirms to stated acceptance criteria and achieves DQOs.

8.4 QUALITY OBJECTIVES AND CRITERIA FOR MEASUREMENT DATA

This section describes quality specifications at two levels: (1) at the level of the decision or study question, and (2) at the level of the measurements used to support the decision or study questions. EPA has developed the Data Quality Objectives (DQO) Process as the Agency's recommended planning process when environmental data are used to select between two alternatives or derive an estimate of contamination. EPA's DQO process is a systematic planning tool designed to ensure that the type, quantity, and quality of measurement data collected are the most appropriate for supporting decisions that will be based on that data. The DQO process will be used, either formally or informally, for all data collection activities to provide the most cost-effective use of program resources. This section describes how the contractor will apply EPA's DQO process to determine the type of data required and presents specific QA objectives for measurement data. Subsections 8.4.1 and 8.4.2 describe the DQOs and data measurement objectives, respectively.

8.4.1 Data Quality Objectives Process

The DQO Process is used to develop performance and acceptance criteria (or data quality objectives) that clarify study objective, define the appropriate type of data, and specify tolerable levels of potential decision errors that will be used as the basis for establishing the quality and quantity of data needed to support decisions. The EPA document developed in 2006, *Guidance on Systematic Planning Using the Data Quality Objectives Process*, EPA QA/G-4, provides a standard working tool for project managers and planners to develop DQO for determining the type, quantity, and quality of data needed to reach defensible decisions or make credible estimates. It replaces EPA's August 2000 document, *Guidance for the Data Quality Objectives Process,* EPA QA/G-4, that considered decision-making only.

The EPA document, *Superfund Lead-Contaminated Residential Sites Handbook*, Office of Solid Waste and Emergency Response (OSWER) 9285.7-50 (EPA, 2003a), was developed by the EPA in 2003 to promote a nationally consistent decision-making process for assessing and managing risks associated with lead-contaminated residential sites across the country.

The EPA document, *Guidance Manual for the IEUBK Model for Lead in Children*, OSWER 9285.7-15-1 (EPA, 1994b) published in 1994 has been developed to assist the user in providing appropriate input to the Integrated Exposure Uptake Biokinetic IEUBK Model for Lead. The IEUBK Model is designed to model exposure form lead in air, water, soil, dust, diet, and paint and other sources with pharmacokinetic modeling to predict blood lead levels in children 6 months to 7 years.

The EPA document, *Systematic Planning: A Case Study for Hazardous Waste Investigations,* EPA QA/CS-1 created in 2006 shows the use of the DQO Process in the form of a case study. For projects that require data collection, the contractor will follow EPA's DQO process as described in the above guidance documents.

The DQO Process is used to establish performance or acceptance criteria, which serve as the basis for designing a plan for collecting data of sufficient quality and quantity to support the goals of a study. The DQO Process consists of seven iterative steps that are documented in Figure 8.1 and described

below. While the interaction of these steps is portrayed in Figure 8.1 in a sequential fashion, the iterative nature of the DQO Process allows one or more of these steps to be revisited as more information on the problem is obtained.

Each step of the DQO Process defines criteria that will be used to establish the final data collection design. The first five steps are primarily focused on identifying qualitative criteria, such as:

- the nature of the problem that has initiated the study and a conceptual model of the environmental hazard to be investigated;
- the decisions or estimates that need to be made and the order of priority for resolving them;
- the type of data needed; and
- an analytic approach or decision rule that defines the logic for how the data will be used to draw conclusions from the study findings.

The sixth step establishes acceptable quantitative criteria on the quality and quantity of the data to be collected, relative to the ultimate use of the data. These criteria are known as performance or acceptance criteria, or DQOs. For decision problems, the DQOs are typically expressed as tolerable limits on the probability or chance (risk) of the collected data leading you to making an erroneous decision. For estimation problems, the DQOs are typically expressed in terms of acceptable uncertainty (e.g., width of an uncertainty band or interval) associated with a point estimate at a desired level of statistical confidence.

In the seventh step of the DQO Process, a data collection design is developed that will generate data meeting the quantitative and qualitative criteria specified at the end of Step 6. A data collection design specifies the type, number, location, and physical quantity of samples and data, as well as the QA and QC activities that will ensure that sampling design and measurement errors are managed sufficiently to meet the performance or acceptance criteria specified in the DQOs. The outputs of the DQO Process are used to develop a QA Project Plan and for performing Data Quality Assessment.

All seven steps of the DQO process may not be applicable to all environmental data collection activities. Examples include activities where specific decisions cannot be identified or studies that are exploratory in nature. In these situations, the contractor will use the steps of the DQO process that are applicable to help plan the data collection effort.

The DQO process is not complete without a final evaluation, after sample collection and analysis has been completed, of whether DQOs were met. This evaluation, called data quality assessment (DQA), is described in Section 11.0 of this SAP.

Each of the above steps is discussed in detail in the following sections.

8.4.1.1 Step 1: State the Problem

The purpose of this step is to describe the problem to be studied so that the focus of the study will be unambiguous.

The problem to be solved is the removal of pre-selected zones of surface soil from residential properties at the OLS in order to remediate lead concentrations to below the action level of 400 mg/kg established for bare soils in 40 CFR 765.65.

8.4.1.2 Step 2: Identify the Decision

The primary focus of this effort is to determine the depth of soil removal necessary to protect residents from lead contamination. Secondary focuses of the analytical program include determining the waste disposal status of soil wastes, determining that backfill soils and sod meet project specifications, and using air samples to determine that project activities do not pose a threat to the health of site workers and occupants or to the environment. Specifically, the contractor must address the following questions:

- After removal of soil to depth between 1 and 12 inches, is the concentration of lead greater than 400 mg/kg in the soil at the bottom of the excavation zone? Note: Gardens must be excavated until reaching a residual soil lead concentration of less than 400 mg/kg at depths of 24 inches or less, or until reaching a residual soil lead level of less than 1,200 mg/kg at depths greater than 24 inches.
- After removal of soil to a depth greater than 12 inches, is the concentration of lead above 1,200 mg/kg in the soil at the bottom of the excavation zone? (This question applies only if the answer to Question #1 is "Yes".)
- Does the soil used for backfill and restoration meet the criteria established in the PWS?
- Does the sod used for restoration meet the criteria established in the PWS?
- Is the excavated waste soil acceptable for disposal as a non-hazardous waste?
- Is the run-off management from the contaminated soil stock pile protective of human health and the environment?
- Is the management of the particulate concentrations in air during soil excavation, soil handling, and soil dumping protective of human health and the environment?

8.4.1.3 Step 3: Identify the Inputs to the Decision

The purpose of this step is to identify the information that needs to be obtained and the measurements that need to be taken to resolve the decision statements. Based on the investigation questions, the following information is required:

- Analytical data from the soil samples (as required) collected at the site.
- Analytical data from the clean fill and sod sampling.
- Analytical data from the contaminated soil stockpile and the associated runoff assessment.
- Analytical data from the air sampling program.
- Documentation of proper field methods and sample collection procedures.

The quantification of data quality will be evaluated based on the analytical data collected by the contractor.

8.4.1.4 Step 4: Define the Boundaries of the Investigation

This step defines the spatial boundaries of the investigation. The OLS is located in the Omaha Metropolitan area. The total area of the site is approximately 27 square miles, which includes

approximately 40,000 residences and more than 125,000 residents. The specific residences to be remediated (and the zones at the properties) have already been identified by the EPA.

8.4.1.5 Step 5: Develop a Decision Rule

Only analytical data that have been reviewed and identified as acceptable, in accordance with this SAP, may be used to support a decision to continue or discontinue excavation.

Specific decisions will be based on the following rules.

1. *After removal of soil, is the concentration of lead greater than 400 mg/kg in the soil at the bottom of the excavation zone (guidelines differ for garden zones)?* This decision will be defined by the on-site results of five-point composite samples collected from soils at the bottom of the excavation zone and any associated off-site confirmation results (four point composite if from a drip zone). The presence of lead above 400 mg/kg at the bottom of any excavation zone will trigger additional excavation. If the concentration of lead at the bottom of an excavation zone is below 400 mg/kg after the initial removal, remediation will be considered complete in that zone. Section 3.0 describes this decision making process in detail.
2. *After removal of 12 inches of soil, is the concentration of lead above 1,200 mg/kg in the soil at the bottom of the excavation zone?* This decision will be defined by the on-site results of five-point composite samples collected from soils at the bottom of the excavation zone and any associated off-site confirmation results (four point composite if from a drip zone). The presence of lead above 1,200 mg/kg at the bottom of any excavation zone will trigger additional excavation until the concentration of lead at the bottom of the excavation is less than 1,200 mg/kg. If the concentration of lead at the bottom of the excavation zone is below 1,200 mg/kg after excavation, remediation will be considered complete in that zone.
3. *Does the soil used for backfill and restoration meet the criteria established in this SAP and/or the PWS?* The results of composite samples collected from the clean backfill source material will define this decision. The soil used for backfill must meet the acceptance criteria established in this SAP and/or the PWS. Soils that do not meet these criteria will not be accepted for use as clean fill.
4. *Does the sod used for restoration meet the criteria established in this SAP and/or the PWS?* The results of composite samples collected from the sod will define this decision. The sod used must meet the acceptance criteria established in this SAP and/or the PWS. Sod that does not meet these criteria will not be accepted for use.
5. *Is the excavated waste soil acceptable for disposal as a non-hazardous waste?* The results of the 20 aliquot composite sample collected from the waste soil piles, as discussed in Section 7.2, will define this decision. If the TCLP lead concentrations in the samples are below the selected landfill's permit requirements, the waste soil will be considered acceptable for disposal as a non-hazardous waste.
6. *Is the run-off management from the soil waste stockpile protective of human health and the environment?* The results of the samples collected from the area around the contaminated soil stockpile will define this decision. If the lead level results are in excess of 400 mg/kg, EPA will be notified within 24 hours and the appropriate action deemed as deemed by the EPA will be carried out as instructed.

7. *Is the management of the particulate concentrations in air during soil excavation, soil handling, and soil dumping protective of human health and the environment?* The air sampling results from the personal, property and contaminated stockpile sampling, as discussed in Section 4.0, will define this decision. If air concentrations of particulates are above the criteria established in the Site Safety and Health Plan (SSHP), the engineering controls discussed in the SSHP must be implemented.

8.4.1.6 Step 6: Specify Tolerable Limits on Decision Errors

The decision-maker's tolerable limits on decision errors, which are used to establish performance goals for the data collection design, are specified in this step. Decision makers are interested in knowing the true value of the constituent concentrations. Since analytical data can only estimate these values, decisions that are based on measurement data could be in error (decision error). There are two reasons why the decision maker may not know the true value of the constituent concentration, these are:

1. Concentrations may vary over time and space. Limited sampling may miss some features of this natural variation because it is usually impossible or impractical to measure every point of a population. Sampling design error occurs when the sampling design is unable to capture the complete extent of natural variability that exists in the true state of the environment.
2. Analytical methods and instruments are never absolutely perfect; hence a measurement can only estimate the true value of an environmental sample. Measurement error refers to a combination of random and systematic errors that inevitably arise during the various steps to the measurement process.

The combination of sampling design and measurement error is the total study error. Since it is impossible to completely eliminate total study error, basing decisions on sample concentrations may lead to a decision error. The probability of decision error is controlled by adopting a scientific approach in which the data are used to select between one condition (the null hypothesis) and another (the alternative hypothesis). The null hypothesis is presumed to be true in the absence of evidence to the contrary. The statements below describe the null hypotheses for this project.

1. Subsurface soil concentrations of lead will be greater than 400 mg/kg at the bottom of each excavation zone after the initial excavation (<12 inches).
2. Subsurface soil concentrations of lead will be greater than 1,200 mg/kg at the bottom of each excavation zone after 12 inches have been excavated.
3. Stockpiled waste soils will be determined to be hazardous waste.
4. Stockpiled waste soils will contaminate the area surrounding the waste pile pad.
5. Clean backfill soils will be unsuitable for use at remediated properties.
6. Sod will be unsuitable for use at remediated properties.
7. Soil removal and stockpiling activities will cause air particulate concentrations to exceed allowable levels.

Decision errors may occur through two scenarios. A false positive or "Type I" decision error would be to conclude that the null hypothesis is true, when in fact, it is not. The consequences of

this decision error would be to incur unnecessary expense to remediate contamination that does not exist; to dispose of non-hazardous material as hazardous waste; to refuse acceptable backfill material and/or sod; and to implement engineering controls for dust suppression when such controls are not needed.

A false rejection or "Type II" decision error would be to conclude that the null hypothesis is false, when in fact it is true. In that case, the error would be to assume that a measured concentration is not greater than the appropriate screening level when, in fact, it is. The consequences of this decision error would be to not remediate the full extent of contamination; dispose of hazardous material as non-hazardous waste; to accept unsuitable backfill material and/or sod; and to fail to implement engineering controls for dust suppression when such controls are needed.

Neither type of error is desirable; however, for this project a Type II error is less acceptable (worse case) than a Type I error because a Type II error could result in elevated human health risk whereas, a Type I error could result in spending additional funding for further remediation of a clean site. The closer the reported concentration is to the remediation goal, the higher the probability that an incorrect decision will be made and, therefore, there is a "gray region" surrounding the action level that should be recognized during data evaluation. This decision making process is discussed in detail in Section 3.
Both types of decision errors are limited by the decision rules and the requirement that decisions be based only on data that have been accepted through the data review process. It should be noted that the primary objective, to remediate surface soils at pre-selected properties, has been determined prior to remediation activities. Consequently, performance of remediation activities will successfully reduce risk to property residents even if a false rejection error is made after the first round of removal.

8.4.1.7 Step 7: Optimize the Design for Obtaining Data

Data from each phase of work at the OLS will be evaluated to determine whether changes in the sampling or analytical methodology are warranted. If it is decided that changes are warranted, the changes will be incorporated into this SAP by an addendum (see Appendix A), as appropriate.

8.4.2 Data Quality Objectives and Criteria for Measurement Data Under the Remedial Program

The overall quality assurance objective for the remedial investigations is to develop and implement procedures for field sampling, COC, laboratory analysis, and reporting that provide technically and legally defensible results to be used to delineate the nature and extent of contamination, evaluate contaminant migration, assess ecological and human health risk, and support the remedial decision-making process.

The remedial program process is to conduct investigations (i.e., Remedial Investigations) for gathering data for the baseline risk assessment for human health and the ecology, and to determine the extent of contamination. The sampling activities that are emphasized in this SAP are the collection of soil and air analysis.

The purpose of soil sampling is to determine if the soils present a threat to human health and the environment due to the presence of lead, and identify the extent that this contamination may impact remedial decision-making. The primary objectives of soil sampling are to: (1) characterize the nature and extent of contamination deposited by historic lead smelting/refining activities in soils downgradient or downwind from known waste sources (2) provide information to allow the risks to human health from exposure to contamination in these areas to be evaluated; (3) determine the chemical stressors that may affect vegetation establishment and/or risk to other ecological receptors; (4) determine whether the level of soil contamination qualifies for remedial action; (5) determine the soil lead levels at the base of the excavation after soil removal; and (6) collect data to support the IEUBK Model (EPA, 1994b and EPA, 2002b).

The purpose of air sampling is to document worker exposure, determine if surrounding properties are being impacted by excavation activities, and assess waste stock pile management practices.

Soil samples will be collected from residential yards, public areas, alleys, street easements, and road rights-of-way. Characterization samples will be discrete samples of surface soils taken at 0 to 1 inches. This depth is necessary to evaluate both the surface horizons for human health and ecological receptors, and the subsurface root-zone to determine the limitations and potential toxicity to plants and soil organisms. The samples should also be properly sieved to determine the metals concentration in the fine fraction of the surface soils. Composites should consist of aliquots collected from the same depth.

8.4.3 Quality Assurance Objectives for Measurement Data

The project data quality objective is to provide valid data of known and documented quality to determine the levels of lead contamination for comparison to benchmarks. Quality assurance objectives are usually discussed in terms of accuracy, precision, sensitivity, completeness, representativeness and comparability. Sample collection and field measurement activities will be performed based on SOPs discussed throughout Section 9.0 and described in Section 3.0. Analytical results for laboratory blanks, duplicates and QC samples, as well as field blanks and field duplicates will be evaluated to determine bias and representativeness.

The overall QA objective is to develop and implement procedures for field sampling, chain-of-custody, laboratory analysis, and data reporting that will provide results that will facilitate sound decision-making to protect human health and the environment, support regulatory findings, and that are legally defensible in a court of law. Specific procedures for sampling, chain-of-custody, laboratory instrument calibration, laboratory analysis, reporting of data, internal QC, audits, preventive maintenance of field equipment, and corrective action are described in other sections of this SAP. The purpose of this section is to address the level of QC effort and the specific QA objectives for sensitivity, accuracy, precision, representativeness, completeness, and comparability of data.

All analytical data will be evaluated for compliance with QC limits. Typically, when analytical data do not meet the QC limits, corrective action might be initiated and the data might be qualified or rejected. Corrective action may include stopping the analysis; examining instrument performance, sample preparation, and analysis information; recalibrating instruments; re-

preparing and reanalyzing samples; and informing the contractor's contract administrator, contractor's quality assurance manager, and the contractor's project manager of the problem.

The following subsections address the level of QC effort and general objectives for accuracy, precision, sensitivity, completeness, representativeness and comparability of data.

8.4.3.1 Sensitivity

Sensitivity is based on the minimum concentration that a substance can be measured and reported with 99 percent confidence that the concentration is greater than zero. This is generally expressed in the form of the MDL or quantitation limit for the analytical method selected. The equation used to calculate MDL is presented in Section 9.0.

The lowest concentration that can be reliably achieved within the specified limits of precision and accuracy during routine operating conditions is termed MQL. The MQL is generally 3 to 5 times greater than the MDL. Pertaining solely to non-FPRXF data, the sample quantitation limit (SQL) is the quantity based on sample dilution where the MQL is multiplied by a dilution factor. If the SQL is higher than the MQL for any analysis resulting from causes other than high analyte concentrations, the project manager will discuss corrective actions with the laboratory manager and quality control officer.

8.4.3.2 Precision

Precision is a measure of the variability of a measurement system. Precision is typically estimated by means of duplicate and replicate measurements and is expressed in terms of RPD. Equations for calculating RPD are presented in Section 9.0 of this SAP. For field sampling, precision is increased by following SOPs and by collecting all samples using the same sampling procedures. Field QC samples that are collected to measure precision include field duplicate samples (i.e., transport and field handling bias) and include collocated samples (i.e., sampling and measurement precision). Field measurement precision is monitored by taking replicate measurements. Field measurement precision is increased through proper operation and maintenance of field equipment.

Precision for laboratory analyses will be measured by collecting and analyzing the following types of samples: field split samples, matrix spike/ matrix spike duplicate (MS/MSD) samples for organic and inorganic analyses, matrix duplicate samples (also known as laboratory batch duplicate samples) for inorganic analyses, and laboratory control samples (LCS).

A RPD goal of $^{+}$/- 25 percent (for example: 75% to 125%) will be used for both field and lab analyses.

8.4.3.3 Accuracy

Accuracy is the degree of agreement between an observed value and an accepted reference value. Accuracy is typically expressed as percent recovery (%R) from spiked samples or bias with respect to a reference standard. The use of spiked samples permits a constant check on method

accuracy and provides an indication of the degree of matrix effect. Equations to calculate accuracy in terms of %R are presented in Section 9.0 of this Sitewide SAP.

In the laboratory, a minimum of one known reference standard or LCS should be analyzed for every 20 samples. Overall analytical accuracy should be assessed on a batch-specific basis by evaluating the %R for each analyte in the LCS against the established QC limits. Acceptable measurement accuracy is also dependent on the sample matrix. The laboratory's internally generated %R QC criteria for MS/MSDs should be used to assess the potential for matrix interferences. Acceptable QC limits for %R are 75 percent to 125 percent for LCSs, method-defined for surrogates, and laboratory-defined for MS/MSDs. Chemical analytical data will be validated for accuracy using surrogates, MS/MSDs, and LCSs, as applicable.

Accuracy for field sampling will be increased by establishing a sound sampling strategy and following appropriate SOPs. The SOPs followed will be documented in the QAPP addendum (see Appendix A). The field QC samples are collected to measure accuracy include trip blanks, field blanks, and equipment rinsate blanks. In general, the accuracy of field measurements will be increased by following appropriate SOPs and through proper calibration and maintenance of equipment. QC measures used to monitor the accuracy of field measurements include checking instrument responses against calibration standards.

Accuracy for laboratory analyses will be assessed by collecting and analyzing the following types of QC samples: MS/MSD samples for organic analyses, MS/MSD and matrix duplicate samples for inorganic analyses, and laboratory QC check samples. Additional volumes for MS/MSD samples and matrix duplicate samples are collected in the field. Other QC check samples used to assess accuracy are prepared in the laboratory. These laboratory check samples may include blank spikes, surrogate spikes, method blanks, reagent blanks, instrument blanks, calibration blanks, laboratory control samples, standard reference materials, and independent check standards.

8.4.3.4 Representativeness

Representativeness expresses the degree to which sample data accurately and precisely represent (a) a characteristic of a population, (b) parameter variations at a sampling point, and/or (c) an environmental condition. Representativeness is a qualitative parameter that is most concerned with the proper design of the sampling plan and the absence of cross-contamination. Good representativeness should be achieved through:

1. Careful, informed selection of sampling sites.
2. Selection of testing parameters and methods that adequately define and characterize the extent of possible contamination and meet the required parameter reporting limits.
3. Proper gathering and handling of samples to avoid interference and prevent contamination and loss.
4. Collection of a sufficient number of samples to allow characterization.

Representativeness is a consideration that should be employed during all sample location and collection efforts and should be assessed qualitatively by reviewing field procedures and reviewing actual sampling locations versus planned locations.

8.4.3.5 Comparability

This QA parameter is qualitative in signifying the confidence with which one data set can be compared with another. The sample data should be comparable to other measurement data for similar samples and sampling conditions. This parameter is achieved through standard sample collection techniques, analyses, and reporting the analytical results in appropriate units.

Generally, comparability will be attained by achieving the QA objectives for sensitivity, accuracy, precision, completeness, and representativeness given in this QAPP. Following field and laboratory procedures consistently for individual investigations will also achieve comparability of data. EPA-approved standard field procedures such as those discussed in Section 9.0 of this QAPP will be used to the extent possible. EPA-approved laboratory methods such as those listed in the contract laboratory program (CLP) statements of work (SOW) and in SW-846 will be used to increase the comparability of laboratory analytical data generated under this contract.

8.4.3.6 Completeness

Completeness is a measure of sample collection usability and whether the data quality has been met. Completeness is a measure of the amount of valid data obtained from a measurement system compared to the total number of measurements necessary to achieve a specified level of confidence in decision-making. Completeness of sample collection is the ratio of the samples actually collected to the number of samples planned to be collected. The typical goal for most sample collection events is 95 percent. The completeness of usable data is the ratio of data points that are not rejected to the total number of data points. The completeness of quality data is the ratio of data points that are qualified to the total number of data points. The goals for these components are 95 percent (usable) and 80 percent (quality). The EPA project manager, in consultation with the Superfund branch chief, will determine if the completeness goals have been met for the field and lab data. If changes to the QAPP are necessary based on site-specific conditions, these will be documented in a QAPP revision for review and approval.

Following completion of analytical testing, the percent completeness will be calculated according to the equation presented in Section 9 of this QAPP.

8.5 DATA CATEGORIES

The DQOs for the activities performed at the site should ensure that environmental data obtained meet the needs of the study and can be used with confidence to support specific decisions (both administrative and regulatory). DQOs specify the quality of data required from a particular activity to support specific decisions. Specific DQOs from the list of those outlined under this QAPP will be identified and documented in accordance with *Guidance on Systematic Planning using the Data Quality Objectives Process*, EPA QA/G-4, published by EPA in 2006 (EPA, 2006a).

8.5.1 Superfund Data Categories

Two Superfund data categories have been established, which are referred to as: 1) Screening data with or without definitive confirmation results; and 2) Definitive data. These categories segregate environmentally related measurement data into two groups, which are based primarily on increasing levels of confidence in the precision and accuracy of the analytical results. Screening data without definitive confirmation results are considered to be data of unknown quality and are preliminary in nature. Screening data with definitive confirmation results comprise data of known quality that are quantitatively "verified" and for which the analyte identification is "definitively" confirmed. Definitive data include all measurements that are performed through analyte-specific EPA-approved methodologies that definitively identify and quantify the analyte of interest. Screening results will be used to select the type and location of analytical samples, including those that would be used for definitive confirmation. The data quality available from current field screening technologies is acceptable for this purpose.

For the site activities, either of the data categories may be used to determine the necessity for further action. The appropriate category that will be used will be determined by the EPA project manager and will be dependent on the specific activity and required data use.

8.5.2 Screening Data With/Without Definitive Confirmation Results

The screening category is a broad classification that includes measurements that can be non-quantitative to semi-quantitative, or involve only probable identification of a compound class. This category will be appropriate for data collection activities that involve rapid, non-rigorous measurement or analytical procedures and limited QA/QC requirements. The screening methods will be used to make quick assessments of the types and levels of pollutants. Screening will often be employed during preliminary site characterization and/or delineation of the extent of contamination across the site. However, screening level data is not acceptable for RA activities at the OLS. The use of screening techniques will generally be confined to sites where the types of contamination are either known or suspected and/or where additional data are needed to expand on existing information.

8.5.3 Definitive Data

The most exhaustive category is definitive data, which is appropriate when rigorous, EPA-approved methods of analysis and comprehensive QA/QC procedures are necessary. Definitive data are analyte-specific, with confirmation of analyte identities and concentrations. Data may be generated at the site or at an off-site location, as long as the QA/QC requirements are satisfied. For the data to be definitive, either analytical or total measurement error must be determined.

8.6 SPECIAL TRAINING REQUIREMENTS

All of the contractor's personnel performing field activities at the OLS must have successfully completed an initial 40-hour hazardous waste operations training course and, thereafter, an 8-hour annual refresher course. This training must comply with OSHA regulations found in 29 CFR 1920.120(e). All of the contractor's personnel serving in a supervisory role (like the field team

leader) must have an additional 8 hours of site supervisor training to comply with OSHA 29 CFR 1910.120(e)(4).

The personnel responsible for sampling and other field activities must have adequate experience to perform the tasks assigned to them. All field personnel will read and familiarize themselves with all pertinent documents, including this QAPP. Field personnel will be cognizant of the importance and level of QC that must be maintained in order to produce the most accurate information possible. In order to ensure that all project activities are performed in accordance with SOPs, good practices and safety requirements, proper training of all project personnel must be maintained and documented. Field work cannot be performed that meets the required levels of quality and safety unless all project personnel are properly trained and are experienced in performing their job functions. Familiarization with air sampling equipment and FPXRFs (including an understanding of basic radiation safety) is necessary.

All employees' training documentation will be maintained at the contractor's home office. During field activities, copies of all relevant training documentation will be available on site.

Specialized training or certification related to environmental data collection might be required if (1) specifically called for in a TO or PR, and (2) identified as necessary by a contractor in responding to a TO or PR. In these situations, the contractor will address training and certification needs in the QAPP addendum. The QAPP addendum will identify contractor personnel that meet the special training or certification requirements; provide documentation of the training or certification; and describe how these personnel will be assigned to the project. If contractor personnel do not meet special training or certification requirements, the QAPP addendum will briefly describe how the necessary skills will be acquired and applied to the project.

8.7 DOCUMENTATION AND RECORDS

This section describes the requirements for data reporting that are expected of contractor field personnel and laboratories that submit field and laboratory measurement data. It is the responsibility of the regional quality assurance manager to ensure that the latest version of the approved SAP is used. Requirements for data validation reports, data quality assessment reports, or other QC reports that are prepared or compiled by contractors are not covered here but are described in subsequent sections.

This SAP provides data reporting requirements for each physical or chemical field and laboratory method that is conducted during the investigation. Data reporting requirements for each field and laboratory method will depend on the DQOs and on the intended uses of the resulting data. Reporting requirements must be clearly specified as part of any request for analytical services and are closely linked to data validation requirements. For example, for most inorganic analytical methods, and for metals in particular, no adequate degree of data validation can be performed without the raw data. This SAP specifies the data that must be reported such that (1) data validation requirements can be satisfied, and (2) attainment of DQOs can be verified.

8.7.1 Property Files

Record keeping and tracking of all information and forms accumulated for each property is a key element for the OLS. This documentation may be needed in case of property owner disputes or legal action over removal and restoration activities. Each individual property file will contain (at a minimum) the following:

- Property sketch, which will be provided by EPA
- Signed Access Agreement form (including any arrangements negotiated with property owner)
- Sampling data and results including any confirmation sampling
- Any photographs or video taken of the property during the excavation, restoration or sampling activities
- Signed Property Owner Satisfaction Surveys
- Documentation of Property Closeout.

8.7.2 Field Logbooks and Photographic Documentation

A field logbook (prepared by the contractor's project manager, subcontractor's project manager, or field team leader) will be maintained to record all pertinent activities associated with the sampling event. The observations and data will be recorded with waterproof ink and kept in a bound, weatherproof field logbook with consecutively numbered pages. Specific sampling information will be recorded on Field Sampling Data Sheets (an example is shown in Appendix F). Each entry into the field logbook will record the following information:

- Names of personnel present during sampling activities.
- Date, time and weather conditions.
- Equipment calibration.
- Analyses performed in the field and in fixed laboratories.
- QA/QC samples collected.
- Photo log with the number (according to the roll and frame count) or file name if digital camera is used, time and a detailed description of each photo taken to record site conditions during the sampling event.

Changes or deletions in the field logbook or sample collection field sheets will be lined out with a single strike mark, initialed and dated, and remain legible. Sufficient information will be recorded to allow the sampling event to be reconstructed without relying on the collector's memory. Each day, the person making entries in the field logbook, will sign each page with recorded information, at the end of the day. Anyone making entries in another person's field book will sign and date those entries.

Daily quality control reports (DQCRs) will be completed for each day of sampling activity by the contractor or subcontractor to supplement the information recorded in the field logbook. The DQCRs will be signed and dated by individuals making entries. A copy of the respective daily calibration logbook pages(s) will be attached to each day's DQCR. An example of DQCR is included in Appendix G. The DQCR will be provided to the EPA Project Manager.

8.7.3 Laboratory Documentation

Site-specific task assignments and/or discussions during scope-of-work/sampling strategy meetings will allow EPA to specify the format and content for the data package as well as the desired reporting format. The types of data deliverables that may be required include the following:

- A case narrative, including a statement of samples received, a description of any deviations from the specified analytical method, explanations of data qualifiers applied to the data, and any other significant problems encountered during analysis. The narrative will describe all QC nonconformance experienced during sample analysis, along with the corrective actions taken.
- A table that cross-references field and laboratory sample numbers.
- The chain-of-custody forms pertaining to each sample delivery group or sample batch analyzed.
- A laboratory report showing traceability to the sample analyzed and containing the following information: project identification; field sample number; laboratory sample number; sample matrix description; dates and times of sample collection, receipt at the laboratory, sample preparation, and analysis; analytical method description and reference citation; individual parameter results with concentration units (including second column results or second detector results, or other confirmatory results, where appropriate); quantitation limits achieved; and dilution or concentration factors.
- The data summary forms and QC summary forms for sample results, surrogate results, blank results, field QC sample results, MS/MSD results, MS results, initial and continuing calibration results, confirmatory results, LCS results, and other QC sample results.
- The laboratory control charts.
- The raw data such as chromatograms, peak areas, retention times for gas chromatography (GC) and high performance liquid chromatography (HPLC) analyses, mass spectra for GC/MS analyses, and laboratory bench sheets.
- The MDL and instrument detection limit results.

Additional data deliverables may also be required depending on DQOs or the particular field and laboratory methods of concern.

Contractor project managers, in conjunction with the contractor's quality assurance manager, have the primary responsibility for defining project-specific data reporting requirements. These requirements, the turnaround time for receipt of the data deliverables specified, and any project-specific requirements for retention of samples and laboratory records, should be clearly defined in requests for analytical services. Subcontractor's laboratory quality assurance managers are responsible for ensuring that all laboratory data reporting requirements in the QAPP are met.

The contractor will retain all project documents for a time period specified by EPA in the contract or until EPA requests transfer or disposition of the documents.

8.7.4 Chain-of-Custody Documentation

All samples collected for shipment to a fixed laboratory will be tracked from the time the samples are collected until laboratory data are issued. Information on the custody, handling, transfer, and transport of samples to the off-site laboratory will be recorded on a COC form as shown in Appendix H. The sampler will be responsible for filling out the COC form. The sampler will sign the COC when relinquishing the samples to anyone else.

A COC form will be completed daily for each set of samples collected, and will contain the following information:

- Sampler's signature and affiliation
- Project name
- Sample identification numbers
- Date and time of collection
- Sample type
- Analyses requested
- Number, size and type of containers
- Preservation method
- Signature of persons relinquishing custody, including date, and time
- Signature of persons accepting custody, including date and time
- Method of shipment

The above elements are included in the latest version of EPA Region 7 SOP Nos. 2420.4C "Field Chain of Custody for Environmental Samples" and 2420.5D "Identification, Documentation and Tracking of Samples". Laboratory QA/QC records and sample results will be included in the required report. All COC forms will be kept in the EPA individual site files (hard copy) and made available for the public file. All public record files are subject to the EPA Records Retention Plan as outlined in the EPA Quality Management Plan (QMP).

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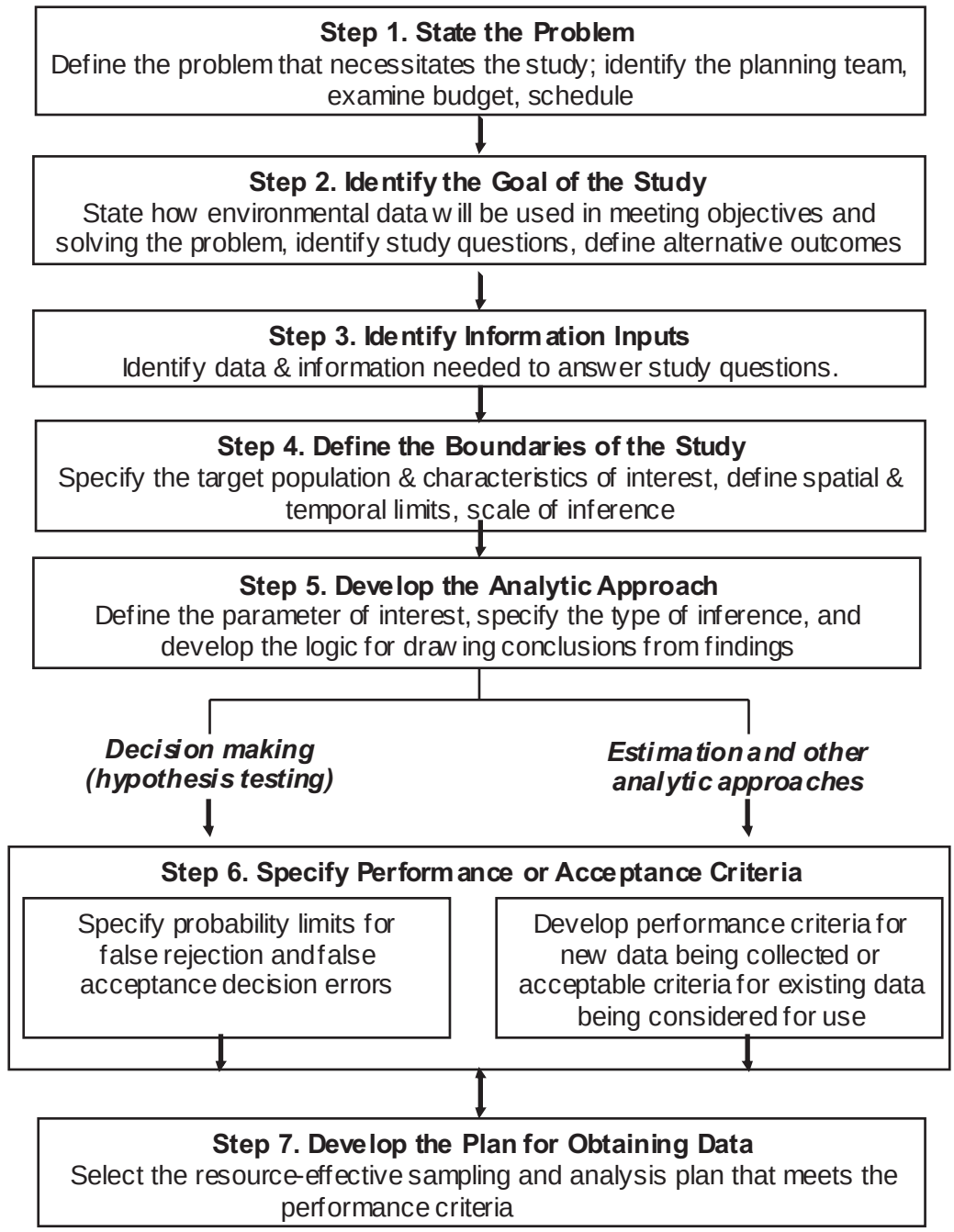
FIGURE



X:/EPA009/Omaha_Lead SAP/
Data_Quality_Objective_Process.cdr
Created: 09/04/2007
Project: EPA009-018-03-03

HydroGeoLogic, Inc

Figure 8.1
Data Quality Objective Process

9.0 MEASUREMENT AND DATA ACQUISITION

This section of the QAPP includes the ten QAPP elements required by EPA QA/R-5 (EPA, 2001) to address all aspects of data generation and acquisition. These QAPP elements ensure that appropriate methods for sampling, analysis, measurement and analysis, data collection or generation, data handling, and QC are identified and followed. The ten QAPP elements related to measurement and data acquisition are:

- Sampling process design (Section 9.1)
- Sampling methods requirements (Section 9.2)
- Sample handling and custody requirements (Section 9.3)
- Analytical methods requirements (Section 9.4)
- Quality control requirements (Section 9.5)
- Inspection and equipment testing, inspection, and maintenance requirements (Section 9.6)
- Instrument and equipment calibration and frequency (Section 9.7)
- Inspection and acceptance requirements for supplies and consumables (Section 9.8)
- Non-direct measurements (data acquisition) requirements (Section 9.9)
- Data management requirements (Section 9.10)

9.1 SAMPLE PROCESS DESIGN

The goal is the removal of pre-selected zones of surface soil from residential properties at the OLS in order to remediate lead concentrations to below the action level of 400 mg/kg in the upper 12 inches and less than 400 mg/kg at depths greater than 12 inches established for soils in the May 13, 2009 Final Record of Decision for the Omaha Lead Site. The FSP (Part I of this SAP) describes the field activities. The total number of samples that need to be collected to achieve this goal is not known, but is based on the number of residences requiring soil remediation and the depth to which removal must proceed. A summary of the samples required is listed in Table 9.1.

9.2 SAMPLING METHOD REQUIREMENTS

Sampling methods, equipment, containers, preservatives, holding times, and overall field management that the remediation contractor will follow are described below.

9.2.1 General Sampling Methods

To the extent possible, the Contractor will rely on EPA-approved methods for sample collection and field measurements. EPA-approved sampling methods that are selected for use for the OLS site will be referenced in the QAPP addendum. Guidance documents containing EPA-approved sampling SOPs include the following:

- OSWER Publication 9360.4-02. January 1991. Compendium of ERT Soil Sampling and Surface Geophysics Procedures. EPA/540/P-91/006. Interim Final (EPA, 1991a).
- OSWER Publication 9360.4-05. May 1992. Compendium of ERT Air Sampling Procedures. PB92-963406 (EPA, 1992a).
- OSWER Publication 9360.4-07. January 1991. Compendium of ERT Waste Sampling Procedures. EPA/540/P-91/008 (EPA, 1991b).
- OSWER Directive 9360.4-10. December 1995. Superfund Program Representative Sampling Guidance Volume 1: Soil. EPA/540-R-95/141 (EPA, 1995a).

- OSWER Directive 9360.4-04. May 1992. Compendium of ERT Field Analytical Procedures (EPA, 1992b).
- OSWER Publication 9285.7-50. August 2003. Superfund Lead-Contaminated Residential Sites Handbook (EPA, 2003a).
- Environmental Protection Agency. Pollution Prevention and Toxics. March 1995. Residential Sampling for Lead: Protocols for Dust and Soil Sampling. EPA 747-R-95-001 (EPA, 1995b). Note: A copy of this SOP is not contained in Appendix B. A copy could not be obtained.
- Portable XRF Analyzer, EPA Region 7. SOP No. 4231.1707.
- Soil Sampling at Lead-Contaminated Residential Sites. EPA Region 7. SOP No. 4230.19A (EPA, 2007b).
- Waste Pile Sampling. EPA Region 7. SOP 4231.2017 (EPA, 1994d).
- X-MET™ 880 Field Portable x-Ray Fluorescence Operating Procedure. EPA Region 7. SOP No. 4232.1707 (EPA, 1994e).
- Spectrace 9000 Field Portable X-Ray Fluorscence Operation Procedure. EPA Region 7. SOP No. 4232.1713 (EPA, 1995c).
- Protocols for the Region 7 Lead-Contaminated Residential Yard Soil Cleanup Actions Procedures and Sequencing. EPA Region 7. SOP No. 4220.03A (EPA, 2007a).

9.2.2 Sampling Equipment, Preparation and Decontamination

Sampling equipment required for the field program (including environmental sampling, health and safety monitoring, equipment and personal decontamination, and general field operations) are presented in the FSP portion of this SAP.

Field preparatory activities include review of this SAP and pertinent SOPs, procurement of field equipment, laboratory coordination, confirmation of site access, as well as a field planning meeting attended by field personnel and QA staff before each day's sampling.

All equipment used to collect and prepare samples will be new or decontaminated prior to sampling by washing with a soap solution (such as Alconox), rinsing the washed equipment with potable water, and rinsing the equipment with distilled water. Disposable equipment should be used whenever possible to reduce decontamination efforts.

9.2.3 Sample Methods, Containers, Preservation and Holding Times and Quantitation Limits

Samples collected will consist of clean backfill samples, sod samples, soil waste stockpile samples and rinsate samples. As discussed in Section 3.3 of the FSP, confirmation soil samples for cleanup activities will not be collected; however, laboratory confirmation soil sampling may resume if deemed appropriate by the EPA. All sample collection procedures are outlined in Sections 3.0 through 7.0 of the FSP.

QC samples will be collected, handled, and shipped in accordance with these procedures. A summary of EPA extraction, digestion and analytical procedures are given in Tables 9.2 and 9.3. Sample containers, preservation and holding time requirements required for each sample from the OLS are listed in Tables 9.4 through 9.11.

Sample containers will be purchased pre-cleaned and treated according to EPA Specifications and Guidance for Contaminant-Free Sample Containers (EPA, 1992c), and should not be reused. The containers should be stored in clean areas to prevent exposure to contaminants.

Holding times are storage times allowed between sample collection and sample extraction or analysis (depending on whether the holding time is an extraction or analytical holding time) when the designated preservation and storage techniques are employed. Holding times listed in the tables begin at the time of sample collection.

The quantitation limits provided in Tables 9.4 and 9.6 through 9.11 are the minimum levels that the laboratory should report analytical results without a qualifier when an analyte is detected. The laboratory can typically detect analytes at concentrations of up to an order of magnitude lower than the quantitation limits. For VOCs, when a positive detection is less than the quantitation limit, the value may be reported and qualified as an estimated concentration.

9.3 SAMPLE HANDLING AND CUSTODY REQUIREMENTS

Custody and documentation for field and laboratory work are described below, followed by a discussion of corrections to documentation.

9.3.1 Field Sample Custody and Documentation

The information contained on the sample label and the COC record must match. The purpose and description of the sample label and the COC record are discussed in the following sections. All identification and tracking procedures for samples will follow EPA Region 7 SOP 2420.5D, *Identification, Documentation, and Tracking of Samples*.

9.3.1.1 Sample Labeling and Identification

An alphanumeric coding system will uniquely identify each sample collected. The location of each sample, as well as time and date of sample collections and requested analyses, will be recorded on a field sheet completed for each sample.

9.3.1.2 Chain-of-Custody Requirements

COC procedures will follow the requirements set forth in EPA Region 7 SOP 2420.4C, *Field Chain of Custody for Environmental Samples*, March 1994. The COC record is employed as physical evidence of sample custody and control. This record system provides the means to identify, track, and monitor each individual sample from the point of collection through final data reporting. A COC must be completed for each sample shipment to the off-site laboratory and must contain the following information:

- Sampler's signature and affiliation
- Project name
- Sample identification numbers
- Date and time of collection
- Sample type
- Analyses requested
- Number, size and type of containers
- Preservation method

- Signature of persons relinquishing custody, including date, and time
- Signature of persons accepting custody, including date and time
- Method of shipment

The original COC will be shipped with the samples to the off-site laboratory. A copy of the COC will be kept for the project files. If more than one shipping container is used for a day's shipment, a separate COC record will be completed for each shipping container.

9.3.1.3 Sample Packaging and Shipping

Samples will be packaged and shipped in accordance with EPA SOP 2420.4C. The samples should be prepared for shipment as follows:

1. Complete and sign the COC and indicate on it the estimated time that the shipping container will be picked up by the courier service or if hand delivered to the laboratory, and the estimated time that the shipping container will be received by the laboratory.
2. Wrap each sample container with bubble wrap or foam and tape securely in place to protect the sample containers from breaking.
3. Place bubble wrap or foam in the bottom of the shipping container.
4. Place the samples in the shipping container.
5. Fill the remaining volume of the container with bubble wrap or foam as necessary.
6. Enclose the original COC in a sealable plastic bag and secured to the inside of the shipping container lid.
7. Seal the container closed with packaging tape.
8. Seal the shipping container with a minimum of two chain-of-custody seals affixed to the container in such a way that the container cannot be opened without breaking the custody seals.
9. Cover the seals with clear plastic tape.
10. Transport or ship to the off-site laboratory as expeditiously as possible.

Note that as long as the COC record is sealed in the shipping container and the custody seals remain intact, commercial carriers will not be required to sign the COC record.

9.3.1.4 Field Logbooks and Records

Field logbooks will be maintained by the field team. The site manager is responsible for maintenance and document control of the field logbooks.

9.3.2 Laboratory Custody Procedures and Documentation

Laboratory custody procedures should be implemented by laboratory personnel following internal laboratory procedures and requests. Upon receipt at the laboratory, each sample shipment should be inspected to assess the condition of the shipping container and the individual samples. This inspection should include measuring the temperature of the cooler (if cooling is required) to document that the temperature of the samples is within the acceptable criteria (4±2°C) and verifying sample integrity. The pH of the samples should be measured, if preserved. The enclosed COC record(s) should be cross-referenced with all of the samples in the shipment. Laboratory personnel should then sign these COC records and copies provided to the contractor should be placed in the project file. The sample custodian may continue the COC record process by assigning

a unique laboratory number to each sample on receipt. This number, if assigned, will identify the sample through all further handling. It is the laboratory's responsibility to maintain internal logbooks and records throughout sample preparation, analysis, data reporting, and disposal.

9.3.3 Corrections to and Deviations from Documentation

A single strikeout initialed and dated is required to document changes in the logbooks. The correct information should be entered in proximity to the erroneous entry. All deviations from the guiding documents will be recorded in the logbooks. Any major deviations will be documented and provided to the QC manager.

9.4 ANALYTICAL METHODS REQUIREMENTS

The laboratory QA program and analytical methods are addressed below.

9.4.1 Laboratory Quality Assurance Program

Samples collected during this project will be analyzed in accordance with standard EPA-approved analytical procedures if available. The laboratory will adhere to all applicable QC requirements stated in the method. EPA methods for lawn and garden nutrients are not available. Sampling and analysis for these parameters will adhere to the standards and QC requirements set forth by the North Central Region for soil nutrient analysis as stated in *Recommended Chemical Soil Test Procedures for the North Central Region*, revised in 1998 (NCR-13, 1998).

9.4.2 Methods

The analytical methods anticipated to be used for chemical analysis and the associated holding times are shown in Table 9.4. The lawn and garden fertility testing parameters and methods for the clean backfill are listed in Table 9.5 and are taken from *Recommended Chemical Soil Test Procedures for the North Central Region* (NCR-13, 1998).

9.5 QUALITY CONTROL REQUIREMENTS

Field, laboratory, and internal office QC requirements are discussed below.

9.5.1 Field Quality Control Requirements

Field QC samples will be collected and analyzed to assess the quality of data generated from sampling activities. These samples may include trip blanks, field blanks, equipment rinsate blanks, field duplicates, field split samples, matrix spike samples, and matrix spike duplicate samples. Field QC measurements may include field replicate measurements and checks of instrument responses against QC standards.

Trip blanks, field blanks, and equipment blanks should be free of contaminants. If contaminants are detected, the data from the environmental samples may be qualified as per data validation procedures discussed in Section 11.0.

Trip blanks are used to assess the potential for sample contamination during handling, shipment, and storage. Trip blanks will consist of VOC analysis vials filled with ASTM Type II water at

the laboratory. The trip blanks are sealed and transported to the field; kept with empty sample bottles and then with the investigative samples throughout the field effort; and returned to the laboratory for analysis with the investigative samples. Trip blanks are never opened in the field. One trip blank will be included within every shipping cooler of liquid samples to and from the field to be analyzed for VOCs to detect any cross-contamination during handling and transport.

Field blanks are samples of the same or similar matrix as the actual investigative samples that are exposed to the sampling environment or equipment at the time of sampling. They are used to assess contamination resulting from ambient conditions. Field blanks are required for liquid matrices. For aqueous samples, field blanks consist of analyte-free water such as degasified organic-free water for VOC analysis, HPLC water for SVOC analysis, and deionized or demineralized water for inorganic analyses. Field blanks are generally not required for solid matrices but may be collected on a case-by-case basis. Typically, one field blank is collected for every 10 or fewer liquid investigative samples.

Equipment rinsate blanks are collected when sampling equipment is used. These blanks assess the cleanliness of sampling equipment and the effectiveness of equipment decontamination. Equipment rinsate blanks are collected by pouring analyte-free water over surfaces of cleaned sampling equipment that contact sample media. Equipment rinsate blanks are collected after sampling equipment has been decontaminated but prior to being reused for sampling. Equipment rinsate blanks are typically collected for each type of decontaminated sampling equipment.

Field duplicate samples are independent samples collected as close as possible in space and time to the original investigative sample. Immediately following collection of the original sample, the field duplicate sample is collected using the same collection method. Care should be taken to collect the field duplicate sample as close to the location of the original sample as possible. Field duplicate samples can measure how sampling and field procedures influence the precision of an environmental measurement. They can also provide information on the heterogeneity of a sampling location. One field duplicate sample will be collected (sequentially) at a frequency of 1 for every 10 investigative samples. A minimum of one field duplicate sample should be taken, even if less than 10 samples are collected. Field duplicates will be analyzed at the fixed laboratory for the same parameter as the primary sample analyzed at the site. These results will be used to evaluate the representativeness of the sample.

Field split samples are usually a set of two or more samples taken from a larger homogenized sample. The larger sample is usually collected from a single sampling location, but can also be a composite sample. Field split samples can be sent to two or more laboratories and are used to provide comparison data between the laboratories. Regulatory agencies involved in a project may request that field split samples be collected to monitor how closely laboratories are meeting site-/project-specific QA objectives.

MS/MSD samples are typically collected for analysis by organic methods, and also often for analysis by inorganic methods. Solid MS/MSDs usually require no extra volume. Each liquid MS/MSD sample is a single sample, usually collected from a single sampling location at triple the normal sample volume. In the laboratory, MS/MSD samples and MS samples are spiked with known amounts of analytes. Analytical results of MS/MSDs are used to measure the precision and accuracy of the laboratory organic (or inorganic) analytical program and MSs are used to measure the accuracy of the inorganic analytical program. Each of these QC samples is

typically collected and analyzed at a frequency of one for every 20 investigative samples per matrix.

QC checks for field measurements will consist primarily of initial and continuing calibration checks of field equipment. When applicable, QC check standards independent of the calibration standards will be used to check equipment performance. For example, when checking the accuracy of field equipment such as pH meters, a standard buffer solution independent of the calibration standards may be used. Precision of field measurements will usually be checked by taking replicate measurements. To the extent possible, contractors will use EPA-approved field methods. If approved methods are not available, contractor SOPs will be referenced in the project work plan and/or site-/project-specific QA/QC documents. The types and frequencies of field QC measurements and the QC limits for these measurements will be specified in the project's QA/QC documents.

9.5.2 Laboratory Quality Control Requirements

The laboratory QA/QC elements including laboratory spikes and blanks will be performed in accordance with the latest versions for EPA analytical methods SOPs and EPA Region 7 SOP No. 2430.12E, "*Regional Laboratory Quality Control Policy*", or equivalent SOP supplied by the contractor. The EPA Project Manager will be responsible for verifying that copies of the referenced SOPs are available and that the SOPs are being followed by conducting periodic site visits to the field, mobile lab and fixed laboratory. When the contractor has identified the subcontractor for the mobile and fixed laboratory, copies of the laboratory's SOPs will be acquired and added as an addendum to the site-/project specific QAPP.

Rinsate water samples will require fixed laboratory confirmation. In general, fixed laboratory analysis will be performed for all critical samples needed to establish primary targets, support attribution, and/or otherwise used for site scoring.
All laboratories must adhere to a QA program that is used to monitor and control all laboratory QC activities. Each laboratory must have a written QA manual that describes the QA program in detail. The laboratory Quality Assurance Manager is responsible for ensuring that all laboratory internal QC checks are conducted according to the laboratory's QA manual and the requirements of this QAPP.

Laboratory QC procedures and requirements may include the preparation and analysis of laboratory control samples (LCS), method blanks, MS and MSD samples, surrogate spikes, and standard reference materials or independent check standards. QC checks that are most frequently required are discussed in the following sections.

9.5.2.1 Laboratory Control Samples

Laboratory control samples (LCS) are well-characterized, laboratory-generated samples that will be used to monitor the laboratory's day-to-day performance of analytical methods. LCSs can include laboratory duplicate samples, laboratory spike samples, or method blanks. The results of LCS analyses are compared to well-defined laboratory control limits to determine whether the laboratory system is in control for the particular method. If the system is not in control, corrective action is implemented. Corrective action can include stopping the analysis; examining

instrument performance or sample preparation and analysis information; and determining whether re-preparation or reanalysis is warranted.

9.5.2.2 Method Blanks

Method blanks, also known as analytical process or preparation blanks, are analyzed to assess the level of background interference or contamination that exists in the analytical system and that may lead to the reporting of elevated concentration levels or false-positive or false-negative data. One method blank is typically analyzed for every 20 samples collected. For batches smaller than 20 samples, one method blank is analyzed with every batch of samples processed.

A method blank consists of reagents specific to the analytical method that are carried through every aspect of the analytical procedure, including sample preparation, cleanup, and analysis. Results of the method blank analysis are evaluated in conjunction with other QC information to determine the acceptability of the data generated for that batch of samples. Ideally, the concentration of target analytes in the method blank should be below the method or instrument detection limit for that analyte. For some common laboratory contaminants, detection of a higher concentration may be allowed.

If the blank for any analysis is not within control limits, the source of contamination must be investigated, and appropriate corrective action must be taken and documented. Investigation includes an evaluation of the data to determine the extent and effect of the contamination on sample results. If a method blank indicates analytes above the method or instrument detection limits, an investigation should be conducted to determine whether any corrective action could eliminate an ongoing source of target analytes.

Refer to the individual analytical methods and the appropriate data validation guidance documents for detailed information regarding blank frequencies of analyses, acceptance criteria for blanks, and corrective actions for out-of-compliance blank results.

9.5.2.3 Matrix Spikes, Matrix Spike Duplicates, and Matrix Duplicates

A matrix spike (MS) is an environmental sample to which known concentrations of target analytes have been added. The MS is used to evaluate the effect of the sample matrix on the accuracy of the analysis. If the number of target analytes is large, target analytes are divided into two to three spike standard solutions. Each spike standard solution must be alternately used. The MS, in addition to an unspiked aliquot, is taken through the entire analytical procedure, and the recovery of the analytes is calculated. Results are expressed as %R. One MS is typically analyzed for every 20 investigative samples prepared in one batch for inorganic analyses.

An MS/MSD is an environmental sample divided into two separate aliquots, each of which is spiked with known concentrations of target analytes. The two spiked aliquots, in addition to an unspiked sample aliquot, are analyzed separately, and the results are compared to determine the effects of the matrix on the precision and accuracy of the analysis. Results are expressed as relative percent difference (RPD) and %R and are compared to control limits that have been established for each analyte. If results fall outside control limits, corrective action must be performed. One MS/MSD is typically analyzed for every 20 investigative samples prepared in one batch for organic or inorganic analyses.

A matrix duplicate sample is an environmental sample divided into two separate aliquots at the laboratory. Once the sample is divided they are considered to be two separate samples. Thus, they are analyzed separately. The results are compared to determine the effects of the matrix on analytical precision. Results are expressed as RPD and are compared to control limits established for each analyte. If results fall outside control limits, corrective action must be performed. One matrix duplicate sample is typically analyzed for every 20 investigative samples prepared in one batch for inorganic analyses.

9.5.2.4 Surrogate Spikes

Surrogates are organic compounds similar to the analytes of interest in chemical behavior but that are not normally found in environmental samples. Surrogates are added to samples prior to being extracted to assess the efficiency of the extraction procedure and bias introduced by the sample matrix. Results are reported in terms of %R. Individual analytical methods may dictate sample reanalysis based on surrogate criteria.

Surrogate recoveries will primarily be used by the laboratory to assess the overall efficiency in implementing the analytical method. Obvious problems with sample preparation and analysis (such as evaporation to dryness, a leaking septum, or other problems) that can lead to poor surrogate spike recoveries must be ruled out prior to attributing low surrogate recoveries to matrix effects.

9.5.2.5 Standard Reference Materials and Independent Check Standards

Standard reference materials and independent check standards can be used to evaluate the accuracy of an analytical system. The source, traceability, certification of purity, and concentration of these materials and standards must be documented. The "true" known concentrations of standard reference materials and independent check standards is then compared to results obtained from the analytical system to evaluate the accuracy of the system.

9.5.3 Common Data Quality Indicators

This section describes how QA objectives for precision, accuracy, completeness, and sensitivity are measured, calculated, and reported. For some investigations, additional equations might also be needed (for example, equations for calculating mass balances, emission rates, and confidence ranges).

9.5.3.1 Precision

Precision of many analyses is assessed by comparing analytical results of MS/MSD sample pairs for organic and inorganic analyses, field duplicate samples, field split samples, laboratory matrix duplicate samples, and replicate measurements. If calculated from two measurements, precision is normally measured as RPD:

$$RPD = \left[\frac{2\ x\ (C_1 - C_2)}{(C_1 + C_2)} \right] \times 100$$

where: RPD = Relative percent difference
C1 = Larger of the two observed measurement values
C2 = Smaller of the two observed measurement values

For field measurements such as pH, where the absolute variation is more appropriate, precision is often reported as the absolute range (D) of duplicate measurements:

$$D = \left| m_1 - m_2 \right|$$

where: D = Absolute range
m1 = First measurement value
m2 = Second measurement value

9.5.3.2 Accuracy

The accuracy of many analytical methods is assessed using the results of MS/MSD samples for organic and inorganic analyses, MS samples for inorganic analyses, surrogate spike samples, laboratory control samples, standard reference materials, independent check standards, and measurements of instrument responses against zero and span gases. For measurements where spikes are used, %R is often calculated as a measure of accuracy:

$$\%R = 100 \times \left[\frac{(S - U)}{C_{sa}} \right]$$

where: %R = Percent recovery
S = Measured concentration in spiked aliquot
U = Measured concentration in unspiked aliquot (usually equals zero for surrogate spikes)
Csa = Actual concentration of spike added

When a standard reference material (SRM) is used, the following equation is often used to calculate %R:

$$\%R = 100 \times \left[\frac{C_m}{C_{srm}} \right]$$

where: %R = Percent recovery
Cm = Measured concentration of SRM
Csrm = Actual concentration of SRM

For field measurements such as pH, accuracy is often expressed in terms of bias (B) and is calculated as follows:

$$B = M - A$$

where: M = Measured value of SRM
A = Actual value of SRM

9.5.3.3 Completeness

Completeness is defined as follows for most measurements:

$$\%C = 100 \times \left[\frac{V}{n} \right]$$

where: %C = Percent completeness
V = Actual number of measurements judged usable
n = Total number of measurements planned to achieve a specified level of confidence in decision making

9.5.3.4 Sensitivity

The achievement of MDLs depends on instrument sensitivity and matrix effects. Therefore, it is important to monitor the instrument sensitivity to ensure data quality and to ensure that analyses meet the QA objectives for sensitivity stated in the project QA/QC documents. Method sensitivity is typically evaluated in terms of the MDL and is defined as follows for many measurements:

$$MDL = t(n-1,1-x=0.99)s$$

where: MDL = Method detection limit
s = Standard deviation of the replicate analyses
t(n – 1, 1 – x = 0.99) = t-value for a one-sided 99 percent confidence level and a standard deviation estimate with n-1 degrees of freedom
n = Number of measurements
x = Statistical significance level

9.5.4 Internal Quality Control Checks

Internal QC checks will be conducted throughout the project to evaluate the performance of the project team during data generation. All internal QC will be conducted in accordance with standard laboratory procedures and method requirements. All laboratory QC samples will be analyzed using confirmation samples from the OLS, if possible (i.e., laboratory duplicates, MS/MSDs).

All project deliverables will receive internal technical and QA reviews prior to being issued to EPA. In addition, any comments received from EPA will be incorporated into the project document before it is made final. Completed review forms will be maintained in the project file.

9.6 EQUIPMENT MAINTENANCE PROCEDURES

This section outlines testing, inspection, and maintenance procedures for field equipment and instruments and for laboratory instruments. This section includes general requirements applicable to both field and laboratory equipment as well as field-specific and laboratory-specific requirements.

9.6.1 General Requirements

General requirements for testing, inspection, and maintenance procedures are as follows. Testing, inspection, and maintenance methods and frequency will be based on the type of instrument; its stability characteristics; the required accuracy, sensitivity, and precision; it's intended use, considering project-specific DQOs; manufacturer's recommendations; and other conditions affecting measurement or operational control. For most instruments, preventive maintenance is performed according to procedures and schedules recommended in (1) the instrument manufacturer's literature or operating manual or (2) SOPs associated with particular applications of the instrument.

In some cases, testing, inspection, and maintenance procedures and schedules may differ from the manufacturer's specifications or SOPs. This can occur when a field instrument is used to make critical measurements or when the analytical methods associated with a laboratory instrument require more frequent testing, inspection, and maintenance.

Any field or laboratory instrument that is in disrepair or is out of calibration must be segregated, clearly marked, and not used until it is repaired and recalibrated. If an instrument is repeatedly broken or out of calibration, the instrument must be replaced or repaired so that it is in good working order. When the condition of an instrument is suspect, unscheduled testing, inspection, and maintenance must always be conducted. Adherence to these field and laboratory preventive maintenance practices is subject to verification during performance and system audits.

9.6.2 Field Equipment and Instruments

The operator is responsible for (1) thoroughly checking and calibrating each instrument before shipment to the field and (2) including instructions for field calibration, testing, and maintenance of each instrument shipped. Once in the field, the operator will also be responsible for testing, inspection, and maintenance of field instruments and equipment.

Field equipment and instruments will be inspected for damage after arrival in the field. Damaged equipment and instruments will be immediately replaced or repaired. Battery-operated equipment is checked to assure full operating capacity; if needed, batteries are recharged or replaced. Critical spare parts such as tape, paper, batteries, and battery chargers will be kept on site to minimize equipment downtime. Backup instruments, equipment, and additional spare parts will be available on site or within a 1-day shipping period, when possible, to avoid delays in the field schedule.

Following use, field equipment will be properly decontaminated prior to being returned to its source. When the equipment is returned, copies of any field notes regarding equipment problems will be included so that problems are not overlooked and any necessary equipment repairs are carried out.

9.6.3 Laboratory Instruments

All laboratories conducting analyses of samples collected under the contract are required to have a preventative maintenance program covering testing, inspection, and maintenance procedures and schedule for each measurement system and required support activity. This program is

usually documented in the form of SOPs for each analytical instrument to be used. The basic requirements and components of such a program include the following:

Each laboratory will have, as a part of its QA/QC program, a routine preventive maintenance program conducted to minimize the occurrence of instrument failure and other system malfunction.

- Service and repair of instruments, equipment, tools, gauges, and so forth will be performed by an internal group of qualified personnel. Alternatively, scheduled instrument maintenance and emergency repair may be provided by manufacturers' representatives under a repair and maintenance contract.
- Instrument maintenance will be carried out by the laboratory on a regularly scheduled basis. The servicing of critical items should be scheduled to minimize the downtime of the measurement system. A list of critical spare parts for each instrument will be identified by the laboratory and requested from the manufacturer. These spare parts will be stored at the laboratory for availability and use to reduce downtime. The availability of spare parts will be monitored periodically.
- Testing, inspection, and maintenance procedures described in laboratory SOPs will be in accordance with manufacturer's specifications and with the requirements of the specific analytical methods employed.
- All maintenance and service must be documented in service logbooks to provide a history of maintenance records. A separate service logbook should be kept for each instrument. All maintenance records will be traceable to the specific instrument, equipment, tool, or gauge.
- Records produced as a result of testing, inspection, or maintenance of laboratory instruments will be maintained and filed at the laboratory. These records will be available for review by internal and external laboratory system audits under the contract.

9.7 INSTRUMENT CALIBRATION PROCEDURES AND FREQUENCY

Instruments will be calibrated according to manufacturer's specifications. Field instruments will be calibrated prior to each sampling event or as instructed by the manufacturer. Field instruments include but may not limited to the FPXRF and air sampling equipment.

This section describes the procedures for maintaining the accuracy of field equipment and laboratory instruments used for field tests and laboratory analyses. The equipment and instruments should be calibrated before each use or on a scheduled, periodic basis when not in use.

9.7.1 Field Equipment

Equipment used to collect field samples or take field measurements under the contract will be maintained and calibrated with sufficient frequency and in such a manner that the accuracy and reproducibility of results are consistent with the manufacturer's specifications and with project-specific DQOs.

The contractor field team leader is to verify that field sampling and measurement equipment is in good working condition. The manufacturer's operating manual and instructions that accompany the equipment will be consulted to ensure that all calibration procedures are followed.

Field measurements will vary according to project requirements. This SAP identifies and includes the types of field equipment to be used, calibration requirements, SOPs covering equipment calibration procedures, requirements for calibration standards, calibration frequencies, and requirements for maintaining calibration records and traceability.

9.7.2 Laboratory Equipment

All laboratory equipment used to analyze samples collected will be calibrated based upon written SOPs maintained by the laboratory. Calibration records (including the dates and times calibration and the names of the personnel performing the calibration) will be filed at the location where the analytical work is performed and maintained by the laboratory personnel performing QC activities. Calibration records will be subject to QA audits. Most laboratory work will be conducted by a subcontracted laboratory. In all cases, the laboratory subcontractor QA manager is responsible for ensuring that all laboratory instruments are calibrated in accordance with the requirements in this QAPP.

When analyses are conducted in accordance with SW-846 methods, calibration procedures and frequencies specified in the relevant method should be followed as closely as possible. The site-/project-specific QA/QC documents should provide any additional calibration requirements (such as equipment requiring calibration, calibration procedures, requirements for calibration standards and apparatus, requirements for maintaining calibration records and traceability, calibration frequency, acceptance criteria, number of calibration points, and internal or external standards) that deviate from or are not specified in the published EPA-approved method. Such deviations will be outlined in the site-/project-specific QA/QC documents or in an appendix as part of a laboratory SOP.

For analytical methods that are not EPA-approved, a complete SOP including the calibration procedures for the method will be included as an appendix to the appropriate project QA/QC document. Laboratory SOPs describing calibration procedures for such non-standard methods should include the following information:

- Detailed calibration procedure for each instrument used.
- Internal standard or external standard calibration requirements and procedures.
- Calibration requirements for confirmatory results (second column, second detector, mass spectral confirmation, and so forth).
- Frequency of calibration and continuing calibration checks.
- Number of calibration standards used, concentrations, and preparation methods.
- Traceability of calibration standards and continuing calibration check standards.
- Numerical acceptance criteria for initial calibration and continuing calibration checks.
- Corrective action procedures for situations where calibration procedures are not performed properly, or calibration acceptance criteria are not met.
- Instructions for recording calibration information and results, including what information is to be recorded and where it is recorded and stored.

9.8 ACCEPTANCE REQUIREMENTS FOR SUPPLIES

Contractor's project managers have primary responsibility for identifying the types and quantities of supplies and consumables needed. The contractor's project managers are also

responsible for determining acceptance criteria for these items. The contractor's project manager will ensure that any required certification is in place and document this in the field notebook and in the report prepared for EPA.

Supplies and consumables can be received either at a contractor office or at a site. When supplies are received at a contractor office, the contractor project manager or contractor field team leader will sort the supplies according to vendor, check packing slips against purchase orders, and inspect the condition of all supplies before the supplies are accepted for use on a project. If the supplies do not meet the acceptance criteria, deficiencies will be noted on the packing slip and purchase order. In addition, a form will be completed describing the problem and circumstances in full, and noting the purchase order number for the item. The item will then be returned to the vendor for replacement or repair.

Procedures for receiving supplies and consumables in the field are similar to those described above. Upon receipt, items will be inspected by the contractor's project manager or field team leader against the acceptance criteria. Any deficiencies or problems will be noted in the field logbook, and deficient items will be returned for immediate replacement.

9.9 NONDIRECT MEASUREMENT DATA ACQUISITION REQUIREMENTS

Nondirect measurement data could include information from previous investigations at the OLS. The acceptance criteria for such data include a review by someone other than the author. Any nondirect measurement data included in information obtained from the above-referenced sources will determine further action at the OLS site only to the extent that those data can be verified.

9.10 DATA MANAGEMENT

The following paragraphs provide general discussion and requirements for managing data under the contract for EPA. Further detail and requirements will be provided as necessary in the site-/project-specific QA/QC documents, including requirements for data recording, validation, transformation, transmittal, reduction, analysis, tracking, storage, and retrieval. The site-/project-specific QA/QC documents will also provide, as necessary, checklists and standard forms for detecting and correcting errors and preventing the loss of data during data reduction, data reporting, data encoding, and data entry.

Data for the contract will be obtained from a combination of sources, including field measurements and analyses, and subcontractor laboratories. The process of data gathering is a coordination effort and will be conducted by project staff in conjunction with all potential data producers. The data itself will be obtained from the analytical service provider, when appropriate, in the form of an electronic data deliverable in addition to the required hard copy analytical data package. The standard data management software of all analytical data to be submitted electronically by the contractor is SCRIBE. A hardcopy of the data will also be required as part of the site report. The EPA project manager will review the data to ensure accuracy prior to placing into the facility file.

Data tracking is imperative to ensure timely, cost-effective, and high-quality results. Data tracking begins with sample chain-of-custody. When the analytical services provider receives the samples into custody, the provider will send a sample acknowledgment to the contractor.

The sample acknowledgment will confirm the sample receipt, condition, and the required analyses.

Unless otherwise directed by EPA, the contractor will validate all data generated as described in Section 11 of this SAP. As a part of the data validation process, the electronic data deliverables will be reviewed against the hard copy deliverables to ensure accurate transfer of data. In addition, the hard copy will be evaluated for errors in calculation of results. As a result of the data validation, qualifiers will be placed on the data to indicate the data usability. These qualifiers will be placed into the electronic data file. Upon approval of the data set with the appropriate data qualifiers, the electronic data will be released to the project leader for reporting. A complete discussion of data validation procedures is contained in Sections 11.1 and 11.2 of this SAP.

Following data validation and release of data, the contractor project managers will use data to prepare project reports. As a part of the final report quality control review procedures, the data will be further checked by technical reviewers and a Quality Control Coordinator (QCC) to verify its accuracy in the report.

In addition to the site report, all analytical data in the form obtained from the analytical services provider will be archived with the final project file in a secure location. The secure location will house all final project files until they are transferred to EPA.

Table 9.1
Summary of Regular and Quality Control Samples

Sample Type	Minimum Number of Samples
FPXRF Soil Samples (Normal)	1 for every zone excavated
Rinsate Sample	Monthly
Air Sampling – Personal (Normal)	2 sets annually
Air Sampling – Property (Normal)	2 sets annually
Air Sampling – Contaminated Stock Pile (Normal)	2 sets annually
Air Sampling Blanks (Quality Control)	1 per sample group
Clean Backfill Samples (Normal)	1 per source
Clean Backfill Sample Duplicates (Quality Control) (5%)	1 for every 20 confirmation samples
Clean Backfill Sample Matrix Spike/Matrix Spike Duplicate (Quality Control) (5%)	1 for every 20 confirmation samples
Sod Samples (Normal)	1 per source
Sod Sample Duplicates (Quality Control) (5%)	1 for every 20 confirmation samples
Sod Sample Matrix Spike/Matrix Spike Duplicate (Quality Control) (5%)	1 for every 20 confirmation samples
Contaminated Stockpile Runoff Assessment (Normal)	4 per stockpile
Contaminated Stockpile Runoff Assessment Duplicates (Quality Control) (5%)	1 for every 20 confirmation samples
Contaminated Stockpile Runoff Assessment Matrix Spike/Matrix Spike Duplicate (Quality Control) (5%)	1 for every 20 confirmation samples
Contaminated Stockpile Disposal Samples	Variable depending on Landfill permit requirements
Contaminated Stockpile Duplicates (Quality Control) (5%)	1 for every 20 samples
Contaminated Stockpile Matrix Spike/Matrix Spike Duplicate (Quality Control) (5%)	1 for every 20 samples

Notes:
% percent
FPXRF field portable x-ray fluorescence

Table 9.2
EPA Extraction and Digestion Procedures

Method	Parameter
SW1311	Toxicity Characteristic Leaching Procedure
SW3005A	Acid digestion of water samples for metals analysis*
SW3010A	Acid digestion of aqueous samples and extracts for metals analysis*
SW3015	Microwave assisted acid digestion of aqueous samples and extracts for metals analysis*
SW3020A	Acid digestion of aqueous samples and extracts for metals analysis*
SW3050B	Acid digestion for solids, sediments, and sludges for metals analysis
SW3051	Microwave assisted acid digestion of solids, sediments, and sludges for metals analysis
SW3510C	Separatory funnel liquid-liquid extraction
SW3520C	Continuous liquid-liquid extraction
SW3550B	Ultrasonic Extraction
SW5030B	Purge and trap
SW5035	Closed-system purge and trap

Notes:
* For aqueous analyses of total recoverable metals, the entire sample is acidified on collection.

Table 9.3
EPA Analytical Procedures

Methods	Parameter
SW-846 Organic Methods	
SW8260B	Volatile organics (water and soil)
SW8270C	Semivolatile organic compounds (water and soil)
SW8081	Organochlorine Pesticides by Gas Chromatography
SW8082	Polychlorinated Biphenyls (PCBs) by Gas Chromatography
SW-846 Inorganic Methods	
SW6010B	Trace metals by ICP-AE Spectrometry (soil)
SW6020	Trace metals by ICP Mass Spectrometry (water)
SW7471A	Mercury in Solid or Semisolid Waste (soil)

Notes:
ICP-AE inductively coupled atomic emission

Table 9.4
Sampling Requirements, Analytical Methods, and Quantitation Limits for Soil, Air and Rinsate Samples Analyzed for Lead

Analyte	EPA Analytical Method	Sample Container	Preservation	Holding Time	Estimated Quantitation Limit
FPXRF Samples (Soil)					
Lead	6200	FPXRF specific sampling cup	None	6 Months	As Determined in Section 3.1.3.2
Rinsate Sample[1] (Water)					
Total Lead	SW3010A SW6020	1 x 4 oz glass jar	None	6 Months	1.0
Air Samples					
Lead	SW6010B	1 x 4 oz glass jar	None	6 Months	0.3[2]
Contaminated Soil Stockpile Samples (Soil)					
TCLP Lead[4]	SW1311 SW6010B	1 x 8 oz glass jar	None	6 Months	1.0

Notes:
The estimated quantitation limits are recommended limits listed in milligrams per kilogram. The laboratory derived estimated quantitation limits supersede these values and will be reported in the event a disparity occurs.
[1] Rinsate samples will be collected monthly to document decontamination of the sieve used to prepare the FPXRF samples.
[2]The Estimated Quatitation Limit for Lead in the air samples is in micrograms per cubic meter.
[3]The initial and final runoff assessment consists of FPXRF screening of the area surrounding the waste pile pad.
[4]At a minimum the composite samples from the contaminated soil stockpile will be analyzed for TCLP Lead.
EPA Environmental protection Agency
FPXRF Field portable X-ray fluorescence instrument
TCLP Toxicity Characteristic Leaching Procedure

Table 9.5
Sampling Methods for Clean Fill Analyzed for Lawn and Garden Fertility Parameters

Analyte	Method
Nitrate-Nitrogen	Water Extraction Cadmium Reduction or Nitrate Electrode Method
Phosphorus	Bray & Kurtz P-1 Test
Cations (Potassium, Calcium, Magnesium, Sodium)	NCR-13 Exchangeable Potassium Procedure
Sulfate-Sulfur	Monocalcium phosphate extraction Turbidimetric Procedure, IC or ICP
Boron	Hot Water Extraction
Micronutrients (Zinc, Copper, Manganese)	DTPA Extraction AA, ICP, DCP
pH	Soil Water Slurry
Exchangeable Sodium Percentage	NCR-13 Exchangeable Potassium Procedure
Soluble Salts (Soil Salinity)	Saturated Paste Method
Cation Exchange Capacity	Calculated

Notes:
All methods are taken from *Recommended Soil Chemical Test Procedures for the North Central Region*.
AA Atomic Absorption Spectrometer
DCP Direct Current Plasma Atomic Emissions Spectrometer
DTPA Diethylenetriaminepentaacetic Acid
IC Ion Chromatographic Technique
ICP Inductively Coupled Atomic Emission Spectrometer
NCR North Central Region

Table 9.6
Sampling Requirements, Analytical Methods, and Quantitation Limits for Clean Fill and Sod Samples Analyzed for RCRA Metals

Analyte	EPA Analytical Method	Bottle Requirements	Preservative	Holding Times	Estimated Quantitation Limits
Arsenic	SW6010B	250 mL wide-mouth glass jar	Cool to 4ºC	6 months	
Barium					1.0
Cadmium					1.0
Chromium					1.6
Lead					1.0
Silver					2.0
Selenium					1.0
Mercury	SW7471A			28 Days	0.1

Notes:
The estimated quantitation limits are recommended limits listed in milligrams per kilogram. The laboratory derived estimated quantitation limits supersede these values and will be reported in the event a disparity occurs.
˚C Degrees Celsius
EPA Environmental Protection Agency
RCRA Resource Conservation and Recovery Act

Table 9.7
Sampling Requirements, Analytical Methods, and Quantitation Limits for Clean Fill and Sod Samples Analyzed for Low Detection Level Volatile Organic Compounds (VOCs) in Soil by Gas Chromatography/Mass Spectrometry

Analyte	EPA Analytical Method	Bottle Requirements	Preservative	Holding Times	Estimated Quantitation Limits
LDL VOCs in Soil					
Acetone	SW8260B (collect by SW5035)	(2) 40 mL glass vial with Teflon™ lined septum	NaHSO3, Cool to 4°C	14 Days	5.0
Benzene					5.0
Bromochloromethane					5.0
Bromodichloromethane					5.0
Bromoform					5.0
Bromomethane					5.0
2-Butanone					5.0
Carbon Disulfide					5.0
Carbon Tetrachloride					5.0
Chlorobenzene					5.0
Chloroethane					5.0
Chloroform					5.0
Chloromethane					5.0
Cyclohexane					5.0
1,2-Dibromo-3-Chloropropane					5.0
Dibromochloromethane					5.0
1,2-Dibromoethane					5.0
1,2-Dichlorobenzene					5.0
1,3-Dichlorobenzene					5.0
1,4-Dichlorobenzene					5.0
Dichlorodifluoromethane					5.0
1,1-Dichloroethane					5.0
1,2-Dichloroethane					5.0
1,1-Dichloroethene					5.0
cis-1,2-Dichloroethene					5.0
trans-1,2-Dichloroethene					5.0
1,2-Dichloropropane					5.0
cis-1,3-Dichloropropene					5.0
trans-1,3-Dichloropropene					5.0
Ethylbenzene					5.0
2-Hexanone					5.0
Isopropylbenzene					5.0
Methyl Acetate					5.0
Methyl tert-butyl ether					5.0
Methylcyclohexane					5.0

Table 9.7 (continued)
Sampling Requirements, Analytical Methods, and Quantitation Limits for Clean Fill and Sod Samples Analyzed for Low Detection Level Volatile Organic Compounds (VOCs) in Soil by Gas Chromatography/Mass Spectrometry

Analyte	EPA Analytical Method	Bottle Requirements	Preservative	Holding Times	Estimated Quantitation Limits
LDL VOCs in Soil (continued)					
Methylene Chloride	SW8260B (collect by SW5035)	(2) 40 mL glass vial with Teflon™ lined septum	$NaHSO_3$, Cool to 4°C	14 Days	5.0
4-Methyl-2-Pentanone					5.0
Styrene					5.0
1,1,2,2-Tetrachloroethane					5.0
Tetrachloroethene					5.0
Toluene					5.0
1,2,3-Trichlorobenzene					5.0
1,2,4-Trichlorobenzene					5.0
1,1,1-Trichloroethane					5.0
1,1,2-Trichloroethane					5.0
Trichloroethene					5.0
Trichlorofluoromethane					5.0
1,1,2-Trichlorotrifluoroethane					5.0
Vinyl Chloride					5.0
Tert-butlybenzene					5.0
n-propylbenzene					5.0
1,2,4-trimethylbenzene					5.0
1,3,5-trimethylbenzene					5.0
Total Xylenes					5.0

Notes:
The estimated quantitation limits are recommended limits listed in micrograms per kilogram. The laboratory derived estimated quantitation limits supersede these values and will be reported in the event a disparity occurs.

°C degrees Celsius
EPA Environmental Protection Agency
mL milliliter
VOCs volatile organic compounds

Table 9.8
Sampling Requirements, Analytical Methods, and Quantitation Limits for Clean Fill and Sod Samples Analyzed for Semivolatile Organic Compounds (SVOCs) in Soil by Gas Chromatography/Mass Spectrometry

Analyte	EPA Analytical Method	Bottle Requirements	Preservative	Holding Times	Estimated Quantitation Limits
SVOCs in Soil					
1,2,4-Trichlorobenzene	SW8270C	250 mL widemouth jar with Teflon™ lined cap	Cool to 4°C	7 days until extraction. Analyze within 40 days of extraction.	330
1,2-DCB					330
1,3-DCB					330
1,4-DCB					330
2,4-DNT					330
2,6-DNT					330
2-Chloronaphthalene					330
2-Methylnaphthalene					330
2-Nitroaniline					330
3-Nitroaniline					330
3,3'-Dichlorobenzidine					330
4-Bromophenylphenyl ether					330
4-Chloroaniline					330
4-Chlorophenylphenyl ether					330
4-Nitroaniline					330
Acenaphthylene					330
Acenapthene					330
Anthracene					330
Benzo (a) anthracene					330
Benzo (a) pyrene					330
Benzo (b) fluoranthene					330
Benzo (g,h,i) perylene					330
Benzyl alcohol					330
Bis(2-chloroethoxy) methane					330
Bis (2-chlorethyl) ether					330
Bis(2-chloroisopropyl) ether					330
Bis(2-ethylhexyl) phthalate					330
Butyl benzylphthalate					330
Chrysene					330
Di-n-butylphthalate					330
Di-n-octylphthalate					330
Dibenz (a,h) anthracene					330
Dibenzofuran					330
Diethyl phthalate					330

Table 9.8 (continued)
Sampling Requirements, Analytical Methods, and Quantitation Limits for Clean Fill and Sod Samples Analyzed for Semivolatile Organic Compounds (SVOCs) in Soil by Gas Chromatography/Mass Spectrometry

Analyte	EPA Analytical Method	Bottle Requirements	Preservative	Holding Times	Estimated Quantitation Limits
SVOCs in Soil (continued)					
Dimethyl phthalate	SW8270C	250 mL widemouth jar with Teflon™ lined cap	Cool to 4°C	7 days until extraction. Analyze within 40 days of extraction.	330
Fluoranthene					330
Fluorene					330
Hexachlorobenzene					330
Hexachlorobutadiene					330
Hexachlorocyclopentadiene					330
Hexachloroethane					330
Indeno (1,2,3-cd) pyrene					330
Isophorone					330
n-Nitrosodiphenylamine					330
n-Nitrosodi-n-propylamine					330
Naphthalene					330
Nitrobenzene					330
Phenanthrene					330
Pyrene					330
2,4,5-Trichlorophenol					330
2,4,6-Trichlorophenol					330
2,4-Dichlorophenol					330
2,4-Dimethylphenol					330
2,4-Dinitrophenol					330
2-Chlorophenol					330
2-Methylphenol					330
2-Nitrophenol					330
4,6-Dinitro-2-methylphenol					330
4-Chloro-3-methylphenol					330
4-Methylphenol					330
4-Nitrophenol					330
Benzoic Acid					330
Pentachlorophenol					330
Phenol					330

Notes:
The estimated quantitation limits are recommended limits listed in micrograms per kilogram. The laboratory derived estimated quantitation limits supersede these values and will be reported in the event a disparity occurs.

°C degrees Celsius
EPA Environmental Protection Agency
LDL low detection level
mL milliliter
$NaHSO_3$ sodium bisulfate
SVOCs semivolatile organic compounds

Table 9.9
Sampling Requirements, Analytical Methods, and Quantitation Limits for Clean Fill and Sod Samples Analyzed for Pesticides in Soil by Gas Chromatography

Analyte	EPA Analytical Method	Bottle Requirements	Preservative	Holding Times	Estimated Quantitation Limits
Pesticides in Soil					
α-BHC	SW8081A	250 mL widemouth jar with Teflon™ lined cap	Cool to 4°C	14 Days	330
β-BHC					330
δ-BHC					330
γ-BHC (Lindane)					330
α-Chlordane					330
γ-Chlordane					330
4,4'-DDD					330
4,4'-DDE					330
4,4'-DDT					330
Aldrin					330
Dieldrin					330
Endosulfan I					330
Endosulfan II					330
Endosulfan Sulfate					330
Endrin					330
Endrin Aldehyde					330
Heptchlor					330
Heptachlor Epoxide					330
Hexachlorobenzene					330
Methoxychlor					330
Toxaphene					330

Notes:
The estimated quantitation limits are recommended limits listed in micrograms per kilogram. The laboratory derived estimated quantitation limits supersede these values and will be reported in the event a disparity occurs.

°C degrees Celsius
EPA Environmental Protection Agency
mL milliliter

Table 9.10
Sampling Requirements, Analytical Methods, and Quantitation Limits for Clean Fill and Sod Samples Analyzed for Polychlorinated Biphenyls (PCBs) in Soil by Gas Chromatography

Analyte	EPA Analytical Method	Bottle Requirements	Preservative	Holding Times	Estimated Quantitation Limits
PCBs in Soil					
PCB-1016	SW8082	250 mL widemouth jar with Teflon™ lined cap	Cool to 4°C	14 Days	330
PBC-1221					330
PBC-1232					330
PBC-1242					330
PBC-1248					330
PCB-1254					330
PCB-1260					330

Notes:
The estimated quantitation limits are recommended limits listed in micrograms per kilogram. The laboratory derived estimated quantitation limits supersede these values and will be reported in the event a disparity occurs.

°C	degrees Celsius	mL	milliliter
EPA	Environmental Protection Agency	PBC	Polychlorinated Biphenyls

Table 9.11
Sampling Requirements, Analytical Methods, and Quantitation Limits for Clean Fill and Sod Samples Analyzed for Polycyclic Aromatic Hydrocarbons (PAHs)

Analyte	EPA Analytical Method	Bottle Requirements	Preservative	Holding Times	Estimated Quantitation Limits
PAHs in Soil					
Acenaphthene	SW8270D	250 mL widemouth amber jar with TeflonTM lined cap	Cool to 4°C	7 days until extraction. Analyze with 40 days after extraction	330
Acenaphthylene					330
Anthracene					330
Benzo(a)anthracene					330
Benzo(a)pyrene					330
Benzo(b)fluoranthene					330
Benzo(ghi)perylene					330
Benzo(k)fluoranthene					330
Chrysene					330
Dibenzo(a,h)anthracene					330
Fluoranthene					330
Fluorene					330
Indeno(1,2,3-cd)pyrene					330
Naphthalene					330
Phenanthrene					330
Pyrene					330

Notes:
The estimated quantitation limits are recommended limits listed in micrograms per kilogram. The laboratory derived estimated quantitation limits supersede these values and will be reported in the event a disparity occurs.

°C	degrees Celsius	mL	milliliter
EPA	Environmental Protection Agency	PAH	polynuclear aromatic hydrocarbons

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10.0 ASSESSMENT AND OVERSIGHT

This section of the QAPP includes the two QAPP elements required by EPA QA/R-5 (EPA, 2001) to assess and evaluate the management of environmental data collection operations. These QAPP elements provide procedures for conducting appropriate audits and reports and implementing corrective actions as necessary to ensure that the quality of data generated by implementation of this QAPP is adequate. The two QAPP elements related to assessment and oversight are:

- Assessment and response actions (Section 10.1).
- Reports to management (Section 10.2).

10.1 ASSESSMENT AND RESPONSE ACTIONS

The EPA regional quality assurance manager and project managers will evaluate the process and quality of performance. All measured parameters will be observed to ensure that the data meets the QA/QC requirements and other site-/project-specific requirements identified in the TO or PR and/or project work plan documents. Every attempt will be made to subcontract analytical work to a National Environmental Laboratory Accreditation Program (NELAP) certified laboratory. If a non-NELAP certified commercial laboratory is used, assessment and response of the analytical phases will be in accordance with that laboratory's internal QA procedures. When a non-NELAP laboratory is used, deviations to EPA SOP 2440.5E will be documented in a QAPP addendum. The contractors/subcontractors will provide copies of the SOPs for the mobile and fixed laboratory, as part of the contractual agreements and conditions of this QAPP, before providing any actual sampling services.

The EPA project manager may periodically visit the site to observe the field activities and whether field personnel are following the project work plan with SAP and to take corrective action if necessary. This should be documented in the field report as well as the site reports.

Performance and system audits of both field and laboratory activities may be conducted to verify that sampling and analysis are performed in accordance with the procedures and requirements established in this SAP. Non-conforming items identified during an audit will be addressed by corrective action. This section addresses basic audit and corrective action requirements that apply to all work conducted by the EPA contractor.

10.1.1 Performance and System Audits

Both internal performance and system audits may be conducted on the contractor's field operations and subcontractor laboratories under the contract. Performance audits include verification that field sampling activities and measurements and laboratory analyses of performance evaluation samples are being conducted in accordance with the requirements of this SAP. System audits involve a qualitative examination of all components of an environmental data collection system, including records, personnel, and QA management activities.
This section describes the selection of audit personnel, the scope of field and laboratory audits, audit frequencies, and typical audit reports for internal audits initiated by contractor's quality

assurance manager. External performance and system audits initiated by EPA may also be conducted under the contract and would involve similar activities.

10.1.1.1 Audit Personnel

All auditors must be independent of the activities being audited. The contractor's quality assurance manager has the lead role in directing all internal audit activities during an investigation. The contractor's quality assurance manager will select the appropriate personnel to conduct each internal audit and will assign them responsibilities and deadlines for completing their audits. These personnel may include the contractor's quality assurance manager, or other independent auditors. When an audit team is required, the contractor's quality assurance manager selects a lead auditor based on relevant technical expertise and audit experience. The lead auditor is responsible for selecting and preparing the audit team; preparing an audit plan; coordinating and scheduling the audit with the project team, subcontractor, or other organization being audited; participating in the audit; coordinating the preparation and issuance of audit reports and corrective action request forms; and evaluating audit responses and resulting corrective actions.

10.1.1.2 Audit Scope of Work

Performance audits of field activities will be conducted to evaluate compliance with the requirements of this SAP. Field systems audits may include an examination of the following items:

- Sample collection records.
- Sample collection, handling, preservation, packaging, shipping, and custody records.
- Equipment operation, maintenance, and calibration records.

Laboratory performance audits include analysis of blind performance evaluation samples to assess a laboratory's ability to comply with QC control limits. Laboratory systems audits may include evaluation of the following:

- Sample log-in, identification, storage, tracking, and custody procedures.
- Sample and standards preparation procedures.
- Availability of analytical instruments.
- Analytical instrument operation, maintenance, and calibration records.
- Laboratory security procedures.
- Qualifications of analysts.
- Case file organization and data handling procedures.

10.1.1.3 Audit Frequencies

As necessary, the site-/project-specific QA/QC documents will provide a schedule of all planned audits that will be conducted during the investigation. These audits may be required by EPA or planned by the contractor's quality assurance manager. Audit frequency will depend on several factors. In selecting projects for auditing, the contractor's quality assurance manager will consider projects with a large volume of work or those on which EPA has placed a high level of importance. For laboratory audits, the contractor's quality assurance manager will focus on

laboratories performing critical measurements (as determined by DQOs) and on subcontractor laboratories performing work for the first time.

Unscheduled follow-up audits may occur if any deficiencies are discovered during an audit or review. Follow-up audits serve to ensure that all necessary corrective actions have been properly implemented to address deficiencies.

10.1.1.4 Audit Reports

Audit reports will be prepared for performance and system audits of field and laboratory activities and all laboratory performance evaluation studies that are conducted under the contract. Reports will be prepared by the lead auditor responsible for coordinating the audit. Audit reports will identify audit participants, describe the activity audited, summarize audit findings, and detail any deficiencies or deviations from protocol that were discovered during the audits, as well as any corrective actions that are proposed. Any field or laboratory analytical data that is generated during the analysis of blind performance evaluation samples must be validated. The validated data will be included with the audit report. Data validation procedures are discussed in Section 11.2.

Audit reports are distributed to the contractor's quality assurance manager, contractor administrator, contractor's project manager, and the field team leader or the laboratory subcontractor's quality assurance manager, as appropriate. The lead auditor has primary responsibility for ensuring that audits are conducted thoroughly and properly. Contractor's project managers and team field or laboratory subcontractor's quality assurance manager are responsible for implementing corrective actions that result from an audit. The contractor's quality assurance manager is responsible for verifying that recommended corrective actions have been implemented.

10.1.2 Corrective Action

Rapid and thorough correction of QA problems, through an effective corrective action program, minimizes the possibility of questionable data or documentation. The two types of corrective action are immediate and long-term. Immediate corrective actions include correcting procedures, repairing instruments that are working improperly, and correcting errors or deficiencies in documentation. Long-term corrective actions eliminate the sources of problems by correcting systematic errors in sampling and analytical procedures, replacing procedures that produce questionable results, and manipulating similar cause-and-effect relationships.

All QA problems and corrective actions applied are documented to provide a complete record of QA activities. These records assist the contract administrator management team in identifying long-term QA problems and enable application of long-term corrective actions such as personnel training, replacement of instruments, and improvement of sampling and analytical procedures.

The contractor's quality assurance manager has the authority to discontinue or limit environmental data measurements that are compromised until corrective action is complete and data quality is no longer questionable. The contractor's quality assurance manager may also

order the recollection or reanalysis of samples or remeasurement of field parameters since the last documented evidence that the measurement system was in control.

Specific corrective action procedures for sample collection and field measurements and laboratory analyses are discussed below.

10.1.2.1 Sample Collection and Field Measurements

Technical staff and project personnel involved in sample collection or field measurement activities are responsible for initiating routine corrective actions by reporting all suspected technical or QA nonconformance's and deficiencies to the contractor project manager or his/her designee. Corrective actions for sample collection and field measurements may include, but are not limited to, the following:

- Repeating measurements to check for error.
- Checking that instruments are properly adjusted for ambient conditions such as temperature.
- Checking batteries.
- Checking calibration and recalibrating equipment if necessary.
- Replacing the instrument or measurement devices.
- Collecting additional samples.
- Stopping work (if necessary).

10.1.2.2 Laboratory Analyses

Each laboratory that participates as a subcontractor is required to write a SOP summarizing procedures for initiating, developing, approving, implementing, and documenting corrective action. The existence of such a program does not exempt the laboratory from following the corrective action requirements outlined in this QAPP. When errors, deficiencies, or out-of-control situations arise, systematic corrective actions must be taken to resolve problems and restore proper functioning analytical systems. Laboratory personnel and quality assurance managers are alerted that corrective actions may be necessary if any of the following situations arise:

- Sample volumes are not sufficient to perform required analyses.
- QC data are outside the acceptable limits for precision and accuracy.
- Blanks contain contaminants above acceptable levels.
- Undesirable trends are detected in spike recoveries or in the RPD between duplicates.
- Unusual changes in detection limits arise.
- Deficiencies are detected during internal or external audits or from the results of performance evaluation samples.
- Inquiries concerning data quality are received from clients.

If sample volumes are insufficient to complete the required analyses, the laboratory will notify the contractor project manager. The contractor's project manager, contractor's quality assurance manager, and laboratory subcontractor's quality assurance manager will contact the EPA project manager to determine if additional samples need to be collected.

Laboratory corrective action procedures are often initiated at the bench level by the analyst, who reviews the preparation or extraction procedure for possible errors; checks the instrument calibration; checks the spiking levels, calibration solutions, and standards; and checks instrument sensitivity. If the problem persists or cannot be identified, the matter may be referred to the laboratory supervisor, project manager, or quality assurance manager for further investigation. Every effort must be made to determine the cause of the problem so that a permanent solution can be developed and implemented. Once a problem is resolved, full documentation of the corrective action procedure is filed with the project records.

Investigations initiated by laboratory technical or quality assurance personnel that result in corrective actions must be documented and reported to the contractor's quality assurance manager. Documentation of investigations of negative performance on performance evaluation samples and corrective actions taken will be forwarded to the appropriate certifying agencies when required.

10.2 REPORTS TO MANAGEMENT

A draft report may be required to be prepared by the EPA contractor at the completion of the field sampling effort and/or upon receipt of validated laboratory data. The report will inform the EPA project manager of the status of the project; results of performance evaluations and system audits; results of periodic data quality assessments; and significant quality assurance problems and recommended solutions.

Following review by the EPA quality assurance manager, and EPA project manager; the EPA contractor will prepare a final report to be submitted to EPA Region 7, as appropriate.

Effective management of environmental data collection operations requires timely assessment and review of measurement activities. Open communication, interaction, and feedback must also occur among all project participants, including contractor's corporate quality assurance manager, the EPA quality assurance manager or a designated representative, contractor's contract administrator, contractor's project manager, contractor's quality assurance manager, technical staff, and team subcontractors.

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11.0 DATA VALIDATION AND USABILITY

This section of the QAPP includes the three QAPP elements required by EPA QA/R-5 (EPA 2001) to ensure that data is valid and usable for its intended purpose. The three QAPP elements related to data validation and usability are:

- Data review, verification, and validation requirements (Section 11.1).
- Validation and verification methods (Section 11.2).
- Reconciliation with data user requirements (data quality objectives) (Section 11.3).

11.1 DATA REVIEW, VERIFICATION, AND VALIDATION REQUIREMENTS

Data review and verification will be performed by a qualified laboratory analyst as described in the fixed laboratory SOPs. The SOPs from the fixed laboratories will be included in a QAPP addendum (see Appendix A). The EPA project manager will be responsible for the validation and final approval of the data (including field notes) in accordance with the stated project purpose and use of the data. Any anomalies will be documented with corrective actions described and included in the site assessment report.

This section focuses on data review and reduction requirements for work conducted under the contract. Data validation and verification requirements are covered in Section 11.2.

Field personnel will record all raw data from chemical and physical field measurements in a field logbook. Contractor's project managers have primary responsibility for (1) verifying that field measurements were made correctly, (2) confirming that sample collection and handling procedures specified in the site-/project-specific QA/QC documents were followed, and (3) ensuring that all field data reduction and review procedures and requirements are followed. They are also responsible for assessing preliminary data quality and for advising the data user of any potential QA/QC problems with field data. When field data are used in a project report, data reduction methods will be fully documented in the report.

Each laboratory subcontractor will complete data reduction for chemical and physical laboratory measurements and will complete an in-house review of all laboratory analytical results. The laboratory subcontractor's quality assurance manager is responsible for ensuring that all laboratory data reduction and review procedures conform to the requirements in this QAPP. The laboratory subcontractor's quality assurance manager is also responsible for assessing data quality and for advising the contractor's quality assurance manager of possible QA/QC problems with laboratory data.

11.2 VERIFICATION AND VALIDATION METHODS

The data will be validated in accordance with the fixed laboratory SOPs. Field notes will be compared for consistency and the EPA Project Manager will document any anomalies. The EPA Project Manager will inspect the data to provide final review and approval to ensure that the data meets the sampling requirements.

All data that are used to support activities under the contract must be valid for their intended purposes. This section outlines the basic data validation procedures that will be followed for all

field and laboratory measurements. The following subsections identify personnel responsible for data validation and the general data validation process and EPA data validation guidance that will be followed.

11.2.1 Data Validation Responsibilities

The contractor's quality assurance manager, or his/her designee, is responsible for validating all field and laboratory data collected. The laboratory subcontractor will also validate all laboratory data according to their own specific procedures before submitting the data to the contractor. As requested, the contractor will validate all laboratory subcontractor data, unless approved by the EPA project manager. Data validation will be completed by one or more experienced data reviewers. When applicable, QAPP addendums will include the names and qualifications of data reviewers assigned to the project.

11.2.2 Data Validation Procedures

The validity of a set of data is determined by comparing the data with a predetermined set of QC limits. Contractor data reviewers will conduct a systematic review of the data for compliance with established QC limits (for example, sensitivity, precision, and accuracy) based on spike, duplicate, and blank sample results provided by the laboratory. The data review will identify any out-of-control data points or omissions. Contractor data reviewers will evaluate laboratory data for compliance with the following:

- Method and project-specific analytical service requests.
- Holding times.
- Initial and continuing calibration acceptance criteria.
- Field, trip, and method blank acceptance criteria.
- Surrogate recovery.
- Field duplicates, MS/MSD and matrix duplicate acceptance criteria.
- Other laboratory QC criteria specified by the method and the project-specific analytical service request.
- Compound identification and quantitation.
- Overall assessment of data in accordance with project-specific objectives.

The contractor will follow the most current EPA guidelines for completing data validation:

- "Data Validation Standard Operating Procedures for Contract Laboratory Program Routine Analytical Services. Revision 2.1." U.S. EPA Region 7. Science and Ecosystem Support Division. Office of Quality Assurance. (EPA, 1999a).
- "U.S. EPA Contract Laboratory Program National Functional Guidelines for Organic Data Review." Publication 9240.1-05A-P (EPA, 1999b).
- "U.S. EPA Contract Laboratory Program National Functional Guidelines for Inorganic Data Review." Publication 9240.1-05-01. (EPA, 1994a).
- General procedures in the EPA guidelines will be modified as necessary to fit the specific analytical method used to produce the data.

In all cases, data validation requirements will depend on DQO levels, region-specific guidelines, reporting requirements and data deliverables requested from the laboratory.

11.3 RECONCILIATION WITH USER REQUIREMENTS (DATA QUALITY OBJECTIVES)

The EPA project manager will evaluate the data for completeness goal needed to be achieved. If the data quality indicators do not meet the project requirements outlined in the QAPP, the data may be discarded and re-sampling may occur. In case of a failure, the project team will evaluate the cause. If the failure is due to laboratory procedures or equipment, necessary corrective measures will be taken by the EPA quality assurance manager and EPA project manager. If failure is associated with sampling, field procedures will be re-evaluated with any changes documented by the EPA project manager and included in a site report.

The primary purpose of a QA system is to define a process for collecting data that is of known quality, is scientifically valid, is legally defensible, and fully supports any decisions that will be based on the data. To achieve this purpose, this QAPP requires that DQOs be fully defined in Section 9.5. All other parts of the QA system must then be planned and implemented in a manner consistent with the DQOs. The QA system components that follow directly from the DQOs include documentation and reporting requirements (Section 8.7); sample network design and sampling methods (Sections 9.1 and 9.2); analytical methods requirements (Section 9.4); QC requirements (Section 9.5); and data reduction, validation, and reporting methods (Sections 11.1 and 11.2).

Once environmental data have been collected, reviewed, and validated, the data must be further evaluated to determine whether the DQOs identified in the SAP and the QAPP addendum (if necessary) have been met. Contractor will follow EPA's data quality assessment (DQA) process to verify that the type, quality, and quantity of data collected are appropriate for their intended use. The DQA process involves first verifying that the assumptions under which the data collection design and DQOs were developed have been met, or taking appropriate corrective action if the assumptions have not been met. The DQA process then evaluates how well the data collected support the decision that must be made so that scientifically valid and meaningful conclusions can be drawn from the data. To the extent possible, the contractor will follow DQA methods and procedures outlined in EPA documents Data Quality Assessment: A Reviewer's Guide QA/G-9R (EPA, 2006b) and Data Quality Assessment: Statistical Tools for Practitioners QA/G-9S (EPA, 2006c).

If data quality indicators do not meet the project's requirements as outlined in the QAPP, the data may be discarded, and re-sampling and/or re-analysis may be required upon approval of EPA.

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APPENDIX A

REGION 7 QUALITY ASSURANCE PROJECT PLAN FORM

Region 7 Superfund Program
Quality Assurance Project Plan Form

Project Information:

Site Name:			**City:**	**State:**
EPA Project Manager:			**START Project Manager:**	
Approved By:			**Prepared For:** EPA Region 7 Superfund Division	
Title:	START Project Manager	**Date:**		
Approved By:				
Title:	START Project Manager	**Date:**	**Prepared By:**	
Approved By:			**Date:**	
Title:	START QA Manager	**Date:**		
Approved By:				
Title:	EPA Project Manager	**Date:**	**START Contractor:**	
Approved By:			**START Project Number:**	
Title:	EPA Superfund QA Coordinator	**Date:**		

1.0 Project Management:

1.1 Distribution List

EPA--Region 7: _____ EPA Project Manager

_____ Robert Dona, Superfund QA Coordinator

START: _____ Start Project Manager

1.2 Project/Task Organization

1.3 Problem Definition/Background:

Description: This site-specific Quality Assurance Project Plan form is prepared as an addendum to the **Generic Quality Assurance Project Plan for Superfund Integrated Assessment and Targeted Brownfields Assessment Program (Updated: July 2005),** and contains site-specific data quality objectives for the sampling activities described herein.

- ☐ Description attached.
- ☐ Description in referenced report: _____ **Title** _____ **Date**

1.4 Project/Task Description:

- ☐ CERCLA PA
- ☐ CERCLA SI
- ☐ Brownfields Assessment
- ☐ Other (description attached):
- ☐ Pre-CERCLIS Site Screening
- ☐ Removal Assessment

Schedule: Field work is scheduled for _____

- ☐ Description in referenced report: _____ **Title** _____ **Date**

1.5 Quality Objectives and Criteria for Measurement Data:

a. Accuracy:	☐ Identified in attached table.
b. Precision:	☐ Identified in attached table
c. Representativeness:	☐ Identified in attached table.
d. Completeness:	☐ Identified in attached table.
e. Comparability:	☐ Identified in attached table.

Other Description:

*A completeness goal of 100 percent has been established for this project. However, if the completeness goal is not met, EPA may still be able to make site Decisions based on any or all of the remaining validated data.

1.6 Special Training/Certification Requirements:

☐ OSHA 1910
☐ Special Equipment/Instrument Operator (describe below):
☐ Other (describe below):

1.7 Documentation and Records:

☐ Field Sheets ☐ Site Log ☐ Trip Report ☐ Site Maps ☐ Video
☐ Chain of Custody ☐ Health and Safety Plan ☐ Letter Report ☐ Photos

☐ Sample documentation will follow EPA Region 7 SOP 2420.5C.

☐ Other: Analytical information will be handled according to procedures identified in Table 2.

2.0 Measurement and Data Acquisition:

2.1 Sampling Process Design:

☐ Random Sampling ☐ Transect Sampling ☐ Biased/Judgmental Sampling ☐ Stratified Random Sampling
☐ Search Sampling ☐ Systematic Grid ☐ Systematic Random Sampling ☐ Definitive Sampling
☐ Screening w/o Definitive Confirmation ☐ Screening w/ Definitive Confirmation
☐ sample Map Attached

The proposed sampling scheme for ground water from private wells and Geoprobe™ temporary wells, silo water, surface water/sediment, and soil will be biased/judgmental, with definitive laboratory analysis, in accordance with procedures included in the Guidance for Performing Site Inspections Under CERCLA, OSWER Directive #9345.1-05, September 1992, and Removal Program Representative Sampling Guidance, Volume 1: Soil, OSWER Directive 9360.4-10, November 1991. All samples will be submitted for analysis by the EPA Region 7 laboratory. See Appendices A and B for additional site-specific information and maps. The proposed number of samples is a balance between cost and coverage and represents a reasonable attempt to meet the study objectives while staying within the budget constraints of a typical site investigation.

Sample Summary Location	Matrix	# of Samples*	Analysis

*NOTE: Background/QC samples are not included with these totals. See Table 1 for a complete sample summary.

2.2 Sample Methods Requirements:

Matrix	Sampling Method	EPA SOP(s)/Methods

2.3 Sample Handling and Custody Requirements:

- ☐ Samples will be packaged and preserved in accordance with procedures defined in Region 7 EPA SOP 2420.6C.
- ☐ COC will be maintained as directed by Region 7 EPA SOP 2420.4B.
- ☐ Samples will be accepted according to Region 7 EPA SOP 2420.1C.
- ☐ Other (Describe):

2.4 Analytical Methods Requirements:

- ☐ Identified in attached table.
- ☐ Identified in attached Analytical Services Request (ASR) Form
- ☐ Other (Describe):

2.5 Quality Control Requirements:

- ☐ Not Applicable
- ☐ Identified in attached table.
- ☐ In accordance with the ***Generic Quality Assurance Project Plan for Superfund Integrated Assessment and Targeted Brownfields Assessment Program (Updated: July 2005).***
  Describe Field QC Samples to be collected:
- ☐ Other (Describe):

2.6. Instrument/Equipment Testing, Inspection, and Maintenance Requirements :

- ☐ Not Applicable
- ☐ In accordance with the ***Generic Quality Assurance Project Plan for Superfund Integrated Assessment and Targeted Brownfields Assessment Program (Updated: July 2005).***
- ☐ Other (Describe):

2.7 Instrument Calibration and Frequency:

- ☐ Not Applicable
- ☐ Inspection/acceptance requirements are in accordance with the ***Generic Quality Assurance Project Plan for Superfund Integrated Assessment and Targeted Brownfields Assessment Program (Updated: July 2005)***.
- ☐ Calibration of laboratory equipment will be performed as described in the previously referenced SOPs and/or manufacturers' recommendations.
- ☐ Other (Describe):

2.8 Inspection/Acceptance Requirements for Supplies and Consumables:

- ☐ Not Applicable
- ☐ In accordance with the ***Generic Quality Assurance Project Plan for Superfund Integrated Assessment and Targeted Brownfields Assessment Program (Updated: July 2005).***
- ☐ All sample containers will meet EPA criteria for cleaning procedures for low-level chemical analysis. Sample containers will have Level II certifications provided by the manufacturer in accordance with pre-cleaning criteria established by EPA in *Specifications and Guidelines for Obtaining Contaminant-Free Containers.*
- ☐ Other (Describe):

2.9 Data Acquisition Requirements:

- ☐ Not Applicable
- ☐ In accordance with the ***Generic Quality Assurance Project Plan for Superfund Integrated Assessment and Targeted Brownfields Assessment Program (Updated: July 2005).***
- ☐ Previous data/information pertaining to the site (including other analytical data, reports, photos, maps, etc., which are referenced in this QAPP) have been compiled by EPA and/or its contractor(s) from other sources. Some of that data has not been verified by EPA and/or its contractor(s); however, the information will not be used for decision-making purposes by EPA without verification by an independent professional qualified to verify such data/information.
- ☐ Other (Describe):

2.10 Data Management:

- ☐ All laboratory data acquired will be managed in accordance with Region 7 EPA SOP 2410.1C.
- ☐ Other (Describe):

3.0 Assessment and Oversight:

3.1 Assessment and Response Actions:

☐ Peer Review ☐ Management Review ☐ Field Audit ☐ Lab Audit

- ☐ Assessment and response actions pertaining to analytical phases of the project are addressed in Region 7 EPA SOPs 2430.5A and 2430.12D.
- ☐ Other (Describe):

3.1A Corrective Action:

- ☐ Corrective actions will be taken at the discretion of the EPA project manager, whenever there appear to be problems that could adversely affect data quality and/or resulting decisions affecting future response actions pertaining to the site.
- ☐ Other (Describe):

3.2 Reports to Management:

☐ Audit Report ☐ Data Validation Report ☐ Project Status Report ☐ None Required

- ☐ A letter report describing the sampling techniques, locations, problems encountered (with resolutions to those problems), and interpretation of analytical results will be prepared by Tetra Tech START and submitted to the EPA.
- ☐ Other (Describe):

3.2 Reports to Management:

☐ Audit Report ☐ Data Validation Report ☐ Project Status Report ☐ None required

- ☐ A letter report describing the sampling techniques, locations, problems encountered (with resolutions to those problems), and interpretation of analytical results will be prepared by START and submitted to the EPA.
- ☐ Reports will be prepared in accordance with the ***Generic Quality Assurance Project Plan for Superfund Integrated Assessment and Targeted Brownfields Assessment Program (Updated: July 2005)***.
- ☐ Other (Describe):

4.0 Data Validation and Usability:

4.1 Data Review, Validation, and Verification Requirements:

- ☐ Identified in attached table.
- ☐ Data review and verification will be performed in accordance with the ***Generic Quality Assurance Project Plan for Superfund Integrated Assessment and Targeted Brownfields Assessment Program (Updated: July 2005).***
- ☐ Data review and verification will be performed by a qualified analyst and the laboratory's section manager as described in Region 7 EPA SOPs 2430.5A and 2430.12D.

Other (Describe):

4.2 Validation and Verification Methods:

- ☐ Identified in attached table.
- ☐ The data will be validated in accordance with Region 7 EPA SOPs 2430.5A and 2430.12D.
- ☐ The EPA site manager will inspect the data to provide a final review. The EPA site manager will review the data, if applicable, for laboratory spikes and duplicates, laboratory blanks, and the field blank to ensure that they are acceptable. The EPA site manager will also compare the sample descriptions with the field sheets for consistency and will ensure that any anomalies in the data are appropriately documented.

☐ Other (Describe):

4.3 Reconciliation with User Requirements:

- ☐ Identified in attached table
- ☐ If data quality indicators do not meet the project's requirements as outlined in this QAPP, the data may be discarded and re-sampling or re-analysis of the subject samples may be required by the EPA site manager.

☐ Other (Describe):

Table 1: Sample Summary							
Site Name:				**City:**			
START Project Manager:				**Activity/ASR #:**		**Date:**	
No. of Samples	**Matrix**	**Location**	**Purpose**	**Depth or other Descriptor**	**Requested Analysis**	**Sampling Method**	**Analytical Method/SOP**

APPENDIX B

STANDARD OPERATING PROCEDURES

U. S. EPA ENVIRONMENTAL RESPONSE TEAM

STANDARD OPERATING PROCEDURES

X-MET™ 880 FIELD PORTABLE X-RAY FLUORESCENCE OPERATING PROCEDURES

CONTENTS

CONTENTS (Cont'd)

CONTENTS (Cont'd)

CONTENTS (Cont'd)

SUPERCEDES: SOP #1707; Revision 1.0; 11/21/91; U.S. EPA Contract 68-03-3482.

X-MET™ 880 FIELD PORTABLE X-RAY FLUORESCENCE OPERATING PROCEDURES

1.0 SCOPE AND APPLICATION

The purpose of this standard operating procedure (SOP) is to serve as a guide to the start-up, check-out, operation, calibration, and routine use of the X-Met 880 instrument, for field use in screening of hazardous or potentially hazardous inorganics. It is not intended to replace or diminish the use of the X-MET 880 Operating Instructions. The Operating Instructions contain additional helpful information to assist in the optimum instrument utilization and which form the basis on which new and varying applications can later be based.

The procedures contained herein are general operating procedures which may be changed as required, dependent on site conditions, equipment limitations, limitations imposed by the QA/QC procedure or other protocol limitations. In all instances, the ultimate procedures employed should be documented and associated with the final report.

1.1 Principles of Operation

X-Ray Fluorescence Spectroscopy (XRF) is a nondestructive qualitative and quantitative analytical technique used to determine the chemical composition of samples. In a source excited XRF analysis, primary X-rays emitted from a sealed radioisotope source are utilized to irradiate samples. During interaction of the source X-rays with samples, they may either undergo scattering (dominating process) or absorption by sample atoms in a process known as the photoelectric effect (absorption coefficient). This most useful analytical phenomenon originates when incident radiation knocks out an electron from the innermost shell of an atom. The atom is excited and releases its surplus energy almost instantly by filling the vacancy created with an electron from one of the higher energy shells. This rearrangement of electrons is associated with emission of X-rays characteristic (in terms of energy) of the given atom. This process is referred to as emission of fluorescent X-rays (fluorescent yield). The overall efficiency of the process described is referred to as excitation efficiency and is proportional to the product of absorption coefficient and fluorescent yield.

Generally, the X-MET 880 utilizes characteristic X-ray lines originating from the innermost shells of the atoms, K, L, and occasionally M. The characteristic X-ray lines of the K series are the most energetic lines for any element and, therefore, are the preferred analytical lines. The K lines are always accompanied by the L and M lines of the same element. However, being of much lower energy than the K lines, they can usually be neglected for those elements for which the K lines are analytically useful. For heavy elements (such as Ce, atomic number (Z)=58, to U, Z=92), the L lines are the preferred lines for X-MET 880 analysis. The L_α and L_β lines have almost equal intensities and the choice of one or the other depends on what interfering lines might be present. A source just energetic enough to excite the L lines will not excite the K lines of the same element. The M lines will appear together with the L lines.

The X-MET 880 Operating Instructions contain tables that show the energies and relative intensities of the primary characteristic X-ray lines for all the applicable elements, see Section 1.3.

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 (i.e., K absorption edge, L absorption edge, M absorption edge) of the element. 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, and the L absorption edge energy is approximately the sum of the L and M line energies of the particular element.

Energies of the characteristic, fluorescent X-rays are converted (within the detector) into a train of electric pulses, the amplitudes of which are linearly proportional to the energy. An electronic multichannel analyzer (electronic unit) measures the pulse amplitudes which, since they are proportional to original energies of emitted characteristic X-rays, are the basis of a qualitative X-ray analysis. The number of equivalent counts at a given energy is representative of element concentration in a sample basis for quantitative analysis.

1.1.1 Scattered X-rays

The source radiation is scattered from the sample by two physical reactions: coherent or elastic scattering (no energy loss) and Compton or inelastic scattering (small energy loss). Thus, the backscatter (background signal) actually consists of two components with X-ray lines close together, the higher energy line being equal to the source energy. Since the whole sample takes part in scattering, the scattered X-rays usually yield the most intense lines in the spectrum. It is also obvious from the aforementioned that the scattered X-rays have the highest energies in the spectrum and contribute the most part of the total measured intensity signal.

1.2 Sample Types

Solid and liquid samples can be analyzed for elements Al (aluminum) through U (uranium) with proper X-ray source selection. Typical environmental applications are:

- Heavy metals in soil (in-situ or samples collected from the surface or from bore hole drillings, etc.), sludges, and liquids (i.e., Pb in gasoline)
- Light elements in liquids (i.e., P, S and Cl in organic solutions)
- Heavy metals in industrial waste stream effluents
- PCB in transformer oil by Cl analysis
- Heavy metal air particulates collected on membrane filters, either from personnel samplers or from high volume samplers.

2.0 METHOD SUMMARY

The X-MET 880 Portable XRF Analyzer employs radioactive isotopes, such as Fe-55, Cm-244, Cd-109 and Am-241 for the production of primary X-rays. Each source emits a specific energy range of primary X-rays that cause a corresponding range of elements in a sample to produce fluorescent X-rays. When more than one source can excite the elements of interest, the appropriate source(s) is selected according to its excitation efficiency for the elements of interest. See X-MET 880 Operating Instructions for a chart of source type versus element range, Section 1.17.

For measurement, the sample is positioned in front of the source-detector window and exposed to the primary (source) X-rays by pulling a trigger on the probe (or pushing the top of the probe unit back on the sample type probe) which exposes the sample to radiation from the source. The sample fluorescent and backscattered X-rays enter through the beryllium (Be) detector window and are detected in the active volume of a high-resolution, gas-filled proportional counter.

Elemental count rates (number of net element pulses per second) are used in correlation with actual sample compositions to generate calibration models for qualitative and quantitative measurements.

Analysis time is user selectable from 1 to 32767 seconds. The shorter measurement times (30 - 100s) are generally used for initial screening and hot spot delineation, while longer measurement times (100 - 500s) are typically used for higher precision and accuracy requirements.

3.0 SAMPLE PRESERVATION, CONTAINERS, HANDLING AND STORAGE

This SOP specifically describes equipment operating procedures for the X-MET 880; hence, this section is not applicable to this SOP.

4.0 INTERFERENCES AND POTENTIAL PROBLEMS

The total error of XRF analysis is defined as the square root of the sum of squares of both instrument precision and user or application related error. Generally, the instrument precision is the least significant source of error in XRF analysis. User or application related error is generally the more significant source of error and will vary with each site and method used. The components of the user or application related error are the following:

4.1 Sample Placement

This is a potential source of error since the X-ray signal decreases as you increase the distance from the radioactive source. However, this error is minimized by maintaining the same sample distance from the source.

4.2 Sample Representivity

This can be a major source of error if the sample and/or the site-specific or site-typical calibration samples (see Section 4.0) are not representative of the site. Representivity is affected by the soil macro- and micro-heterogencity. For example, a site contaminated with pieces of slag dumped about by a smelting operation will be less homogeneous than a site contaminated by liquid plating waste. This error can be minimized by either homogenizing a large volume of sample prior to analyzing an aliquot, or by analyzing several samples (in-situ) at each sampling point and averaging the results.

4.3 Reference Analysis

Soil chemical and physical matrix effects may be corrected by using Inductively-Coupled Plasma (ICP) or Atomic Absorption (AA) spectrometer analyzed site-specific soil samples as calibration samples. A major source of error can result if the samples analyzed are not representative of the site and/or the analytical error is large. With XRF calibrations based on reference analyses results, the XRF analytical results can be reported in the same units as the calibration samples reference analyses. Results, for example, will be in Contract Laboratory Protocol (CLP) extractable metals if the CLP specified HNO_3/H_2O_2 digestion is used. Results will be in total metals if total (HF) digestion or KOH fusion is used.

4.4 Chemical Matrix Effects (Effects due to the chemical composition of the sample)

Chemical matrix effects result from differences in concentrations of interfering elements. These effects appear as either spectral interferences (peak overlaps) or as X-ray absorption/enhancement phenomena. Both effects are common in soils contaminated with heavy metals. For example, Fe (iron) tends to absorb Cu (copper) K shell X-rays, reducing the intensity of Cu measured by the detector. This effect can be corrected if the relationship between Fe absorption and the Cu X-ray intensities can be modeled mathematically. Obviously, establishing all chemical matrix relationships during the time of instrument calibration is critical. These relationships are modeled mathematically, with X-MET 880 internal software, using ICP or AA analyzed site-specific soil samples as the XRF calibration standards. Additionally, increasing the number of standards and the range of the standard concentrations used may decrease the error in the calibration mathematical modeling. Generally, as rule-of-thumb, a minimum of five calibration samples per element to be analyzed are used to generate reliable X-MET 880 calibration models.

4.5 Physical Matrix Effects (Effects due to sample morphology)

Physical matrix effects are the result of variations in the physical character of the sample. They may include such parameters as particle size, uniformity, homogeneity and surface condition. For example, consider a sample in which the analyte exists in the form of very fine particles within a matrix composed of much courser material. If two separate aliquots of the sample are ground in such a way that the matrix particles in one are much larger than in the other, then the relative volume of analyte occupied by the analyte-containing particles will be different in each. When measured, a

larger amount of the analyte will be exposed to the source X-rays in the sample containing finer matrix particles; this results in a higher intensity reading for that sample and, consequently, in apparently higher measured concentration of that element.

4.6 Modeling Error

The error in the calibration mathematical modeling is insignificant (relative to the other sources of error) IF the instrument's modeling operating instructions are followed correctly (see Section 14.0).

4.7 Moisture Content

If measurement of soils or sludges is intended, the sample moisture content will affect the accuracy of the analysis. The overall error from moisture may be a secondary source of error when the moisture range is small (5-20%), or may be a major source of error when measuring on the surface of soils that are saturated with water.

4.8 Cases of Severe X-ray Spectrum Overlaps

Certain X-ray lines from different elements, when present in the sample, can be very close in energy and, therefore, interfere by producing a severely overlapped spectrum.

The typical spectral overlaps are caused by the K_β line of element Z-1 (or as with heavier elements, Z-2 or Z-3) overlapping with the K_α line of element Z line. This is the so-called K_α/K_β interference. Since the K_α:K_β intensity ratio for the given element usually varies from 5:1 to 7:1, the interfering element, (Z-1), must be present in large concentrations in order to disturb the measurement of analyte Z. The presence of large concentrations of titanium (Ti) could disturb the measurement of chromium (Cr). The TiK_α and K_β energies are 4.51 and 4.93 Kev, respectively. The CrK_α energy is 5.41 Kev. The resolution of the detector is approximately 850 eV. Therefore, large amounts of Ti in a sample will result in spectral overlap of the TiK_β with the CrK_α peak.

Other interferences are K/L, K/M and L/M. While these are less common, the following are examples of a severe overlap:

$$AsK_\alpha/PbL_\alpha\ ,\ SK_\alpha/PbM_\alpha$$

In the arsenic/lead case, lead can be measured from the PbL_β line, and arsenic from either the AsK_α or the AsK_β line; this way the unwanted interference can be corrected. However, due to the severeness of the overlap (energy of AsK_α is almost identical to that of PbL_α), measurement sensitivity is reduced.

4.9 Inappropriate Pure Element Calibration

It is of paramount importance that the pure element calibration, also called "instrument calibration" (see Section 12.0), include all elements that can be present at the site, (i.e., in the samples to be analyzed). This means that even if the element is not a target element, as long as it is present in detectable amounts with the source in use, it must be included in the pure element calibration in order for the X-MET 880 to correct for its potential spectral interference effect on the target element.

5.0 EQUIPMENT/APPARATUS

5.1 Description of the X-Met 880 System

The X-MET 880 analyzer includes a compact, sealed radiation source contained that is in a measuring probe. This probe is connected by cable to an environmentally sealed electronic module.

The analyzer utilizes the method of Energy Dispersive X-Ray Fluorescence (EDXRF) spectrometry to determine the elemental composition of soils, sludges, aqueous solutions, oils, and other waste materials.

Each probe is equipped with a high resolution gas-filled proportional detector, or a high resolution solid state lithium drifted silicon (Si/Li) detector.

The complete X-MET 880 system consists of five alternate configurations.

1. The X-MET 880ES Extended Range X-MET 880 Silicon Detector System includes the Silicon Detector Surface Probe System (SDPS) with a 60 millicurie (mCi) Curium-244* (Cm-244) excitation source and a 30 mCi Americium-241** (Am-241) excitation source.

2. The X-MET 880SH, Standard X-MET 880 System comes with a SAPS probe containing a 60 mCi Cm-244* excitation source.

3. The X-MET 880ER Extended Range X-MET 880 System comes with a DOPS probe that contains a 60 mCi Cm-244* excitation source and a 30 mCi Am-241** excitation source.

4. The X-MET 880AS Alternate Standard Range X-MET 880 System comes with a SAPS probe containing 10 mCi Cd-109*** (in place of the 60 mCi Cm-244).

5. The X-MET 880AE Alternate Extended Range X-MET 880 System includes the DOPS with 10 mCi Cd-109*** plus 30 mCi Am-241**.

* Cm-244 in SDPS probe (or Surface Analysis Probe Set (SAPS) and Double Source Surface Probe Set (DOPS) probes) will allow analysis of all elements from atomic number 19 (potassium) to 35 (bromine) and from atomic number 56 (barium) to atomic number 83 (bismuth).

** The Am-241 excitation source extends the elemental range of the system to include such important priority pollutants as Cd, Ag, and Ba. Am-241 in SDPS, SAPS or DOPS will allow analysis of all elements from atomic number 30 (zinc) to 60 (neodymium), and atomic number 73 (tantalum) to 92 (uranium).

*** Replacing the Cm-244 with Cd-109 provides somewhat improved precision and accuracy for Pb when As is present. Cd-109 in SAPS or DOPS will allow analysis of elements from atomic number 24 (chromium) to 42 (molybdenum) and from 65 (terbium) to 92 (uranium).

Optional sample type probes (for laboratory or mobile lab use only) are available for use when all samples will be contained in X-ray cups. These probes can contain any of the excitation sources described above.

The use of the special optional sample probes, Light Element sample Probe System (LEPS), or the Surface Light element Probe System (SLPS), with the Fe-55 ring source, enables analysis of light elements ranging from atomic number 13 (aluminum), to atomic number 24 (chromium), and heavy elements from 37 (rubidium) to 56 (barium).

The electronic module includes a 256 channel multi-channel analyzer and a high speed, 16/32 bit, Motorola 68000 microprocessor. Up to 32 multi-element analysis programs, called models, can be stored in its memory.

The unit comes factory pre-calibrated based on the Outokumpu Electronics synthetic soil calibration suite, or based on customer supplied standards (see supplemental documentation on factory calibration for calibration data specific to each X-MET 880).

Optional calibrations can be installed at the factory for other soil or waste material types on request.

Additional models tailored to specific needs may be added by the user after attending the X-MET 880 Calibration and Operators Training Course which is conducted by Ouotkumpu Electronics at regular intervals. (Request course description and schedule from Outokumpu Electronics, Langhorne, PA.)

The X-MET 880 can be operated from a 115-volt (or 220-volt) wall outlet or its 12-volt, 10-hour capacity battery, or a standard 12-volt car or truck battery.

The X-MET 880 can be operated in a temperature range from 32 to 140^0 Fahrenheit (F), or may be operated down to -13^0 F with the low temperature option. The freezing point of a discharged battery is 14^0 F.

The probe and electronic unit may be exposed to a light rain. However, additional protection is provided if the system (electronic unit and probe) is contained in the optional water repellant carrying case.

The instrument can be calibrated for up to 10 elements per model, six (6 target elements) of which can provide a readout in the Assay Mode.

In the Assay Mode, up to 30 reference samples per assay model can be used to generate the sample calibration curve in the X-MET 880.

5.2 Equipment and Apparatus List

5.2.1 X-MET 880 Analyzer System

The complete X-MET 880 Analyzer System includes:

- 880 Electronics Module
- Single source SAPS or DOPS or SDPS with optional LEPS or optional Heavy Element Sample Probe Set (HEPS) in place of, or in addition to, the SAPS and/or DOPS, each containing appropriate excitation source(s)
- Pure element standards
- Battery charger
- Battery pack
- X-MET 880 Operating Instructions, X-MET 880 Operator's Manual and any applicable X-MET 880 factory calibration documentation.

5.2.2 Optional Items

- 31 millimeter (mm) diameter sample cups
- XRF polypropylene film, 0.2mm thickness

- Nylon reinforced, water-repellant backpack
- Metal reinforced shipping case with die-cut foam inserts for X-MET 880 and accessories
- Peripheral devices such as the Terminal/Printer data logger, the DMS-1 Data Management System, the ESP extended software package for use with IBM compatible Personal Computer (PC).
- Surface probe shield assembly. Shield assembly must be used when the SAPS or DOPS probes are inverted for measuring sample in XRF cups.

See Outokumpu Electronics X-MET 880 Accessories Price List for additional Outokumpu Electronics options.

For mobile lab or laboratory X-ray sample preparation accessories, such as drying ovens, grinders, sieves, etc., consult general laboratory equipment suppliers.

5.2.3 Limits and Precautions

The probes should be handled in accordance with the following radiological control practices:

1. The probe should always be in contact with the surface of the material being analyzed and the analyzed material should completely cover the probe opening (aperture), when the probe shutter is open. The indicator flag on the side of the DOPS and SAPS probes is green when the shutter is closed and red when it is open.
2. Under no circumstances should the probe be pointed at the operator or surrounding personnel with the shutter open.
3. Do not place any part of the operator's or co-worker's body in line of exposure when the shutter is opened and not fully covered.
4. The SAPS or DOPS probe trigger must be key-locked when not in use.
5. Notify Outokumpu Electronics immediately of any condition or concern relative to the probe structural integrity, source shielding, shutter condition or operability.
6. Notify the appropriate state agency or Nuclear Regulatory Commission (NRC) office (see factory supplied data on radiological safety) immediately of any damage to the radioactive source, or any loss or theft of the device.

7. Labels or instructions on the probe(s) must not be altered or removed.

8. The user must not remove the probe covers or attempt to open the probe.

9. The source(s) in the probe must be leak tested every six (6) months as described in the X-MET 880 Operating Instructions. The Leak Test Certificates must be kept on file and a copy must accompany the instrument at all times.

10. The probe shield assembly must be used when the SAPS or DOPS probe is inverted for measuring samples contained in cups.

11. During operation, keep the probe at least 10 feet from computer monitors and any other source of radio frequency (RF). Some monitors have very poor RF shielding and will affect measurement results.

12. The X-MET 880 should not be dropped or exposed to conditions of excessive shock or vibration.

13. Keep the force on the probe, with the trigger pulled, to less than four (4) pounds to avoid shutter binding.

Additional precautions include:

1. Do not pull on the probe wire to unplug the probe. Grasp the probe plug at the ribbed rubber connector cover and squeeze, then press firmly while plugging, and pull while unplugging the connector.

2. Do not attempt to rotate the handle on the electronic unit unless the release buttons on each side of the handle are depressed.

3. The X-MET 880 should not be operated or stored at an ambient temperature below 32^0F (-13^0F with low temperature version) or above 140^0 F.

4. The battery charging unit should only be used indoors at dry conditions.

5.3 Peripheral Devices

The X-MET 880 may be used with a wide range of peripheral devices for electronic data capture or printed readout as long as they are equipped with input compatible with the RS-232 serial protocol. Such devices include terminals, printers, electronic data loggers, personal computers, etc. Any time a peripheral device is connected to the X-MET 880, all text and commands shown on the X-MET 880 display will be automatically output to and copied by the peripheral.

5.3.1 Communication Cable Connection

Plug the round connector of the RS-232 cable, into the X-MET 880 "IN/OUT" port (the connection just above the probe connection on the electronic unit), and the rectangular (25 pin) connector of the cable to the RS-232 port of the receiving device (serial port).

If the receiving device, serial port, does not have a 25 pin, male, RS-232 C standard connector, THEN it will be necessary to obtain a "gender mender plug" (male-to-female converter), OR, in the case of 9 pin device connectors, a 25 to 9 pin adapter (with or without gender changer, depending on the gender of the connector at the receiving device). All Outokumpu supplied peripherals are delivered with appropriate connections.

5.3.2 Communication Speed

To communicate with the external device, the X-MET 880 MUST be set at the same Baud Rate as the receiving device. The X-MET 880 command for setting or resetting the Baud Rate is CSI (Configure Serial Interface). The CSI command is a sub-command under the EMP (Enter Maintenance Parameters) command, which must therefore precede it. Enter EMP followed by "CONT/YES", THEN enter CSI on the keypad FOLLOWED by "CONT/YES". The X-MET 880 will display

BAUD RATE: XXXX NEW?

Press "CONT/YES" until the desired baud rate (data transfer speed) is in the display, then press "END/NO" to accept the displayed reading. The baud rate can have values 50, 75, 110, 134.5, 150, 200, 300, 600, 1200, 1800, 2400, 4800, 9600 and 19200 baud. Select the baud rate of the peripheral device with which you are communicating.

5.3.3 Character Set

The RS-232 C interface supports all the standard ASCII characters. Upper and lower case letters are equivalent in data transmission. This means that the X-MET 880 will execute any legal command typed in lower case on the peripheral keyboard. (See Section 5.3.5 for some special keys.) The serial data format is:

* 1 start bit (SPACE)
* 8 data bits (ASCII from LSB to MSB)
* 1 stop bit (MARK)

5.3.4 Parity

Parity MUST be set to NO PARITY.

5.3.5 Terminal Emulation

Although most keys on the standard typewriter style keypad (as used on most terminals and computers) are the same as the X-MET 880 keypad, there are, however, some specific keys on the X-MET 880 that require the operator to use an "equivalent" key on the terminal. The following lists correlates the unique X-MET 880 keys and their terminal equivalents:

X-MET 880 KEY	TERMINAL KEY (or key combination)
CONT/YES	ENTER (or RETURN on some devices)
END/NO	ESC
MO**D**EL	CTRL **D**
M**T**IME	CTRL **T**
RECALC	CTRL **R**
<	SHIFT <
^	SHIFT ^
ST**A**RT	CTRL **A**

5.3.6 User Software

Refer to your PC software manual for details on additional settings that may be required for proper interfacing between X-MET 880 and your particular software.

5.4 Instrument Maintenance

5.4.1 Probe Window

Should the probe window become damaged or punctured, it should be replaced as soon as possible to prevent dust and moisture from entering the probe. Replacement window assemblies can be ordered from Outokumpu Electronics. Simply pry out the old window aperture using a small, flat-blade screwdriver, or similar. PRIOR to reinstalling the new aperture, rub a very thin film of silicon grease (or liquid soap, IF silicon grease is not available) around the rubber "O" ring, inside the probe aperture opening, to facilitate re-installation.

The removal of any loose dirt on the probe window should be done with a soft brush or cloth and then blown with air. To remove adhering dirt, a solvent such as methanol or ethanol may be used.

5.4.2 Further Information and Troubleshooting

Refer to the X-MET 880 Operating Instructions (or optional Maintenance Manual) for additional detailed operational and/or maintenance and troubleshooting instructions. IF no solution is not found in either manual, THEN contact Outokumpu Electronics for assistance.

It is recommended that an Instrument Log be maintained for documenting specific corrective actions taken to alleviate any instrumental problems, or to record any service that has been performed.

6.0 REAGENTS

6.1 Site-Specific Calibration Standards (SSCS)

The SSCS must be representative of the matrix to be analyzed by the XRF. They are employed in the "sample calibration" stage of programming the instrument (see Section 7.5.6). They are also employed in the subsequent calibration checks (see Section 7.2 and 9.0) and any re-calibrations that may be performed.

The concentration of the target elements in the SSCS should be determined by independent AA or ICP analysis that meets an acceptable quality for referee data.

Additionally, the concentration in the SSCS of elements adjacent (+/-2 atomic numbers Z) to the Z of the target element should be determined by independent AA or ICP analysis if: 1) they are excited by the source used, and/or 2) their concentrations are unknown or suspected to be greater than ten percent of the target element concentration and/or 3) it is unknown or suspected that their concentration variance is greater than twenty percent in the site matrix, or if this variance (if greater than twenty percent) has a non-linear relationship to the variance of the target element concentration.

For example, the requested target elements are Cd and Sb for a site. Review of the site history indicates that Sn and Ag may be present. The SSCS should be analyzed for Sn and Ag in addition to Cd and Sb, to determine their concentrations and the relationship (linear or non-linear) to the Cd and Sb concentrations in the SSCS samples.

6.1.1 SSCS Sampling

Review Section 4.2 on sample representivity. The SSCS samples must be representative of the matrix to be analyzed by XRF. It does not make sense to collect the SSCS samples in the site containment area if you are interested in investigating off-site contaminant migration. The matrices may be different and could affect the accuracy of the XRF results. If there are two different matrices on-site, collect two sets of SSCS samples.

A full range of target element concentrations is needed to provide a representative calibration curve. Mixing high and low concentration soils to provide a full range of target element concentrations is not recommended due to homogenization problems. The highest and lowest SSCS samples will determine the linear calibration range. Unlike liquid samples, solid samples cannot be diluted and re-analyzed.

The number of SSCS samples needed for calibrating an assay model depends on:

1. The number of target elements (analyte). For each additional target element, increase the number of SSCS samples by five (up to a maximum of 30).

2. The number of elements adjacent to the target elements. For each additional adjacent element known or found to be present in the samples, you should increase the number of SSCS samples by five (up to a maximum of 30) to insure that the calibration model properly corrects for X-ray interferences and spectral overlaps.

Additionally, collect several SSCS samples in the concentration range of interest. If the action level of the site is 500 mg/kg, providing several SSCS samples in this concentration range will tend to improve the XRF analytical accuracy in this concentration range.

Generally, a minimum of 10 and a maximum of 30 appropriate SSCS samples should be taken. A minimum of a 4 oz. sample is required. A larger size sample should be provided to compensate for samples with a greater content of non-representative material such as rocks and/or organic debris. Standard glass sampling jars should be used.

6.1.2 SSCS Preparation

The SSCS samples should be dried either by air drying overnight, or oven drying at less than 105°C. Aluminum drying pans or large plastic weighing boats for air drying may be used. After drying, remove all large organic debris and non-representative material (twigs, leaves, roots, insects, asphalt, rocks, etc.).

The sample should be sieved through a 20-mesh stainless steel sieve. Clumps of soil and sludge should be broken up against the sieve using a stainless steel spoon. Pebbles and organic matter remaining in the sieve should be discarded. The under-sieve fraction of the material constitutes a sample.

Although a maximum final particle size of 20-mesh is normally recommended, a smaller particle size may be desired (see Section 4.5). The sample should be homogenized by dividing the sieved soil into quarters and physically mixing opposite quarters with a clean stainless steel spoon. Re-composite and then repeat the quartering and mixing procedure three times. Place the sieved sample in a clean glass sample jar and label it using both the site name and sample identification information.

The stainless steel sieves should be decontaminated using soap and water and dried between samples.

One or more plastic XRF sample cups should be filled with the sieved soil for each SSCS sample. A piece of .2mm polypropylene film is cut and tensioned, wrinkle-free, over the top of the x-ray sample cup and then sealed using the plastic film securing ring. The cup should be labeled using both the site name and specimen identification information.

Either the XRF sample cup or the balance of the prepared sample is submitted to the approved laboratory for analysis of the requested element(s) by AA or ICP.

6.2 Site Typical Calibration Standards (STCS)

When the goal of the analysis with X-MET 880 is semi-quantitative measurements, such as hot spot delineation or determination of sampling points for a SSCS, then use of a STCS may be the most appropriate method. STCS are SSCS from a different site that have the identical target elements in a similar range and matrix as the site that is to be analyzed. It should be noted that the STCS are not from the site to be analyzed and may generate false positive and negative results.

For example, samples are going to be taken at lead battery manufacturing site for a SSCS. There is no information in the site history on the location or concentration of the Pb contamination. A model calibrated for Pb with a SSCS from another battery breakage site could be used as a STCS to screen this site and locate low, mid and high Pb contamination points for the SSCS sampling.

7.0 PROCEDURE

7.1 Prerequisites

If the X-MET 880 will be used in a location where AC power outlets are conveniently accessible, connect the battery charger to the battery and plug the charger cord into the outlet. The cable probe must be connected before the power is switched on. Plugging and unplugging this cable with the power on can damage the detector.

Verify that the probe shutter is closed by checking the mechanical flag color on the side of the SAPS or DOPS. When the flag is green the shutter is closed and open when it is red.

Connect the probe cable to the connector labeled "PROBE" on the electronic module. Make certain the plug has been firmly pushed in all the way (you will feel and hear a slight "click" as the probe connector locks into position).

Apply power to the X-MET 880 by pressing the "ON" button.

Verify that the display briefly reads:

X-MET 880 VX.X.X (software version) and DATE

SELF TEST COMPLETED, followed by (if the X-MET 880 has been off, more than a few minutes) the message:

GAIN CONTROL: COUNT RATE TOO LOW

will flash intermittently (along with a "beep") on the display. This is normal and will stop approximately 30 seconds after power is on.

Verify that a prompt sign (>) appears in the lower left corner of the display after the gain control message has stopped flashing.

Verify that the upper right corner of the display reads the number of seconds (0 - 32,000s) on the top line and the model number (1 - 32) on the bottom line.

If a "battery low" message appears recharge the battery before proceeding or operate using line voltage.

Allow the X-MET 880 to warm up for approximately 60 minutes.

If the X-MET 880 is being used in a location where the temperature of the environment has changed by more than 5°F, then allow the X-MET 880 to stabilize at the new ambient temperature. Approximately 1 minute stabilization time for each 1°F change in ambient temperature should be allowed.

7.1.1 Gain Control

Allow 5 minutes after temperature stabilization for the X-MET 880 to perform a complete cycle of automatic electronic gain control. The trigger must be released on the probe to activate the gain control operation. Additionally, the prompt sign (>) must be in the display.

The X-ray spectrum contains a number of incident X-ray quantum energies of which there are corresponding channels in the multichannel analyzer. These channels are limited by certain changes in conditions such as the resolution of the detector and temperature coefficients. This means that a proportionally large error from measurement would be obtained, if no compensation was made for these variations.

The elimination of such errors is made possible by monitoring the state of the probe spectrum and compensating for any spectral drift as described in Section 7.1.2.

The state of the detection region is maintained by a feedback gain control system, which operates all the time the probe is in a non-operative position (gain control position, DOPS, SAPS, SLPS placed on their sides, HEPS, LEPS with the lid in the forward position) and the instrument is not under any command (i.e., prompt displayed (>_)).

The gain control routine is accomplished by allowing the X-MET 880 to measure a reference material (usually copper) mounted on the shutter and then maintaining a track on the peak spectral position.

The initial peak position is set up during the initialization of the probe (INI) and from this point the instrument will adjust accordingly, during gain control.

7.1.2 Gain Control Monitoring

During initialization, the initial peak channel is established for the gain control. During gain control, adjustments are made to return the maximum peak (usually copper), after the backscatter peak, to this initial peak channel location. The peak channel is monitored and recorded to verify if the gain control mechanism is working and returning the peak to the correct channel. Failure of the gain control mechanism will result in spectral drift and calculation of incorrect intensities in the element windows or incorrect pure element window calibration. The gain control peak channel should be measured and recorded a the beginning, end, and every 25 to 40 minutes during the following operations:

1. Pure element calibration
2. SSCS Measurements
3. All sample analysis
4. All preoperational check sample measurements.

The gain control peak channel measurement is performed with the probe in the same position as it was during gain control (shutter is closed for the DOPS or SAPS probes and the sample chamber is pulled forward for the HEPS probe). The instrument should be set for a minimum measuring time of 60 seconds. The enter maintenance program command (EMP) is entered followed by "CONT/YES" command. Then the test measurement (TSM) command is entered followed by the "CONT/YES". The peak channel (PKCH), the full width half-maximum (FWHM) of the peak and the counts will be displayed. Record the PKCH and the FWHM in a log book.

The peak channel (N) should not vary more than N±1 during <u>all</u> of the operations listed above.

If the PKCH varies more than one channel, allow the instrument to gain control for another 5.0 minutes. If the peak channel continues to drift after allowing it to gain control several times, contact an Outokumpu representative. DO NOT continue to perform any analysis until the problem has been corrected.

7.2 Preoperational Checks

Select a minimum of one low, mid and high SSCS (used in the model to be checked, not detected) for all target elements for every model to be checked. Select a low SSCS above the typical detection limit for each target element (i.e., typical detection limits for lead are 100-200 mg/kg. Selection of a 300

or 400 mg/kg SSCS would be appropriate). Select a mid SSCS at or near the action level for each target element or an SSCS about 5 or 10 times the low SSCS concentration (i.e., typical action levels for lead are 500-2000 mg/kg. Selection of a 1000 mg/kg SSCS would be appropriate). Select a high SSCS at or near the end of the linear calibration range of each target element.

These SSCS should be measured using the same measuring that will be applied to the sample analysis. However, a minimum of 60 seconds should be used for Model verification and preoperational checks if the instrument is going to be using 15 to 30 second screening analysis.

The low SSCS should be measured ten times, using the anticipated site measuring time, after the model calibration has been completed. These results will be used to calculate a preliminary detection limit (DL) and quantitation limit (QL) as described in Section 15.0. A control range may be calculated using the average of the results plus or minus the detection limit (i.e., a low SSCS has a mean of 200 mg/kg and a DL of 120 mg/kg; the control range would be 80 to 320 mg/kg). The low SSCS will have the largest relative percent variance due to its proximity to the DL.

The mid and high SSCS can be measured as described above and a range calculated using the average plus or minus three times the standard deviation of the results. Generally, three measurements of the standards is sufficient. A control range may be calculated using the average of the three measurements plus or minus twenty-five percent of the average.

These SSCS should be measured at least once whenever the instrument is transported. Additionally, they should be analyzed at the beginning of each analysis day and at the end of the analytical period. All results should be logged in the operator's log book or saved on a computer disk in a report format.

These SSCS may be used for verifying the model as described in Section 7.5.10 and for Quality Assurance and Control as described in Section 9.0.

Results outside the described range indicate that there is an instrument problem.

7.3 Normalization and Standardization

Normalization and standardization should never be performed. These procedures have never been needed at ERT/REAC and have never been performed. It is recommended that the operator recalibrate the model if the calibration is older than four months for a Cd-109 source; six months for an Fe-SS source; and three years for a Cm-244 source. Recalibration is never required for the Am-241 source.

7.4 General Keys and Commands

7.4.1 The "Shift" Key

Prior to any operations using the keypad, determine which keypad function is to be used:

ALPHA = SH showing in display
NUMERIC = SH not showing in display

Then press the "SHIFT" key to change keypad functions. For selecting the model as outlined in Section 7.4.2 below, the SH must not be showing in the display.

7.4.2 The "Model" (Selection) Key

If it is desired to change models, then depress the "MODEL" key. The analyzer will show the current model in the lower right corner of the display. The analyzer active display will read:

MODEL Y?

Where Y is the currently selected model number.

Enter the desired model number using the number keys and press the "CONT/YES" key. The analyzer will display the model type, UNCALIBRATED (no calibration - spectral data only), INTENSITY (pure element intensities only - if the pure element routine has been completed), LIBRARY (identification calibration completed), or ASSAY (chemistry or composition calibration completed), along with the model's name (if assigned a name). Note that the change in model number (and the associated pre-programmed measuring time) has been registered in the lower right corner of the display.

If no new model number is entered and only the "CONT/YES" or "END/NO" key is pressed, the model number remains unchanged.

Example of model selection:

>MODEL		
MODEL 1?	**CONT/YES**	15s
UNCALIBRATED MODEL		1
>		

or

>MODEL			
MODEL 1?	**2**	**CONT/YES**	10s
OLD LIBRARY MODEL PB LEVEL	2		
>			
or			
>MODEL			
MODEL 1?	**32**	**CONT/YES**	100s
OLD ASSAY MODEL SITE X	32		

The factory pre-programmed measurement time for each model can be changed using the MTM, Measurement Time by Model, command. The change will not be executed until the model is exited and re-selected.

7.4.3 The "MTIME" (Measurement Time Selection) Key

If the measurement time needs to be changed, depress the "MTIME" key. The analyzer will show the measuring time in seconds followed by a lower case "s" in the upper right corner of the display. The active display (bottom line) will read:

MEASURING TIME XXX?

Depress the number keys to enter the desired measurement time (15 to 240 seconds are typical measuring time for hazardous waste application - 1 to 32767 is the total range) and depress the "CONT/YES" key. Note the corresponding change in measuring time in the upper right corner of the display.

The measurement time remains unchanged if the "CONT/YES" or "END/NO" keys alone are pressed. The new measurement time replaces the old value in the upper right hand corner of the display.

All uncalibrated models are defaulted to a measuring time of 15 seconds. All calibrated models are defaulted to the pre-programmed (under the MTM command) measurement time.

Example of selection of measurement time:

>**MTIME**		**CONT/YES**
MEASURING TIME 15 ?	**60**	**CONT/YES**

The MTIME command provides a temporary change in the measurement time. The measurement time will return to the model pre-programmed (MTM command) measurement time when a new model is selected.

7.4.4 AMS Command: Data Averaging Mode

Use the AMS command (Average Measurement Service) if an average of several measurements is desired. First, select the desired model (see Section 7.4.2). Then enter the command AMS and depress "CONT/YES". The analyzer will display:

MEASURE

Measure the sample(s). Depress the "END/NO" key to terminate the AMS command and display the average of the measurements.

7.4.5 The "RECALC" Key

Results (spectrum) from the last measurement can be recalculated using another model. This is done by switching to a new model, and pressing the "RECALC" key. If the new model is for two sources, only the result for the Last Source Measurement can be calculated; if this is not sufficient, a new measurement has to be carried out.

7.4.6 The Function Keys "F1 - F5"

The five function keys can be programmed to contain any of the three letter command acronyms. Up to five (5) pre-programmed expressions can be stored in the analyzer's memory and are retained as long as a charged battery is connected. The programming is initiated with the FNC command.

If the keyboard is locked (LOC command), only the "ON, OFF, CONT/YES, END/NO, RECALC, START, and F1 - F5" keys can be used.

7.4.7 STD Command: Standard Deviations

The STD command computes the statistical standard deviations, the error due to counting statistics, from the last measured sample spectrum for the analyzed (target) elements.

The display format is similar to the concentration output:

STDEVS: FE 1.04 CR .361 CU .142 PB .006

This does not reflect the total error of the measurement (accuracy), but only the part due to counting statistics (precision). Generally, it is a good estimate of the instrument's precision. The statistical error is reduced fifty percent for each quadrupling (multiplying by four) of the measurement time.

7.4.8 Other Commands

Many other commands are available on the X-MET 880 and are confirmed and executed with the "CONT/YES key. Refer to the X-MET 880 Operating Instructions for further information.

7.5 Instrument Calibration

7.5.1 DEL Command: Deleting a Model

If all 32 models have (an) old library(s) or assay model(s) in them, then a model must be deleted before proceeding with a new calibration. Enter DEL followed by the model number (1-32) and confirm the action by pressing the "CONT/YES" key. The X-MET 880 will display the selected model number and it's name and ask if this model is to be deleted. Another "CONT/YES" key response deletes the old model clearing the space for a new model. Therefore, deleting a model with the DEL command means that a new pure element and sample calibration will be required.

Example:

To delete model 6:

>**DEL**	**CONT/YES**
WHICH MODEL TO DELETE (1-32) ? **6**	**CONT/YES**
DELETE MODEL 6 CRFECU CHEM ?	**CONT/YES**
DELETED	
>	

An "END/NO" key response to the above will result in the following display:

NOTHING CHANGED

and the model will not be deleted.

7.5.2 Changing a Model's "Maximum Count Rate" and Default Measuring Time

All uncalibrated models have a default maximum count rate limit of 6 Khz (6000 counts per second) and a default measuring time of 15 seconds. Prior to pure element calibration, the maximum count-rate limit should be increased to 15 Khz. Simultaneously, the model default measuring time can be changed to the anticipated calibration time (and after calibration, to the anticipated field or sample analysis time). These changes are performed with the following procedure.

Unlock the maintenance program by typing in the EMP command, followed by the "CONT/YES" key. This enables the maintenance program. Enter the model parameters section of the maintenance program by typing in the PRM command, (Parameters command) followed by the "CONT/YES" key. Enter the number of the model to be accessed (1-32), or accept the number offered by pressing the "CONT/YES" key. The words "UNCALIBRATED MODEL" will be displayed. Press the "CONT/YES" key. The words "GENERAL PARAMETERS" (the general parameters sub-section of the overall model parameters) will be displayed. This sub-section is accepted by pressing the "CONT/YES" key. If the model has already been assigned a name, the name of the model number entered will appear next and the model can be re-named if desired, if not, press the "CONT/YES" key. The model type will appear at the prompt. Enter "CONT/YES".

Entering "END/NO" at this query will change the type of model to one of the three choices: Identification (IDENT), Assay (ASSAY), or Undefined (UNDEF). The wrong computational algorithm will be applied if the incorrect type of model is entered. Answer "END/NO" to the incorrect model type and "CONT/YES" to the correct model type.

The measuring time will be displayed next. Key in the new default measuring time (performs the same function as using the MTM command) or accept the offered time by pressing the "CONT/YES" key. The display will show "Number of channels: XX". Continue past this with the "CONT/YES" key. The title "Flow Check Channel" will appear at the prompt. Press the "CONT/YES" key. The title "Check Count Rate" will appear at the prompt. Press the "CONT/YES" key. The title "Max Count Rate" will appear at the prompt followed by "6 Khz ?". Press the "END/NO" key. This will scroll the display to the next count rate default of 10 Khz. Press the "END/NO" key and the display will show "15 Khz?". To accept the 15 Khz maximum count rate limit press "CONT/YES". The display will show "Safety Limit". Continue past this with the "CONT/YES" key. The title "CHANNEL PARAMETERS?", which is the next sub-section of the PRM command, will appear at the prompt. Enter the "END/NO" key. The title "G-MATRIX?", the next sub-section of the PRM command, will appear at the prompt. Enter the "END/NO" key. The title "ASSAY/IDENT PARAMETERS?", which is the last sub-section of the PRM command, will appear at the prompt. Enter the "END/NO" key. You have exited the PRM file, which is evidenced by the reappearance of the prompt sign ">" on the display.

To re-lock the parameters file type in the XMP command, followed by the "CONT/YES" key. This inhibits access to the maintenance program.

Example of changing a model's Maximum Count Rate limit and Default Measuring Time:

>**EMP**	**CONT/YES**
>**PRM**	**CONT/YES**
MODEL 5 ?	**CONT/YES**
UNCALIBRATED MODEL	**CONT/YES**
GENERAL PARAMETERS ?	**CONT/YES**
Name: ?	**CONT/YES**

Type: UNDEF ?		**CONT/YES**
Meas. Time: 15?	**200**	**CONT/YES**
No. of channels: 0 ?		**CONT/YES**
Flow check channel: 0 ?		**CONT/YES**
Check count rate: 0.00 ?		**CONT/YES**
Max count rate: 6 Khz ?		**END/NO**
Max count rate: 10 Khz ?		**END/NO**
Max count rate: 15 Khz ?		**CONT/YES**
Safety limit %: 0.00 ?		**CONT/YES**
CHANNEL PARAMETERS ?		**END/NO**

Example (Continued)

G MATRIX ?	**END/NO**
ASSAY/IDENT PARAMETERS ?	**END/NO**

>

7.5.3 PUR Command: Pure Element Calibration

7.5.3.1 Introduction

During pure element calibration, the analyzer determines the locations of the channels which will bracket the energy of the element(s) to be measured, including the backscatter peak. The analyzer also simultaneously calculates the element spectral overlap factors, or the values to be used in the "G-Matrix" table to correct each channel for the influence of adjacent element peaks (in the form of spectral overlap).

The pure element samples for the up to 10 elements chosen for calibration (1 to 9 elements plus backscatter) may be measured in any order, however, it is recommended that the elements be measured in order of their respective X-ray energies to facilitate operator review of the channel limit (LIM command) settings (LIM review follows the PUR command).

The elements selected for an assay model should include all elements to be analyzed plus any elements that might interfere, either by spectral overlap or by matrix interference.

The analyzer stores the spectra measured in a main spectra table and computes the correct channel limits for each element, that are assigned to the specific model. Pulses falling between these limits are included in the channel or "window" of the

respective element. The analyzer calculates spectral overlap factors and stores them in the G-MATRIX. This is the information from the spectra that will be required for the deconvolution calculations. Each spectrum and the respective channel limits are automatically labeled with the element, probe number and source used.

The instrument must be stabilized (powered-up for electrical stabilization and kept in a constant temperature environment for thermal stabilization) for 60 minutes, and allowed to gain control for 5.0 minutes prior to the pure element calibration. The instrument must be allowed to gain control for 5.0 minutes every 25 minutes during pure element calibration. The area in which the instrument is being calibrated in must be thermally stable to within ±3°F. A minimum measurement time of 240 seconds is normally used for pure element calibration.

Pure element spectra are stored in a main spectra storage table to facilitate copying to another model. This gives the operator the option of performing pure element calibration in several models without measuring the pure elements each time. Pure element spectra stored in the main spectrum table should not be used unless they have been acquired and stored within the previous eight hours by the same operator that performed the initial pure element calibration.

The model channel limits can be reviewed and re-set manually after pure element calibration using the LIM command. They can be output to a peripheral device by using P command (print) followed by the "CONT/YES" key while in the LIM Table.

7.5.3.2 Operation

Gain control monitoring must be performed as described in Section 7.1.2. Pure element calibration is started by entering the PUR command followed by the "CONT/YES" key. The instrument asks if you wish to "Delete the old spectra?". This is answered by the "CONT/YES" or "END/NO" key. Answering with the "CONT/YES" key erases all spectra in the library, making copying impossible until new spectra have been generated from pure element measurements.

The X-MET 880 asks for a new model name if the model has not been previously calibrated. It is recommended that the operator include a statement in the model name's title concerning the concentration of the XRF readings (e.g., if the assay model is going to measure 0 -20,000 ppm Pb (mg/kg), the assay values will have to be entered as parts per million by weight (ppmw) or mg/kg/10 since the instrument only has 4 places before and after the decimal point for data entry). To ensure all operators are aware of this, it is recommended that in addition to noting

this fact in the XRF logbook (see Section 19.0), the model name reflects this also (i.e., title: BBATY PPM = XRFX10).

Next, the instrument prompts for the first element and asks for it to be measured. The elements can be named either by their one or two letter element symbol or by their atomic number. The scattering sample has to be named by the symbol BS (or atomic number 0).

For DOPS Probe only

If the DOPS probe is being used, the instrument prompts for the source to be used (either source A or B). The operator should determine the appropriate source for the most efficient excitation of the desired target element(s) (see source selection chart in X-MET 880 Operating Instructions) and select the appropriate source(s) for the target element(s). Source A is the Am-241 source and source B is the Cm-244 source (or the Cd-109 source in the 880EA system). Both sources can be used in a model to analyze different target elements. If a dual source method is used, a total maximum of 10 pure element calibrations may be used in the model for both sources (i.e., an even division of pure elements between two sources could result in 4 elements and a backscatter for Source A and 4 elements and a backscatter for Source B for a total of 10 pure elements in the model). Remember to include all elements that might interfere, either by spectral overlap or by matrix effect in each source pure element calibration.

For example, a site requires the soil analysis of cadmium (Cd) and nickel (Ni). Ni analysis requires the use of the Cm-244 source (B). Cd analysis requires the use of the Am-241 source (A). The operator notes that there is also tin (Sn) present in the soil and this may cause spectral overlap in the Cd window due to the fact that Sn is an adjacent element to Cd. The minimum PUR command calibration elements for source A would include

Cd, Sn, Fe, and BS (backscatter peak). Fe is always included in the PUR calibration of all sources because it is always present in soil. The minimum PUR calibration for source B would include Fe, Ni, and Bs. Note that the Fe and BS must be measured twice, once for each source used. The channel limits for an element cannot be interchanged between sources.

For DOPS or SAPS Probes

Place the pure element sample in position and start the measurement by pressing the "START" key (sample type probe) or by pulling the trigger on the surface probe and holding it open until the measurement is completed. On DOPS probe, make

sure the appropriate source is selected by observing the source window display on the side of the DOPS probe and the display panel on the X-MET 880 electronic unit.

Interrupt any measurements that start with an incorrect source by releasing the trigger and pressing the "END/NO" key. Then re-enter the program with the PUR command and respond to the X-MET 880 prompts until you return to the desired channel number. Type in the desired element symbol and re-start the measurement.

It is recommended that the operator examine each pure element sample prior to use to ensure that the pure element plastic or metallic disc is still fixed in place and has not fallen out.

It is recommended that the elements be measured in order of their respective X-ray energies to facilitate operator review of the channel limit (LIM command) settings (LIM review follows the PUR command).

All of the calibration samples will have to be re-measured, if any pure elements are added, deleted, or re-measured with the PUR command after the calibration samples have been measured using the CAL command.

While the measurement is in progress, the remaining measurement time is continuously displayed. When the measurement is completed, the peak channel and the Full-Width at Half-Maximum (FWHM) of the pure element spectrum is displayed and the instrument requests the next pure element. The PUR calibration can be terminated with the "END/NO" key.

Example of pure element calibration of a new model using a DOPS probe (6), with an Am-241 source (A) and a Cm-244 source (B):

>**PUR**	**CONT/YES**
DELETE OLD SPECTRA ?	**E N D / N O** o r
CONT/YES*	

***(CONT/YES** deletes all existing spectra in main spectral library; maximum = 20)

CALIBRATING A NEW MODEL	
NAME? **PLATE PPM=XRFX10**	**CONT/YES**
1. PURE SAMPLE: **CR**	**CONT/YES**
SOURCE A ?	**END/NO**
SOURCE B ?	**CONT/YES**
MEASURE CR6B	**PULL TRIGGER**

MEASURING: PROBE 6 TYPE DOPS (B) 200 SECONDS - measurement, time is counted down; at conclusion display reads:
PEAK CHANNEL: 69 FWHM: 12 (peak channel & channel width)
2. PURE SAMPLE: **MN** **CONT/YES**
SOURCE A ? **END/NO**
SOURCE B ? **CONT/YES**

MEASURE MN6B **PULL TRIGGER**
MEASURING: PROBE 6 TYPE DOPS (B) 200 SECONDS
PEAK CHANNEL: 75 FWHM: 12
3. PURE SAMPLE: **FE** **CONT/YES**
SOURCE A ? **END/NO**
SOURCE B ? **CONT/YES**
MEASURE FE6B **PULL TRIGGER**
MEASURING: PROBE 6 TYPE DOPS (B) 200 SECONDS
PEAK CHANNEL: 81 FWHM: 12
4. PURE SAMPLE: **BS** **CONT/YES**
(scattering sample)
SOURCE A ? **END/NO**
SOURCE B ? **CONT/YES**
MEASURE BS6B **PULL TRIGGER**
MEASURING: PROBE 6 TYPE DOPS (B) 200 SECONDS
PEAK CHANNEL: 255 FWHM: 1
5. PURE SAMPLE: **END/NO**
CALIBRATION FINISHED
>

7.5.3.3 Multiple Models

Pure element calibration can be performed using the pure element spectra stored in the buffer memory called the spectrum table, which holds 20 complete pure element spectra. The pure element spectra stored in the spectrum table can be copied into any model(s) using the PUR command. Each model can accept up to 10 such spectra. These pure element spectra are accessed during PUR calibration for transfer of the appropriate channel limits data as well as for calculating the appropriate G-Matrix overlap factor corrections. Thereafter, the model no longer needs to refer to the spectrum table. Therefore, the 20 element limitation of the spectrum table does not limit the X-MET 880 to the measurement of only 20 elements. If more than 20 spectra are added, the 21st one will overwrite the first one in the spectra table, and so on, but previously calibrated models will not be affected. If calibrating several models with more than 20 total elements using

PUR, it is advisable to begin with those elements that occur only in the first model(s) to be calibrated as they will be overwritten in the spectrum table, but not in the calibrated model(s). Pure element spectra stored in the main spectrum table should not be used unless they have been acquired and stored within the previous eight hours by the same operator that performed the initial pure element calibration in the spectrum table.

7.5.3.4 LIM Command: Examining and Verifying the Channel Limits

The channel limits are printed out and examined using the LIM command once the pure element calibration has been completed. The LIM command first displays the number of channels and asks if the operator wants to examine the individual channel information. The bottom line (active line) of the X-MET 880 display reads: "EXAMINE ?". Pressing the "END/NO" key exits the LIM command. The operator may use "CONT/YES" key to review the data for the first channel; element symbol, probe and source identification, pure element gross count rate and the normalization coefficient. On the second line, the X-MET 880 displays the channel limits. Element channels are advanced in the forward direction with the "CONT/YES" key and backwards with the "^" (up arrow) key. The P command followed by the "CONT/YES" key gives an output in table form to a printer or a terminal (see Section 5.3).

During scanning, new channel limits can be manually entered in place of the old ones. The count-rate for the new channel limits will then change accordingly. Manual channel limit setting is required in certain situations, such as when setting a channel at an L-beta line, (i.e., when the L-alpha line has an overlap with another element channel (such as the case of As (arsenic) K-alpha overlapped with Pb (lead) L-alpha). The pure element spectra must be in the spectrum table in order to manually change the channel limits.

Before proceeding to the next step, the pure element channel limits must be examined and verified for each source. None of the pure element channel limits should overlap or coincide. If any do, the overlapping pure element spectra should be deleted and remeasured. Additionally, the order of the channel limits must be identical to the corresponding X-ray energy order of the elements measured (low to high measurement order facilitates this review). Note in the example below, that the order of the Cr (chromium), Mn (manganese) and Fe (Iron) channel limits corresponds to the atomic number order and the X-ray energy order of the elements. If the channel limits do not follow this pattern, the anomalous pure element spectra should be deleted and remeasured.

Stepping through channel limits is discontinued with the "END/NO" key. After termination of the command, the instrument pure element calibration data (spectral overlap factors called G-Matrix) is re-calculated on the basis of the new channel limits.

Example of examining the channel limits:

> **LIM** **CONT/YES**
4 channels
EXAMINE ? **CONT/YES**

CR6B 820.17 1.000 63 71 NEW ? **P** **CONT/YES**

P command initiates formatted printout, if printer is connected):
(MODEL 5: PLATE PPM=XRFX10)

ELEM	PROBE	COUNT RATE	NORM.FACTOR	LIMITS
CR	6B	820.17	1.000	63 71
MN	6B	1194.64	1.000	72 77
FE	6B	450.06	1.000	78 86
BS	6B	185.18	1.000	255 255

7.5.4 SPE Command: Examining Spectra

The pure element and sample spectra can be output by the SPE command. The last sample spectrum is output by answering "CONT/YES" to the query "LATEST?" (latest measurement). Pure element spectrum are output by answering "END/NO"

to the query "LATEST?". If "END/NO" is selected, the pure element spectrum can be specified the following three ways:

1. The position number in the spectrum table (1-20).
2. The pure element symbol.
3. The atomic number of the pure element (but only if it is larger than 20; otherwise it is interpreted by the instrument as the position number in table).

If the same pure element has been measured with more than one probe, the program will also prompt for the probe number (and for source A or B if DOPS probe is being used) after entry of the symbol or element number. When the pure element has been specified with its position number or symbol, then the probe number and measurement time will be displayed. Display of the channel numbers and counts of two channels at a time can be obtained by

scanning backwards or forwards. Forward scanning is done with the "CONT/YES" key and backward scanning with the "^" (up arrow) key. Keying in the micro-channel number n (0 to 255) displays the counts of micro-channel n. Scanning is discontinued with the "END/NO" key and the instrument will then ask for a new pure element spectrum. Another "END/NO" at this point will terminate the SPE function.

7.5.5 SPL Command: Spectrum Plot Using a Peripheral Device

A spectrum can be plotted with a printer or on a computer terminal by using the command SPL. The spectrum is chosen as in the SPE command. Thereafter, X-MET 880 will prompt for the first and last channels, the output window settings and the lower and upper limits for the scaling of the counts axis. The output "window" means the number of micro-channels which are integrated and printed as one channel of the plot. If the automatic lower and upper limits are not changed, the plot will be scaled according to the highest peak of the spectrum.

7.5.6 CAL Command: Assay Model Sample Calibration

7.5.6.1 Introduction

When the pure element calibration (PUR command) of the analyzer has been carried out, the X-MET 880 is capable of computing the net counts (intensities) in the element channels. In order to proceed from net intensities to concentrations (assay), sample calibration is required. This entails measuring known samples using the CAL calibrate command (assay readout).

Assay models are used for hazardous waste application models since they provide results in a concentration which can be QA/QC'd. Identification models produce identification names of the sample types by the quality of spectral matchings (i.e., LOW PB LEVEL or HIGH PB LEVEL). Identification models are never used for hazardous waste applications because matching low resolution spectra can result in false qualitative and quantitative results. Additionally, identification models will not be addressed in this operating procedure.

For calibration of an assay model, the samples are measured using the CAL command, and the assay model is calculated using X-MET 880 internal software by multi-variable regression analysis. The calculation can be performed either internally, using the analyzer software (with a maximum of 30 calibration samples), or, externally in which case there is no restriction on the number of samples used. This operating procedure will only address internal calculations since ERT/REAC does not have external software.

7.5.6.2 Measurement of Calibration Samples

The instrument must be stabilized (turned-on and warmed- up for 60 minutes) and allowed to gain control for 5.0 minutes prior to the measurement of calibration samples. The area in which the instrument is being calibrated should be thermally stable (maintain ±3°F). A measurement time of 200 seconds or more should be used for measurement of calibration samples. The instrument must be allowed to gain control for 5.0 minutes after measuring every 6th calibration sample, or every 25 minutes, during the CAL sample measurements. Gain control monitoring must be performed as described in Section 7.1.2.

In the sample calibration of an assay model, the X-MET 880 stores the intensities of the calibration samples in a calibration intensities table, from which they can be output using the CIN command, or used directly for the generation of a regression model by the internal regression program of the X-MET 880 (MOD calibration modeling command). The calibration intensities table is an electronic scratch-pad that is erased after each model is completed and then moved to the next model to be calibrated. This table can be used for the calculation of only one model at a time. The maximum size of the calibration intensities table is 30 samples, hence, if a greater number of samples is required, the intensities must be output for external calculation of the regression model. The calibration samples can be measured in an arbitrary order, AS LONG AS THE ORDER IS DOCUMENTED. Therefore, be sure to make a note of the order used and document it in the operator's log book. The calibration intensities table stores the samples by cardinal number (1-30) only, in the order they were measured. The function is started with the CAL command. The X-MET 880 begins by displaying the status of the calibration table:

- NO OLD DATA
- OLD DATA: 30 SAMPLES (TABLE FULL) or
- OLD DATA: n SAMPLES, MODEL XX OVERWRITE?

If the OVERWRITE? question appears, it is reminding the operator to finish the modeling calculations using the calibration intensities table measured in an earlier model. If the earlier model is already completed (modeling is finished using the MOD command) and the calibration coefficients are already calculated, then the "CONT/YES" key should be pressed, to erase the old model scratchpad and move it to the current model. If the earlier model is not completed, then the "END/NO" key should be pressed and calibration calculations finished in the earlier model. NOTE: if "CONT/YES" is pressed only new samples can be measured and the old calibration intensities table is erased.

The X-MET 880 asks for one sample at a time:

MEASURE SAMPLE n

This can be answered in any of the following ways:

Measure the sample	**PULL TRIGGER**
Proceed to Sample m	Enter sample number **m,**
followed by:	**CONT/YES**
Exit from Sample query	**END/NO**

Repeat measurement of a particular sample can be accomplished by means of the sample number. After pressing "END/NO", the last sample to be stored is verified.

LAST SAMPLE n ?

This can be answered with a smaller number if it is desired to omit some of the samples. If the table becomes full, the message TABLE FULL is displayed and the command is terminated.

7.5.6.3 Sample Calibration of a Model Using Both Sources in a DOPS Probe

A given analysis may require the use of both sources in a DOPS probe to cover all the elements to be measured. A single model employing both sources in the DOPS probe can be calibrated as follows:

Every element to be measured by each source must undergo independent pure element calibration for each source. For example:

If BS is going to be used in the calibration of both sources A & B, then the BS sample must be measured by both sources A & B during the pure element calibration (PUR) .

If the model's pure element calibration channel limits contain limits for both sources, then during sample calibration the DOPS probe will automatically switch between sources, as required, to measure the spectra for both sources, and store the intensities in the respective source's element channels. Be sure to continue to hold the trigger open after the 1st source measurement. The X-MET 880 will indicate the completion of the first of the two measurements with a "beep-beep-beep". This is immediately followed by a clicking noise that is the sound of the source changer switching sources. Do not release the trigger until the 2nd source has completed its measurement.

During measurements, always verify that the appropriate source is selected by observing the display on the side of the DOPS probe. Interrupt and end ("END/NO") any measurements started with an incorrect source.

7.5.6.4 Deletion of the Calibration Intensities Table

The calibration intensities table can only be deleted by going to a different model to obtain the "OVERWRITE" message. The operator can then either go back to the previous model or stay with the new model to start a new calibration intensities table.

Example:

>**MODEL**		**CONT/YES**
MODEL 4 ?		**CONT/YES**
>**CAL**		**CONT/YES**
OLD DATA: 11 SAMPLES, MODEL 2 (means previous calibration is in Model 2)		
OVERWRITE?		**CONT/YES**
MEASURE SAMPLE 1		**PULL TRIGGER**
MEASURING: PROBE 6 TYPE DOPS (B)		
200 SECONDS		
MEASURE SAMPLE 9		**END/NO**
LAST SAMPLE 8?		**CONT/YES**
8 SAMPLES		
>		

If it is discovered that after measuring eight samples, sample # 4 was incorrect, then measuring the correct sample will automatically overwrite the previous # 4 sample data in the table.

>**CAL**		**CONT/YES**
OLD DATA:		8 SAMPLES
MEASURE SAMPLE 9	**4**	**CONT/YES**
MEASURE SAMPLE 4		**PULL TRIGGER**
MEASURING: PROBE 6 TYPE DOPS (B)		
200 SECONDS		
MEASURE SAMPLE 5		**END/NO**
LAST SAMPLE 8 ?		**CONT/YES**
8 SAMPLES		
>		

7.5.7 ASY Command: Input of Calibration Sample Assay Values

The chemical metal analysis (assay) results of the SSCS are entered using the ASY command. The X-MET 880 starts by prompting for the target elements for which concentrations are required (these are known as the DEPENDENTS). This is answered by entering the element symbols (XX) of all the desired elements for readout (up to six) using the "SPACE" key in between the symbols, but not following the last entry. To enter the element symbols typed onto the display press "CONT/YES". The instrument will then start asking for each sample assay value to be entered. Type in each assay value for each sample followed by "CONT/YES", until all the calibration samples assay values have been entered. The concentrations can be changed afterwards if required by stepping backward with the "^" (up-arrow). If concentration values for newly measured samples, or additional elements, are desired to be added later, then re-enter the ASY command and press "CONT/YES" until the first assay value is on the lower line of the display. Next, type a (/) followed by the sample number you desire to jump to. The use of a (/) mark in front of the sample number advances the assay table directly to the sample number entered. From any location in the assay table, P followed by "CONT/YES" gives output in tabular form to a printer or a terminal.

CAUTION: Do not enter a P "CONT/YES" command while in the first section of the ASY command, the entry of DEPENDENTS, as in this section a P will be interpreted as the element phosphorus. Should this be done accidently, the instrument will ask for re-verification that the element P (phosphorus) is desired in place of the previously selected element(s). At this point the operator would simply re-enter the correct element symbols prior to pressing "CONT/YES" which accepts the offered element (P). IF the symbol P is inadvertently accepted as the element, by acknowledging it with "CONT/YES" twice, it will then be necessary to re-enter ASY and the correct elements symbols and re-install all the assay values.

NOTE: Only assay values with less than four significant figures in front of the decimal point can be entered into this table. Therefore, if any samples contain assay values with greater than 4 significant figures in front of the decimal point ALL assay values for all elements in the suite should be divided by some power of 10 (such as 10, 100, 1000, or 10,000) prior to entry into the assay table. For example, one sample in the suite is assayed at 33,999 mg/kg. The decimal fraction resulting from the division of 10 would convert this value to 3,399.9. Therefore, the value 3,399.9 would be entered into the assay table and all other element assays in the calibration suite would likewise be divided by 10 prior to entry into the assay table.

Example 1: Entering Cr and Cu assays, for eight calibration samples that have been measured using the CAL command.

>**_ASY**		**CONT/YES**
OLD DEPENDENTS: NO OLD DEPENDENTS		**CONT/YES**
NEW: **CR CU**		**CONT/YES**
NEW DEPENDENTS?: CR CU		**CONT/YES**
1. SAMPLE CR: 0.0000 ?	**10.97**	**CONT/YES**
1. SAMPLE CR: 10.9700		
1. SAMPLE CU: 0.0000 ?	**34.23**	**CONT/YES**
1. SAMPLE CU: 34.2300		
2. SAMPLE CR: 0.0000 ?	**8.56**	**CONT/YES**
2. SAMPLE CR: 8.5600		
2. SAMPLE CU: 0.0000 ?	**42.29**	**CONT/YES**
....		
8. SAMPLE CR: 0.0000	**22.39**	**CONT/YES**
8. SAMPLE CR: 22.3900		
8. SAMPLE CU: 0.0000 ?	**15.00**	**CONT/YES**
(Terminates assay command automatically)		
8*2 ASSAYS		
>		

Example 2: Changing dependents from the old ones Cr, Cu, to new: Cr, Cu and Fe. NOTE: This will not erase the previous assay table data, but will open up space to add Fe data.

>**ASY**		**CONT/YES**
OLD DEPENDENTS: CR CU		
NEW ? **CR CU FE**		**CONT/YES**
1. SAMPLE CR: 10.9700 ?		**CONT/YES**
1. SAMPLE CR: 10.9700		
1. SAMPLE CU: 34.2300?		**CONT/YES**
1. SAMPLE CU: 34.2300		
1. SAMPLE FE: 0.0000 ?	**40.2**	**CONT/YES**
1. SAMPLE FE: 40.2000		
2. SAMPLE CR: 34.2300?		**CONT/YES**
2. SAMPLE CR: 34.2300		
....		
8. SAMPLE CR: 22.3900		**CONT/YES**
8. SAMPLE CU: 15.0000 ?		**CONT/YES**
8. SAMPLE FE: 0.0000 ?	**51.00**	**CONT/YES**
(Terminates assay command automatically)		
8*3 ASSAYS		
>		

7.5.8 MOD Command: Generating the Regression Model

The MOD command of the X-MET 880 initiates the program for calculation of multi-variable regression model coefficients. The program offers regression calculations for the DEPENDENTS (assay values) given in the ASY command (i.e., those elements whose symbols were entered as DEPENDENTS).

7.5.8.1 Setting the Dependents

>MOD	**CONT/YES**
REGRESSION FOR CR ?	**END/NO**
REGRESSION FOR FE ?	**CONT/YES**

Upon an affirmative answer ("CONT/YES"), the INDEPENDENTS (elements' intensities) for the element(s) in question are requested: i.e., those elements that may affect the slope of the dependent element are entered with the element symbol, to be included in the regression calculation.

7.5.8.2 Setting the Independents

DEFINE INDEPENDENTS:
Stop indep input by END-key

1. indep: ?	**FE**	**CONT/YES**
2. indep: ?	**CR**	**CONT/YES**
3. indep: ?		**END/NO**

At this stage it is possible to scan forward with the "CONT/YES" command and backwards with the "^" (up arrow) command: "-" deletes an independent. "END/NO" terminates input, computes the regression model and displays the quality-of-fit of the regression line.

The simplest independent expressions are recommended at first. The element can be specified either by its symbol or its element number. The other permissible forms of expression are as follows using iron, chromium and backscatter as examples:

FE	
FE/ or FE/BS	BS = scatter intensity
FE*CR	
FE*CR/	means FE*CR/(BS*BS)
BS/	means 1/BS
FE*BS/	means FE/(BS*BS)
BS*BS	means 1/(BS*BS)

If independents have been entered incorrectly, i.e., using elements that are not included in the instrument calibration, the X-MET 880 responds by beeping and then listing the possible elements for inclusion as independents (only those that were included in the pure element calibration) and then displays a repetition of the query:

"beep"
USE CR FE CU
1. indep:

7.5.8.3 Reviewing the Regression Fit Parameters

After defining the independents, "END/NO" terminates the independent input and calculates the internal regression fit. The figure-of-merit parameters are then displayed:

R	= correlation coefficient
S	= standard deviation around regression line
F (M,N)	= F-test value of regression with degrees of freedom M,N
M	= number of independents
N	= number of samples M-1

Example:

R=0.975 S=1.55 F=(2.5)=4.25
RESIDUALS ?

7.5.8.4 Examining the Residuals Table

The X-MET 880 then asks if it is desired to examine the residuals (i.e., the comparison of concentrations calculated by the model with the assay values entered). If these are not required, reply "END/NO"; otherwise "CONT/YES" leads to scanning of the residuals. The notation is as follows:

ASSAY = concentration entered (from the AA, ICP or referee analysis)
ESTIM. = concentration calculated with the model
RESID. = ASSAY - ESTIM
ST.RES = RESID/standard deviation S

Example:

NO	ASSAY	ESTIM	RESID	ST.RES
1	30.300	29.875	0.425	0.274

Forward scanning is done with the "CONT/YES" command, backward scanning with the "^" (up-arrow) command; entering the number of a calibration sample followed by "CONT/YES" leads directly to that sample in a table. P "CONT/YES" sends the output, in table form, to the printer or other peripheral device, D "CONT/YES" sends a calibration Data plot diagram to the printer or other peripheral device.

"END/NO" terminates the scanning.

7.5.8.5 Deleting Points

The scanning of residuals is followed by the question:

DELETE POINTS:

If it is required to delete samples from the regression model calculation, enter the numbers of such samples, i.e., 3 SPACE 5 and press "CONT/YES". This will calculate a new regression model and display the new figure-of- merit data for this new regression model. This display will be followed by the RESIDUALS ? query. A "CONT/YES" response leads to scanning the new residual table (using the new regression model calculated without the deleted points). This table may be scanned also. "END/NO" terminates the scanning and returns to the DELETE POINTS: prompt. Additional points may be deleted at this time.

NOTE: If the new residual table is scanned forward using the "CONT/YES" command and backward using the "^" (up arrow) command, the deleted point(s) will simply be missing in the residual table.

If the new residual table is output in tabular form using the P "CONT/YES" command, then the calibration table knows the samples by cardinal number (1-30) only. The residual table's sample numbers will change for points with cardinal numbers larger than those which are deleted. For example: Using a calibration table with 20 samples, an operator deletes point number 5 and returns to the residual table. The new residual table will contain points 1-19. Points 1-4 in the "output" new residual table will correspond to the cardinal numbers 1-4 in the calibration table. Points 5-19 correspond to the original calibration cardinal numbers 6-20. Therefore, if the operator wants to delete sample number 9 in the "output" residual table, sample (calibration cardinal) number 10 must be entered to the DELETE POINTS: prompt after terminating the residual scanning.

The next new residual table will contain residuals for points 1-18 now. Points 1-4 in this new residual table correspond to the calibration cardinal numbers 1-4. Points 5-8 in the new residual table correspond to calibration cardinal numbers 6-9. Points 10-18 in the new residual table correspond to calibration cardinal numbers 11-20.

This loop will continue until the reply to the DELETE POINTS: prompt is "CONT/YES" or "END/NO". The X-MET 880 response to an incorrect input is "GIVE POINT NUMBERS".

If the residuals are not examined, or the response to the "DELETE POINTS" prompt is "CONT/YES" or "END/NO", the next question is COEFFICIENTS AND T VALUES?.

7.5.8.6 Examining Coefficients and T Values

To the question:

COEFFICIENTS AND T-VALUES ?

a response of "CONT/YES" will allow you to scan the coefficients and T-Values for each independent used in the regression model. Forward scanning is done with the "CONT/YES" command, backward scanning with the "^" (up-arrow) command.

INTERCEPT	= 1.23 E-4		
SLOPE 1	= 3.827E-2	T = 15.60	**CONT/YES**
SLOPE 2	=-1.00837	T = -9.86	

The slope and t-value for slope 1 correspond to the first independent (element). The slope and t-value for slope 2 correspond to the second independent, etc.

"END/NO" terminates the scanning. **P "CONT/YES"** gives output in table form:

COEFFICIENTS AND T-VALUES ?
CONT/YES
INTERCEPT = -1.1433235E+1
SLOPE 1 = 7.6707910E+2 T = 83.40 **P CONT/YES**

COEFFICIENTS AND T-VALUES FOR *NI*

	ITC	NI/	NI*FE
SL	-11.433235	767.079104	-0.000234
T VALUE	83.401736	-7.457542	

7.5.8.7 Reiterating the Model or Exiting

After terminating the scanning with the "END/NO" command, the query CHANGE INDEPENDENTS? is displayed. If the model created for the element does not require changing, reply "END/NO". This will terminate the regression for the dependent (element) you have been working on. You will be asked if you want to enter the next dependent regression ("CONT/YES" if yes) or you will pass it and go to the next element (END/NO).

If the reply "CONT/YES" is entered, all points deleted at previous stages will be reinstated, and the dependent regression returns to the independents query:

Example: 1. indep: FE ?

In this way, it is possible to stay in a loop (in a dependent regression), reiterating the model with new independents, computing a new regression, possibly deleting points and computing a new regression again, until the best possible regression model is found (see Section 14.2).

When all elements in the model are finished, exit the MOD program with "END/NO" which returns the display to the prompt (>_).

7.5.9 Model Optimization Methodology

7.5.9.1 Basic Theory

The X-MET 880 uses the so-called empirical calibration approach, that is, the calibration equations are developed based on intensities measured on known calibration samples. The general calibration formula is patterned after the intensity correction model of Lucas-Tooth and Price. The generalized calibration equation for the analyte i in a sample can be written as follows:

$$C_i = I_i*(K_i + \underset{j=1}{SUM}(K_{ij}*I_j)) + B_i + \underset{j\neq i}{SUM}(B_{ij}*I_j)$$

Terms: 1 2 3 4

The model assumes that the analyte concentration is a function of the intensities from elements in the sample. While this model is limited to relatively narrow

concentration ranges, it has an important advantage that only the analyte need be chemically assayed in order to develop it.

If no matrix effects are present in the sample, the calibration equation would have only part 1 and 3, and would be a simple straight line equation.

The second term modifies the slope of the calibration line, according to the amounts of other elements present in the sample, as determined by their intensities. This correction is needed when the matrix element is close in energy to the analyte, so that it strongly absorbs or excites its radiation (matrix interference). This slope modification is also useful when the matrix element varies over such a large concentration range that it significantly changes the effective matrix absorption of the incident or emitted x-rays. The square term, $I_i * I_i$, would be the correction for self-absorption.

The fourth term reflects the change in background intensity under the analyte peak. This term is significant if matrix element(s) vary enough to alter the matrix scattering and its power to change the general shape of the background, or more commonly, if the matrix element has a spectral peak that overlaps the analyte peak. The overlapping peak may also be an escape peak. This situation often arises in alloy analysis where strong peaks of iron, chromium or nickel can produce escape peaks (2.9 keV), which are to the low energy side of their fluorescent peaks (caused by argon filling).

7.5.9.2 Parameters Used in Regression Modeling

R - COEFFICIENT OF CORRELATION. This calculation tells how well the given calibration equation explains the variation of data. Its value varies between 0 and 1. The closer its value is to 1.000 (i.e., a one-to- one correlation between the calculated element assay values and the corresponding element intensity values), the better the calibration model is. It should have a value greater than .65 to be meaningful. The R value is a function of the number of independents, the number of data points, and usually increases as those numbers increase.

NOTE: The R coefficient alone, however good, cannot be the only basis of determining the quality or goodness of the model. Often in the case of too few data points and/or over-complicating the calibration equation (too many independents), one can get a "perfect" R = 1.000, which may be meaningless.

S - STANDARD ERROR OF THE FIT. Also known under the term "Sum of Squares for Error" (SSE), or "Root Mean Square Error" (RMS). This is one standard deviation of the spread of data points around the fitted calibration line. The greater its value (error), the more scattered the data points. If two lines are drawn parallel to the calibration line at plus S and minus S distance from it, the band created around the calibration line will contain about 68% of all the data. The value of S is expressed in the units of the dependent variable being fit (generally concentration units as in the case - mg/kg of soil). During the modeling one should try to minimize this parameter. It should be noted that if the dependent mg/kg assay values are divided by 10 or 100 before being entered into the assay (ASY) table, the S value displayed must be multiplied by the same factor to get mg/kg values.

F - TEST VALUE. This is a statistic which is expressed as the ratio of the sum of squares explained by the given regression model, to the sum of squares not explained by the model (our familiar parameter S). If its value is low, say 5 to 10, then the model is not reliable, and may not be stable if a different data set is used for its calibration. Alternatively, we might say that a low F value indicates the inability of a proposed regression model to predict the correlation between concentrations and intensities of the element being fit. Assuming that an average calibration set has 10 samples, the value of the F statistics should not be smaller than 10 for us to say with 95% confidence that the given model is a valid one. F is a function of the number of data points, the number of independents and S. It usually decreases if the number of variables increases. It is a dimensionless number.

T - TEST VALUE. This statistic is a test of significance for the calculated slope. It is expressed as the ratio of the slope value to the estimate of error of that slope. For example, a T-value equal to 10, means that the error on the estimate of the slope is one tenth (or 10%) of the slope itself, which is significant. For an average case of ten calibration samples the absolute value of T should not be smaller than 2.5 for us to say with 95% confidence that the given slope is statistically valid. Therefore, the rule of thumb would be to eliminate from the model all independents featuring a T value less than |2.5| (±2.5). The T test is a dimensionless number and has the same sign as its slope.

STDEV - STANDARD DEVIATION DUE TO COUNTING STATISTICS. This is the error caused by the random nature of radioactive decay and counting statistics. It is, by definition, equal to the square root of the total number of counts accumulated within a given period of time. Alternatively, standard deviation of intensity I is equal to the square root of intensity I, divided by the square root of the time the intensity was measured for. It is seen that by extending the measurement

time by a factor of 4, one can reduce the standard deviation of counting (error) by a factor of 2. This error is the only one easily controlled. It should be noted that if the dependent mg/kg assay values are divided by 10 or 100 prior to entry into the assay (ASY) table, the STDEV value displayed must be multiplied by the same factor to get mg/kg values.

7.5.9.3 Iterative Process of Building the Model

STEP 1

Always start with the intensity of the element of interest as the first independent variable. Next, depending on the nature of the sample matrix, try the intensity of the analyte ratioed to the intensity of the backscattered (BS) radiation (XX/BS). This usually works for sulfur in oil, chlorine in oil, metals in soil, or any element in a light (or highly scattering) matrix. If the analyte range is relatively wide (such as several percent), try the square term of the analyte intensity (XX*XX)-(example: Pb in soil in the range of 0 to 10%). For each regression note the values of R, S, and F. The focus should be to MINIMIZE S as this parameter is closely related to the accuracy of the method. It can be shown mathematically that R and F are functions of S.

Therefore, by concentrating on S we usually drive R and F in the desired direction, that is high. Save the regression which yields the smaller S. However, keep in mind that two values of S, for example one of .12 and another of .11, ARE FOR ALL PRACTICAL PURPOSES THE SAME. The difference between the S's should be at least about 20% to be a criterion for selection. IF the S's are similar then select the model with the higher F and/or the higher R.

Examine the table of residuals. Make note of the points which are clearly off. The last column of the table tells how many S's below or above (+/-) the curve the given point is. About 68% of the data points should have a STRES (last column) value smaller than the absolute 1, 95% smaller than the absolute 2, and 99.9% (for practical reasons all points) smaller than the absolute 3.

Here is a worked example:

Total number of data points is 17 (17 samples). Therefore, about 12 points (.68*17 = 11.56 or 12 points) should have a STRES (the last column) value between -1.0 and +1.0.

About 16 points (.95*17 = 16.15 or 16 points) should have a STRES value between -2.0 and +2.0. This also means that four points may fall between -2.0 and -1.0 and between +1.0 and +2.0.

At the most, only one data point should have an absolute value between 2.0 and 3.0.

The number of points in each band may vary at least by one from the predicted number due to randomness.

If any point is distant by more than ±(3S) from the line, it becomes a candidate for deletion. However, the reason for deviation should be established first. Often it is the incorrect entry of the referee assay (ASY command) for this point. Sometimes it just may be a bad measurement which can be easily corrected by remeasuring the sample(s). It may also be a case of the (outlying) sample being incompatible (i.e., different matrix) with the rest of the set. If all available information indicates that none of the above applies, then it is probably best to delete the point from the regression. If deleting the "outlier" results in significant improvement of the model, then usually it is justified.

Often all data points will be within the ±(3S) band, but more of them than expected will exhibit large deviations from the line. In most cases this is caused by incompleteness of the model. Examining the composition of those samples which deviate too much may reveal that those particular samples contain significant amounts of some other element(s) which was not yet accounted for in the model. This situation provides a hint as to which independent to include in the next round of the modeling process. Examine the table of residuals and the plot of the data. Is any point, or group of points, indicating curvature of the calibration line which could be corrected by including in the model a square term (XX•XX)?

STEP 2

Now is the time to add to our first independent term the next one. It may already be obvious what to do from the examination of the table of residuals as mentioned above in STEP 1. However, there are some guidelines to keep in mind. The matrix element with the highest concentration should be examined, by including its intensity in the model as the next variable. If that element happens to be close in energy to the analyte peak, then the non-linear term in the form of the product of the analyte and interfering element intensities (XX*YY) may prove to be helpful. Check the S, R, and F values again. If the S does not improve, the independent

should be rejected and another one tried. To assess progress in reducing the S parameter, it is convenient to use the ratios of S's and F's from the two models. If one model is better than the other one then:

$F_{better}/F_{worse} > (S_{worse}/S_{better})^2$,

where "better" refers to the smaller S value.

Examine the table of residuals and the plot of the data. Examine the t test values of the slopes. Reject independent(s) which show T smaller than +/-2.5.

Exit the modeling and enter the STD command. The X-MET 880 should still have in its buffer memory the spectrum of the last measured calibration sample. Therefore, it should output on the display the values of the standard deviations due to the counting statistics for all of the already modeled elements. Check and make a note of those STD's. THEY CANNOT BE LARGER THAN THE S ERROR(S) OBTAINED DURING MODELING. If they are larger, then the model is over-corrected and should be changed by rejecting the variable (independent) with the lowest t value. The S error, by definition, includes the standard deviation, and cannot (be smaller than any of its components.

Continue the iterative process of modeling by repeating STEP 2 until no further improvement seems feasible.

7.5.9.4 Concluding Remarks

Examination of the table of residuals during the iterative process is very important. During the examination of the table of residuals, keep the following in mind:

1. The results displayed in the table are in the same concentration as the values entered into the assay (ASY) table (i.e., values entered in the assay table were mg/kg/10. Therefore, the values in the table of residuals must be multiplied by 10 to get mg/kg.

2. A calibration based on several low concentration SSCS and one or two high concentration SSCS will report a very low S value. This low S value may be meaningless with respect to the analytical accuracy of samples with concentrations between the low and high SSCS. The possibility of large analytical errors for samples in this concentration range should be brought to the attention of the task leader or project manager prior to mobilization if SSCS cannot be obtained for this concentration range.

3. Development of a model with SSCS concentrations greater than one or two percent will generally result in increased analytical error in the region of the detection limit and the site action level (500 to 2000 mg/kg). Development of a low (DL to 1 or 2 percent concentration) and a high (one to ten percent concentration) concentration models usually solves this problem. The recalculation function (see "RECALC" Key Section) can be used to recalculate concentrations from spectra generated in the other model.

4. Pay particular attention to the SSCS with assay values in the critical analytical region. The critical analytical region is located between the DL and four or five times the site action level. The goal of the operator should be to bring the estimated values off the SSCS in the region as close to their assay values as possible. Ideally, half of these SSCS should have estimated values higher then their assay values and half should have estimated values lower then their assay values. Select the model with the majority of the SSCS estimated values above their assay values if an even distribution is impossible. The goal is to provide the most accurate analysis in the critical analytical region because this is where managers are going to make removal/remedial/health and safety/sampling decisions. Additionally, high-biased or false positive data is typically preferred over low-biased or false negative data.

   A higher S value and an independent with a|T| value <2.5 can be tolerated if the model provides improved accuracy in the critical analytical region. Additionally, SSCS in the critical analytical region should be deleted only if there is a large number of SSCS in the region and the dilution of the point provides significant improvement (greater than 20 percent) in the estimated values of the SSCS in the region.

   Select as the final model the one with the best (smallest) S and the best (most accurate) estimated values for SSCS in the critical analytical region. If two models have a very similar S, select the one with greater value of F. If by including in the model one more independent(s), only a small improvement of S is obtained, consider keeping the model with the larger S but with smaller STD value. In most cases, the smaller the number of independents in the model, the better (smaller error) the STD value.

   Use common sense. Remember that simpler is better. It is always easier to correct or knowingly change the model which has the smaller number of parameters. Do not over-correct. If the number of calibration samples is too small, it is just not possible to develop a good model. The rule of thumb is that if you fit the equation with k independents, the number of

samples in the model should be at least twice as much, that is N>=2•K. If the calibration set has only 6 samples and you fit the calibration equation with 5 terms (independents), then you will get a "perfect fit" with R=1.000, S=0.000 and F=****. However, this "forced fit" will yield a calibration equation which will not work in practice, as it is not mathematically representing the real-life scenario. This is a case of over-defining the model on the grounds of statistical analysis.

As a rule, do not bother to include as independents those intensities whose element concentrations in samples are very small compared to the concentration range of the analyte.

There are some exceptions from the rules we tried to spell out above. One of the most significant exceptions is the calibration for an element which is very strongly affected by the presence of all matrix elements. A classical example is the analysis of phosphorus in phosphate rock. Phosphorus x-rays are absorbed by calcium, silica, potassium, sulfur, all of which may be present in the sample in quantities that call for each to be included in the calibration. Usually, for that calibration the t values of all of the slopes are quite marginal. However, none of the variables can be rejected without significant degradation of the model parameters.

When testing the final model for accuracy, keep in mind when comparing the measured values to the given ones (X-MET 880 computed), that if the difference seems too large, perhaps the measured sample was also off during the calibration and modeling. THEREFORE, COMPARE THE MEASURED RESULT WITH THE VALUE OBTAINED DURING MODELING (in the table of residuals) RATHER THAN WITH THE GIVEN ONE. If the difference between the S value and the STD value (for the same measurement time) is significant, then the S error should be used as the criterion for judging any discrepancies between the measured and the given values. This applies if S is about twice the corresponding STD.

7.5.10 Verifying the Accuracy of the Regression Model

The Regression Model should be verified whenever possible, prior to sample analysis, to ensure that everything was done properly. Ideally, all SSCS used (not deleted) in the model should be measured by the model to verify its accuracy. As a minimum, one low, mid and high SSCS should be used to verify the model as described in Section 7.2. Read Section 9.0 on recommended Quality Assurance and Control methods.

7.5.11 Documenting the Regression Model

After the regression model has been completed for all the dependents the results should be captured to a peripheral device such as a printer or a PC floppy disk using ProComm+, or other communication software (see Section 5.3).

8.0 CALCULATIONS

The X-MET 880 is a direct readout instrument. The element concentrations displayed in the readout are identical to the assay value concentration entered during instrument calibration (see Section 7.5.7).

9.0 QUALITY ASSURANCE/QUALITY CONTROL

9.1 Precision

The precision of the method is monitored by reading the low SSCS selected as described in Section 7.2 at the start and end of sample analysis and after approximately every tenth sample. The low concentration sample is analyzed by the instrument for the normal field analysis time, and the results are recorded in a log book. The standard deviation for each dependent element is calculated (using the N-1 formula).

9.1.1 Preliminary Detection Limit (DL) and Quantitation Limit (QL)

A preliminary DL and QL is needed to give the operator an indication of the instruments capability out in the field. A low SSCS sample is selected as described in Section 7.2. Models with multiple dependent elements may require the use of more than one standard to obtain low concentration values for each element.

The sample is measured ten times without moving it, using the anticipated field analysis measuring time. The standard deviation of the mean for each dependent element is calculated (using the N-1 formula).

If the standard deviation has a fractional component, round up to the next whole number prior to calculating the DL and QL.

The definition of the DL is three times the calculated standard deviation value.

The definition of the QL is 10 times the calculated standard deviation value.

9.1.2 Field Detection Limit (FDL) and Field Limit of Quantitation (FLOQ)

The precision of the method is monitored in the field by reading the low SSCS selected as described in Section 7.2 at the start and end of sample analysis and after approximately every tenth sample. The low concentration sample is analyzed by the instrument for the normal

field analysis time, and the results are recorded in a log book. The standard deviation for each dependent element is calculated (using the N-1 formula) using the measurements from the entire sampling period.

If the standard deviation has fractional component, round up to the next whole number prior to calculating the FDL and FLOQ.

The definition of the FDL is three times the calculated standard deviation value.

The definition of the FLOQ is 10 times the calculated standard deviation value.

9.2 Reporting Results

All raw XRF data should be reported including the individual results of multiple analyses of samples and sampling points. The average and standard deviation (using the N-1 formula) of each multiple analysis should also be reported.

A "reported" value for each analysis or average of multiple analyses should be messaged in the following manner.

1. First round the value to the same degree of significance contained in the SSCS sample assay values (usually 2).

2. All values less than or equal to the FDL are reported not detected (ND).

3. All values greater than the FDL and less than or equal to the FLOQ are flagged (usually with a "J" next to the reported value) and noted as such.

4. All values above the FLOQ and within the linear calibration range are reported as is.

5. All values above the linear calibration range (greater than the highest SSCS used in the model) are flagged (usually with a "*" next to the reported value) and noted as such.

9.3 Accuracy

The accuracy of the method is monitored and verified by sending an XRF analyzed sample or sample cup out for AA or ICP analysis at an independent laboratory.

Although AA and ICP are generally recognized as having good accuracy and precision over the concentration range typical of metals contamination in soil, it is most important to recognize the possibility of real differences in the composition of samples sent in for comparative analysis, due to heterogeneity of the soil. It is recommended that the prepared sample cups be sent in for analysis if the samples were prepared in this manner.

Another very important source of potential difference between XRF and AA or ICP results is incomplete digestion of the leaching technique. Since XRF is a total elemental technique, any comparison with referee results must account for the possibility of variable extraction depending upon the extraction method used and its ability to dissolve the mineral form in question.

9.3.1 Additional QA/QC

Additional QA/QC plans may call for monitoring for potential instrumental drift by measuring a mid-range calibration sample at regular intervals (such as every 20th sample) in order to validate the previous measurements. The use of such measures insures that the instrument is continuing to provide the same level of accuracy throughout the entire series of samples at a given site. Should, for example a cold front blow through during a series of measurements, it is required that the X-MET 880 be allowed a gain control period of five minutes to compensate for temperature effects on the detector gain. The need for such a gain control cycle would become apparent based on any inaccuracies noted during the QA/QC sample tests.

9.3.2 Matrix Considerations

Other types of QA/QC verification should include verification that the instrument calibration is appropriate for the specific site to be assessed. This should include verification of the potentially multiple soil matrix types that may exist at a site. Matrix variations that affect the XRF include large variations in calcium content, such as may be encountered when going from siliceous to calcareous soils, as well as variations in iron content.

10.0 DATA VALIDATION

10.1 Active Calibration Model Documentation Method

Immediately following a calibration, the Calibration Intensities Table and the Assay Table are both active; all model information can be captured at this point. Once a new model is started these tables are overwritten and can no longer be documented. In this case, go directly to the Alternate Documentation Method described in Section 10.2.

If using a PC, first set the program to capture to disk. Enter the LIM command. Entering P "CONT/YES" will output the channel limits in table form to the peripheral device for capture.

Enter the "CIN" command next. Entering P "CONT/YES" will output the calibration intensities from the sample calibration in table form to the peripheral device for capture.

Enter the "ASY" command next. Entering P "CONT/YES" after the first sample will output the assay values entered, in table form, to the peripheral device for capture.

Enter the "MOD" command next. For each regression modeled:

1. Scroll down through all independents used with "CONT/YES" and then exit with the "END/NO" command.

2. Press "CONT/YES" to enter the Residuals Table.

3. Re-delete the points deleted and print out the residual table by entering P "CONT/YES". Next enter D "CONT/YES" to capture the calibration plot.

4. Press "END/NO" to exit the Residuals Table, and "END/NO" again to pass the "Delete Points:" query and step to the COEFFICIENTS AND T VALUES.

5. Enter "CONT/YES" to enter the Coefficients and T values table and then P "CONT/YES" to capture the table.

6. Enter "END/NO" to exit the coefficients and T values table and display the question CHANGE INDEPENDENTS?.

7. Enter "END/NO" to go to the next DEPENDENT and repeat steps a - g.

After capturing the above calibration information, print a copy (if captured to floppy disk) and make a photocopy (if captured directly to a thermal printer, the thermal paper will fade after a few months) and staple the copy into the XRF log book for future reference. On the next page, staple or tape a copy of the Assay Table results for future reference. Additionally, hand write in the margin of the Assay Table, the ID name or number of the calibration samples corresponding to the X-MET 880 order, which is the order in which the calibration samples were measured using the CAL command (next to the sample sequence # on the Assay Table form).

10.2 Alternate Calibration Model Documentation Method

If using a PC, first set the program to capture to disk. For models that no longer contain an active Calibration Intensities Table or Assay Table, the balance of the model calibration data can be captured with one command. The Enter Maintenance Program command, EMP, followed by "CONT/YES" and then the Parameters command, PRM, followed by "CONT/YES", will result in the X-MET 880 display:

MODEL XX?

Enter the model number to be documented and press "CONT/YES". The X-MET 880 will display:

GENERAL PARAMETERS?

Enter P "CONT/YES" and all required data for the model will be output to the data capture device.

After capturing the above calibration information, print a copy (if captured to floppy disk), make a photocopy (if captured directly to a thermal printer, the thermal paper will fade after a few months) and staple the copy into the XRF log book for future reference.

10.3 Confirmation Samples

Confirmation samples are recommended at a minimum rate of 10%. Confirmation samples are required if QA2 data objectives have been established for site activities. Ideally, the sample cup that was analyzed by XRF should be the same sample that is sent for AA/ICP analysis. When confirming an in-situ analysis, collect a sample from a six-inch by six-inch area for both an XRF measurement and confirmation analysis.

The XRF and metals results are analyzed with a regression analysis using either SAS™ or Statgraphics™ software with the intercept forced through zero. The correlation factor between XRF and AA/ICP data should be 0.7 or greater.

10.4 Recording Results

Record all results and monitoring activities in a laboratory or field notebook.

NOTE: Alternatively electronic data capture may be used. See Section 5.0.

11.0 HEALTH AND SAFETY

When working with potentially hazardous materials, follow USEPA, OSHA, corporate and/or any other applicable health and safety practices.

12.0 REFERENCES

1. Outokumpu X-MET 880 Portable XRF Analyzer Operating Instructions, Revision A, October, 1989.

2. Outokumpu Notes and General Information on the Regulatory Requirements for owners of the Outokumpu X-MET 880.

3. Outokumpu Supplemental Documentation on the factory calibration of X-MET 880 (if calibrated at Outokumpu prior to delivery), including Table of Check samples and Normalizing Samples for Assay Models.

4. "X-ray Fluorescence Analysis of Environmental Samples", Dzubay, T., Ed, Ann Arbor Science, 1977, p. 310.

5. "Advancements in Portable XRF Technologies for On-Site Hazardous Waste Screening", Pasmore, J., Piorek S., and McLaughlin, J.

6. "Portable X-ray Fluorescence as a Screening Tool for Analysis of Heavy Metals in Soils and Mine Wastes", Chappell, R., Davis, A., Olsen, R. Proceedings Conference Management of Uncontrolled Hazardous Waste Sites, Washington, D.C., 1986, p 115.

7. "A New Calibration Technique for X-ray Analyzers Used in Hazardous Waste Screening, Piorek, S., Rhodes, J., Proceedings 5th National RCRA/Superfund Conference, April 1988, Las Vegas, NV.

8. "Data Quality Objectives for Remedial Response Activities", EPA\540\G-87\004, March 1987.

9. "Portable X-ray Survey Meters for In-Situ Trace Element Monitoring of Air Particulates", Rhodes, J., Stout, J., Schlinder, J., and Piorek, S., American Society for Testing and Materials, Special Technical Publication 786, 1982, pp. 70 - 82.

10. "In-Situ Analysis of Waste Water Using Portable Pre-concentration Techniques and a Portable XRF Analyzer", Piorek, S., Rhodes, J., Presented at the Electron Microscopy and X-ray Applications to Environmental and Occupational Health Analysis Symposium, Penn. State Univ., Oct. 14 - 17, 1980.

11. "Hazardous Waste Screening Using a Portable X-ray Analyzer", Piorek, S., Rhodes, J., Presented at the Symposium on Waste Minimization and Environmental Programs within D.O.D., American Defense Preparedness Assoc., Long Beach, CA., April 1987.

12. "Field-Portable X-Ray Fluorescence", U.S. EPA/ERT Quality Assurance Technical Information Bulletin, Vol. 1, No. 4, May 1991.

SPECTRACE 9000 FIELD PORTABLE X-RAY FLUORESCENCE OPERATING PROCEDURES

SOP#: 1713
DATE: 01/26/95
REV. #: 0.0

1.0 SCOPE AND APPLICATION

The purpose of this Standard Operating Procedure (SOP) is to serve as a guide to the start-up, check out, operation, calibration, and routine use of the Spectrace 9000 field portable x-ray fluorescence instrument for field use in screening hazardous or potentially hazardous inorganic materials. It is not intended to replace or diminish the use of the Spectrace 9000 Operating Instructions. The Operating Instructions contain additional information for optimizing instrument performance and for utilizing different applications.

The procedures contained herein are general operating guidelines which may be changed as required, depending on site conditions, equipment limitations, limitations imposed by Quality Assurance\Quality Control (QA\QC) procedure or other protocol limitations. In all instances, the procedures finally employed should be documented and included in any or all final reports. Mention of trade names or commercial products does not constitute endorsement or recommendation for use.

1.1 Principles of Operation

X-ray Fluorescence (XRF) spectroscopy is a non destructive qualitative and quantitative analytical technique used to determine the chemical composition of samples. In a source excited XRF analysis, primary X-rays emitted from a sealed radioisotope source are utilized to irradiate samples. During interaction with samples, source X-rays may either undergo scattering (dominating process) or absorption by sample atoms in a process known as the photoelectric effect (absorption coefficient). This phenomenon originates when incident radiation knocks out an electron from the innermost shell of an atom creating a vacancy. The atom is excited and releases its surplus energy almost instantly by filling the vacancy with an electron from one of the higher energy shells. This rearrangement of electrons is associated with the emission of X-rays characteristic (in terms of energy) of the given atom. This process is referred to as emission of fluorescent X-rays (fluorescent yield). The overall efficiency of the fluorescence process is referred to as excitation efficiency and is proportional to the product of the absorption coefficient and the fluorescent yield.

1.1.1 Characteristic X-rays

The Spectrace 9000 utilizes characteristic X-ray lines originating from the innermost shells of the atoms: K, L, and occasionally M. The characteristic X-ray lines of the K series are the most energetic lines for any element and, therefore, are the preferred analytical lines. The K lines are always accompanied by the L and M lines of the same element. However, with energies much lower than those of the K lines, they can usually be neglected for those elements for which the K lines are analytically useful. For heavy elements such as cerium (Ce) (atomic number [Z]=58), to uranium (U, Z=92), the L lines are the preferred lines for analysis. The L_{α} and L_{β} lines have almost equal intensities, and the choice of one or the other depends on what interfering lines might be present. A source just energetic enough to excite the L lines will not excite the K lines of the same element. The M lines will appear together with the L lines.

The Spectrace 9000 Operating Instructions contain a table that identifies the X-rays (K or L) and elements measured for each excitation source.

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 (e.g., K absorption edge, L absorption edge, M absorption edge). 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, and the L absorption edge energy is approximately the sum of the L and M line energies of

the particular element.

Energies of the characteristic fluorescent X-rays are converted (within the detector) into a train of electric pulses, the amplitudes of which are linearly proportional to the energy. An electronic multichannel analyzer (electronic unit) measures the pulse amplitudes, which is the basis of a qualitative X-ray analysis. The number of counts at a given energy is representative of element concentration in a sample and is the basis for quantitative analysis.

1.1.2 Scattered X-rays

The source radiation is scattered from the sample by two physical processes: coherent or elastic scattering (no energy loss), and Compton or inelastic scattering (small energy loss). Thus, source backscatter (background signal) actually consists of two components with X-ray lines close together. The higher energy line is equal to the source energy. Since the whole sample takes part in scattering, the scattered X-rays usually yield the most intense lines in the spectrum. Furthermore, the scattered X-rays have the highest energies in the spectrum and, therefore, contribute most of the total measured intensity signal.

1.2 Sample Types

Solid and liquid samples can be analyzed for elements aluminum (Al) through uranium (U) with proper X-ray source selection and instrument calibration. Typical environmental applications are:

- Heavy metals in soil (in-situ or samples collected from the surface or from bore hole drillings, etc.), sludges, and liquids (e.g., lead (Pb) in gasoline)
- Light elements in liquids (e.g., phosphorus [P], sulphur [S], and chlorine [Cl] in organic solutions)
- Heavy metals in industrial waste stream effluents
- PCB in transformer oil by Cl analysis
- Heavy metal air particulates collected on membrane filters, either from personnel samplers or from high volume samplers.
- Lead (Pb) in paint

2.0 METHOD SUMMARY

The Spectrace 9000 Portable XRF Analyzer employs three radioactive isotope sources: iron-55 (Fe-55), cadmium-109 (Cd-109), and americium-241 (Am-241) for the production of primary X-rays. Each source emits a specific set of primary X-rays which excite a corresponding range of elements in a sample. When more than one source can excite the element of interest, the appropriate source is selected according to its excitation efficiency for the element of interest. See page 1-2 of the Spectrace 9000 Operating Instructions for a chart of source type versus element range.

The sample is positioned in front of the source-detector window and sample measurement is initiated which exposes the sample to primary radiation from the source. Fluorescent and backscattered X-rays from the sample enter through the beryllium (Be) detector window and are counted in the high resolution mercuric iodide (HgI_2) detector.

Elemental concentrations are computed using a Fundamental Parameter (FP) algorithm of the form:

$$\text{Concentration} = R \times S \times (1 + \text{SUM}\{A_n \times C_n\})$$

"R" is the measured analyte X-ray intensity relative to the pure element; "S" is a calculated sensitivity coefficient. The quantity SUM{} is a summation of "n"-element absorption-enhancement terms containing calculated alpha-coefficients and iteratively computed element concentrations. The Spectrace 9000 utilizes FP XRF calibrations derived from theoretical considerations (as opposed to empirical data). The menu-driven software in the Spectrace 9000 supports multiple XRF calibrations called "applications." Each application is a complete analysis configuration including elements to be measured, interfering elements in the sample, and a set of FP calibration coefficients.

The measurement time of each source is user-selectable. The shorter source measurement times (15 - 30s) are generally used for initial screening and hot spot delineation, while longer measurement times (30 - 500s) are typically used for higher precision and accuracy requirements.

3.0 SAMPLE PRESERVATION, CONTAINERS, HANDLING AND STORAGE

This SOP specifically describes equipment operating procedures for the Spectrace 9000; hence, this section is not applicable to this SOP.

4.0 INTERFERENCES AND POTENTIAL PROBLEMS

The total method error for XRF analysis is defined as the square root of the sum of squares of both instrument precision and user or application related error. Generally, the instrument precision is the least significant source of error in XRF analysis. User- or application-related error is generally more significant and will vary with each site and method used. The components of the user or application related error are the following.

4.1 Sample Placement

This is a potential source of error because the X-ray signal decreases as the distance from the radioactive source is increased. However, this error is minimized by maintaining the same distance for each sample.

4.2 Sample Representivity

In order to accurately characterize site conditions, samples collected must be representative of the site or area under investigation. Representative soil sampling ensures that a sample or group of samples accurately reflects the concentration of the contaminant(s) of concern at a given time and location. Analytical results from representative samples reflect the variation in pollutant presence and concentration range throughout a site. Variables affecting sample representativeness include: (1) geologic variability, (2) contaminant concentration variability, (3) collection and preparation variability, and (4) analytical variability. Attempts should be made to minimize these sources of variability. For additional information on representative sampling, refer to the "Removal Program Representative Sampling Guidance, Volume 1 - Soil."[(1)]

4.3 Reference Analysis

Soil chemical and physical matrix effects may be corrected by using site-specific soil samples which have been analyzed by Inductively-Coupled Plasma (ICP) or Atomic Absorption (AA) spectroscopy as calibration samples. A major source of error can result if these samples are not representative of the site and/or if the analytical error is large. Additionally, when comparing XRF results with reference analyses results, the efficiency of the sample digestion reference analysis should be considered. Some digestion methods may breakdown different sample matrices more efficiently than others.

4.4 Chemical Matrix Effects (Due to the Chemical Composition of the Sample)

Chemical matrix effects result from differences in concentrations of interfering elements. These effects appear as either spectral interferences (peak overlaps) or as X-ray absorption/enhancement phenomena. Both effects are common in soils contaminated with heavy metals. For example, iron (Fe) tends to absorb copper (Cu) X-rays, reducing the intensity of Cu measured by the detector. This effect can be corrected mathematically through the use of FP coefficients.

4.5 Physical Matrix Effects (Due to Sample Morphology)

Physical matrix effects are the result of variations in the physical character of the sample. They may include such parameters as particle size, uniformity, homogeneity, and surface condition. For example, consider a sample in which the analyte exists in the form of very fine particles within a matrix composed of much courser material. If two separate aliquots of the sample are prepared in such a way that the matrix particles in one are much larger than in the other, then the relative volume of analyte occupied by the analyte-containing particles will be different in each. When measured, a larger amount of the analyte will be exposed to the source X-rays in the sample containing finer matrix particles; this results in a higher intensity reading for that sample and, consequently, an apparently higher measured concentration for that element.

4.6 Application Error

Generally, the error in the application calibration model is insignificant (relative to the other sources of error) **PROVIDED** the instrument's operating

instructions are followed correctly. However, if the sample matrix varies significantly from the design of the application, the error may become significant (e.g., using the soils application to analyze a 50 percent iron mine tailing sample).

4.7 Moisture Content

Sample moisture content will affect the analytical accuracy of soils or sludges. The overall error may be secondary when the moisture range is small (5-20 percent), or it may be a major source of error when measuring the surface of soils that are saturated with water.

4.8 Cases of Severe X-ray Spectrum Overlaps

When present in the sample, certain X-ray lines from different elements can be very close in energy and, therefore, can interfere by producing a severely overlapped spectrum.

The typical spectral overlaps are caused by the K_β line of element Z-1 (or as with heavier elements, Z-2 or Z-3) overlapping with the K_α line of element Z. This is the so-called K_α/K_β interference. Since the $K_\alpha:K_\beta$ intensity ratio for the given element usually varies from 5:1 to 7:1, the interfering element, Z-1, must be present in large concentrations in order to disturb the measurement of analyte Z. The presence of large concentrations of vanadium (V) could disturb the measurement of chromium (Cr). The V K_α and K_β energies are 4.951 and 5.427 Kev, respectively. The Cr K_α energy is 5.41 Kev. The resolution of the detector is approximately 270 eV. Therefore, large amounts of V in a sample will result in spectral overlap of the V K_β with the Cr K_α peak (see Figure 1, Appendix A) and the measured X-ray spectrum will include TOTAL counts for Cr plus V lines.

Other interferences arise from K/L, K/M, and L/M line overlaps. While these are less common, the following are examples of severe overlap:

As K_α/Pb L_α, S K_α/Pb M_α

In the arsenic (As)/lead case, Pb can be measured from the Pb L_β line, and arsenic from either the As K_α or the As K_β line; this way the unwanted interference can be corrected. However, due to the limits of mathematical corrections, measurement sensitivity is reduced. Generally, arsenic concentrations can not be efficiently calculated in samples with Pb:As ratios of 10:1 or more. This may result in zero arsenic being reported regardless of what the actual concentration is.

The Spectrace 9000 uses overlap factors to correct for X-ray spectral overlaps for the elements of interest for a given application.

5.0 EQUIPMENT / APPARATUS

5.1 Description of the Spectrace 9000 System

The analyzer utilizes the method of Energy Dispersive X-Ray Fluorescence (EDXRF) spectrometry to determine the elemental composition of soils, sludges, aqueous solutions, oils, and other waste materials.

The Spectrace 9000 analyzer includes three compact, sealed radiation sources contained in a measuring probe: Fe-55, Cd-109, and Am-241. The analyzer software automatically selects which sources to use as well as measurement time for each source based on stored information for each application. The probe is equipped with a high resolution HgI_2 detector, which is connected by cable to an environmentally sealed electronic module.

The electronic unit provides internal non volatile memory for storage of 120 spectra and 300 multi-element analysis reports. An RS-232 serial port is provided for downloading data and spectra to a peripheral device. The multi-element analysis reports and the 2000-channel spectra can be displayed on the instrument's LCD panel. The replaceable and rechargeable internal battery provides for field-portable operation.

The Spectrace 9000 is supplied with three factory-installed FP-based applications (calibrations). The "Soil Samples" application is for analysis of soils where the balance of the sample (that portion not directly measured by the instrument) is silica (SiO_2). The "Thin Film" application is for analysis of thin films such as air monitoring filters or wipes. Finally, the "PbK in Paint" application is for analyzing Pb in paint films and is reasonably independent of the type of substrate. Spectrace Instruments will also develop calibrations to meet new user application requirements (e.g., adding elements to the present "Soil Samples" application). The PC-based Spectrace 9000 Application Generater software may also be used to

develop new applications.

The Spectrace 9000 can be powered from a 115-volt (or 220-volt) wall outlet or from its 4-hour capacity battery. It can be operated in temperatures ranging from 32 to 120° Fahrenheit (F). Furthermore, the probe and electronic unit may be exposed to a light rain. However, additional protection is provided when the system (electronic unit and probe) is contained in the optional water repellant carrying case.

5.2 Equipment and Apparatus List

5.2.1 Spectrace 9000 Analyzer System

The complete Spectrace 9000 Analyzer System includes:

- Analyzer unit for data acquisition, processing, and display
- Hand-held probe including:
  - High-resolution HgI_2 detector
  - Three excitation sources(^{55}Fe, ^{109}Cd, ^{241}Am)
  - Safety cover
- Probe laboratory stand with the following:
  - Base for table top use
  - Safety shield over sample
  - Positioning fixtures for standard 30-mm and 40-mm X-ray sample cups
- Interconnecting cable
- RS-232C Serial I/O Interface cable
- Two blank check samples
- Pure element check samples
- Battery charger
- Battery pack
- System carrying/shipping case
- Spectrace 9000 Operating Instructions, application software, and utilities software. The application software is specific to each unit and cannot be interchanged between different units. The software is identified by the serial number of the unit.

5.2.2 Optional Items

- 31-mm diameter sample cups
- XRF polypropylene film, 0.2 mil thick
- Field carrying case
- Peripheral devices such as a printer and IBM compatible Personal Computer (PC)
- Spare probe window assembly
- Spare battery pack, charger, and charger adaptor (required to charge spare battery outside of electronic unit)

See the Spectrace 9000 Accessories Price List for additional options.

For mobile lab or laboratory X-ray sample preparation accessories (such as drying ovens, grinders, sieves, etc.), consult general laboratory equipment suppliers.

5.2.3 Limits and Precautions

The probes should be handled in accordance with the following radiological control practices.

1. The probe should always be in contact with the surface of the material being analyzed, and that material should completely cover the probe opening (aperture) when the sources are exposed. Do not remove a sample or move the probe while the indicators show **SOURCE ON**.

SOURCE ON indicators are:

- the message on the screen "**SOURCE ON**"
- the flashing light at the base of the probe.

2. When the sources are exposed, under no circumstances should the probe be pointed at the operator or surrounding personnel.

3. Do not place any part of the operator's or co-worker's bodies in line of exposure when the sources are exposed or partially covered.

4. The probe must be covered with the safety cover or laboratory safety shield when not in use.

5. Spectrace Instruments must be notified immediately of any condition or concern relative to the probe's structural integrity, source shielding, source switching condition, or operability.

6. The appropriate state agency or the Nuclear Regulatory Commission (NRC) office must be notified immediately of any damage to the radioactive source, or any loss or theft of the device (see factory supplied data on radiological safety).

7. Labels or instructions on the probe(s) must not be altered or removed.

8. The user must not attempt to open the probe.

9. The source(s) in the probe must be leak-tested every 6 months as described in the Spectrace 9000 Operating Instructions. The leak test certificates must be kept on file, and a copy must accompany the instrument at all times.

10. The probe laboratory safety shield assembly must be used when the probe is inverted for measuring samples contained in cups.

11. During operation, the probe must be kept at least 10 feet from computer monitors and any other source of radio frequency (RF). Some monitors have very poor RF shielding and will affect measurement results.

12. The Spectrace 9000 should not be dropped or exposed to conditions of excessive shock or vibration.

13. The electronic unit should be left on whenever the battery charger is connected to it. If the electronic unit is shut off with the battery charger plugged in, the battery may be damaged due to overcharging.

Additional precautions include:

1. The probe cable must never be pulled while unplugging the probe. The probe plug should be grasped at the ribbed metal connector and squeezed and pulled gently while the connector is unplugged. The connector must never be forced when plugging in the connector.

2. The handle of the electronic unit must not be rotated unless the release buttons on each side of the handle are depressed.

3. The Spectrace 9000 should not be stored at an ambient temperature below -4°F or above 110EF.

4. The battery charging unit should only be used indoors in dry conditions.

5. Battery packs should be changed only in dry conditions.

5.3 Peripheral Devices

The Spectrace 9000 may be used with a wide range of peripheral devices for electronic data capture or printed readout as long as they are compatible with the RS-232 serial I/O protocol. Such devices include terminals, printers, electronic data loggers, personal computers, etc.

5.3.1 Communication Cable Connection

Plug the 25-pin connector of the RS-232 Serial I/O cable into the Spectrace 9000 25-pin connector (the connection just below the display screen on the electronic unit) and the 9-pin connector of the cable into the serial port of the receiving device.

5.3.2 Communication Port Setup

To communicate with an external device, the Spectrace 9000 **MUST** be set at the same baud rate, word length, and parity as the receiving device. The Spectrace 9000 allows you to select various configurations for these parameters in the communication (Comm.) port setup portion of the More submenu (which can be accessed from the main menu).

The default COM setup for application and utilities software is 9600,N,8,1.

5.3.3 User Software

Refer to your PC software manual for details on additional settings that may be required for proper interfacing between the Spectrace 9000 and your particular software.

5.4 Instrument Maintenance

5.4.1 Probe Window

Should the probe window become damaged or punctured, it should be replaced as soon as possible to prevent dust and moisture from entering the probe. Replacement window assemblies can be ordered from Spectrace Instruments. Note the location of the window aperture; it is closer to one end of the window plate. Simply unscrew the old window plate, press any corner of it, and remove it. Stretch the O-ring for 10 seconds, and lay it back in the groove. The O-ring must lie flat in the groove in order for the new window plate to be installed. Install the new window assembly in the same manner as the old. If the surface of the window plat is not flush with the face of the probe, the O-ring has probably come out of the grove. Remove the assembly, and try the same procedure again.

5.4.2 Further Information and Troubleshooting

Refer to the Spectrace 9000 Operating Instructions for additional detailed operational and/or maintenance and troubleshooting instructions. If no solution is found in the manual, contact Spectrace Instruments for assistance.

An instrument log should be maintained to document specific corrective actions taken to alleviate any instrumental problems, or for recording any service that has been performed.

6.0 REAGENTS

Generally, calibration standards are not necessary for site screening and extent of contamination analyses with the Spectrace 9000. Optionally, an application (only the Soil Sample application will be discussed here) can be optimized or verified to be 1:1 proportional to another analytical (reference) method (see Section 9.3 and 10.1). This can be done by analyzing a suitable set of Site-Specific Calibration Standards (SSCS) or Standard Reference Materials (SRMs) and performing a regression analysis on the reference (dependent) and the Spectrace 9000 results (independent) for each element of concern. SCSS and SRMs must be representative of the sample matrix to be analyzed by XRF, for example, National Institute of Standards and Technology (NIST) SRMs 2709, 2710, and 2711 for the soil application. In an application, any element's calibration can be adjusted by entering the desired slope and offset (intercept) in the Adjust Calibration menu. If any element's calibration has been adjusted in an application, "adj" will appear on the results screen. An adjusted element calibration can always be changed back to the initial slope and offset values of 1 and 0, respectively.

7.0 PROCEDURE

7.1 Prerequisites

If the Spectrace 9000 will be used in a location where AC power outlets are conveniently accessible, connect the battery charger to the electronic unit and plug the charger cord into the outlet. The probe cable must be connected before switching on the power. Plugging and unplugging this cable with the power on can damage the detector.

To connect the battery, set the electronics unit on its face and use a flat blade screwdriver to loosen the two one-quarter turn fasteners on the back. Remove the battery pack. Inside, find the cord with the red cap covering the three-pronged plug. Remove the cap and plug it into the battery pack. Put the battery pack into the unit and tighten the fasteners.

Apply power to the Spectrace 9000 by pressing the <ON> button. The electronic unit may not come on with the battery charger hooked up if the battery has been totally drained. The drained battery may require a 10 minute charge prior to startup. In a few seconds the display shows the version of software. If necessary, adjust the contrast knob located on the underside of the front display. This knob can be turned so far that the display appears blank.

The initial screen displays for about 10 seconds and then a prompt will ask if the time and date are set correctly. The date **MUST** be set correctly otherwise serious errors in source-decay compensation can result. Additionally, results tables include the time

and date of analysis. The main menu appears after the time and date screens.

If a "battery low" message appears, recharge or change the battery before proceeding, or operate the unit using line voltage.

Allow the Spectrace 9000 to warm up for approximately 30 minutes after it has been turned on before performing analysis.

7.1.1 Gain Control

Automatic gain compensation is a feature of both Soil and Thin Samples applications, which allows operation of the instrument over a wide range of ambient temperatures and from one day to another without standardization. To maintain gain control compensation, it is necessary to occasionally operate with a minimum acquisition time of 50 seconds on the Cd-109 source. If the automatic gain control fails or is out of range, an error message will appear on the screen. If the error message continues to appear after repeat analyses, then the Cd-109 measurement time should be checked and/or an energy calibration should be performed. If the problem continues, contact Spectrace Instruments for help.

7.1.2 Setting Data and Spectrum Store/Send Mode

The Set store/send modes option is located in the More screen which can be accessed from the main menu. Data and/or Spectrum storage must be enabled for automatic on-board storing to occur. Sufficient memory is available to store up to 300 sets of analysis results and up to 120 spectra (40 samples since each sample has three spectra). When the available memory is full, the respective spectra or results storage mode is automatically disabled. The spectra or results memory must be cleared (deleted) and the respective store mode enabled before results and/or spectra can be stored again.

7.2 General Keys and Menu Software

This section outlines the general keys and basic menu software. Flow charts which describe the menu structure in detail are located on pages 4-13 through 4-17 in the Spectrace 9000 Operating Instructions.

7.2.1 The Keyboard

The row of numeric keys under the LCD screen performs functions defined by labels (a menu) written to the bottom line of the display by the Spectrace 9000 software. As the operator moves through the various menus, the keys are redefined to provide an efficient user interface.

The keypad to the right of the screen is used for numeric entry. The <Cont/Pause> key (referred to as the <Cont>) is used:

- to enter information as an <Enter> key
- to begin an analysis
- to pause an analysis in progress

The left arrow <7> key is used to edit entries before pressing <Cont>.

7.2.2 The Measure (Ready) Screen

This main menu selection displays the application name, revision date, measurement time for each source, and accesses other options (see flow diagrams in Spectrace 9000 Operating Instructions).

7.2.3 The Choose an Application Screen

This main menu selection lists the applications currently loaded in the unit. Applications are selected and source measurement times may be modified in this screen (see flow diagrams in Spectrace 9000 Operating Instructions).

7.2.4 The Review Stored Results Screen

This main menu selection lists the stored results. *Up* and *Down* scroll are used on many screens. When *Up* and *Down* are displayed, pressing the <0> (zero) key will toggle to *PgUP* and *PgDN* for rapid movement through long lists. Stored results may be reviewed, deleted, or downloaded to the COM port (see flow diagrams in Spectrace 9000 Operating Instructions).

7.2.5 The Review Stored Spectra Screen

This main menu selection lists the stored spectra which may be deleted or transmitted to the COM port (see flow diagrams in Spectrace 9000 Operating Instructions). You cannot display spectra under this

screen. Spectra may be displayed in the *Examine Spectrum* portion of the More screen (accessed from the main menu) or in the *Examine Spectrum* selection from the Results screen under the *More Options* menu selection.

7.2.6 The More (Other Functions) Screen

This main menu selection lists the following functions:

- *Set clock/calendar*
- *Comm. port setup*
- *Set store/send modes*
- *Application maintenance*
- *Examine spectrum*

7.2.7 The Results Screen

The Results screen is displayed at the end of the analysis. If the automatic *Store Results* mode is enabled, you will be prompted for sample identification (*ID*) before the Results screen is displayed. *Up* or *Down* scrolls the screen to view more results. When *Up* and *Down* are displayed, pressing the <0> (zero) key will toggle to *PgUP* and *PgDN* for rapid movement through long lists. *Send* transmits results to the COM port. *Store* prompts for an ID and then stores results in memory. *Measr* will immediately begin another analysis cycle. *Opts* displays the first of two screens listing special options under the Results screen (the second screen is located under *More Opts* of the first screen. See flow diagrams in Spectrace 9000 Operating Instructions). The most frequently used functions are the *Examine Spectrum* and *Enable/Disable Display Thresholds* located on the second screen of options.

7.3 Preoperational Checks

7.3.1 Energy Calibration Check

An energy calibration should be performed after an instrument is shipped and periodically (approximately 2 weeks) to ensure proper energy calibration. The *Energy Calibration* function is located in the *Options* section of the Measure Screen. You will be prompted to place the safety shield on the probe and then initiate a 600- second analysis that will update the X-ray energy calibration.

The energy calibration check is performed in the field daily and after an energy calibration to verify proper energy calibration. To perform an energy calibration check, place the safety shield on the probe. Select the *Soil Samples* application and measure the safety shield using a minimum acquisition time of 60 seconds for each source. Save the results and spectra for documentation. Select *Opts*, *More Options*, and then *Examine Spectrum*. Examine the spectrum of each source. Locate and record the centroid KeV (using the x12 horizontal magnification) for each of the following peaks:

Source	Peak	Theoretical (KeV)	Specification (KeV)
Cd-109	Pb L-alpha	10.54	± 0.040
	Pb L-beta	12.61	± 0.040
	Pb L-gamma	14.76	± 0.040
	Source line	22.10	± 0.040
Fe-55	S K-alpha	2.31	± 0.020
	Source line	5.89	± 0.020
Am-241	Pb L-alpha	10.54	± 0.050
	Pb L-beta	12.61	± 0.050
	Source line	59.5	± 0.200

Perform an *Energy calibration* (see Spectrace 9000 Operating Instructions) and then do another energy calibration check if any of the peaks fail to meet specification. The energy calibration check should be performed once at the beginning of the day, after an energy calibration, after loading an application, and whenever the instrument exhibits a persistent drift.

7.3.2 Resolution Check

The resolution check examines the detector's ability to resolve X-ray energies. This should be performed once at the beginning of the day. Select the *Soil Samples* application, and measure a sample of iron using a minimum acquisition time of 60 seconds for the Cd-109 source. Save the results and spectra for documentation. Select *Examine spectrum* under the More Options section of the Results screen. Examine the Cd-109 spectrum. Locate and record the maximum peak counts (must be >1000 counts)of the iron K-alpha peak (6.4 KeV) using the x12 horizontal magnification (see Figure 2, Appendix A). Divide the maximum peak counts by two. Examine the right (high energy) side of the peak and record the counts and KeV of the channel with counts less than or equal to one-half the maximum peak count value (channel

B, Figure 2). Examine the left (low energy) side of the peak and record the counts and KeV of the channel with counts less than or equal to one-half the maximum peak count value (channel A, Figure 2). Subtract the left-side KeV from the right-side KeV (KeV at B - Kev at A, Figure 2). The difference should be less than 0.300 KeV. If the unit fails to meet this specification, call Spectrace Instruments for assistance.

7.3.3 Blank (Zero) Sample Check

The blank (Zero) sample check is performed to monitor the instrument's zero drift in the selected application. The blank sample check and the *Aquire Background Data* operation (discussed below) only apply to the application currently selected. This should be done once at the beginning of the day, after an energy calibration, after loading an application, and whenever the instrument exhibits a persistent drift on a blank or low-level sample.

Mount the probe in the laboratory stand and select the *Soil Samples* application. Disable the display thresholds. This will permit results less than one standard deviation (STD) to be displayed (even negatives). Measure the quartz blank provided with the unit (or a "clean" sand sample) using a minimum acquisition time of 60 seconds for each source. Review the results table. All elemental results for target elements with atomic number 24 (Cr) and higher in the periodic table should be within 3 standard deviations of zero ($0 \pm 3 @ |STD|$); all non-target element results should be within 5 standard deviations ($0 \pm 5 @ |STD|$). Repeat the measurement if the unit fails to meet these specifications. If several elements continue to be significantly out of these specifications, check the probe window and the blank sample for contamination or perform the *Acquire background data* operation located in the Measure (Ready) screen option. Perform the blank (Zero) sample check again. Save the results and spectra for documentation. Enable the display thresholds prior to sample analysis after the blank sample check procedure is completed.

7.3.4 Target Element Response Check

The purpose of the target element response check is to ensure that the instrument and the selected application are working properly prior to performing sample analysis. This check should be performed at the beginning of the day. Use low, mid, and high samples, or standards with known concentrations for some or all of the target elements to be checked. Select a low sample near the quantitation limit of the target elements. Select a mid sample near the site action level and a high sample near the maximum concentration of the target elements expected on site.

These samples should be measured using the same source acquisition times that will be used for sample analysis. Save the sample check results and spectra for documentation.

7.4 Selecting Source Measuring Time

The source measuring time may be modified under the Measure screen. **Zero (seconds) measuring time should never be selected for any source for any application**. Generally, the element detection limit is reduced by 50 percent for every four-fold (x4) increase in source measuring time. Although counting statistics improve as measurement time increases, the practical limit for typical applications is 600 to 800 seconds. The elements are grouped together according to the radioisotope used for their excitation with typical minimum detection limits shown in Sections 7.4.2. and 7.4.3.

Automatic gain compensation is a feature of both the Soil and Thin Samples applications which allows operation of the instrument over a wide range of ambient temperatures and from one day to another without standardization. To maintain this gain control compensation, it is necessary to occasionally operate with a minimum acquisition time of 50 seconds on the Cd-109 source.

The *Real/live* option toggles between real time (true clock time) and live time (total time the instrument is counting). The latter adds time to the analysis to correct for the time the system is busy processing pulses.

7.4.1 Minimum Source Measuring Times

A minimum measuring time (real or live) of 15 seconds for the Fe-55 source, 30 seconds for the Cd-109 source, and 10 seconds for the Am-241 source is recommended when using the Soil Samples application. Measuring times for a source that excites a target element can be increased if lower detection limits are required.

When using the Thin Samples application, the

measuring time for any source may be reduced to 10 seconds if the source does not excite a target element since this application does not correct for interelement effects. If a source excites a target element, a minimum measuring time (real or live) of 60 seconds for the Fe-55 source, 60 seconds for the Cd-109 source, and 120 seconds for the Am-241 source is recommended.

A minimum of 60 seconds is recommended for the Cd-109 source when using the PbK in Paint application.

7.4.2 Typical Minimum Detection Limits (MDLs) for the Soil Samples Application

For source measuring times of 60 seconds, typical element MDLs (in milligram per kilogram, mg/kg) for the Soil Samples application are:

Source	Element	MDL (mg/kg)
Fe-55	Potassium (K)	325
	Calcium (Ca)	150
	Titanium (Ti)	110
	Chromium (CrLo)	180
Cd-109	Chromium (CrHi)	525
	Manganese (Mn)	410
	Iron (Fe)	225
	Cobalt (Co)	205
	Nickel (Ni)	125
	Copper (Cu)	90
	Zinc (Zn)	70
	Mercury (Hg)	60
	Arsenic (As)	50
	Selenium (Se)	35
	Lead (Pb)	30
	Rubidium (Rb)	10
	Strontium (Sr)	10
	Zirconium (Zr)	10
	Molybdenum (Mo)	10
Am-241	Cadmium (Cd)	180
	Tin (Sn)	100
	Antimony (Sb)	65
	Barium (Ba)	20

NOTE: These typical MDLs are provided as an aid for selecting source measurement times; observed values for a given situation may vary depending on the matrix of the soil standard used to calculate MDLs, age of sources, moisture content, and other factors discussed in Section 4.

Generally, the detection limit is reduced by 50 percent for every four-fold (x4) increase in source measuring time. Additionally, more elements may be added to the Soil Samples application. Contact Spectrace Instruments for information about modifications to applications.

7.4.3 Typical Minimum Detection Limits (MDLs) for the Thin Samples Application

For source measuring times of 200 seconds for the Fe-55 and Cd-109 sources, and 800 seconds for the Am-241 source, typical element MDLs (in microgram per square centimeter, $\mu g/cm^2$) for the Thin Samples application are:

Source	Element	MDL ($\mu g/cm^2$)
Fe-55	Potassium (K)	0.40
	Calcium (Ca)	0.20
	Titanium (Ti)	0.15
	Chromium (CrLo)	0.40
Cd-109	Chromium (CrHi)	0.90
	Manganese (Mn)	0.65
	Iron (Fe)	0.65
	Cobalt (Co)	0.50
	Nickel (Ni)	0.30
	Copper (Cu)	0.65
	Zinc (Zn)	0.40
	Mercury (Hg)	0.45
	Arsenic (As)	0.40
	Selenium (Se)	0.15
	Lead (Pb)	0.50
	Rubidium (Rb)	0.10
	Strontium (Sr)	0.10
	Zirconium (Zr)	0.15
	Molybdenum (Mo)	0.10
Am-241	Cadmium (Cd)	2.5
	Tin (Sn)	2.5
	Antimony (Sb)	1.5
	Barium (Ba)	0.70

NOTE: These typical MDLs are provided as an aid for selecting source measurement times; observed values for a given situation may vary depending on the thin sample standard used to calculate MDLs, age of sources, and other factors discussed in Section 4.

Generally, the detection limit is reduced by 50 percent

for every four-fold (x4) increase in source measuring time. Use of thick filters or filters with high background or contamination will result in higher MDLs and require a background subtraction. Additionally, more elements may be added to the Thin Samples application. Contact Spectrace Instruments for information about modifications to applications.

7.5 Sample Handling and Presentation

When making XRF measurements, be sure to maintain constant measurement geometry in order to minimize variations in analysis results. Document any anomalies in measurement geometry, sample surface morphology, moisture content, sample grain size, and matrix (see Section 4.0).

7.5.1 Soil Samples

Soil samples may be analyzed either in-situ or in prepared X-ray sample cups. The Soil Samples application assumes the sample to be infinitely thick. For in-situ measurements this is almost always the case. However, for sample cup measurements it is advisable to fill the cup nearly full and tap it on the bench to compact the soil. This ensures that the sample is as uniformly thick as possible from analysis to analysis. The Spectrace 9000 laboratory stand and safety shield should be used when analyzing sample cups.

An area for in-situ analysis should be prepared by removing large rocks and debris. The soil surface should be rendered flat and compact prior to analysis. The Spectrace 9000 probe should be held firmly on the ground to maximize instrument contact with the ground. The probe should not be moved during analysis. Analysis of water saturated soils should be avoided. A thin layer of 0.2-mil polypropylene XRF film may be mounted on the surface probe to minimize contamination. Use of varying thicknesses of plastic (bags) have been shown to interfere in the light element (low atomic number) measurement and may affect the FP calibration of the other element concentrations.[2] Additionally, plastic may contain significant levels of target element contamination.

Course-grained soil conditions or nuggets of contaminated material may preclude a truly representative sample and adversely affect the analysis results (typically by under reporting the target element). Such samples should be prepared before analysis. Preparation consistency is important to minimize variation in analytical results.

This application is specifically designed for soil with the assumption that the balance of the material is silica. If samples with a much lighter (lower atomic number) balance are analyzed, the results will typically be elevated by a factor of two to four. Contact Spectrace Instruments for help in analysis of different matrices.

7.5.2 Thin (Filter) Samples

The Thin Samples application is for analysis of thin samples such as filters or wipes. The detection limits are affected by the thickness of the substrate. Best results are obtained on the thinnest substrates. Always use the probe safety cover when measuring thin samples. This is not only for user safety, but also ensures a controlled background environment and provides a reference signal for the automatic gain control. Probe safety covers should never be interchanged between instruments.

Filters and wipes should be prescreened before use to establish background and contamination levels. Care should be used to prevent zinc oxide contamination from disposable gloves. Small 37-mm filters can be mounted between two layers of 0.2-mil thick polypropylene XRF film on 40-mm XRF cups for analysis. Larger filters can be placed on the probe with a sheet of 0.2-mil thick polypropylene XRF film between the filter and probe to prevent the window from being contaminated. Then the probe safety cover may be placed over the filter prior to analysis. Filters should be presented loaded side down and wrinkle free.

7.5.3 Lead in Paint

The area selected for analysis should be smooth, representative and free of surface dirt. The Spectrace 9000 probe should be held firmly on the surface to maximize instrument contact. The probe should not be moved during analysis.

When used for specimen application (e.g., on paint chips or nonbacked films) remember to use the probe safety cover. In the PbK Application, you should also position a thick neutral sample, such as the quartz disk (blank), behind the specimen before closing the safety lid. Otherwise, the PbK X-rays excited in the safety

cover will be sensed by the detector. In this application, do not perform the *Acquire background data* option from the list of options under the Ready screen.

8.0 CALCULATIONS

The Spectrace 9000 is a direct readout instrument that does not require any calculations.

9.0 QUALITY ASSURANCE/ QUALITY CONTROL

9.1 Precision

The precision of the method is monitored by reading a low- or mid-target element concentration sample (or SSCS or SRMs selected as described in Sections 6.0) at the start and end of sample analysis and after approximately every tenth sample. (A daily total of seven measurements is recommended.) Determining the precision around the site action level can be extremely important if the XRF results are to be used in an enforcement action. Therefore, selection of a sample with a target element concentration at or near the site action level or level of concern is recommended. The sample is analyzed by the instrument for the normal field analysis time, and the results are recorded. The standard deviation for each target element is calculated. The relative standard deviation (RSD) of the sample mean can be used to calculate precision. The RSD should be within ± 20 percent for the data to be considered adequately precise.[3]

9.1.1 The Method Detection Limit (MDL) and Method Quantitation Limit (MQL)

The MDL and MQL may be calculated from the measurement of either a low or blank sample, (or a SSCS or SRMs selected as described in section 6.0), at the start and end of sample analysis, and after approximately every tenth sample (a daily total of seven measurements is recommended). Alternatively, the quartz blank or "clean" sand may be used if a blank soil or sediment sample is unavailable.

Disable the display thresholds. This will permit results less then one standard deviation (STD) to be displayed (even negatives). Measure the sample using the same application and measuring time used for the samples. Enable the display thresholds prior to analyzing the next sample.

The sample standard deviation of the mean for each target element is calculated. If the standard deviation has a fractional component, round up to the next whole number prior to calculating the MDL and MQL.

The definition of the MDL is three times the calculated standard deviation value.

The definition of the MQL is 10 times the calculated standard deviation value.

9.2 Reporting Results

All raw XRF data should be reported including the individual results of multiple analyses of samples and sampling points. The average and concentration range of each multiple analysis should also be reported.

A "reported" value for each analysis or average of multiple analyses should be processed in the following manner.

1. Round the value to the same degree of significance contained in the SSCS or SRM sample assay values (usually two) if the element's calibration has been adjusted (see Section 6.0). Round to 2 significant figures for sample results. DO NOT round results for standards used to determine MDL or RSD values (use raw data).

2. Report all values less than the MDL as not detected (ND).

3. Flag and note all values greater than or equal to the MDL and less than the MQL (usually with a "J" next to the reported value).

4. Report all values equal to or greater than the MQL and within the linear calibration range (if the element's calibration has been adjusted [see section 6.0]).

5. Flag and note all values above the linear calibration range (greater than the highest SSCS used in the calibration adjustment procedure) if SSCS were used and the

calibration was adjusted.

9.3 Accuracy

Accuracy, relative to a specific digestion method and elemental analysis procedure, is determined by submitting an XRF analyzed sample (prepared sample cups may be submitted) for AA or ICP analysis at a laboratory.

The on-site analysis of soils by XRF instrumentation should be considered a screening effort only (QA1 data). Data derived from the instrument should be used with discretion. Confirmatory analyses on a subset of the screening samples (minimum 10 percent) can be used to determine if the XRF data meets QA2 data objectives. The confirmation samples should ideally be selected randomly from the sample set and include a number of samples at or near the critical level. The results of the metal analysis (dependent) and the XRF analysis (independent) are evaluated with a regression analysis. The correlation factor (R^2) should be 0.7 or greater.[3]

XRF results may be multiplied by the slope prior to substitution for metal analysis results in contouring, kriging programs, or removal volume estimates. Correcting the XRF results based on confirmatory analyses should only be undertaken after careful consideration. It must be understood that the confirmatory analysis (AA or ICP) is an estimate of the concentration of metal contamination and is dependent upon the specific instrumentation and sampling methodology used. Since XRF is a total elemental technique, any comparison with referee results must account for the possibility of variable extraction, dependent upon the digestion method used and its ability to dissolve the waste or mineral form in question.

9.3.1 Matrix Considerations

Other types of QA/QC verification should include verification that the instrument calibration is appropriate for the specific site to be assessed. This includes verification of potential multiple soil matrix types that may exist at a site. Matrix differences which affect the XRF measurement include large variations in calcium content, which may be encountered when going from siliceous to calcareous soils, as well as large variations in iron content.

10.0 DATA VALIDATION

10.1 Confirmation Samples

Confirmation samples are recommended at a minimum rate of 10 percent and are required if QA2 data objectives have been established for site activities.[3] Ideally, the sample cup that was analyzed by XRF should be the same sample that is submitted for AA/ICP analysis. When confirming an in-situ analysis, collect a sample from a 6-inch by 6-inch area for both an XRF measurement and confirmation analysis.

The XRF and metals results are analyzed with a regression analysis using a statistical program such as SAS® or Statgraphics® with the intercept calculated in the regression. The correlation factor between XRF and AA/ICP data must be 0.7 or greater for QA2 data objectives.[3]

10.2 Recording Results

Record all results and monitoring activities in a laboratory or field notebook. Alternatively, record results electronically on a hard drive or floppy disk.

10.3 Downloading Stored Results and Spectra

Results (analytical reports) and spectra which have been stored in the Spectrace 9000 internal memory should be downloaded and captured in disk files on a PC (see section 5). Spectrace Instruments provides software for this purpose. Additionally, they provide software to prepare results or spectra for importing into a spreadsheet. Refer to the instructions provided with the programs for details on their operation.

Alternatively, other software with terminal data logging capabilities may be used to capture results and spectra to disk files.

After capturing results to a file, print a copy and save both the disk files and the printout for future reference and documentation purposes.

11.0 HEALTH AND SAFETY

When working with potentially hazardous materials, follow U.S. EPA, OSHA, corporate and/or any other

applicable health and safety practices.

12.0 REFERENCES

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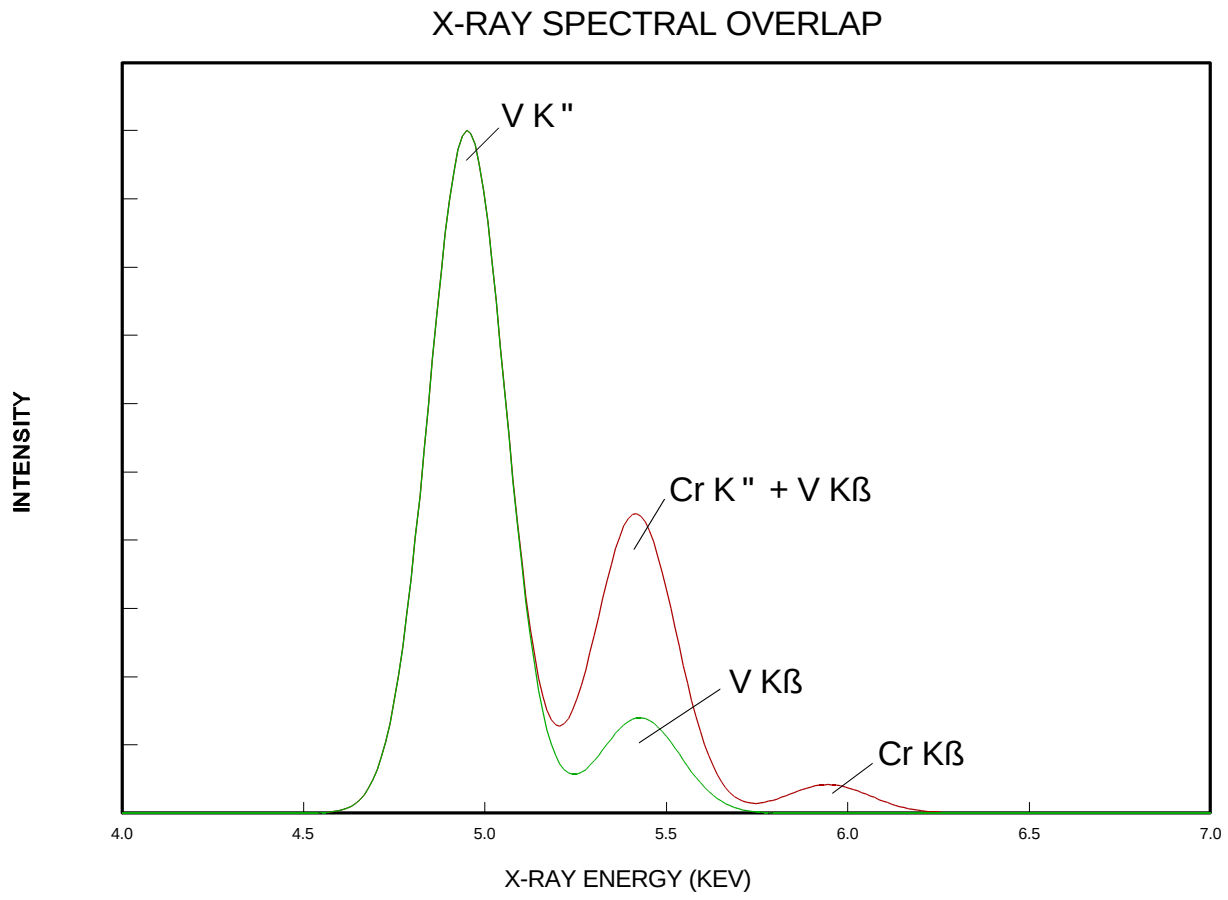
APPENDIX A

Figures

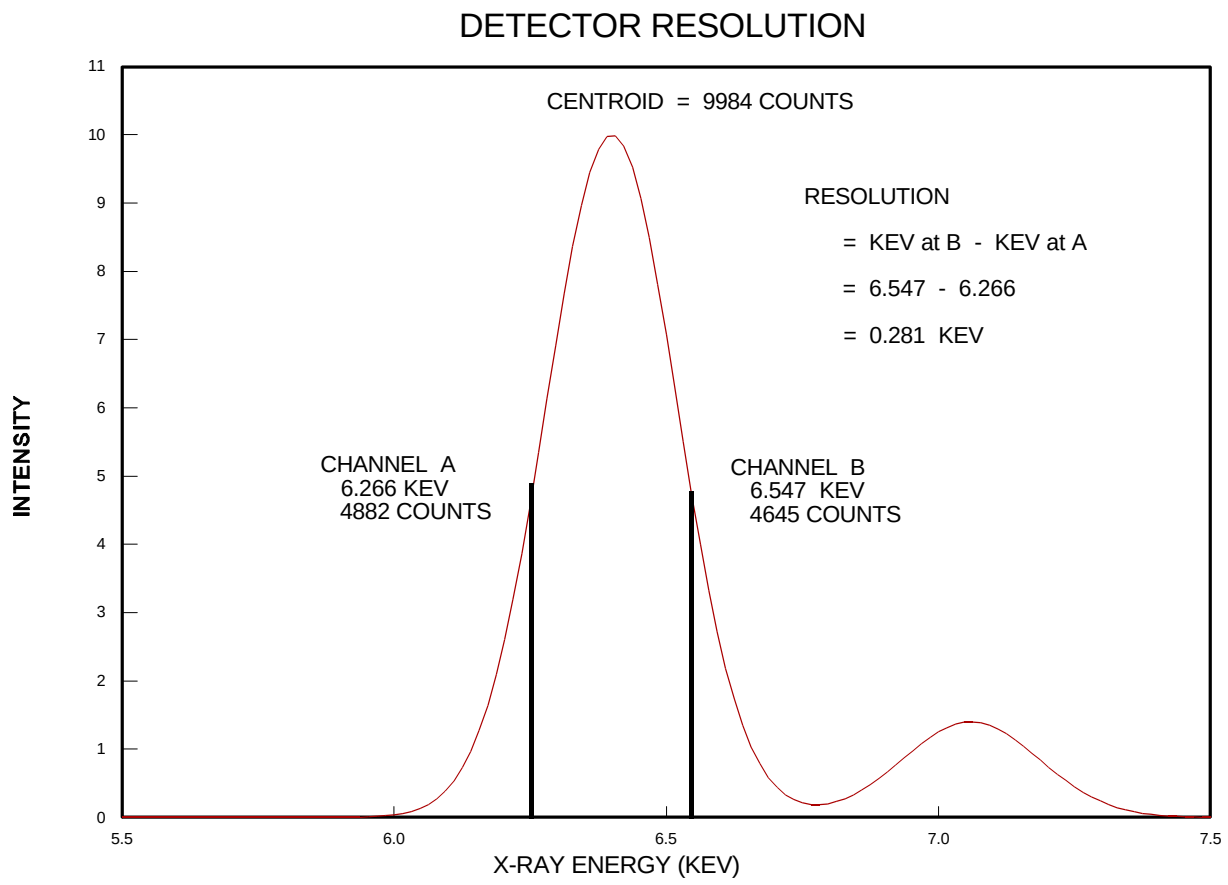
FIGURE 1. X-Ray Spectral Plot Showing Overlap of Vanadium K$_{ß}$ X-Rays in the Chromium K$_{"}$ Measurement Region.



APPENDIX A (CON'T)

Figures

FIGURE 2. Iron X-Ray Spectrum Illusrating Detector Resolution Measurement



U. S. EPA ENVIRONMENTAL RESPONSE TEAM

STANDARD OPERATING PROCEDURES

SOIL SAMPLING

CONTENTS

SUPERCEDES: SOP #2012; Revision 0.0; 11/16/94; U.S. EPA Contract 68-C4-0022.

1.0 SCOPE AND APPLICATION

The purpose of this standard operating procedure (SOP) is to describe the procedures for the collection of representative soil samples. Sampling depths are assumed to be those that can be reached without the use of a drill rig, direct-push, or other mechanized equipment (except for a back-hoe). Analysis of soil samples may determine whether concentrations of specific pollutants exceed established action levels, or if the concentrations of pollutants present a risk to public health, welfare, or the environment.

These are standard (i.e., typically applicable) operating procedures which may be varied or changed as required, dependent upon site conditions, equipment limitations or limitations imposed by the procedure. In all instances, the actual procedures used should be documented and described in an appropriate site report.

Mention of trade names or commercial products does not constitute U.S. Environmental Protection Agency (EPA) endorsement or recommendation for use.

2.0 METHOD SUMMARY

Soil samples may be collected using a variety of methods and equipment depending on the depth of the desired sample, the type of sample required (disturbed vs. undisturbed), and the soil type. Near-surface soils may be easily sampled using a spade, trowel, and scoop. Sampling at greater depths may be performed using a hand auger, continuous flight auger, a trier, a split-spoon, or, if required, a backhoe.

3.0 SAMPLE PRESERVATION, CONTAINERS, HANDLING, AND STORAGE

Chemical preservation of solids is not generally recommended. Samples should, however, be cooled and protected from sunlight to minimize any potential reaction. The amount of sample to be collected and proper sample container type are discussed in ERT/REAC SOP #2003 Rev. 0.0 08/11/94, *Sample Storage, Preservation and Handling*.

4.0 INTERFERENCES AND POTENTIAL PROBLEMS

There are two primary potential problems associated with soil sampling - cross contamination of samples and improper sample collection. Cross contamination problems can be eliminated or minimized through the use of dedicated sampling equipment. If this is not possible or practical, then decontamination of sampling equipment is necessary. Improper sample collection can involve using contaminated equipment, disturbance of the matrix resulting in compaction of the sample, or inadequate homogenization of the samples where required, resulting in variable, non-representative results.

5.0 EQUIPMENT

Soil sampling equipment includes the following:

- Maps/plot plan
- Safety equipment, as specified in the site-specific Health and Safety Plan
- Survey equipment or global positioning system (GPS) to locate sampling points
- Tape measure
- Survey stakes or flags
- Camera and film
- Stainless steel, plastic, or other appropriate homogenization bucket, bowl or pan
- Appropriate size sample containers
- Ziplock plastic bags
- Logbook
- Labels
- Chain of Custody records and custody seals
- Field data sheets and sample labels
- Cooler(s)
- Ice
- Vermiculite
- Decontamination supplies/equipment
- Canvas or plastic sheet
- Spade or shovel
- Spatula
- Scoop
- Plastic or stainless steel spoons
- Trowel(s)
- Continuous flight (screw) auger
- Bucket auger
- Post hole auger
- Extension rods
- T-handle
- Sampling trier
- Thin wall tube sampler
- Split spoons
- Vehimeyer soil sampler outfit
  - Tubes
  - Points
  - Drive head
  - Drop hammer
  - Puller jack and grip
- Backhoe

6.0 REAGENTS

Reagents are not used for the preservation of soil samples. Decontamination solutions are specified in ERT/REAC SOP #2006 Rev. 0.0 08/11/94, *Sampling Equipment Decontamination*, and the site specific work plan.

7.0 PROCEDURES

7.1 Preparation

1. Determine the extent of the sampling effort, the sampling methods to be employed, and the types and amounts of equipment and supplies required.

2. Obtain necessary sampling and monitoring equipment.

3. Decontaminate or pre-clean equipment, and ensure that it is in working order.

4. Prepare schedules and coordinate with staff, client, and regulatory agencies, if appropriate.

5. Perform a general site survey prior to site entry in accordance with the site specific Health and Safety Plan.

6. Use stakes, flagging, or buoys to identify and mark all sampling locations. Specific site factors, including extent and nature of contaminant, should be considered when selecting sample location. If required, the proposed locations may be adjusted based on site access, property boundaries, and surface obstructions. All staked locations should be utility-cleared by the property owner or the On-Scene-Coordinator (OSC) prior to soil sampling; and utility clearance should always be confirmed before beginning work.

7.2 Sample Collection

7.2.1 Surface Soil Samples

Collection of samples from near-surface soil can be accomplished with tools such as spades, shovels, trowels, and scoops. Surface material is removed to the required depth and a stainless steel or plastic scoop is then used to collect the sample.

This method can be used in most soil types but is limited to sampling at or near the ground surface. Accurate, representative samples can be collected with this procedure depending on the care and precision demonstrated by the sample team member. A flat, pointed mason trowel to cut a block of the desired soil is helpful when undisturbed profiles are required. Tools plated with chrome or other materials should not be used. Plating is particularly common with garden implements such as potting trowels.

The following procedure is used to collect surface soil samples:

1. Carefully remove the top layer of soil or debris to the desired sample depth with a pre-cleaned spade.

2. Using a pre-cleaned, stainless steel scoop, plastic spoon, or trowel, remove and discard a thin layer of soil from the area which came in contact with the spade.

3. If volatile organic analysis is to be performed, transfer the sample directly into an appropriate, labeled sample container with a stainless steel lab spoon, or equivalent and secure the cap tightly. Place the remainder of the sample into a stainless steel, plastic, or other appropriate homogenization container, and mix thoroughly to obtain a homogenous sample representative of the entire sampling interval. Then, either place the sample into appropriate, labeled containers and secure the caps tightly; or, if composite samples are to be collected, place a sample from another sampling interval or location into the homogenization container and mix thoroughly. When compositing is complete, place the sample into appropriate, labeled containers and secure the caps tightly.

7.2.2 Sampling at Depth with Augers and Thin Wall Tube Samplers

This system consists of an auger, or a thin-wall tube sampler, a series of extensions, and a "T" handle (Figure 1, Appendix A). The auger is used to bore a hole to a desired sampling depth, and is then withdrawn. The sample may be collected directly from the auger. If a core sample is to be collected, the auger tip is then replaced with a thin wall tube sampler. The system is then lowered down the borehole, and driven into the soil to the completion depth. The system is withdrawn and the core is collected from the thin wall tube sampler.

Several types of augers are available; these include: bucket type, continuous flight (screw), and post-hole augers. Bucket type augers are better for direct sample recovery because they provide a large volume of sample in a short time. When continuous flight augers are used, the sample can be collected directly from the flights. The continuous flight augers are satisfactory when a composite of the complete soil column is desired. Post-hole augers have limited utility for sample collection as they are designed to cut through fibrous, rooted, swampy soil and cannot be used below a depth of approximately three feet.

The following procedure is used for collecting soil samples with the auger:

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

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

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

4. After reaching the desired depth, slowly and carefully remove the auger from the hole. When sampling directly from the auger, collect the sample after the auger is removed from the hole and proceed to Step 10.

5. Remove auger tip from the extension rods and replace with a pre-cleaned thin wall tube sampler. Install the proper cutting tip.

6. Carefully lower the tube sampler down the borehole. Gradually force the tube sampler into the soil. Do not scrape the borehole sides. Avoid hammering the rods as the vibrations may cause the boring walls to collapse.

7. Remove the tube sampler, and unscrew the drill rods.

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

9. Discard the top of the core (approximately 1 inch), as this possibly represents material collected before penetration of the layer of concern. Place the remaining core into the appropriate labeled sample container. Sample homogenization is not required.

10. If volatile organic analysis is to be performed, transfer the sample into an appropriate, labeled sample container with a stainless steel lab spoon, or equivalent and secure the cap tightly. Place the remainder of the sample into a stainless steel, plastic, or other appropriate homogenization container, and mix thoroughly to obtain a homogenous sample representative of the entire sampling interval. Then, either place the sample into appropriate, labeled containers and secure the caps tightly; or, if composite samples are to be collected, place a sample from another sampling interval into the homogenization container and mix thoroughly.

    When compositing is complete, place the sample into appropriate, labeled containers and secure the caps tightly.

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

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

7.2.3 Sampling with a Trier

The system consists of a trier, and a "T" handle. The auger is driven into the soil to be sampled and used to extract a core sample from the appropriate depth.

The following procedure is used to collect soil samples with a sampling trier:

1. Insert the trier (Figure 2, Appendix A) into the material to be sampled at a 0^{o} to 45^{o} angle from horizontal. This orientation minimizes the spillage of sample.

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

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

4. If volatile organic analyses are required, transfer the sample into an appropriate, labeled sample container with a stainless steel lab spoon, or equivalent and secure the cap tightly. Place the remainder of the sample into a stainless steel, plastic, or other appropriate homogenization container, and mix thoroughly to obtain a homogenous sample representative of the entire sampling interval. Then, either place the sample into appropriate, labeled containers and secure the caps tightly; or, if composite samples are to be collected, place a sample from another sampling interval into the homogenization container and mix thoroughly. When compositing is complete, place the sample into appropriate, labeled containers and secure the caps tightly.

7.2.4 Sampling at Depth with a Split Spoon (Barrel) Sampler

Split spoon sampling is generally used to collect undisturbed soil cores of 18 or 24 inches in length. A series of consecutive cores may be extracted with a split spoon sampler to give a complete soil column profile, or an auger may be used to drill down to the desired depth for sampling. The split spoon is then driven to its sampling depth through the bottom of the augured hole and the core extracted.

When split spoon sampling is performed to gain geologic information, all work should

be performed in accordance with ASTM D1586-98, "Standard Test Method for Penetration Test and Split-Barrel Sampling of Soils".

The following procedures are used for collecting soil samples with a split spoon:

1. Assemble the sampler by aligning both sides of barrel and then screwing the drive shoe on the bottom and the head piece on top.

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

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

4. Record in the site logbook or on field data sheets the length of the tube used to penetrate the material being sampled, and the number of blows required to obtain this depth.

5. Withdraw the sampler, and open by unscrewing the bit and head and splitting the barrel. The amount of recovery and soil type should be recorded on the boring log. If a split sample is desired, a cleaned, stainless steel knife should be used to divide the tube contents in half, longitudinally. This sampler is typically available in 2 and 3 1/2 inch diameters. A larger barrel may be necessary to obtain the required sample volume.

6. Without disturbing the core, transfer it to appropriate labeled sample container(s) and seal tightly.

7.2.5 Test Pit/Trench Excavation

A backhoe can be used to remove sections of soil, when detailed examination of soil characteristics are required. This is probably the most expensive sampling method because of the relatively high cost of backhoe operation.

The following procedures are used for collecting soil samples from test pits or trenches:

1. Prior to any excavation with a backhoe, it is important to ensure that all sampling locations are clear of overhead and buried utilities.

2. Review the site specific Health & Safety plan and ensure that all safety precautions including appropriate monitoring equipment are installed as required.

3. Using the backhoe, excavate a trench approximately three feet wide and approximately one foot deep below the cleared sampling location. Place excavated soils on plastic sheets. Trenches greater than five feet deep must be sloped or protected by a shoring system, as required by OSHA regulations.

4. A shovel is used to remove a one to two inch layer of soil from the vertical face of the pit where sampling is to be done.

5. Samples are taken using a trowel, scoop, or coring device at the desired intervals. Be sure to scrape the vertical face at the point of sampling to remove any soil that may have fallen from above, and to expose fresh soil for sampling. In many instances, samples can be collected directly from the backhoe bucket.

6. If volatile organic analyses are required, transfer the sample into an appropriate, labeled sample container with a stainless steel lab spoon, or equivalent and secure the cap tightly. Place the remainder of the sample into a stainless steel, plastic, or other appropriate homogenization container, and mix thoroughly to obtain a homogenous sample representative of the entire sampling interval. Then, either place the sample into appropriate, labeled containers and secure the caps tightly; or, if composite samples are to be collected, place a sample from another sampling interval into the homogenization container and mix thoroughly. When compositing is complete, place the sample into appropriate, labeled containers and secure the caps tightly.

7. Abandon the pit or excavation according to applicable state regulations. Generally, shallow excavations can simply be backfilled with the removed soil material.

8.0 CALCULATIONS

This section is not applicable to this SOP.

9.0 QUALITY ASSURANCE/QUALITY CONTROL

There are no specific quality assurance (QA) activities which apply to the implementation of these procedures. However, the following QA procedures apply:

1. All data must be documented on field data sheets or within site logbooks.

2. All instrumentation must be operated in accordance with operating instructions as supplied by the manufacturer, unless otherwise specified in the work plan. Equipment checkout and calibration

activities must occur prior to sampling/operation, and they must be documented.

10.0 DATA VALIDATION

This section is not applicable to this SOP.

11.0 HEALTH AND SAFETY

When working with potentially hazardous materials, follow U.S. EPA, OHSA and corporate health and safety procedures, in addition to the procedures specified in the site specific Health & Safety Plan..

12.0 REFERENCES

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U.S. Environmental Protection Agency. 1984 Characterization of Hazardous Waste Sites - A Methods Manual: Volume II. Available Sampling Methods, Second Edition. EPA-600/4-84-076.

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.

ASTM D 1586-98, ASTM Committee on Standards, Philadelphia, PA.

APPENDIX A
Figures
SOP #2012
February 2000

FIGURE 1. Sampling Augers

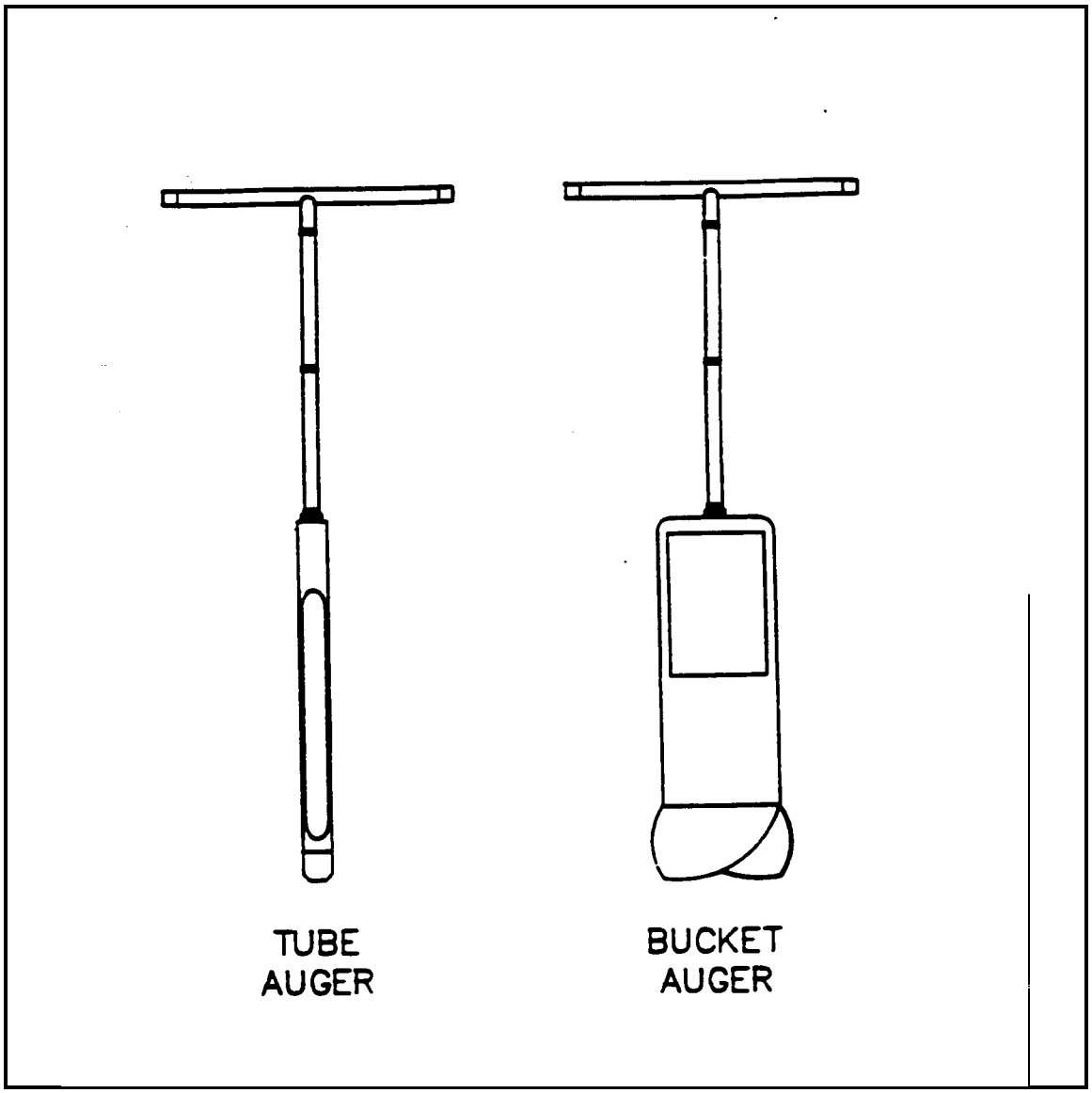
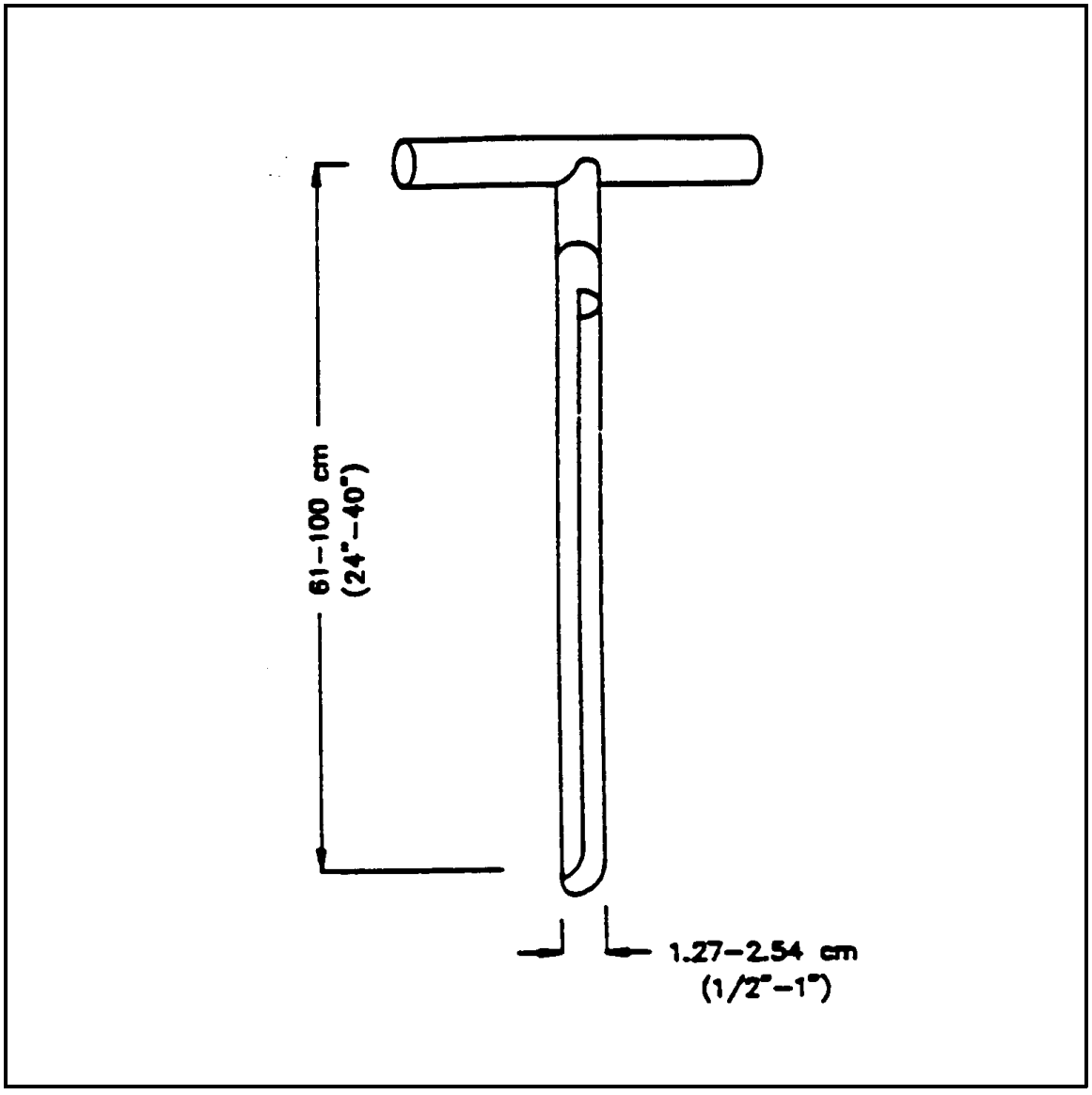


FIGURE 2. Sampling Trier



WASTE PILE SAMPLING

1.0 SCOPE AND APPLICATION

The objective of this standard operating procedure (SOP) is to outline the equipment and methods used in collecting representative samples from waste piles, sludges or other solid or liquid waste mixed with soil.

These are standard (i.e., typically applicable) operating procedures which may be varied or changed as required, dependent on site conditions, equipment limitations or other procedure limitations. In all instances, the ultimate procedures employed should be documented and associated with the final report.

Mention of trade names or commercial products does not constitute U.S. EPA endorsement or recommendation for use.

2.0 METHOD SUMMARY

Stainless steel shovels, trowels, or scoops should be used to clear away surface material before samples are collected. For depth samples, a decontaminated auger may be required to advance the hole, then another decontaminated auger used for sample collection. For a sample core, thin-wall tube samplers or grain samplers may be used. Near surfaces, samples can be collected with a clean stainless steel spoon or trowel.

All samples collected, except those for volatile organic analysis, should be placed into a Teflon lined or stainless steel pail and mixed thoroughly before transfer to appropriate sample container.

3.0 SAMPLE PRESERVATION, CONTAINERS, HANDLING, AND STORAGE

Chemical preservation of solids is generally not recommended. Refrigeration to 4°C is usually the best approach, supplemented by a minimal holding time, depending on contaminants of concern.

Wide mouth glass containers with Teflon lined caps are typically used for waste pile samples. Sample volume required is a function of the analytical requirements and should be specified in the work plan.

4.0 INTERFERENCES AND POTENTIAL PROBLEMS

There are several variables involved in waste sampling, including shape and size of piles, compactness, and structure of the waste material. Shape and size of waste material or waste piles vary greatly in areal extent and height. Since state and federal regulations often require a specified number of samples per volume of waste, the size and shape must be used to calculate volume and to plan for the correct number of samples. Shape must also be accounted for when planning physical access to the sampling point and the equipment necessary to successfully collect the sample at that location.

Material to be sampled may be homogeneous or heterogeneous. Homogeneous material resulting from known situations may not require an extensive sampling protocol. Heterogeneous and unknown wastes require more extensive sampling and analysis to ensure the different components (i.e. layers, strata) are being represented.

The term "representative sample" is commonly used to denote a sample that has the properties and composition of the population from which it was collected and in the same proportions as found in the population. This can be misleading unless one is dealing with a homogenous waste from which one sample can represent the whole population.

The usual options for obtaining the most "representative sample" from waste piles are simple random sampling or stratified random sampling. Simple random sampling is the method of choice unless: (1) there are known distinct strata; (2) one wants to prove or disprove that there are distinct

strata; or (3) one is limited in the number of samples and desires to statistically minimize the size of a "hot spot" that could go unsampled. If any of these conditions exist, stratified random sampling would be the better strategy.

Stratified random sampling can be employed only if all points within the pile can be accessed. In such cases, the pile should be divided into a three-dimensional grid system with, the grid cubes should be numbered, and the grid cubes to be sampled should be chosen by random number tables or generators. The only exceptions to this are situations in which representative samples cannot be collected safely or where the investigative team is trying to determine worst case conditions.

If sampling is limited to certain portions of the pile, a statistically based sample will be representative only of that portion, unless the waste is homogenous.

5.0 EQUIPMENT/APPARATUS

Waste pile solids include powdered, granular, or block materials of various sizes, shapes, structure, and compactness. The type of sampler chosen should be compatible with the waste. Samplers commonly used for waste piles include: stainless steel scoops, shovels, trowels, spoons, and stainless steel hand augers, sampling triers, and grain samplers.

Waste pile sampling equipment check list:

- Sampling plan
- Maps/plot plan
- Safety equipment, as specified in the Health and Safety Plan
- Compass
- Tape measure
- Survey stakes or flags
- Camera and film
- Stainless steel, plastic, or other appropriate homogenization bucket or bowl
- Appropriate size sample jars
- Ziplock plastic bags
- Logbook
- Labels
- Chain of Custody records and seals
- Field data sheets
- Cooler(s)
- Ice
- Decontamination supplies/equipment
- Canvas or plastic sheet
- Spade or shovel
- Spatula
- Scoop
- Plastic or stainless steel spoons
- Trowel
- Continuous flight (screw) augers
- Bucket auger
- Post hole auger
- Extension rods
- T-Handle
- Thin-wall tube sampler with cutting tips
- Sampling trier
- Grain sampler

6.0 REAGENTS

No chemical reagents are used for the preservation of waste pile samples; however, decontamination solutions may be required. If decontamination of equipment is required, refer to the Sampling Equipment Decontamination SOP, and the site specific work plan.

7.0 PROCEDURES

7.1 Preparation

1. Review all information available on the waste pile and expected or unknown contaminants.

2. Determine the extent of the sampling effort, the sampling methods to be employed, and the types and amounts of equipment and supplies required.

3. Obtain necessary sampling and monitoring equipment.

4. Decontaminate or pre-clean equipment, and ensure that it is in working order.

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

6. Perform a general site survey prior to site entry in accordance with the site specific Health and Safety Plan.

7. Use stakes or flagging to identify and mark

all sampling locations. Specific site factors, including extent and nature of contaminant should be considered when selecting sample locations. If required, the proposed locations may be adjusted based on site access, property boundaries, and surface obstructions.

7.2 Sample Collection

7.2.1 Sampling with Shovels and Scoops

Collection of samples from surface portions of the pile can be accomplished with tools such as spades, shovels, and scoops. Surface material can be removed to the required depth with this equipment, then a stainless steel or plastic scoop, or equivalent can be used to collect the sample.

Accurate, representative samples can be collected with this procedure depending on the care and precision demonstrated by sample team members. Use of a flat, pointed mason trowel to cut a block of the desired material can be helpful when undisturbed profiles are required. A stainless steel scoop, lab spoon, plastic spoon, or equivalent will suffice in most other applications. Care should be exercised to avoid the use of devices plated with chrome or other materials. Plating is particularly common with implements such as garden trowels.

The following procedure is used to collect the surface samples:

1. Carefully remove the top layer of material to the desired sample depth with a pre-cleaned spade.

2. Using a pre-cleaned stainless steel scoop, plastic spoon, trowel, or equivalent remove and discard a thin layer of material from the area which came in contact with the spade.

3. If volatile organic analysis is to be performed, transfer the sample into an appropriate, labeled sample container with a stainless steel lab spoon, or equivalent, and secure the cap tightly. Place the remainder of the sample into a stainless steel, plastic, or other appropriate homogenization container, and mix thoroughly to obtain a homogenous sample representative of the entire sampling interval. Then, either place the sample into appropriate, labeled containers and secure the caps tightly; or, if composite samples are to be collected, place a sample from another sampling interval into the homogenization container and mix thoroughly. When compositing is complete, place the sample into appropriate, labeled containers and secure the caps tightly.

7.2.2 Sampling with Bucket Augers and Thin-Wall Tube Samplers

These samplers consist of a series of extensions, a "T" handle, and a bucket auger or thin-wall tube sampler (Appendix A, Figure 1). The auger is used to bore a hole to a desired sampling depth, and is then withdrawn. The sample may be collected directly from the bucket auger. If a core sample is to be collected, the auger tip is then replaced with a thin-wall tube sampler. The sampler is then lowered down the borehole, and driven into the pile to the completion depth. The sampler is withdrawn and the core collected from the thin-wall tube sampler.

Several augers are available. These include: bucket, continuous flight (screw), and post hole augers. Bucket augers are better for direct sample recovery since they provide a large volume of sample in a short time. When continuous flight augers are used, the sample can be collected directly from the flights, which are usually at five (5) foot intervals. The continuous flight augers are satisfactory for use when a composite of the complete waste pile column is desired. Post hole augers have limited utility for sample collection as they are designed to cut through fibrous, rooted, swampy areas.

The following procedure will be used for collecting waste pile samples with the bucket augers and thin-wall tube samplers:

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

2. Clear the area to be sampled of any surface debris. It may be advisable to remove the first three to six inches of surface material for an area approximately six inches in radius around the drilling location.

3. Begin augering, periodically removing and depositing accumulated materials onto a plastic sheet spread near the hole. This prevents accidental brushing of loose

material back down the borehole when removing the auger or adding drill rod extensions. It also facilitates refilling the hole, and avoids possible contamination of the surrounding area.

4. After reaching the desired depth, slowly and carefully remove the auger from the borehole. When sampling directly from the auger, collect the sample after the auger is removed from the borehole and proceed to Step 10.

5. Remove auger tip from drill rods and replace with a pre-cleaned thin-wall tube sampler. Install proper cutting tip.

6. Carefully lower the tube sampler down the borehole. Gradually force the tube sampler into the pile. Care should be taken to avoid scraping the borehole sides. Avoid hammering the drill rod extensions to facilitate coring as the vibrations may cause the borehole walls to collapse.

7. Remove the tube sampler, and unscrew the drill rod extensions.

8. Remove the cutting tip and the thin-wall tube sampler.

9. Discard the top of the core (approximately one-inch), as this represents material collected before penetration of the layer of concern. Place the remaining core into the appropriate labeled sample container. Sample homogenization is not required.

10. If volatile organic analysis is to be performed, transfer the sample into an appropriate, labeled sample container with a stainless steel lab spoon, or equivalent and secure the cap tightly. Place the remainder of the sample into a stainless steel, plastic, or other appropriate homogenization container, and mix thoroughly to obtain a homogenous sample representative of the entire sampling interval. Then, either place the sample into appropriate, labeled containers and secure the caps tightly; or, if composite samples are to be collected, place a sample from another sampling interval into the homogenization container and mix thoroughly. When compositing is complete, place the sample into appropriate, labeled containers and secure the caps tightly.

11. If another sample is to be collected in the same hole, but at a greater depth, reattach the bucket auger to the drill and assembly, and follow steps 3 through 11, making sure to decontaminate the bucket auger and thin-wall tube sampler between samples.

7.2.3 Sampling with a Trier

This sampling device consists of a trier, and a "T" handle. The trier is driven into the waste pile and used to extract a core sample from the appropriate depth.

The following procedure will be used to collect waste pile samples with a sampling trier:

1. Insert the trier (Appendix A, Figure 2) into the material to be sampled at a 0E to 45E angle from horizontal. This orientation minimizes spillage of the sample. Extraction of the samples might require tilting of the sample containers.

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

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

4. If volatile organic analysis is to be performed, transfer the sample into an appropriate, labeled sample container with a stainless steel lab spoon, plastic lab spoon, or equivalent and secure the cap tightly. Place the remainder of the sample into a stainless steel, plastic, or other appropriate homogenization container, and mix thoroughly to obtain a homogenous sample representative of the entire sampling interval. Then, either place the sample into appropriate, labeled containers and secure the caps tightly; or, if composite samples are being collected, place samples from the other sampling intervals into the homogenization container and mix thoroughly. When compositing is complete, place the sample

into appropriate, labeled containers and secure the caps tightly.

7.2.4 Sampling with a Grain Sampler

The grain sampler (Appendix A, Figure 3) is used for sampling powdered or granular wastes or materials in bags, fiber drums, sacks, similar containers or piles. This sampler is most useful when the solids are no greater than 0.6 cm (1/4") in diameter.

This sampler consists of two slotted telescoping brass or stainless steel tubes. The outer tube has a conical, pointed tip at one end that permits the sampler to penetrate the material being sampled. The sampler is opened and closed by rotating the inner tube. Grain samplers are generally 61 to 100 cm (24 to 40 in.) long by 1.27 to 2.54 cm (1/2 to 1 in.) in diameter and are commercially available at laboratory supply houses.

The following procedures will be used to collect waste pile samples with a grain sampler:

1. With the sampler in the closed position, insert it into the granular or powdered material or waste being sampled from a point near a top edge or corner, through the center, and to a point diagonally opposite the point of entry.

2. Rotate the sampler inner tube into the open position.

3. Wiggle the sampler a few times to allow material to enter the open slots.

4. Place the sampler in the closed position and withdraw from the material being sampled.

5. Place the sampler in a horizontal position with the slots facing upward.

6. Rotate the outer tube and slide it away from the inner tube.

7. If volatile organic analysis is to be performed, transfer the sample into an appropriate, labeled sample container with a stainless steel lab spoon, plastic lab spoon, or equivalent and secure the cap tightly. Place the remainder of the sample into a stainless steel, plastic, or other appropriate homogenization container, and mix thoroughly to obtain a homogenous sample representative of the entire sampling interval. Then, either place the sample into appropriate, labeled containers and secure the caps tightly; or, if composite samples are to be collected, place a sample from another sampling interval into the homogenization container and mix thoroughly. When compositing is complete, place the sample into appropriate, labeled containers and secure the caps tightly.

8.0 CALCULATIONS

This section is not applicable to this SOP.

9.0 QUALITY ASSURANCE/ QUALITY CONTROL

There are no specific quality assurance activities which apply to the implementation of these procedures. However, the following QA procedures apply:

1. All data must be documented on field data sheets or within site logbooks.

2. All instrumentation must be operated in accordance with operating instructions as supplied by the manufacturer, unless otherwise specified in the work plan. Equipment checkout and calibration activities must occur prior to sampling/operation, and they must be documented.

10.0 DATA VALIDATION

This section is not applicable to this SOP.

11.0 HEALTH AND SAFETY

When working with potentially hazardous materials, follow U.S. EPA/OSHA and corporate health and safety procedures.

12.0 REFERENCES

Test Methods for Evaluating Solids Waste (SW-846), Third Edition, Vol. II Field Manual U.S. EPA Office of Solid Waste and Emergency Response, Washington, D.C. November, 1986.

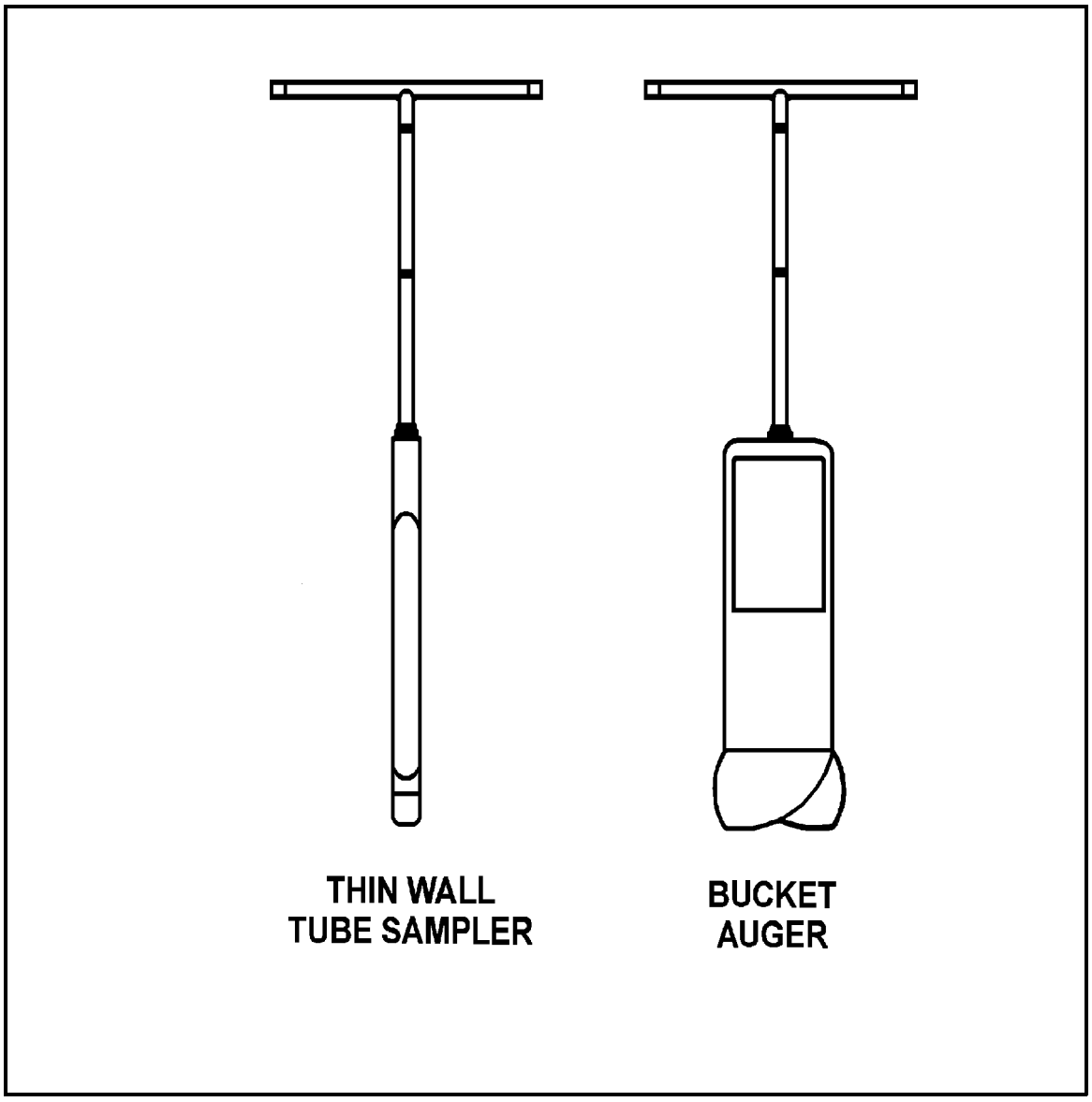
Engineering Support Branch Standard Operating Procedures and Quality Assurance Manual, U.S. Environmental Protection Agency, Region IV, April 1, 1986.

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

APPENDIX A

Figures

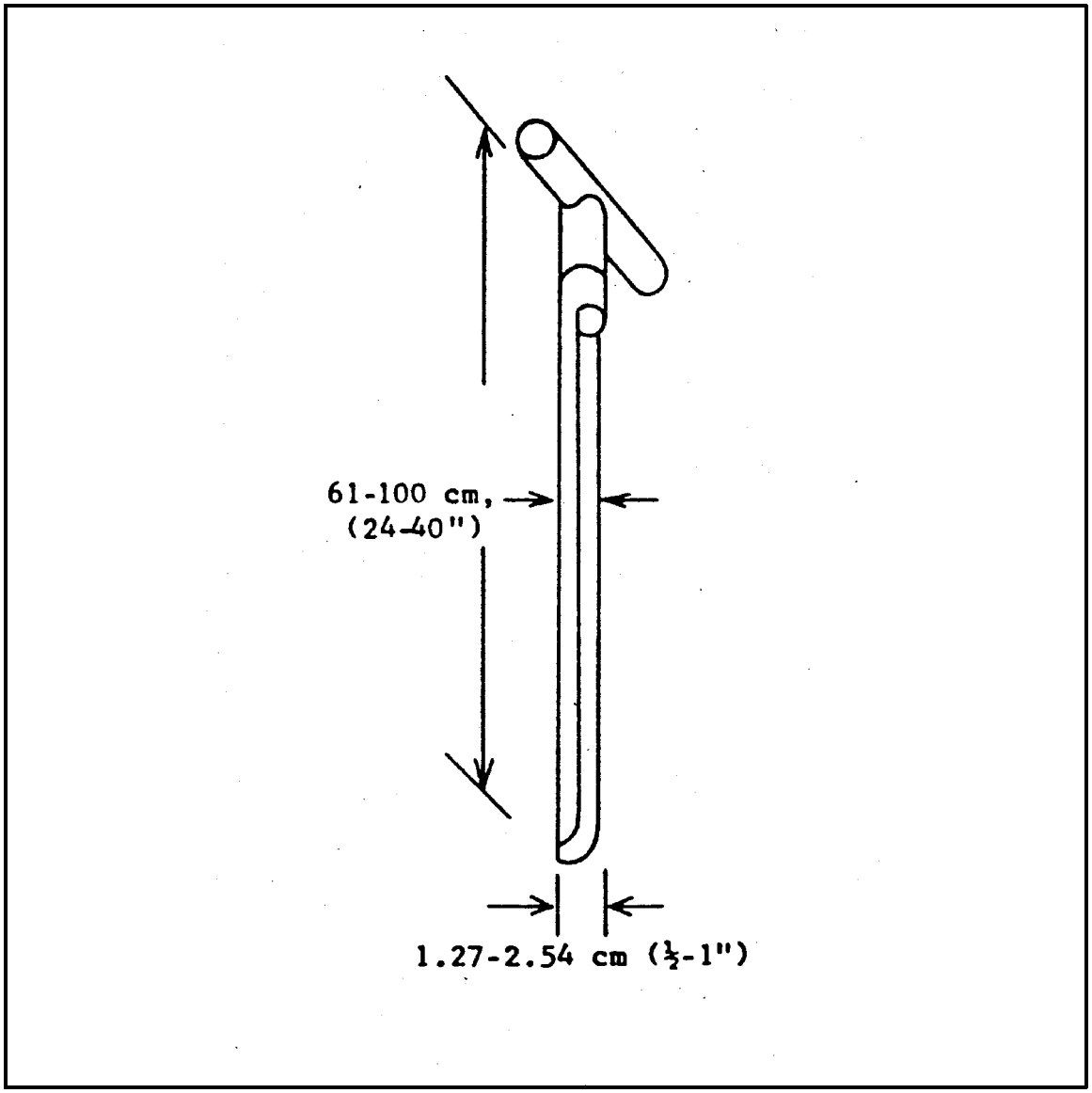
FIGURE 1. Sampling Augers



APPENDIX A (Cont'd)

Figures

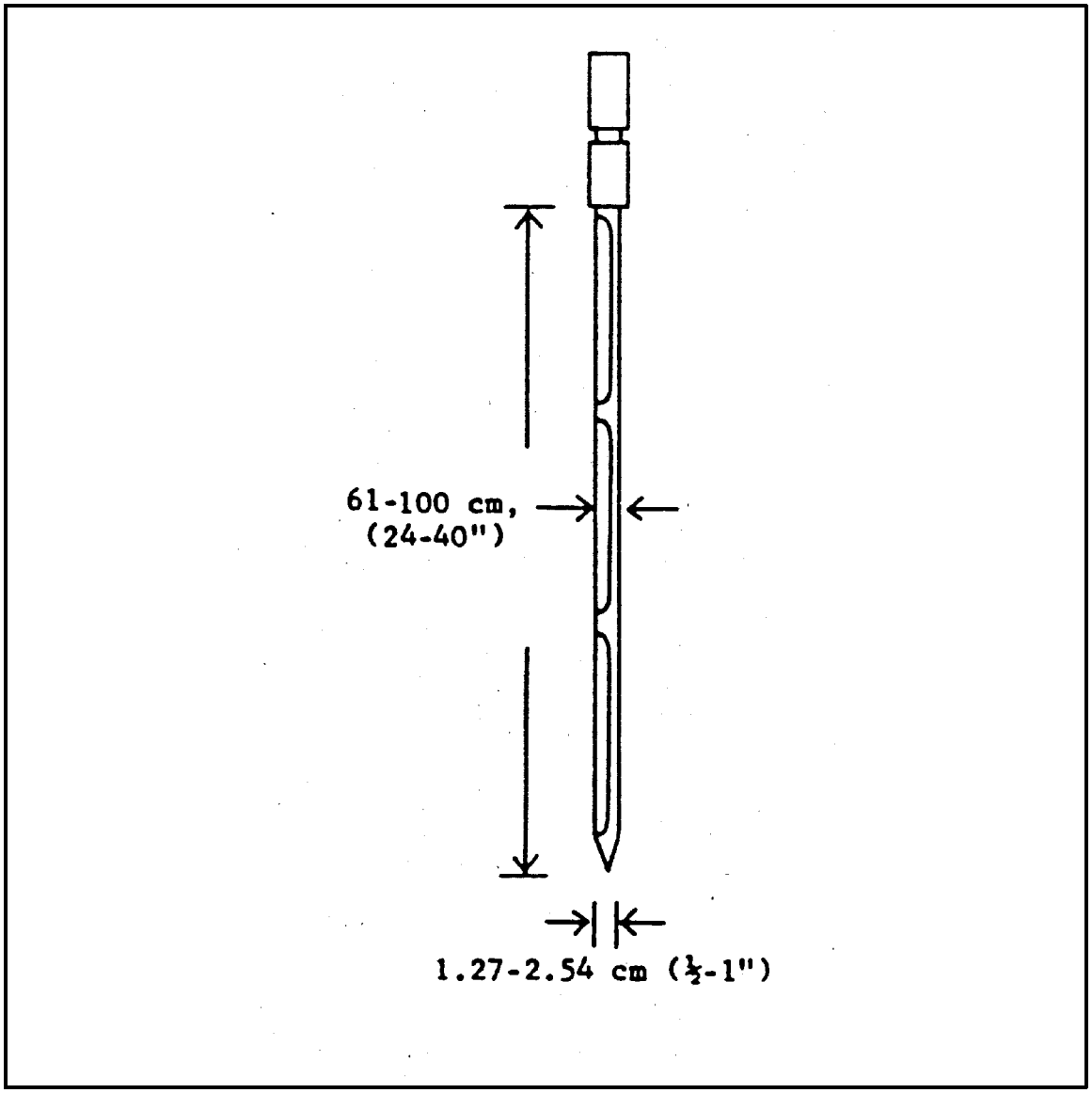
FIGURE 2. Sampling Trier



APPENDIX A (Cont'd)

Figures

FIGURE 3. Grain Sampler



AIR SAMPLING FOR METALS [NIOSH Method 7300, Elements]

SOP#: 2119
DATE: 10/07/94
REV. #: 0.0

1.0 SCOPE AND APPLICATION

The purpose of this standard operating procedure (SOP) is to define the proper sample collection technique for air sampling for elements (metals), as well as delineate the typical working range of the method and indicate potential interferences. Elements covered by this method include the metals listed in Table 1 (Appendix A).

These are standard (i.e., typically applicable) operating procedures which may be varied or changed as required, dependent upon site conditions, equipment limitations or limitations imposed by the procedure. In all instances, the ultimate procedures employed should be documented and associated with the final report.

Mention of trade names or commercial products does not constitute U.S. Environmental Protection Agency (U.S. EPA) endorsement or recommendation for use.

2.0 METHOD SUMMARY

Air sampling for elements (metals) involves passing a known quantity of air across a mixed cellulose ester (MCE) filter. The particulate phase of the air, with a nominal size of greater than or equal to 0.8 microns (µm), is trapped in the filter.

This method requires air sampling utilizing 37 millimeter (mm), 3-stage cassettes loaded with 0.8 um MCE filters and support pads. The approximate minimum and maximum sample volumes required for detection of the metals of interest are listed in Table 1 (Appendix A).

3.0 SAMPLE PRESERVATION, CONTAINERS, HANDLING, AND STORAGE

No preservatives or special storage conditions are required. However, the samples should be stored with the filter upright and transported at or near ambient conditions to prevent significant deterioration of the samples. When transporting and handling the samples, prevent impact and vibrations which would dislodge particulates from the filters.

4.0 INTERFERENCES AND POTENTIAL PROBLEMS

A potential problem with the sampling method is over-loading of the filter. This can disrupt flow, consequently producing falsely low analytical results. Periodic checking of the filter and pump can predict this condition and sample cassettes can be changed during the sampling period. The multiple filters would be analyzed as one sample with the total volume indicated on the Chain of Custody record.

5.0 EQUIPMENT/APPARATUS

The following equipment is required for air sampling for elements:

- Low or medium volume air pumps
- Tygon tubing
- 0.8 µm MCE filters with support pads
- 37 mm 3-stage cassettes
- Hose-barb filter adapters
- Air flow calibration standard (calibrated rotameter or bubble meter)
- Screw driver set
- Air Sampling Worksheets and sample labels
- Chain of Custody records
- Particulate monitoring equipment (RAM)
- Protective clothing
- Whirl bags

6.0 REAGENTS

This section is not applicable to this SOP.

7.0 PROCEDURE

7.1 Preparation

1. Determine the extent of the sampling effort, the sampling methods to be employed, and the types and amounts of equipment and supplies needed.

2. Obtain and organize the necessary sampling and monitoring equipment.

3. Decontaminate or pre-clean equipment, and ensure that it is in working order. Precalibrate sampling equipment, if possible.

4. Prepare scheduling and coordinate with staff, client, and regulatory agency, if appropriate.

5. Perform a general site survey prior to site entry in accordance with the site-specific Health and Safety Plan.

6. Use stakes, flagging tape, or other appropriate means to mark all sampling locations. If necessary, the proposed locations may be adjusted based on site access, property boundaries, and surface obstructions.

7. Make an estimate of the airborne concentrations of the elements of concern. It may be possible to extrapolate the concentration of particulates by assuming similar percentages of metals are present in the airborne particulates as in the soils. However, it should be noted that this is only a rough estimate. If estimation of the airborne concentration of metals is not possible, then sample volumes should remain within the limits recommended in Table 1 (Appendix A).

8. Arrange for sample analysis by an appropriately certified laboratory and check with the laboratory for any special requirements (e.g., additional lot blanks).

7.2 Calibration

Calibrate the required number of sampling pumps in the following manner:

1. Assemble the calibration train as shown in Figure 1 (Appendix A) using a representative 37 mm, 3-stage filter cassette loaded with a 0.8 µm MCE filter and support pad (outlet plug removed), tygon tubing, a hose-barb filter adapter, a rotameter, and an air sampling pump. Depending on the required flow rate, a low volume or a medium volume sampling pump may be required. Refer to Figure 2 (Appendix A) for an illustration of the components of the filter cassette.

2. Turn on the pump and adjust the flow using the flow adjust mechanism until the float ball on the rotameter is aligned with the rotameter's precalibrated flow rate value. A sticker on the rotameter should indicate this value.

3. Affix a sticker to the pump indicating flow rate and media.

7.3 Sampling

1. Assemble the sampling trains with clean filter cassettes (Figures 3 and 4, Appendix A).

2. Verify the pump calibration by removing the inlet plug from the cassette, attaching a rotameter with Tygon tubing and turning on the sampling pump. Ensure that all connections are tight. Record the actual flow rate on the Air Sampling Worksheet. Replace the inlet plug until ready to sample.

3. Set the sampling pump timer (low volume pumps) for the appropriate sampling time as determined by the Work Assignment Manager, or record the elapsed timer readings (medium volume pumps) on the Air Sampling Worksheet. This will be dictated by the type of sampling pump being utilized.

4. Deploy the sampling pumps as indicated in the sampling plan, following site health and safety procedures.

5. Remove the cassette cap or inlet plug from the cassette. Sampling for elements can be conducted with the cassettes open-faced (cassette cap removed) or closed-faced (only inlet port plug removed). Open-faced is preferred because it permits an even loading of the filter cassette and should be used

whenever high particulate concentrations are expected. This allows greater particulate loading of the filter. However, either method is acceptable since the entire filter is used during sample analysis. Closed-faced sampling is typically performed when there is a possibility that the sample may be shaken and particulates may be lost.

6. Turn on the sampling pump and allow it to run for the sampling period determined by the Work Assignment Manager.

7.4 Post Sampling

1. Verify the sampling period by reading the sample run time (low volume pumps) or by checking the elapsed time on the counter (medium volume pumps). Record the sampling time on the Air Sampling Worksheet and turn off the pump.

2. Verify the pump calibration by attaching a rotameter with Tygon tubing and turning on the sampling pump. Record the actual flow rate on the Air Sampling Worksheet. Insert the inlet plug.

3. Remove the sampling cassette from the sampling train and insert the outlet plug.

4. Complete the Air Sampling Worksheet and calculate the sample volume.

5. Label the sample and place it in a whirl bag for transport to the laboratory for analysis.

6. Prepare the samples (including QC samples) for transport by packing them in a shipping container with bubble wrap or styrofoam pieces. Complete a Chain of Custody record in accordance with applicable Chain of Custody Procedures.

8.0 CALCULATIONS

The total volume of a sample is calculated by multiplying the total sample time by the flow rate. The total volume for each sample must be indicated on the Chain of Custody Record.

9.0 QUALITY ASSURANCE/ QUALITY CONTROL

The following general QA procedures apply:

1. All data must be documented on Air Sampling Worksheets or within site logbooks.

2. All instrumentation must be operated in accordance with operating instructions as supplied by the manufacturer, unless otherwise specified in the work plan. Equipment checkout and calibration activities must occur prior to sampling/operation and they must be documented.

The following specific QC activities apply:

1. Provide one field blank per sampling event or per 20 samples, whichever is greater. The field blank should be handled in the same manner as the sampling cassette (remove/replace cap and plug, and transport) except that no air is drawn through it.

2. Collect one collocated sample per sampling event or per 10 samples, whichever is greater. Collocated samples are two samples collected adjacent to each other during the same time period at the same flow rates.

3. Include a minimum of two lot blanks per manufacturer's lot of sampling cassettes utilized per sampling event. Consult with the analytical laboratory to determine if additional lot blanks are required.

10.0 DATA VALIDATION

Results of the QA/QC samples will be evaluated for contamination. This information will be utilized to qualify the environmental sample results accordingly with the project's data quality objectives.

11.0 HEALTH AND SAFETY

When working with potentially hazardous materials, follow U.S. EPA, OSHA, or corporate health and safety procedures.

12.0 REFERENCES

[(1)]NIOSH Manual of Analytical Methods, NIOSH Method 7300, Elements (ICP) (Issued 02/15/84).

APPENDIX A

Table

TABLE 1. Metal Concentrations are Anticipated to be at or Near the Threshold Limit Value (TLV)

Element (Symbol)	Minimum Air Volume to be collected - Liters	Maximum Air Volume to be collected - Liters
Silver (Ag)	250	2000
Aluminum (Al)	5[1]	100[1]
Arsenic (As)	5	2000
Beryllium (Be)	1250	2000
Calcium (Ca)	5	200
Cadmium (Cd)	13	2000
Cobalt (Co)	25	2000
Chromium (Cr)	5	1000
Copper (Cu)	5	1000
Iron (Fe)	5	100
Lithium (Li)	100	2000
Magnesium (Mg)	5	67
Manganese (Mn)	5	200
Molybdenum (Mo)	5	67
Sodium (Na)	13	2000
Nickel (Ni)	5	1000
Phosphorus (P)	25[1]	2000[1]
Lead (Pb)	50	2000
Platinum (Pt)	1250	2000
Selenium (Se)	13	2000
Tin (Sn)	5	500
Tellurium (Te)	25	2000
Titanium (Ti)	5	100
Thallium (TI)	25	2000
Vanadium (V)	5	2000
Tungsten (W)	5[1]	200[1]
Yttrium (Y)	5	1000
Zinc (Zn)	5	200
Zirconium (Zr)	5	200

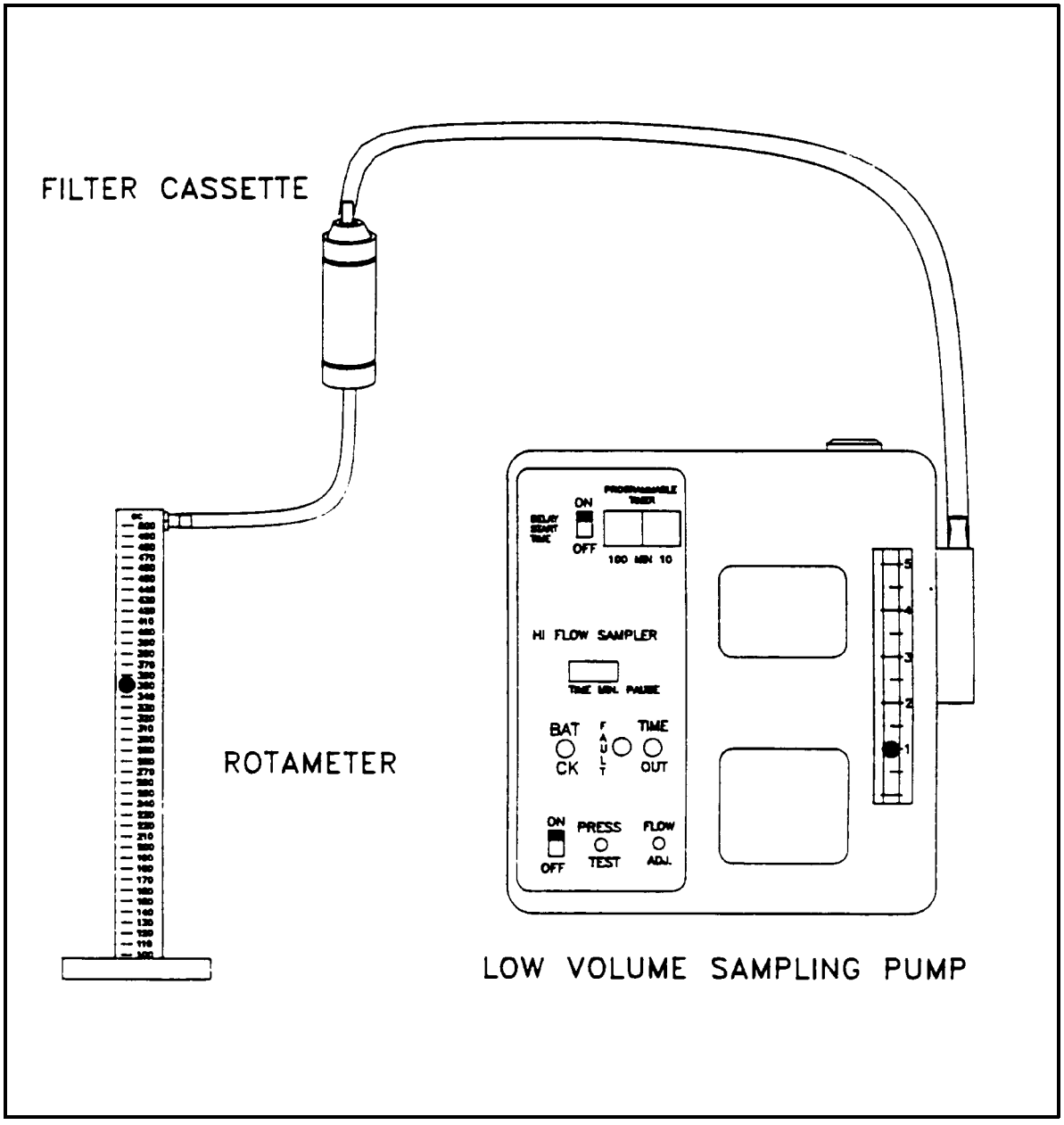
NOTE: Do not exceed a filter loading of approximately 2mg total dust.

[1] Greater volumes may be required if the anticipated concentration is less than the ACGIH TLV.

APPENDIX B

Figures

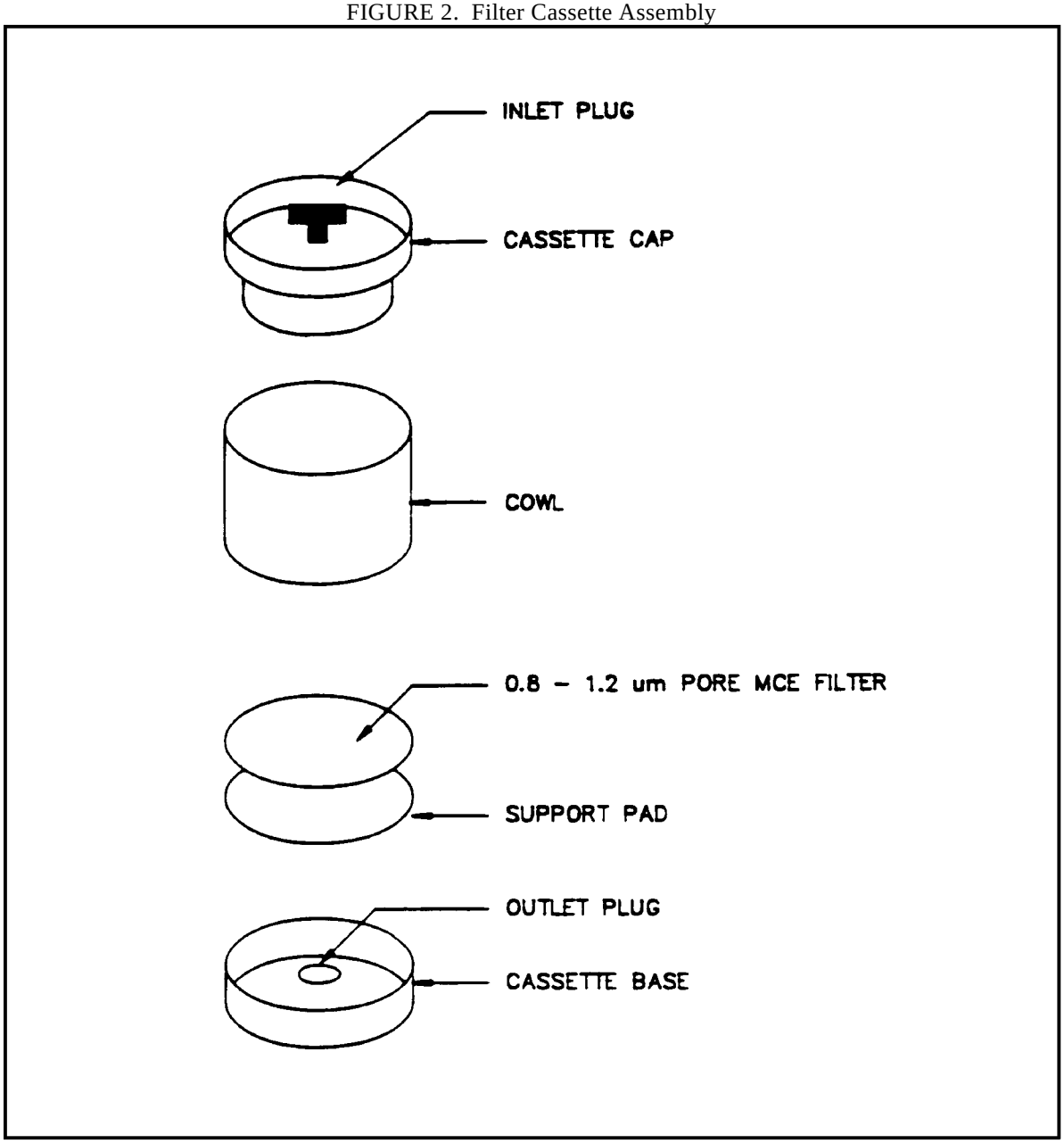
FIGURE 1. Calibration Train with Low Volume Sampling Pump



APPENDIX B (Cont'd)

Figures

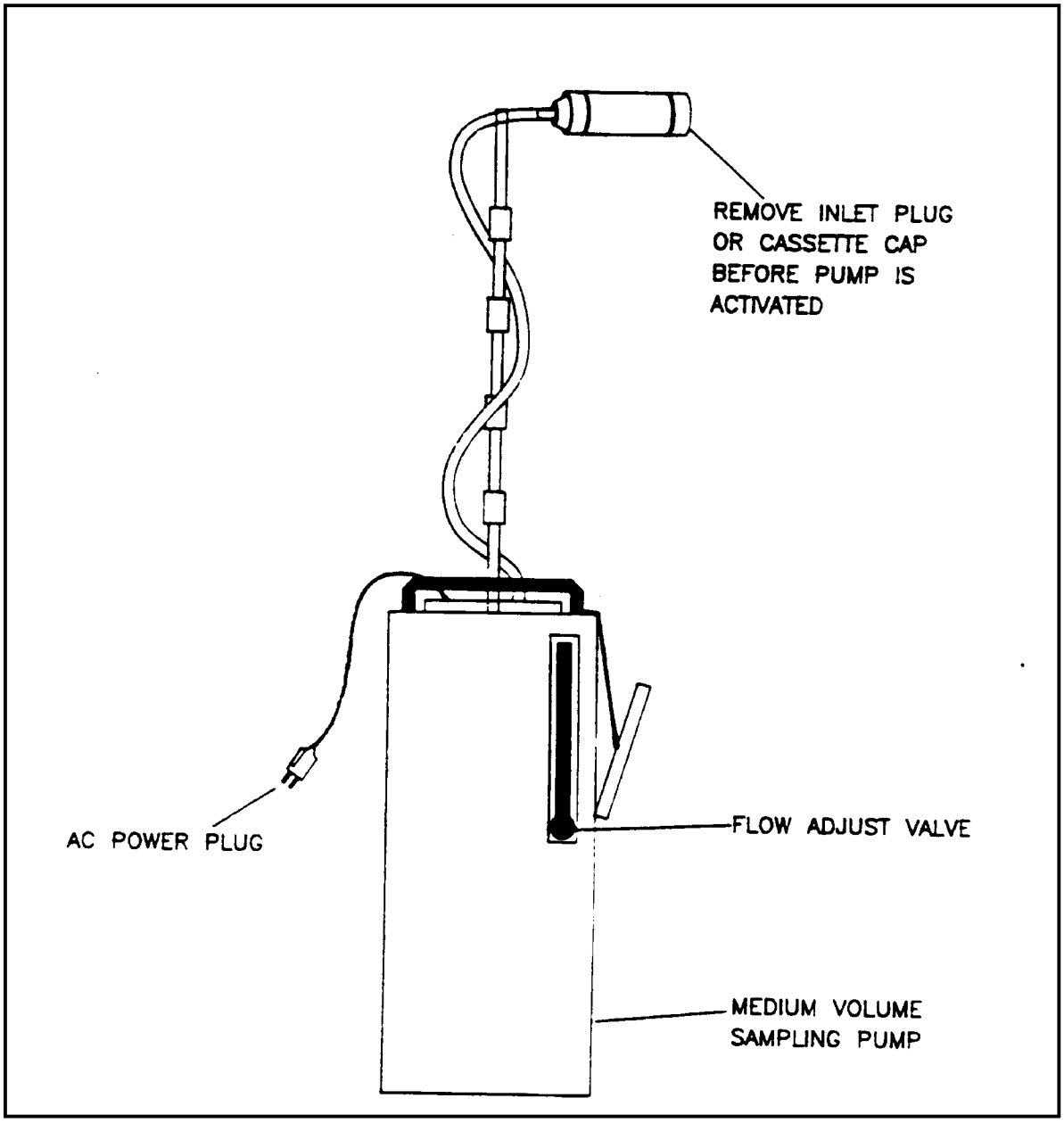
FIGURE 2. Filter Cassette Assembly



APPENDIX B (Cont'd)

Figures

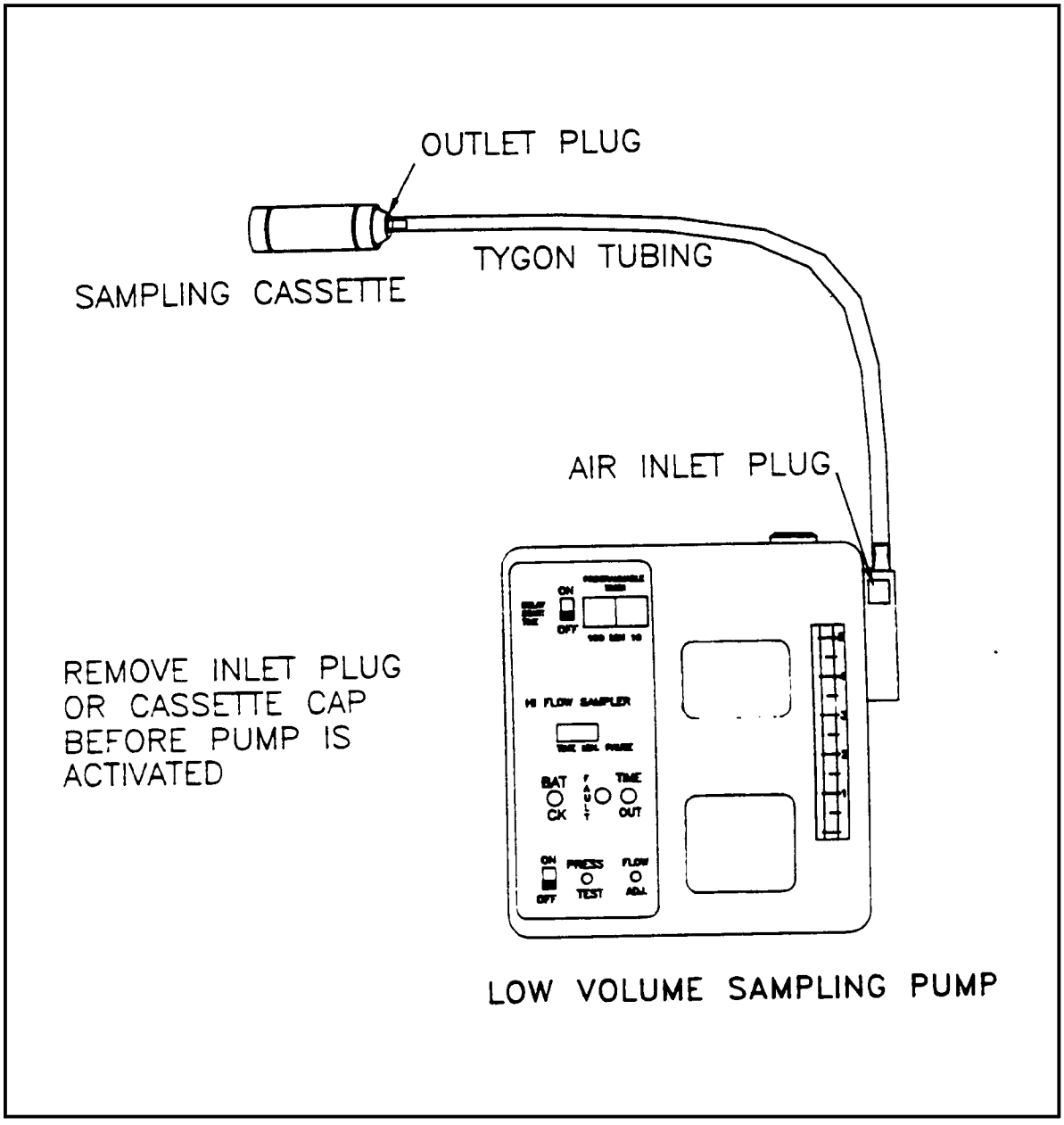
FIGURE 3. Medium Volume Sampling Train



APPENDIX B (Cont'd)

Figures

FIGURE 4. Sampling Train with Low Volume Sampling Pump



STANDARD OPERATING PROCEDURE

No. 2314.1A

Hi-Vol Operation

May 13, 1985

NOTE: This SOP is a pre-existing Environmental Monitoring & Compliance Branch SOP (SOP No. FB010B) which has been incorporated into the Environmental Services Division Operations & Quality Assurance Manual with a new SOP Number.

APPROVED:

John R. Heliz — NOV 28 1989
Chief, Emergency Planning & Response Branch — Date

Thomas T Holloway — 11/17/89
Chief, Environmental Monitoring & Compliance Branch — Date

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Chief, Laboratory Branch — Date

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Recertified:

Branch	EMWC	EMWC	EMWC	EMWC/Hinkhan	
Date	7/05/93	2-1-00	4/3/03	10/28/05	

OPERATING PROCEDURE FOR
TOTAL SUSPENDED PARTICULATE MONITOR

1 SCOPE AND APPLICATION

1.1 This procedure outlines the operation of a Total Suspended Particulate Monitor referred to as a hi-vol. This procedure includes filter preparation, calibration of the orifice and flow recording device, maintenance and quality control.

1.2 Principle of Operation

1.2.1 Air is drawn into a covered housing and through a filter by a high-flow-rate blower at 1.00m^3/min to 1.70m^3/min (35-60 ft^3/min) that allows total suspended particulates (TSP) with diameters of <100 um (stokes equivalent diameter) to collect on the filter surface. The mass concentration (ug/m^3) in ambient air is computed by measuring the mass of TSP collected and the volume of air sampled.

2 FILTER PREPARATION

2.1 The 8 by 10 inch glass fiber filters are purchased by the Environmental Monitoring and Support Laboratory, Research Triangle Park, North Carolina. These filters meet Federal specifications.

2.1.2 The filters are prenumbered by the contract supplier on only one corner.

2.2 The filters must be visually inspected, using a lighted box, for pin holes, weak spots, dirt or other foreign material embedded in the fiber. Both sides should be inspected and the flawed filters discarded.

2.3 Equilibrate the filters in a conditioning environment (i.e., a dessicator) for 24 hours before weighing them.

2.3.1 The conditioning environment must average between 20° and 25°C and not vary more than $\pm$ 30°C with a relative humidity (RH) <50 percent without variation of more than $\pm$ 5 percent RH.

2.4 The balance must be checked prior to weighing filters.

2.4.1 Following the manufacturers instructions check the balance using a standard class-S weight between 3 and 5 g. Record the weight. If the weight differs more than $\pm$ 0.5 mg from the standard weight the balance must be repaired and recalibrated before filters are weighed.

2.5 Record the room temperature and relative humidity.

2.6 Weigh each flat filter to the nearest mg and record the filter number, date, weight and atmospheric conditions in the filter notebook. Only a few months worth of filters should be weighed at any one time.

2.6.1 Reweigh 8 percent of each batch of filters weighed. Record the weight, indicating that it is a reweigh. If the reweigh differs by ± 5 mg for any reweighed filter, recheck the balance and weigh each filter again recording all data.

2.6.2 Store the filters, in numerical order, in the original box or in manila folders.

3 ELAPSED - TIME METER

3.1 All elapsed-time meters must be checked when new against a timepiece of known accuracy.

3.2 Recheck the meters whenever they appear to be giving faulty readings.

3.3 A gain or loss of 2 minutes or more for a 24-hour period warrants adjustment or replacement of the indicator.

3.4 Record all checks or adjustments in the hi-vol notebook.

4 ON-OFF TIMER

4.1 All timers must be checked to be certain that they turn the sampler on and off within ± 15 minutes of the assigned time of day.

5 ORIFICE CALIBRATION

5.1 Calibrate the orifice annually using the calibration procedure detailed in the "Hi-Vol Sampler Audit Procedure," SOP No. 2317.2A.

5.2 If any nicks or dents are observed on the orifice or plates recalibrate the orifice before use.

6 HI-VOL SAMPLER CALIBRATION

6.1 Samplers should be calibrated when first purchased, after major maintenance, if the flow rate measuring device is changed or repaired, or if any one audit point deviates more than ± 7 percent from the calibration curve.

6.2 Samplers with a flow recorder.

6.2.1 Assemble the sampler with the calibration orifice clamped to the filter holder. Attach a water or oil manometer to the calibration orifice with flexible tubing. Attach the flow recorder to the pressure tap of the hi-vol motor housing.

6.2.2 Turn the sampler on and allow the motor to warm up for at least 5 minutes at 115v. If a stepdown transformer is used during normal operation, have the transformer in operation during calibration.

6.2.3 Record the flow recorder number, hi-vol motor number, and the date.

6.2.4 Put a clean recorder chart on the recorder and check the recorder for proper operation. Zero the pen if necessary.

6.2.5 Turn the motor on and record the manometer and flow recorder readings after they stabilize.

6.2.6 With the motor off, change load plates and repeat step 6.2.5 for each load plate.

6.2.7 Calibrate and record the air flow rates from the hi-vol orifice calibration curve for each flow recorder reading. Record the barometric pressure (mm Hg) and the temperature (°C).

6.2.8 Calculate the sampler calibration curve, slope (m) and intercept (b), by linear regression analysis of the corrected recorder readings (x) versus the standard sample flow rates (y).

7 SAMPLING: SET UP, COLLECTION, ANALYSIS

7.1 Prior to the sampling day, place a preweighed filter into a cassette making sure it is centered evenly and the filter number is down so that the sample will be collected on the un-numbered side of the filter.

7.1.2 In the filter notebook, record the filter number, the sample date and initial elapsed time meter reading. Also include the site ID number and the hi-vol motor number.

7.1.3 Check the on-off timing device for the proper setting and check the elapsed-time meter.

7.1.4 Place a recording chart on the flow recorder, checking the zero and the pen.

7.2 At the conclusion of the sampling period record the final elapsed time meter reading. Before removing the cassette, turn the hi-vol motor on and check the final flow for consistency with the initial flow.

7.2.1 Record the date and the sampling time indicated on the elapsed time meter in the filter notebook. Note any abnormalities.

7.2.2 Remove the filter cassette and replace the cover. Remove the flow recording trace.

7.2.3 It may be more convenient to remove the filter from the cassette inside a building.

7.2.4. When removing the filter grasp it gently at the ends.

7.2.5 Fold it lengthwise with the exposed side in. Place the filter in the envelope or folder. Replace in the conditioning environment until it is weighed.

7.3 Obtain the average temperature and average pressure for the sampling period by any means listed below.

7.3.1 The average temperature and pressure may be obtained by telephoning the national Weather Bureau in the area.

7.3.2 A temperature recording device may be attached to the hi-vol and the low and high reading for the sampling period averaged.

7.4 Visually inspect the filters for damage and remove any embedded insects with teflon-tipped tweezers.

7.4.1 Record any abnormalities in the filter notebook.

7.4.2 Equilibrate the filters in a conditioning environment for 24-hours or 48-hours if the filters are damp.

7.4.3 Check the balance as described in section 2.4.

7.4.4 Record the date, temperature and relative humidity.

7.4.5 Weigh the exposed filters to the nearest milligram removing only one at a time from the conditioning environment. Record the weight in the filter notebook. Reweigh 8% of each group of filters as described in section 2.6.1.

7.5 Sample Calculations

7.5.1 Calculate the sample air volume (v):

$$v = Qt$$

v = air volume sampled (m^3)
Q = average sampling rate (m^3/min @ STP)
t = sampling period (elapsed time) (min)

Determine Q from the recorder chart. If the flow rate varies less than 0.11 m^3/min during the sampling period, read the flow rate from the chart at 2-hour intervals and take the average value. For greater flow rate variations, use hourly flow rate readings. Then correct the chart reading to standard temperature and pressure, and calculate the average sampling rate using the hi-vol calibration curve.

7.5.2 Calculate the TSP concentration (SP):

$$SP = \frac{(W_f - W_i)\, 10^6}{V}$$

SP = concentration of TSP (ug/m^3)
W_f = weight of exposed filter (g)
W_i = tare weight of filter (g)
v = air volume sampled (m^3)

7.6 Sample Reporting

7.6.1 Data will be reported according to the current SAROAD format.

8 MAINTENANCE

8.1 Sample Motor

8.1.1 Motor brushes should be replaced after 400-500 hours of operation. (115 v) Follow the manufacturer's instructions for replacement.

8.1.2 Replace the motor brushes sooner than 400 hours of operation if they are worn out, so that no damage to the motor will occur.

8.1.3 Inspect the armature and replace if necessary.

8.1.4 Recalibrate the motor after the initial break-in period of about 1 to 2 hours.

8.1.5 Record any maintenance performed.

8.2 Gaskets

8.2.1 Check the cassette gasket for wear when collecting the sample filters. Also check both motor gaskets for wear and/or deterioration while performing any maintenance on the motor.

8.2.2 Replace if needed, cleaning the surface throughly, sealing the gasket with rubber cement.

8.2.3 Record any maintenance performed.

8.3 Flow Transducer and Recorder

8.3.1 These devices require no routine maintenance. If they malfunction, repair or replace them.

8.4 Filter Support

8.4.1 Check the sampling head for leaks before installation.

8.4.2 Assemble the head to the hi-vol and apply a filter for resistance

9 QUALITY CONTROL

9.1 Quarterly

9.1.1 Calibrate hi-vol motors as described in Section 6 prior to use.

9.1.2 Check on-off timers, recorders and motor brushes prior to motor calibration. Examine, clean, calibrate or repair as needed.

9.1.3 Audit 25 percent of hi-vols.

9.1.4 Audit hi-vol motors after use, before they are replaced.

9.2 Semi-annually

9.2.1 Calibrate the elapsed time meters.

9.3 Annually

9.3.1 Calibrate the orifice used for hi-vol calibration.

9.4 Routinely

9.4.1 Follow the maintenance schedule when applicable.

9.4.2 Collocated samplers must be operated at two sampling sites according to the TSP procedure. They shall be located where the highest and second highest geometric mean is expected.

HI-VOL MAINTENANCE

DATE								
SITE I.D.								
MOTOR NUMBER								
MOTOR BRUSHES								
ARMATURE								
GASKETS								
RECORDER								
FILTER SUPPORT								
CALIBRATE ELAPSED TIME METER								
CALIBRATE HI-VOL								
CALIBRATE ORIFICE								

SOP 2341.1A

ELAPSED TIME METER CALIBRATION SHEET

CALIB. TIMEPIECE READING			DATE OF CALIBRATION		ELAPSED TIME INDICATOR SERIAL NO.	TEST PERIOD	ELAPSED TIME INDICATOR READING			ELAPSED TIME METER CORREC-TION FACTOR	CALIB. TIME SPAN (min.)	SIGNATURE
Start	Stop	Span	Start	Stop			Start	Stop	Span			

HI-VOL FILTERS

DATE	BALANCE ROOM Temp. °C	Humidity	BALANCE CHECK S Weight	Weight	FILTER NUMBER	INITIAL WEIGHT	REWEIGH

DATE	BALANCE ROOM Temp. °C	Humidity	BALANCE CHECK S Weight	Weight	FILTER NUMBER	FINAL WEIGHT	REWEIGH

PARTICULATE SAMPLES

STATION SAROAD NO.	SAMPLER IDENTIFICATION	SAMPLE DATE	FILTER NUMBER	SAMPLING TIME Initial Lapse Meter	SAMPLING TIME Final Lapse Meter	SAMPLING TIME Sample Time (min.)

FILTER NUMBER	AMBIENT CONDITIONS Avg. Temp. (°C)	AMBIENT CONDITIONS Avg. Press. (mmHg)	SAMPLE FLOW RATE Avg. Chart Reading	SAMPLE FLOW RATE Sample Flow Rate (m^3/min)	SAMPLE WEIGHT (g)	SAMPLE AIR VOL. (m^3)	PART. CONCEN. (ug/m^3)

HI-VOL SAMPLER CALIBRATION DATA SHEET

Site Name _____ Initial Sampling Date _____

Motor Number _____ Final Sampling Date _____

Recorder Number _____ Total Sampling Time (Hrs.) _____

INITIAL CALIBRATION						FINAL CALIBRATION					
	Flow Recor. Readings		Orifice Mand. Reading				Flow Recor. Readings		Orifice Mand. Reading		
Calib. Point	Ambient Cond. R	Corr. to St. Cond. R_s	Ambient Cond. $\triangle H$	Corr. to Std. Cond. X	Flow Rate Q (m^3/min.)	Calib. Point	Ambient Cond. R	Corr. to Std. Cond. R_s	Ambient Cond. $\triangle H$	Corr. to Std. Cond. X	Flow Rate Q (m^3/min.)

Date _____ Orifice Number _____

Temperature (°C) _____

Pressure (mm Hg) _____

Signed _____

Date _____ Orifice Number _____

Temperature (°C) _____

Pressure (mm Hg) _____

Signed _____

STANDARD OPERATING PROCEDURE

No. 2420.1E

SAMPLE RECEIPT AND LOG-IN

March 31, 2006

by

Nicole Roblez
ENSV/RLAB/CATS

APPROVED:

Peer Reviewer — Date: 3-30-06

Laboratory Manager — Date: 30 March 2006

Independent QA Reviewer — Date: 3/31/06

Recertified:

Reviewer					
Date					

TABLE OF CONTENTS

ATTACHMENTS:

A. PURPOSE AND APPLICABILITY

The purpose of this Standard Operating Procedure (SOP) is to establish a uniform policy and procedure for the receipt and log-in of environmental samples shipped by Environmental Protection Agency (EPA) field personnel or their contractors to the Environmental Services Division (ENSV) Regional Laboratory (RLAB). The policies and procedures contained in this SOP are applicable to all ENSV personnel and EPA contractors.

B. SUMMARY OF METHOD

Sample shipments, normally in ice chests (coolers), are delivered on a daily basis by either field personnel or air courier. In some instances, samples are shipped via over night carrier (e.g. Federal Express (FedEx) or Airborne Express) to the ENSV RLAB.

The Regional Sample Control Coordinator (RSCC) (or designated backup) receives all samples. The RSCC's sample custodial duties reside in the Contracts and Technical Support (CATS) Section, RLAB/ENSV. The RSCC (or designated backup) is available to receive samples Monday through Friday (excluding holidays) from 7:30 a.m. until 4 p.m. Deliveries on days and times other than these require scheduling through the RLAB Program Manager. However, RLAB does maintain a secured refrigerator in the receiving area for night and weekend deliveries allowing the Project Manager to temporarily store samples until they can relinquish them to the RSCC (or designated backup). When samples are left in that refrigerator, it is the Project Manager's responsibility to notify the RSCC (or designated backup) that samples are stored there (e.g., voice mail message or written note).

The RSCC (or designated backup) coordinates delivery of sample shipments with field personnel. When samples arrive, the sample collector, Purchasing and Receiving, or other ENSV personnel will contact the RSCC (or designated backup) who will then proceed to the sample receiving area.

During this time, either the Purchasing and Receiving person (or designated backup) or RSCC (or designated backup) will perform the radioactive materials cooler/sample survey according to SOP 2420.15, Management of Radioactive Materials in the Laboratory Environment.

Personnel delivering samples to the RLAB are to observe the radioactive materials survey and sample receipt procedures for the purpose of alleviating any questions and/or problems relating to the radioactive materials survey, samples or paperwork.

No samples are to be "dropped-off" without the appropriate notification to the RSCC (or designated backup). Any such samples will not be processed until the Project Manager contacts the RSCC (or designated backup).

All samples delivered must have an associated Analytical Services Request (ASR) form on file (Attachment 1 contains a copy of the ASR form that is currently in use). The only exception to this will be for emergency spills or special enforcement actions that cannot legitimately be pre-planned. Acceptance of such samples will be on a case-by-case basis with specific approval of the RLAB Program Manager. For samples of this nature, the Project Manager must initiate and be in the process of routing the ASR for signature/acceptance concurrent to sample delivery.

C. DEFINITIONS\ACRONYMS

ANSETS	Analytical Services Tracking System
ASR	Analytical Services Request
BODs	Biological Oxygen Demand
CATS	Contracts and Technical Support
CLP	Contract Laboratory Program
COC	Chain Of Custody Record
ENSV	Environmental Services Division
EPA	Environmental Protection Agency
EPA TO#	REAP EPA Task Order Number
F2L	CLP Forms2Lite5.1
FedEx	Federal Express
R7LIMS	Laboratory Information Management System
PM	Project Manager
PO	Project Officer
Project	Sampling effort
QA	Quality Assurance
QAPP	Quality Assurance Project Plan
QC	Quality Control
REAP	Region 7 Environmental Analysis Program
ESAT	Region 7 Environmental Services Assistance Team
RLAB	Regional Laboratory

RQAM	Regional Quality Assurance Manager
RSCC	Regional Sample Control Coordinator
RSTC	Region 7 Science and Technology Center
SMO	Sample Management Office
SOP	Standard Operating Procedure
SRN	Sample Receipt Notification
Tags	Sample container labels
TAT	Turn-around Time
VOAs	Volatiles

D. PERSONNEL QUALIFICATIONS

Personnel performing this task should have a basic knowledge of RLAB sample management procedures and the computer software utilized.

1. Responsibilities of Personnel Performing this Task:

   a. Project Manager

      (1) The Project Manager submits a completed ASR to the RLAB at least 30 days before the projected sampling delivery date.

      (2) The Project Manager collects and ships properly labeled, preserved, and packaged samples to ENSV in a timely fashion.

      (3) The Project Manager is responsible for the accuracy and completeness of all accompanying paperwork. If any changes are required as a result of the sampling (e.g., sample number changes, additional analytes, samples not collected, quality control (QC) code additions, etc.), the Project Manager must see that these corrections are made on all paperwork. All changes made to the paperwork (sample tags, field sheets) must also be made to the information contained in the Laboratory Information Management System (LIMS). It is the responsibility of the Project Manager to supply correct information so that the RSCC (or designated backup) can reconcile the samples in LIMS. Whenever possible, any changes are made prior to the delivery of the samples. The Project Manager coordinates the changes through the appropriate person as indicated. If necessary, the RSCC (or designated backup) will assist the Project Manager when changes are noted prior to

sample collection/delivery, concurrent to sample delivery or after. The Project Manager is available to help resolve any problems with his samples or designates someone to do this for him in his absence. This requires that when delivering samples, the Project Manager stays with the RSCC (or designated backup) to answer any questions. Samples are not to be dropped off without notification to the RSCC (or designated backup) or sample receiving personnel. The Project Manager calls the RSCC (or designated backup) close to the anticipated delivery time of samples sent in by air courier (e.g., FedEx) to confirm that samples have arrived and to answer any questions the RSCC (or designated backup) may have. In the case that the Project Manager is not available and has no alternate, RLAB contacts the Project Manager's supervisor for any necessary reconciliation information in order to resolve problems quickly and initiate sample processing.

b. Regional Sample Control Coordinator

(1) The RSCC (or designated backup) performs the radioactive materials cooler/sample survey according to SOP 2420.15, Management of Radioactive Materials in the Laboratory Environment if the Purchasing and Receiving person (or designated backup) has not performed this task.

(2) The RSCC (or designated backup) verifies the presence of all samples, checks all documentation, and signs the Chain Of Custody Record (COC) after all paperwork is complete and accurate.

(3) The RSCC (or designated backup) works with the Project Manager to obtain correct information and put the amended information into LIMS.

(4) The RSCC (or designated backup) notifies the Project Manager of problems which prevent acceptance of the samples by ENSV. RLAB maintains all samples received in a secure location including those pending reconciliation of problems.

(5) The RSCC (or designated backup) is responsible for the proper storage, tracking and/or distribution of the samples to the appropriate in-house and contract laboratories (this includes while the sample is in transit to the contract laboratory facility). Refer to SOP 2420.2 for sample storage procedures.

(6) The RSCC (or designated backup) logs samples into LIMS and prepares an electronic sample receipt notification to inform RLAB analytical personnel that the samples have been delivered to the RSTC and the location of the refrigerators in which they have been placed.

E. PROCEDURAL STEPS

1. Sample Receipt Procedure

a. The RSCC (or designated backup) is notified of sample receipt through periodic checks of the sample receiving area and through notification by sampling or ENSV personnel. The RSCC (or designated backup) identifies the project and ASR number from the COC and field sheets, and locates a copy of the ASR.

(1) If all paperwork is sealed with the samples in the cooler (often taped to the inside lid), the RSCC (or designated backup) must open the cooler(s) (see Section E.4.), in order to identify the ASR number.

(2) Except under extraordinary circumstances, the RSCC (or designated backup) must have a copy of the completed and approved ASR before samples can be received. If an ASR has not been received, the RSCC (or designated backup) notifies the Project Manager and assists him with information on how to complete and route the ASR.

b. The RSCC (or designated backup) performs the radioactive materials cooler/sample survey according to SOP 2420.15, Management of Radioactive Materials in the Laboratory Environment if the Purchasing and Receiving person (or designated backup) has not performed this task

c. The RSCC (or designated backup) notes the presence (or absence) and condition of custody tape seals on the outside of the cooler(s). A notation of this information is made in the file.

d. A completed COC (Attachment 2) must accompany each shipment of samples. The RSCC (or designated backup) requests that when a COC is not present, incomplete, or incorrect, that an amended or new one be submitted by the Project Manager or his designee.

e. The RSCC (or designated backup) removes all samples from the cooler(s) and notes the condition of each sample. Sample descriptions such as intact, broken, air bubbles present (for volatiles), or above 4 degrees Celsius (ice melted) are used to describe the sample condition. The sample condition is noted on the COC and field sheets (Attachment 4). Any additions or notes made on the COC or field sheets should be initialed and dated next to the correction or entry by the person making them. Any corrections should be done by placing a single line through the error and then noting the correct information. All corrections must be dated and signed by the person making the correction. Under no circumstances is information to be obliterated or whited out.

The Project Manager will be asked to make the necessary changes or corrections on the paperwork. In instances when the Project Manager is on site, he may designate the RSCC (or designated backup) to make minor changes for him. This may extend to changing a sample number or analysis listed, but does not include generating and completing all field paperwork. The RSCC (or designated backup) makes the indicated changes and sends the Project Manager a copy (no copy can be made of changes to sample tags on containers) of all paperwork changes. The Project Manager must respond back to the RSCC (or designated backup) as soon as possible if the corrections made are not as he intended. If no response is received within four working days, the RSCC (or designated backup) assumes that the Project Manager concurs.

f. If a sample is broken and there are no other containers from which the analysis can be done, the RSCC (or designated backup) initiates a memo of Non-Reportable Results (Attachment 5). This memo is sent to the Project Manager and the RLAB Data Coordinator to alert them to the fact that no data will be forthcoming for that sample/analysis. In addition, the RSCC (or designated backup) personally notifies the Project Manager of the sample loss so that the sample can be retaken at the Project Manager's

discretion. A notation is made on the COC to indicate any broken containers.

In the case of broken or leaking samples, all packing material (bubble or foam wrap, vermiculite, broken glass, etc.) and sample residue in the cooler are considered hazardous trash until proven otherwise through laboratory analysis of the samples.

g. The RSCC (or designated backup) verifies specific items on each of the samples and associated documents of the shipment. This includes determining if all documents/samples are consistent and accurate with regard to one another. It also includes identifying discrepancies in the use of EPA sample numbers. This circumstance usually occurs when the sampler adds additional unplanned samples. The Project Manager takes care of any necessary paperwork reconciliation to include assigning new EPA sample numbers if the ones identified are inappropriate.

(1) The RSCC (or designated backup) verifies the following specific information on the COC with respect to the sample container labels and field sheets:

(a) EPA Project/ASR number
(b) EPA sample numbers
(c) Number and type of sample containers
(d) Sample matrix

(2) The RSCC (or designated backup) verifies the following specific information on the sample containers with respect to the COC and field sheets:

(a) EPA Project/ASR number
(b) EPA sample numbers
(c) Type of containers
(d) Analysis
(e) Preservation

The RSCC (or designated backup) also observes the amount of sample received and determines if a sufficient quantity of sample was submitted to conduct the requested analyses. If an obviously insufficient sample was submitted, the RSCC (or designated backup) initiates a Notice of Non-Reportable Results form that is

forwarded to the Project Manager and Data Coordinator to inform them that no results will be forthcoming for that sample/analysis. A notation is also made on the COC. The RSCC (or designated backup) personally contacts the Project Manager and informs him of the situation so that he can retake the sample. In some instances, it is not determined until after the sample is in the laboratory that there is insufficient sample to conduct any or all of the tests requested. In these instances, RLAB informs the Project Manager as soon as possible of the existing situation. In some cases where some tests can be performed, but not all, the Project Manager determines the order and priority of tests for the sample.

(3) The RSCC (or designated backup) verifies the following specific information on the field sheets with respect to the COC and sample container labels:

(a) EPA Project/ASR number
(b) EPA sample numbers
(c) Matrix
(d) Container
(e) Analysis name
(f) Preservation

In addition to these items, the RSCC (or designated backup) notes any comments that may be inconsistent with the analysis requested (e.g., field filtered for total metals), information necessary to report the data (e.g., sample area or volume information for wipe and air samples), and if the field sheet is signed by the sampler in the space indicated.

h. When the information on the COC, field sheets, and sample tags is verified as complete and correct, the RSCC (or designated backup) checks the appropriate box at the bottom of the COC, "sealed" (custody taped) or "unsealed" (no custody tape present), and signs each COC and records the date and time of sample transfer. This action completes the sample receipt process and RLAB officially accepts custody of the samples. The yellow copy (or a photocopy) of the signed COC is given or mailed to the Project Manager. The original (white copy) is retained in the ENSV sample activity file maintained by RLAB.

2. Sample Log-In Procedure

a. From the ASR, the RSCC (or designated backup) identifies whether samples will be analyzed in-house (RLAB) or analyzed through the contract laboratory services (e.g., CLP or REAP). The samples are placed in the appropriate walk-in refrigerators located in the secured sample storage room (L-55) with the following exceptions: in-house volatile (VOA) samples are put in the refrigerator in the VOA analysis laboratory (L-30 for EPA and L-32 for ESAT, respectively). In-house samples requiring immediate analysis because of short holding times (Biological Oxygen Demand (BODs), chromium VI, or for other reasons, are delivered directly to the analyst by the RSCC (or designated backup).

b. The in-house sample receipt notification (SRN) is sent to RLAB personnel. The REAP and CLP sample receipt notification (Attachment 6) is only sent to the CATS staff. The information contained in the SRN includes ASR number, Project ID, due date (as assigned on the ASR by the RLAB Manager or designated person), the sample numbers, matrix, and the analyses requested. In the remarks area of each message type where the samples are stored, whether there will be more samples arriving, or not, who has the laboratory assignment, and the date when samples were collected. The sending of sample receipt notification instructions are outlined in section E.4 of this SOP.

(1) If the activity is being field shipped directly to a CLP or REAP laboratory, it is imperative that the Project Manager work closely with the RSCC (or designated backup). The Project Manager must return ENSV's copy of all paperwork (COC, field sheets, CLP shipment records, and/or REAP shipping documents) as soon as the samples are shipped to the CLP or REAP laboratory. The Project Manager works with the RSCC (or designated backup) to reconcile any discrepancies identified on the paperwork. The RSCC (or designated backup) logs the samples into R7LIMS when reconciliation of the paperwork is complete.

(2) The procedures for submitting ENSV's copy of the paperwork for samples sent directly to the REAP laboratory includes working closely with the REAP Project Officer (PO). Each Delivery Order must be coordinated and handled on an individual basis. The Project Manager or field personnel must fax ENSV's copy of all of the paperwork (COC, field sheets, and REAP shipping documents)

as soon as the samples are shipped to the REAP laboratory. The RSCC (or designated backup) and REAP PO (or designated backup) contacts the REAP laboratory informing them of the sample shipment.

c. The RSCC (or designated backup) makes the necessary entries into LIMS, reconciling the LIMS data with the sample paperwork and logs in the samples. Instructions for sample log-in are outlined in section E.3. of this SOP.

d. At the conclusion of all log-in activities for the shipment, the RSCC (or designated backup) promptly transmits the ENSV's copy of the field sheets and COC to the RLAB Data Coordinator. The Data Coordinator (or designated backup) files these documents according to the ASR number in the ENSV sample activity file maintained by RLAB.

e. If any hazardous trash is generated in the receipt of the samples, it is put in a polyethylene containment bag, tied, and labeled. The label includes the name of the sampling site, ASR number, and the date. The hazardous trash is turned over to the Sample Disposal Coordinator (or designated backup) for temporary storage in Sample Holding/Disposal (L-57).

f. The RSCC (or designated backup) places all uncontaminated used packing material from the cooler(s) in the dock-area trash cans, empties all water and ice from the cooler(s), and stacks the cleaned-up cooler(s) in the dock area. If there are directions to ship back specific cooler(s), the RSCC (or designated backup) coordinates their return.

3. Procedures for Sample Log-In Into R7LIMS

a. Confirm sample information (e.g., Project/ASR numbers, sample number(s))
b. Double click R7LIMS icon
c. Type Username and press enter
d. Type Password and press enter
e. Click once on Data Manager
f. Under ASR/Samples, double click Browse ASRs
g. Arrow to the ASR number and click to select
h. Click once on Log-In Samples
i. Type sample information from the field sheets

j. If the samples are assigned to a CLP laboratory, click once on Get SMO number. Confirm the next available CLP sample number on the list provided in cubicle 202B.
k. Click once on Log-In button
l. Check each sample to verify that the Received Date is the same for each sample (including the PE sample which has already been defined in the system). The computer uses the received date to create each particular F2L download file
m. Check the sample container list, and edit as necessary, to assure that it matches the COC
n. Click Exit when all samples are logged in

4. Instructions for Sample Receipt Notification to Regional Laboratory Personnel

a. Double click R7LIMS icon
b. Type Username and press enter
c. Type Password and press enter
d. Click once on Data Manager
e. Under ASRs/Samples, double click Browse ASRs
f. Using the mouse, click once and highlight the ASR number
g. Click once on SRN button
h. At lab, arrow down to Receipt Date tab needed
i. Under Report Sample, arrow down to Numbers tab
j. Click once in the Comments box and type the following information in the Comments section:
   (1) The lab for which the samples are assigned (e.g., REAP, CLP, EPA, or ESAT);
   (2) The refrigerators or freezers where the samples will be stored for in-house analyses (e.g., 1st or 2nd walk-in refrigerators in L-55, EPA volatiles refrigerator in L-30 and the ESAT volatiles refrigerator in L-32)
   (3) Whether this SRN set completes or does not complete the specific ASR.
k. Click once on Generate Receipt
l. The computer will pull up an on-screen preview of the SRN in Acrobat Adobe Reader
m. Click once on Email
n. The computer will pull up an Email Address Screen
o. Click once on Addresses
p. The computer will pull up a Select Email To Information screen

q. Select the appropriate RLAB staff to receive the SRN by clicking once on the appropriate staff member
r. Click once on the single arrow
s. Repeat until all appropriate personnel have been selected
t. Click once on Close
u. Click once in the Subject box and type lab assignment based on the information contained in the ASR, (e.g., EPA/REAP SRN -ASR 2199(not)complete)
v. Click once on Store SRN and send Email
w. A message box will be generated and state that the Email was sent.
x. Click once on OK
y. The computer will pull up a screen that will show the number of SRNs sent for that ASR. Click once on Close
z. An Acrobat Report Viewer screen will appear. Click once on Close
aa. A Sample Receipt Notice screen will appear. Click once on Exit.

5. Instructions for Generation of the COC Record for REAP

a. The RSCC (or designated back-up) or ESAT contractor will receive the COC and field sheets with the sample shipment. The information on these forms is compared to the condition and actual samples in the shipment to verify that the information is recorded correctly.

b. The RSCC (or designated backup) or ESAT contractor takes the shipment paperwork (Attachment 3) and records the sample information onto an EPA COC Record (FORM-7-EPA-9262). The RSCC (or designated backup) or ESAT contractor records the Task Order Number, EPA Reference Number, and name of the REAP laboratory at the top of the EPA COC Record. The Project Manager, ASR/Project ID, Sample Collection Date and number of pages are recorded in the appropriate boxes. Information pertaining to the number and type of sample containers, media and QC codes are recorded for each sample number on this form. The total amount of samples and containers and number of ice chests is recorded in the block labeled Description of Shipment. The shipment carrier and shipping document number is noted in the block labeled Mode of Shipment. The RSCC (or designated backup) or ESAT contractor must sign the COC and note the date and time that the shipment is to be relinquished to the sample carrier. The RSCC (or designated backup) must check mark whether the shipment has been sealed or unsealed. The white copy is placed with the shipment paperwork which is

sealed in a document holder attached to the inside lid of the cooler. The yellow carbon copy is placed in the RSCC's in-box.

6. Instructions for the Creation of F2L files for CLP Shipments

a. Double click on R7LIMS icon
b. Type Username and press enter
c. Type password and press enter
d. Click once on Data Manager
e. Under Samples Results, double click Single ASR Criteria
f. Click once on All
g. Click once on Browse Single ASR
h. Click and highlight the particular ASR to be downloaded to an F2L file.
i. Click once on the F2L Download. The computer will pull up a screen reading Download Forms2Lite Site File.
j. At Sample, click Received
k. Click CLP
l. At Date Received, click once on the log-in date for the needed shipment ("control-click" can be used if multiple dates are needed)
m. Click once on the Select File at Output File
n. At Output File, type k:\ASR#F2Ldwnld.F2L (e.g., k:\2127F2Ldwnld.F2L)
o. Click once on Download. A message will appear stating that the file has been downloaded to k:\ASR#F2Ldwnld.F2L (e.g., k:\2127F2Ldwnld.F2L)
p. Click once on OK
q. Click once on Exit to return to Browse Single ASR Results
r. Click once on Exit to return to Browse Main Menu
s. Click on File to exit R7LIMS
t. Double click F2Lite5.1 icon
u. A Forms2Lite5.1 box will appear. Click once on Cancel
v. Click once on File at the upper left hand corner of the screen
w. Click once on Import/Export
x. Click once on Import a F2L.5.x file
y. Click once on Next
z. Click once on Browse on Select File: Screen on the Import Export Wizard. Select date to Import (Make sure that the Import Measurements Data and the Import Archived TR Date do not have a check mark)
aa. Click once on arrow at Look in:
bb. Arrow down to k:\ drive
cc. Click once on file to be imported (e.g., 2127F2Ldwnld.F2L)
dd. The appropriate ASR to be downloaded will appear in File Name
ee. Click once on Open

ff. Click once on Finish
gg. Click once on OK after import has been completed
hh. Click once on File in upper left hand corner
ii. Click once on Exit to exit from Forms2Lite5.1

7. Instructions for the Generation of the Traffic Report (Attachment 7) and Sample Labels in Forms2Lite5.1

a. Click once on File
b. Click once on Open Site
c. Arrow to the downloaded Site and click to select
d. The computer will pull up a screen stating Step One: Site Information-(Site Name)
e. The information from the site should already be downloaded into the system with the exception of the Case Number. Type in the case number
f. Click Next
g. The computer will pull up a screen stating Step Two: Select Sampling Team
h. Click Next
i. Step Three: Select Analysis. The particular analysis should already be downloaded into the system
j. Click Next
k. The computer will pull up Step Four: Station/Location Information. The information should already be in the system except for the Concentration. At Concentration click the down arrow and select the appropriate concentration used in the field sheets for the site
l. Click Next
m. The computer will pull up Step Five: Assign Bottles. In the Select Station/Location, use the down arrow to select the sample(s) to be assigned Quality Control samples. The sample number(s) should appear in the Assigned Analysis With Sample Number table
n. Click Lab QC Type.
o. Click the down arrow and click the appropriate QC type
p. Click Next. A Forms II Lite query will appear to verify that QC has been assigned
q. Click OK
r. Click Next
s. Step Six: Assign Lab
t. Click down arrow at Lab Code
u. Click appropriate lab for a particular analysis(es) according to the assignment in the RSCC's LIMS ASRs & Sample Receipt notebook

v. All samples for that particular lab should appear in the Assigned Samples to Labs table
w. Repeat this procedure until all samples are assigned to the appropriate laboratories
x. Click Next
y. Step Seven: Assign Carrier
z. Click down arrow at Carrier
aa. Click the appropriate Carrier
bb. Type in the Date Shipped
cc. Type in the Airbill number
dd. Click the Lab header at the top of the table to sort by a particular lab when shipments are going to more than one lab
ee. Click and highlight the samples going to a particular lab
ff. Click Assign
gg. Repeat this procedure for each airbill and its associated analyses/lab until all samples are assigned
hh. Click Finish
ii. The computer will pull up Print/View a Specific TR screen
jj. Verify whether the shipment is complete or not complete from the COC delivered from the field. If the shipment is complete, double click the Complete box and query will change from No to Yes
kk. At the Select a View, click Lab Copy
ll. Click Print (this copy is retained to be shipped with the samples). Sign and date the document(s) in the section marked Relinquished By and initial for the Sampler in the Sampler Signature section
mm. Click Region Copy
nn. Click Print (this copy is given to the RSCC or designated backup). Sign and date the document(s) in the section marked Relinquished By and initial for the Sampler in the Sampler Signature section.
oo. Click Back until Step Five: Assign Bottles screen
pp. Click Generate Labels
qq. Click All Samples for Site
rr. Click Generate Label
ss. Click Edit Label
tt. Arrow to appropriate Avery number that matches the dimensions of the label to be used for the sample tags
uu. Click Next
vv. Verify that the Font Name is Arial
ww. Verify that the Font Weight is Normal
xx. Verify that the Font Size is 10
yy. Verify that the Text Color is black

zz. Click Next
aaa. Verify that the prototype label information is correct
bbb. Click Next
ccc. The computer will pull up the query, "What name would you like for your label?"
ddd. Type the string ASR#initialsshipmentdate (without spaces, e.g., 2171lh1024)
eee. Click Finish
fff. A preview of the sample tags will appear. Verify that the sample information is correct
ggg. Click Print
hhh. Select the printer designated for sample tag generation
iii. Click Print
jjj. Verify that the sample tag information is correct
kkk. Click the X box at the top right-hand corner of the screen
lll. Click Close
mmm. The computer will query, "Would you like to exit this Site?" Click Yes

8. Procedures to Export an F2L Site File and E-mail Instructions

a. Click on File at the upper left-hand corner of the screen
b. Click Import/Export
c. Click to highlight Export to a File at the Import Export Wizard screen
d. Click Next
e. Arrow to Site and click to highlight
f. Click Finish
g. At File Name, type the following string without spaces: R7ASR#Case#initials(not)complete.F2L, (e.g., R721723125lhnotcomplete.F2L)
h. Click Save
i. A computer message will appear stating that the file has been exported
j. Click OK
k. Click File
l. Click Exit
m. The computer will exit Forms2Lite
n. Double click LOTUS Notes icon
o. Type in password and press enter
p. Click OK
q. Click Mail
r. Click New Memo

s. Type in the names of the RSCC, CLP Program Officer and the Sample Management Office coordinator
t. Press tab and type the following information in the Subject line: R7case#ASR#complete.F2L or R7case#ASR#notcomplete.F2L (without spaces) e.g., R731292202notcomplete.F2L
u. Press tab until the cursor is in the body of the message
v. Click File
w. Click Attach
x. Type: k:\ and press Enter
y. Choose the file on the k:\ drive
z. Click Send
aa. Exit LOTUS NOTES

F. QUALITY ASSURANCE AND QUALITY CONTROL

All sample information is verified as correct through comparison of the field sheets, sample tags, and shipping documentation (such as, traffic reports, and/or COC). All documents must be consistent prior to acceptance and properly maintained throughout the shipping and analytical data files process. Any discrepancies are resolved through consultation with the Project Manager or RSCC (or designated back-up).

G. REFERENCES

1. US EPA, Region 7, "RLAB Analytical Data Management Procedures" Environmental Services Division Operations and Quality Assurance Manual, SOP 2410.1

2. USEPA, Region 7, "Management of Radioactive Materials in the Laboratory Environment", SOP 2420.15

Attachment 1

US EPA Region 7 Analytical Services Request (ASR)

Project ID: ASR Number: Projected Delivery Date:

Project Dest:
City: State: Program:
Site Name: Site OU
Site ID: CERCLIS ID:
GPRA PRC:

Project Manager:
Organization: Phone Number:

Contact:
Organization: Contact Phone:

ASR Purpose:
Comments:

Is this activity currently or potentially a criminal investigation?
Has a QAPP for the requested services been approved?

For health, safety and environmental compliance are any samples expected to contain:

Dioxin > 1ppb:
RCRA Listed Wastes:
Toxic/Hazardous Chemicals >1000ppm:

No. of Samples	Reg No	CNS List	Conc of Interest	Expected Conc

Special Analytical Requirements or Comments:

Date Submitted: By: ASR Status:
Date Accepted: By: RLAB Turn Around Time: Days
Date Transmitted: By: ANOP Turn Around Time: Days

Attachment 2

CHAIN OF CUSTODY RECORD
ENVIRONMENTAL PROTECTION AGENCY REGION VII

ACTIVITY LEADER(Print)	NAME OF SURVEY OR ACTIVITY	DATE OF COLLECTION DAY MONTH YEAR	SHEET of

CONTENTS OF SHIPMENT

SAMPLE NUMBER	TYPE OF CONTAINERS: CUBITAINER	BOTTLE	BOTTLE	BOTTLE	VOA SET (2 VIALS EA)	SAMPLED MEDIA: water	soil	sediment	dust	other	RECEIVING LABORATORY REMARKS/OTHER INFORMATION (condition of samples upon receipt, other sample numbers, etc.)
	NUMBERS OF CONTAINERS PER SAMPLE NUMBER										

DESCRIPTION OF SHIPMENT	MODE OF SHIPMENT
_____ PIECE(S) CONSISTING OF _____ BOX(ES)	_____ COMMERCIAL CARRIER: _____
_____ ICE CHEST(S); OTHER _____	_____ COURIER
	_____ SAMPLER CONVEYED (SHIPPING DOCUMENT NUMBER)

PERSONNEL CUSTODY RECORD

RELINQUISHED BY (SAMPLER)	DATE	TIME	RECEIVED BY	REASON FOR CHANGE OF CUSTODY
☐ SEALED ☐ UNSEALED			☐ SEALED ☐ UNSEALED	
RELINQUISHED BY	DATE	TIME	RECEIVED BY	REASON FOR CHANGE OF CUSTODY
☐ SEALED ☐ UNSEALED			☐ SEALED ☐ UNSEALED	
RELINQUISHED BY	DATE	TIME	RECEIVED BY	REASON FOR CHANGE OF CUSTODY
☐ SEALED ☐ UNSEALED			☐ SEALED ☐ UNSEALED	

Attachment 3

REAP SHIPPING DOCUMENT
(ARDL)

SHIPPING INFORMATION

TASK ORDER#: 0154 SHIPMENT DATE: 08/20/2003

EPA REFERENCE#: 2111A03 SHIPMENT NO.: 1

SAMPLE INFORMATION

SAMPLE #	MATRIX	PARAMETERS	DATE COLLECTED
2111-1	Water	Explosives	08/19/2003
2111-2	Water	Explosives	08/19/2003

REMARKS:

Attachment 4

Sample Collection Field Sheet

U.S. EPA Region VII
Kansas City, KS

ASR Number: Sample Number: QC Code: Matrix: Tag ID:

Project ID: Project Manager:
Project Desc:
City: State:
Program:
Site Name: Site ID: Site OU:

Location Desc: _____

External Sample Number: _____

Expected Conc: (or circle one: Low Medium High) Date Time (24 Hr)

Latitude: ___ ___ ___ Sample Collection: Start ___/___/___ ____:____
Longitude: ___ ___ ___ End ___/___/___ ____:____

Laboratory Analyses:

Container	Preservative	Holding Time	Analysis
1 - 1 Liter Cubitainer	HNO3 acidify, 4 Deg C	180 Days	Metals in Water by ICP

Sample Comments:

Sample Collected By: _____

Attachment 5

Date

MEMORANDUM

SUBJECT: Notice of Non-Reportable Sample Results

FROM:

TO: Primary Data File ASR Number: _____/Site Name: _____)

Data will not be reported for the following samples:

Sample Number	Parameter	Reason

cc: EPA Project Manager

Attachment 6

Sample Receipt Notice

11/07/2003 10:52

ASR Number: 2216
Samples Received: 11/07/2003
RLAB T-A-T: 30
Criminal: No
Project Id: WGP99
Project Desc: Webster City WWTF

Lab: (All)
Report Sample: Numbers

Req No	Analysis	Matrix	Analyst Lab	Pri	Sec	Samples
1	NH3-N W	Water	EPA	DAD		6-__
1	Met Water	Water	EPA		DAD	6-__

Comments:

USEPA Contract Laboratory Program
Organic Traffic Report & Chain of Custody Record

Case No: 31796 **R**
DAS No:

Region:	7
Project Code:	RS07WC/2054
Account Code:	50102D
CERCLIS ID:	IAD984601039
Spill ID:	07WC
Site Name/State:	Alberty City SBA sampling/IA
Project Leader:	Jeff Pritchard
Action:	Remedial Action
Sampling Co:	Tetra-Tech. EMI

Date Shipped:	6/5/2003
Carrier Name:	FedEx
Airbill:	841407300500
Shipped to:	Datachem Laboratories, Inc. 960 West LeVoy Drive Salt Lake City UT 84123 (801) 266-7700

Chain of Custody Record — **Sampler Signature:** JP

Relinquished By	(Date / Time)	Received By	(Date / Time)
1	6/5/03		
2			
3			
4			

ORGANIC SAMPLE No.	MATRIX/ SAMPLER	CONC/ TYPE	ANALYSIS/ TURNAROUND	TAG No./ PRESERVATIVE/ Bottles	STATION LOCATION	SAMPLE COLLECT DATE/TIME	INORGANIC SAMPLE No.	QC Type
G0HA8	Municipal Water Supply/ Jeff Pritchard	L/G	LDL VOA (21)	20544525 (HCL), 20544526 (HCL), 20544527 (HCL), 20544528 (HCL) (4)	2054-101/MW#2	S: 6/3/2003 8:40		--
G0HA9	Municipal Water Supply/ Jeff Pritchard	L/G	LDL VOA (21)	20544529 (HCL), 20544530 (HCL), 20544531 (HCL), 20544532 (HCL) (4)	2054-102/MW#3	S: 6/3/2003 8:52		--
G0HB0	Municipal Water Supply/ Jeff Pritchard	L/G	LDL VOA (21)	20544533 (HCL), 20544534 (HCL), 20544535 (HCL), 20544536 (HCL) (4)	2054-103/MW-9B	S: 6/3/2003 10:45		--
G0HB1	Surface Water/ Jeff Pritchard	L/G	LDL VOA (21)	20544537 (HCL), 20544538 (HCL), 20544539 (HCL), 20544540 (HCL) (4)	2054-104/FSKS	S: 6/3/2003 11:06		--
G0HB2	Municipal Water Supply/ Jeff Pritchard	L/G	LDL VOA (21)	20544541 (HCL), 20544542 (HCL), 20544543 (HCL), 20544544 (HCL) (4)	54-105FB/LDL VOA Trip Bl	S: 6/3/2003 11:20		Trip Blank
G0HB3	PE Water/ Jeff Pritchard	L/G	LDL VOA (21)	20544545 (HCL), 20544546 (HCL), 20544547 (HCL), 20544548 (HCL) (4)	2054-106/CLP PE sample	S: 6/3/2003 8:35		PE

Shipment for Case Complete? Y	**Sample(s) to be used for laboratory QC:** G0HA8	**Additional Sampler Signature(s):**	**Chain of Custody Seal Number:**
Analysis Key: LDL VOA = Low Con.VOA(OLC03.2)	**Concentration:** L = Low, M = Low/Medium, H = High	**Type/Designate:** Composite = C, Grab = G	**Shipment Iced?** _____

TR Number: 7-304918220-060503-0001

REGION COPY

PR provides preliminary results. Requests for preliminary results will increase analytical costs.
Send Copy to: Sample Management Office, 2000 Edmund Halley Dr., Reston, VA. 20191-3400 Phone 703/264-9348 Fax 703/264-9222

EPA SOP 2420.4C –
Field Chain of Custody for Environmental Samples,
December 2, 2003

STANDARD OPERATING PROCEDURE

No. 2420.4C

FIELD CHAIN OF CUSTODY FOR ENVIRONMENTAL SAMPLES

December 2, 2003

by Nicole Roblez

ENSV/RLAB/CATS

APPROVED:

Peer Reviewer — Date: 12/3/03

Manager, Regional Laboratory — Date: 4 Dec 03

Harold D. Brown — Independent QA Reviewer — Date: 12/08/03

Recertified

Reviewer					
Date					

TABLE OF CONTENTS

Attachments

A. Purpose

The purpose of this Standard Operating Procedure (SOP) is to establish uniform policies and procedures for use by field personnel to maintain an accurate written record of environmental samples from the time of collection through their acceptance by a laboratory for analysis. The custody procedures utilized within the laboratory for receiving samples and maintaining custody through the analytical processes are not covered in this SOP. See "Storage and Security of Environmental Samples", SOP 2420.2 for custody procedures utilized within the Regional Laboratory (RLAB).

B. Applicability

The policies and procedures outlined in this SOP are applicable to all Environmental Services Division (ENSV) personnel, Environmental Protection Agency (EPA), state/local agencies, and/or EPA contractors who collect environmental field samples for analyses by the RLAB or contract laboratories.

C. Summary of Procedures

As a requirement of any activity which may be used to support litigation proceedings, the validity of any data introduced into evidence must be clearly demonstrated. In the case of samples collected in support of an enforcement case, it must be clearly documented that the sample introduced into evidence is, in fact, the same sample collected and/or that the analytical data offered into evidence accurately represent the environmental conditions at the time of sample collection. It is imperative that there is adequate proof to demonstrate that transfer, storage or analysis, and that the analytical results were obtained from the same sample collected. Therefore, an accurate written record must be maintained to track the possession and handling Chain Of Custody Record (COC) (see Attachment 2) of each sample from the moment of collection through analysis and its introduction into evidence.

By definition, a sample is in "custody" if:

1. It is in one's actual physical possession; or
2. It is in one's view, after being in one's physical possession; or
3. It is locked up so no one can tamper with it, after being in one's physical possession; or
4. It is placed in a designated secured area

D. Definitions/Acronyms

ASR	Analytical Services Request
CLP	Contract Laboratory Program
COC	Chain of Custody Record

ENSV	Environmental Services Division
EPA	Environmental Protection Agency
LIMS	Laboratory Information Management System
PM	Project Manager
PO	Project Officer
QC	Quality Control
RECAP	Region 7 Environmental Collection and Analysis Program
ESAT	Environmental Services Assistance Team
RLAB	Regional Laboratory
RSCC	Regional Sample Control Coordinator
SOP	Standard Operating Procedure
SRN	Sample Receipt Notice
Tags	Sample container labels
UPS	United Parcel Service
VOA	Volatiles

E. Personnel Qualifications

Personnel performing this task should have a basic knowledge of the RLAB sample and records management procedures.

F. Responsibilities

1. Project Manager

a. The Project Manager submits a completed Analytical Services Request (ASR) to the RLAB 30 days before initiation of the sampling activity.

b. The Project Manager or designee (i.e., field contractor) ships and/or delivers properly collected, preserved, labeled, and packaged samples to the RLAB.

c. The Project Manager or designee (i.e., field contractor) is responsible for the accuracy and completeness of all accompanying paperwork. If any changes are required as a result of the sampling (e.g., sample number changes, additional analyses, samples not collected, quality control (QC) code additions), the Project Manager or designee (i.e., field contractor) must see that these corrections are made on all paperwork.

All changes made to the paperwork (COC, sample tags, or field sheets) must also be made to the information contained in the LIMS. It is the responsibility of the Project Manager or designee to supply correct information so that the Regional Sample Control Coordinator (RSCC) can

properly process the samples into the LIMS. Whenever possible, any changes are made prior to the delivery of the samples. If necessary, the RSCC will assist the Project Manager when changes are noted prior to sample collection/delivery, concurrent to sample delivery or after.

d. The Project Manager must be available to help resolve any problems with the samples or must designate someone to do this for them in their absence. This requires that when delivering samples, the Project Manager or designee stays with the RSCC to answer any questions. Samples must not be just dropped off (unless after normal business hours).

The Project Manager or designee calls the RSCC close to the anticipated delivery date and/or time that samples are sent by courier (i.e., Federal Express) to confirm that samples have arrived and to answer any questions the RSCC may have.

2. RSCC

a. The RSCC opens the ice chest (cooler) and utilizing the Infrared Digital Thermometer, checks the cooler temperature and records the temperature (in degrees Celsius) in the last row of the "Receiving Laboratory Remarks/Other Information" column on the COC (see Attachment 2).

b. The RSCC verifies the presence of all samples, checks all documentation and signs the COC after all paperwork is complete and accurate.

c. The RSCC works with the Project Manager to obtain correct information and puts the amended information into the LIMS.

d. The RSCC notifies the Project Manager of problems which prevent acceptance of the samples by ENSV. RLAB maintains all samples received in a secure location including those pending reconciliation of problems.

e. The RSCC logs samples into the LIMS and is responsible for the proper storage, tracking and/or distribution of the samples to the appropriate contract laboratories (this includes while the sample is in transit to the contract laboratory facility). The RSCC prepares an electronic Sample Receipt Notice (SRN) message for each activity received by the RLAB and routes it appropriately to the Environmental Services Assistance Team (ESAT), the Contract Laboratory Program (CLP) PO, or the Region 7 Environmental Collection and Analysis Program (RECAP) PO, CATS PM, ANOP PM, and appropriate back-up personnel.

G. Procedures

1. In order to ensure adequate control and documentation of collected samples, the number of personnel handling the samples from the time of collection through delivery to RLAB should be limited.

2. The following actions must be accomplished in order to ensure that the relationship between the physical sample and the description of the sample is clearly, completely and accurately established, and that the custody of the sample is initiated from the time of actual sample collection.

   a. A unique number is assigned to each sample (see "Identification, Documentation, and Tracking of Samples", SOP No. 2420.5) in order to relate the descriptive information to a physical sample. If a sample consists of several containers for analysis of different parameters from the same physical sample, the same number is used for each portion of the original sample.

   b. A sample tag (sample container label) is securely attached to each container at the time of collection for specific instructions for filling out the sample tag (see "Identification, Documentation and Tracking of Samples", SOP No. 2420.5).

   c. Custody of the sample is initiated at the time of collection by ensuring that the sample is in the sample collector's physical possession or view at all times, or is stored in a locked place where no one can tamper with it.

   The sample collector is responsible for the collected samples until they are delivered to the RLAB.

3. Samples may be delivered to RLAB by the sampler or EPA contractor via courier or commercial carrier.

   a. Sampler or EPA contractor-conveyed samples are those transported and delivered to RLAB. The coolers may be sealed or unsealed, but the sampler or EPA contractor must ensure that they are secured in the transport vehicle when he/she is not physically with the vehicle.

   b. Samples may be delivered via courier (e.g., Greyhound). The cooler and sample containers must be transported with the lids secured. The transfer of possession of the samples must be recorded from the sampler or EPA contractor to RLAB.

c. Samples may be shipped via commercial carrier (e.g., Federal Express, Airborne, United Parcel Service (UPS)) from the field to RLAB. The cooler and sample containers must be sealed at the time of shipment.

4. Samples are considered to be sealed when they are packaged in such a manner that would prohibit tampering or readily reveal any tampering, if it occurred.

a. A custody seal (see Attachment 1) may be used to secure the individual sample container, as appropriate to meet specific regulatory program requirements. These custody seals must be signed and dated by the sampler or EPA contractor when used to seal individual sample containers.

b. The use of a custody seal must be used to secure the openings of boxes, plastic bags, ice chests or coolers containing samples. These custody seals must be signed and dated by the sampler or EPA contractor when used to seal the shipping containers.

5. The COC (see Attachment 2) is initiated at the time of sample collection and must accompany all samples. The COC is utilized to document the transfer of a sample from the sampler or EPA contractor through receipt by the RSCC or designated back-up at RLAB.

RLAB instructions for the completion of the COC are outlined in Attachment 3.

a. The transfer of possession of the samples would occur when the sampler or EPA contractor delivers the samples to RLAB, gives them to the courier who will deliver the samples to RLAB, or packs the samples in a sealed shipping container for shipment to RLAB via commercial carrier.

b. The original and yellow copy of the COC will accompany the samples to RLAB. When the samples are conveyed by the sampler or EPA contractor, the COC may be hand carried. When the samples are delivered via courier or commercial carrier, the COC must be placed in a plastic document enclosure which is enclosed in the shipping container.

6. When samples are delivered to RLAB after duty hours, the samples and the COC will be placed in the refrigerator located on the back dock until acceptance by the RSCC or designated backup in accordance with the procedures outlined in "Storage and Security of Environmental Samples", SOP No. 2420.2.

7. Once RLAB has accepted the samples, the responsibility for custody of the samples transfers to the RLAB personnel. Custody of the samples is maintained through analysis in accordance with the laboratory's internal control procedures.

8. The original of the completed COC is obtained by RLAB for inclusion with the permanent site activity files, and included with the final data transmittal sent to the Project Manager.

9. The yellow copy of the completed COC is returned to the Project Manager for inclusion in their appropriate activity files after all samples, for a given activity, have been accepted.

10. The custody seals or evidence tape associated with the specific samples or sample shipments are not retained.

H. Quality Assurance/Quality Control

A written tracking record (COC) is maintained from the time that the sample is collected to its transfer from the collection site to its laboratory destination. This record is used to demonstrate that sample possession has been secured and limited. Signed and dated custody seals placed over the access points of the sample shipment demonstrate that the contents of the samples have not been tampered with or compromised.

I. References

1. US EPA, Region 7,"RLAB Procedures for Sample Receipt and Log-In", Environmental Services Division Operations and Quality Assurance Manual, SOP 2420.1

2. US EPA, Region 7, "Identification, Documentation, and Tracking of Samples", Environmental Services Division Operations and Quality Assurance Manual, SOP 2420.5

3. US EPA, Region 7, "Storage and Security of Environmental Samples", Environmental Services Division Operations and Quality Assurance Manual, SOP 2420.2

Attachment 1

RLAB Custody Seal

Attachment 2

CHAIN OF CUSTODY RECORD
ENVIRONMENTAL PROTECTION AGENCY REGION VII

ACTIVITY LEADER(Print)	NAME OF SURVEY OR ACTIVITY	DATE OF COLLECTION (DAY / MONTH / YEAR)	SHEET ___ of ___

CONTENTS OF SHIPMENT

SAMPLE NUMBER	TYPE OF CONTAINERS: CUBITAINER	BOTTLE	BOTTLE	BOTTLE	VOA SET (2 VIALS EA)	SAMPLED MEDIA: water	soil	sediment	dust	other	RECEIVING LABORATORY REMARKS/OTHER INFORMATION (condition of samples upon receipt, other sample numbers, etc.)
	NUMBERS OF CONTAINERS PER SAMPLE NUMBER										

DESCRIPTION OF SHIPMENT

_____ PIECE(S) CONSISTING OF _____ BOX(ES)

_____ ICE CHEST(S): OTHER _____

MODE OF SHIPMENT

_____ COMMERCIAL CARRIER: _____

_____ COURIER

_____ SAMPLER CONVEYED

_____ (SHIPPING DOCUMENT NUMBER)

PERSONNEL CUSTODY RECORD

RELINQUISHED BY (SAMPLER)	DATE	TIME	RECEIVED BY	REASON FOR CHANGE OF CUSTODY
☐ SEALED ☐ UNSEALED			☐ SEALED ☐ UNSEALED	
RELINQUISHED BY	DATE	TIME	RECEIVED BY	REASON FOR CHANGE OF CUSTODY
☐ SEALED ☐ UNSEALED			☐ SEALED ☐ UNSEALED	
RELINQUISHED BY	DATE	TIME	RECEIVED BY	REASON FOR CHANGE OF CUSTODY
☐ SEALED ☐ UNSEALED			☐ SEALED ☐ UNSEALED	

7-EPA-9262(Revised 5/85)

Attachment 3

Instructions For Completing A Chain Of Custody Record

(Note: Each numbered item explains what is to be entered into that particular block moving from left to right, top to bottom of the document.)

1. Activity Leader. Enter the first initial and last name of the EPA Project Manager.

2. Name of Survey or Activity. Enter the activity number and/or Analytical Services Request (ASR) number (e.g., ERN07/900) for which the samples were collected.

3. Date of Collection. Enter the day, month, and year the samples were collected.

4. Sheet. Enter 1 of 1 unless there are more than one total sheets describing the shipment. If multiple sheets, enter the consecutive number of each sheet of the total number of sheets (e.g., 1 of 3, 2 of 3, 3 of 3).

5. Contents of the Shipment.

   a. Enter the specific sample numbers, number of sample type containers per sample number and sample media in the appropriate column

      (1) The ASR number and the individual sample numbers composing the shipment are entered in the "Sample Number" column (e.g., 2222-2). If more than one sheet is required, continue on additional sheets. For shipments of a large group of samples, it would be more appropriate and efficient to complete a separate sheet for each shipping container.

      (2) The types of containers for each sample number are entered in the columns provided. The size should be entered above the container type, as appropriate. For Volatiles, the "VOA Set" refers to two=40 ml vials contained in the cubitainer which are collected for volatile organics analyses. The container types are modified, as necessary or appropriate, to describe sample containers.

      (3) The sampled media for each sample number will be indicated by placing an "X" in the appropriate column. If the sample media is not listed, the actual media sampled should be entered in the "Other" column (e.g., wipe, sludge, air, biota, fish, etc.).

      (4) The "Receiving Laboratory Remarks/Other Information" is to be used by the RLAB to indicate any problems with the shipment or condition of the samples upon receipt; e.g., custody seal on sample container or shipping container broken, a sample container broken in transit, a sample lost due to leakage during shipment, etc. The temperature of the shipping coolers(s) are to be recorded in the lower area of this column. This column may also be used to record other sample numbers for cross-referencing purposes (e.g., external sample number).

b. After entering all of the above information, the total contents of the shipment should be indicated by marking out any remaining lines in this section. This can be accomplished either by drawing a line across the next line after the last entry and entering "None to Follow" or "Activity/ASR Complete," or by drawing a line across the next blank line or diagonally across the remaining lines in the section and entering "None to Follow" or "Activity/ASR Complete."

6. Description of Shipment. Enter the total number of pieces (e.g., samples or sample containers) packed in the total number of shipping containers (e.g., ice chests, boxes or other, which comprise the total shipment)(e.g., 12 pieces in 2 ice chests or 24 pieces in 2 boxes).

7. Mode of Shipment. Indicate the mode by which the samples are shipped to the RLAB by placing an "X" in the appropriate line preceding the specific mode in this block. If the shipment is via commercial carrier, the name of the carrier and the shipping document number (e.g., airbill) should be entered in the appropriate lines provided. This information may be entered by the sample shipper (sampler or individual to whom the sampler relinquished the samples), or the shipment receiver (lab sample custodian), as appropriate.

8. Personnel Custody Record. This portion of the form provides the record of changes of custody of the shipment (sample or group of samples)from the sample collector to the laboratory. To provide an adequate written record, all of the blocks should be completed as described below.

a. The sample collector will sign the first "Relinquished By" block when the samples are presented to another individual or commercial carrier.

(1) An "X" should be entered in the appropriate block to indicate whether the shipment is sealed or unsealed with a piece of completed custody seal tape, the date and time when the samples are relinquished should be entered in the appropriate blocks, and the reason for change of custody (e.g., transport to lab, receipt by lab, etc.) should be entered in the appropriate block.

(2) If the sampler is presenting the samples to a commercial carrier for shipment, the name of the carrier should be entered in the next available "Received By" block. The signature of a representative of the carrier is not required.

b. Each individual who received the shipment of samples will sign the next available "Received By" block and enter an "X" in the appropriate block to indicate whether the samples were received sealed or unsealed with a piece of completed custody seal tape. If the samples were shipped via commercial carrier, the individual receiving the samples (e.g., sample custodian at the RLAB) should enter the date and time the samples were received and the reason for change of custody (e.g., receipt by the RLAB) in the appropriate blocks.

c. Each successive individual who relinquishes custody of the samples will sign the next available "Relinquished By" block, enter an "X" in the appropriate block to indicate whether the sample shipment is sealed or unsealed with a piece of completed custody seal tape, enter the date and time when custody is relinquished and enter the reason for change of custody in the appropriate blocks.

STANDARD OPERATING PROCEDURE

No. 2420.5D

IDENTIFICATION, DOCUMENTATION, AND TRACKING OF SAMPLES

December 16, 2003

by

Harry Kimball

ENSV/RLAB/CATS

APPROVED:

Peer Reviewer — Date: 12/19/03

Manager, Regional Laboratory — Date: 19 Dec 03

Independent QA Reviewer — Date: 12/19/03

Recertified

Reviewer					
Date					

Table of Contents

A. PURPOSE AND APPLICABILITY

The purpose of this standard operating procedure (SOP) is to establish uniform procedures for assigning sample numbers, labeling sample containers, documenting the sample collection process, and for tracking samples.

The collection of samples is an essential step in the process for obtaining information on a variety of environmentally-related conditions and situations. Because the analytical results of samples are used extensively to support regulatory decisions, statutory actions, environmental and health assessments, and litigation proceedings, a critical component of the sample collection process is the proper identification, documentation, and tracking of each sample collected.

The procedures outlined herein are applicable to all samples received by the Region 7 Laboratory (RLAB) for analysis (either in-house analysis or out-source contract lab analysis) and to laboratory-generated quality control (QC) samples. The Regional Sample Control Coordinator (RSCC), or their surrogate, shall ensure, at the time of sample receipt, that samples received by RLAB conform to the identification and documentation requirements of this SOP. This SOP should be provided to all individuals (EPA, state, and tribal staff, plus their contractors) collecting samples for delivery to RLAB to facilitate compliance with these procedures.

B. SUMMARY OF PROCEDURE

1. The identification and documentation of each sample is required in order to provide tangible evidence that shows the data resulting from sample analysis is linked directly to the sample collected. The basic mechanism used to establish this critical link between samples collected and analytical data is the assignment of a unique sample identifier to each sample collected, with supporting written information to document the sampling process. In addition to providing the means for establishing the relationship between samples and analytical results, the assignment of unique sample identifiers provides a means for tracking samples through the analytical data generation process.

2. Sample identification is achieved by labeling each field collected sample with a unique sample identifier. Samples contained in multiple sample containers will bear the same unique sample identifier on each container, plus, each container will be uniquely identified (usually by analysis). Quality control is an integral part of the process of obtaining reliable information about environmental samples, therefore, field and laboratory quality control samples will be uniquely identified in an appropriate and consistent manner.

3. Sample documentation is accomplished by recording the appropriate information about the sample on a field sheet which bears the sample's unique sample identifier. If samples are delivered to RLAB with sample identifiers that are not consistent with the unique sample identifiers described in this SOP, the RSCC will assign the requisite unique sample identifiers and record the original sample identifier in the LIMS "External Sample Number" field. Laboratory QC samples are documented on the sample prep and/or analysis log.

4. Sample tracking is accomplished by using the Region 7 Laboratory Information Management System (LIMS). The LIMS is used to identify and track the status of all samples analyzed by the EPA Region 7 Laboratory and its contractors. The current LIMS is a product called R7LIMS. R7LIMS and any future LIMS products will follow the sample identification scheme defined in this SOP. Additionally, the LIMS can generate field sheets and tags (sample labels) to facilitate identification and documentation of field collected samples (see SOP 2420.13, "RLAB Procedures for Preparation of Field Sheets and Tags"). The physical location of samples is tracked by chain-of-custody procedures.

5. Because the identification and documentation of samples establishes the foundation for substantiating reported analytical data, it is important that the individuals who collect and/or generate samples follow the procedures contained in this SOP. The procedures contained in SOP No. 2420.4, "Field Chain-of-Custody for Environmental Samples," should be used in conjunction with this SOP to provide complete field sample documentation.

C. DEFINITIONS

The following definitions of commonly-used terms relating to types of samples and sampled matrices are provided for clarification in the sample identification process:

1. Sample. The word 'sample' is an often overworked term. It can refer to a sample collected in the field, a portion of a field sample that has been spiked with additional analytes (matrix spike sample), or a sample generated entirely within the laboratory, such as a method blank. The term 'sample' most often refers to a Field Sample that is of one matrix collected from a specific point (or area if spatially composited) at a specific time (or period of time if temporally composited). A sample may be divided into several different containers, each for a different type of analysis and possibly requiring different methods of preservation (see SOP 2420.6, "Sample Container Selection, Preservation and Holding Times"). It is common for all of these containers to be collectively referred to as being a (one) sample and for all of them to bear the same unique sample identifier.

2. Field Sample. A representative portion of an environmental matrix (e.g. air, soil, water, etc.) collected from a specific location at a specific time to obtain information regarding environmental conditions and/or effects, process operations and material contents. Field Samples are actual portions of a matrix collected to determine its physical, chemical, or biological constituents and are distinguished from samples used for quality control (QC) purposes. Although QC samples collected in the field are in a sense field samples, the term Field Sample is used to denote a non-QC sample and is sometimes referred to as a "real" or "regular" Field Sample. Field Samples include those collected to evaluate background conditions and are categorized as grab, composite or continuous samples.

   a. Grab Sample. A discrete portion of a matrix collected at a specific location at one instance in time (this period of time is typically defined as not exceeding 15 minutes to allow adequate time for sample collection under most field situations). This type of sample is representative of the environmental condition at the time of collection. This type of sample is commonly used for in-situ determinations and for obtaining information on constituents that require special handling or may be lost if sampled in another manner.

   b. Composite Sample. A portion of a matrix consisting of a mixture of two or more discrete portions (grab samples) collected from a specific location over a period of time or from a specific area (multiple locations) at one time or over a period of time. This type of sample is a representative average of the environmental condition for a definable area and/or period of time. This type of sample is commonly used for assessing environmental conditions.

   c. Continuous Sample. As the name implies, it is a representative portion of a matrix collected in an uninterrupted manner for a period of time. This type of sample is normally associated with in-situ determinations and is, therefore, not usually collected for submittal to a laboratory for analysis. Continuous samples are most commonly used for collecting data of air and water media; e.g., flow, pH, temperature, etc.

3. Split Sample. As the name implies, it is a sample that is separated or split from the total amount of material sampled and sent to a different laboratory for analysis. Soil matrix samples are homogenized then split to ensure uniformity. The Split samples are used to independently verify laboratory analysis.

4. Extract. An extract is the result of the extraction process. The sample extract is labeled by extraction personnel.

5. Digestate. A digestate is the result of the digestion process. The sample digestate is labeled by digestion personnel.

6. Quality Control Sample. Prepared in the laboratory, in the field, or combination thereof, a QC sample is incorporated into sample collection and/or analysis activities as a means of evaluating the quality of analytical results obtained from Field Samples. This type of sample may be a field-collected sample (e.g. duplicate sample) or a laboratory-generated sample, depending on its intended purpose, to evaluate and/or substantiate analytical results. Additional information on the use of QC samples for calculating data quality may be found in SOP No. 2410.15, "Estimating and Documenting Data Quality". The following types of QC samples are commonly encountered in sampling events and should be sufficient to categorize most QC samples:

   a. Duplicate Sample. It is recognized that there are several interpretations of this term. For the purpose of calculating data quality, there are essentially two types of duplicate samples: field and laboratory, as described below.

      (1) Field duplicate samples refer to two Field Samples collected simultaneously from the same location(s) under identical conditions. A duplicate grab sample consists of collecting two Field Samples at the same location and time. A duplicate composite sample consists of two Field Samples containing multiple grab samples each collected at the same location and time. If automatic samplers are used to collect composite samples, the collection of duplicate composite samples would require two automatic samplers to be collocated and set to collect the individual portions or aliquots at the same times. The dividing (also referred to as "splitting") of a single sample into two portions will be considered field duplicate samples in those situations where the preferred method of simultaneous collection cannot be met due to field conditions (e.g. the media being sampled is non-homogeneous like some soils, gravel, etc.).

      (2) Laboratory duplicate samples refer to equivalent aliquots taken from a single sample received by a laboratory for analysis as unique samples. The process of obtaining the duplicate aliquots should be preceded by ensuring the sample is well mixed.

   b. Blank Sample. A sample that is presumed to be free of contamination from constituents of concern and is designed to detect contamination due to the sampling and/or analysis process (collection, preservation, handling, sampling environment, extraction, analysis, etc.).

(1) Field Blank. Includes all blank samples which are prepared in or enter the field environment and include trip blanks, equipment blanks, bottle or container blanks, reagent or preservative blanks and tubing blanks. Ideally, a field blank for most analytical parameters should be exposed to the sampling, preservation and handling process used to collect the physical samples, but this may not always be possible (e.g. the field blanks for volatile organics are only transported unopened to and from the sampling environment). The type of field blank should be identified, as well as the group of Field Samples with which it is associated, in the appropriate sample documentation.

(a) Trip Blank. It is a sample that is presumed to be free of contamination from constituents of concern, and is carried into the field and returned while being exposed to the same field conditions which the sample containers experience during the sample shipping process.

(b) Tubing/Equipment Blank. It is a sample free from constituents of concern (normally deionized water that is distilled) and is pumped through or otherwise introduced into the sampling equipment. The process results in exposure of the sample to any constituents of concern which might be contained in or on the surfaces of the sampling equipment.

(c) Preservation Reagent Blank. It is a sample which is originally free from constituents of concern (normally distilled deionized water) and to which the preservative (acid or other chemical) is added in the same concentration and quantity as normally added to a sample. The purpose is to determine if any contaminants of concern exist in the preservative used.

(d) Container Blank. A sample originally free from constituents of concern (normally distilled deionized water) which is introduced into randomly chosen containers at the time of sampling. The purpose of this blank is to determine the existence of contaminants of concern in the sampling containers.

(2) Method Blank. A laboratory QC sample used to assess the level of contamination in the analytical system. A method blank is,

typically, a portion of a clean matrix that is taken through the entire sample preparation and analysis process.

c. Laboratory QC Sample. A variety of QC samples are used by an analytical laboratory for internal QC purposes. For the purpose of sample identification, all such samples prepared by the laboratory for internal use are classified under this category. Commonly used laboratory QC samples include lab duplicate samples, method blanks, lab control samples, matrix spikes, and lab fortified blanks.

d. Performance Evaluation Sample. A sample that contains a known amount of a chemical constituent or parameter and is introduced for analysis to assess the accuracy of the analytical method. The actual content of the PE sample, either in regard to specific constituents and/or concentrations of constituents, is normally unknown to the receiving analytical laboratory.

e. Performance Testing Sample. Similar to a performance evaluation sample except that it is provided by a NELAC (National Environmental Laboratory Accreditation Conference) certified PT sample provider. Results of the analysis of these samples are used for NELAC accreditation purposes.

f. Some additional Field Samples may be thought of as QC samples due to the location or method of sample collection. These are labeled the same as, and analyzed the same as, other Field Samples.

(1) Rinsate Sample. This type of sample is used to evaluate the effectiveness of field decontamination procedures for sampling equipment. The sample is obtained by collecting the rinse water that is poured over the sampling equipment after decontamination has been completed (the water is normally distilled ionized water prepared in the laboratory and carried to the field).

(2) Background Sample. In some investigations, samples are collected to determine what is representative of the environment for constituents of concern. These samples, normally called background samples, are Field Samples which are collected off-site or upstream of an area that is affected by a contaminant of concern, but are not expected to contain any or significant amounts of the contaminant of concern.

7. Matrix. The matrix (also known as 'media') refers to the substance from which the sample was obtained and/or of which the sample consists. Since the sampled matrix has a direct bearing on how a sample is preserved and on the selection of

the method to analyze the sample, the identification of the matrix is an important aspect of sample documentation.

a. RLAB Matrix. The RLAB matrix is the matrix name used by RLAB to identify the matrix of the sample. It is the matrix used in the LIMS and in the RLAB Methods.

(1) Air. All samples collected to evaluate or analyze the chemical and physical contents of the air, both indoor and outdoor. The resulting sample may be in different forms depending on the method of collection (e.g. Tenex tube, canister, PUF, etc.).

(2) Solid. All samples obtained of soils, sediments, sludge, dust, and any other solid material.

(3) Tissue. All samples obtained of living organisms; e.g., plants or vegetation, fish, animals, etc., either whole or portions thereof.

(4) Waste. All samples obtained of media that do not logically fit under one of the other specifically defined matrices or contain exceedingly high concentrations of analytes. (Previously referred to as "Hazardous/Other".) Examples of these type samples are wipe samples, drum samples, non-aqueous liquid samples, product or formulation samples and mixed media samples.

(5) Water. All samples obtained of aqueous liquid, e.g., wastewater, surface water, drinking water, groundwater, etc.

b. NELAC Matrix. NELAC has its own list of Quality System Matrices. These matrices are referenced in the RLAB Methods, but are not used in the LIMS or for sample definition/identification.

(1) Aqueous. Any aqueous sample excluded from the definition of Drinking Water matrix or Saline/Estuarine source. Includes surface water, groundwater, effluents, and TCLP or other extracts.

(2) Drinking Water. Any aqueous sample that has been designated a potable or potential potable water source.

(3) Saline/Estuarine. Any aqueous sample from an ocean or estuary, or other salt water source such as the Great Salt Lake.

(4) Non-aqueous Liquid. Any organic liquid with <15% settleable solids.

(5) Biological Tissue. Any sample of a biological origin such as fish tissue, shellfish, or plant material. Such samples shall be grouped according to origin.

(6) Solids. Includes soils, sediments, sludges and other matrices with >15% settleable solids.

(7) Chemical Waste. A product or by-product of an industrial process that results in a matrix not previously defined.

(8) Air and Emissions. Whole gas or vapor samples including those contained in flexible or rigid wall containers and the extracted concentrated analytes of interest from a gas or vapor that are collected with a sorbent tube, impinger solution, filter, or other device.

D. PERSONNEL QUALIFICATIONS

Personnel collecting and/or delivering samples to RLAB should have a basic knowledge and understanding of RLAB sample management procedures including chain-of-custody (SOP 2420.4). RLAB personnel receiving samples must be knowledgeable of the sample log-in process (SOP 2420.1, "Sample Receipt and Log-in"). Personnel defining samples in the LIMS must be familiar with using the LIMS (SOP 2410.20, "R7LIMS Functions and Security") and have an R7LIMS account.

E. SAMPLE IDENTIFICATION

1. Each sample is identified by a unique sample identifier which is assigned to it.

   a. This identifier is used to distinguish an individual sample from all other samples and is used on all documentation relating to collection, handling, analysis and reporting the analytical results of an individual sample.

   b. Since a sample is normally analyzed for a number of different chemical constituents or parameters that require different sample containers and preservation techniques, the same unique sample identifier will be assigned to each portion of the original sample split among individual sample containers. For example, if a sample is split among three individual sample containers in order to properly preserve each portion for the specific parameter or group of parameters to be analyzed, each of the

individual sample containers would be identified by the same unique sample identifier.

2. The unique sample identifier consists of three parts: the Analytical Services Request Number (ASR Number), Sample Number, and Quality Control Code (QC Code). These are frequently written together, separated by hyphens. The unique sample identifier is sometimes (confusingly) simply referred to as the sample number.

   a. ASR Number - This is the number automatically assigned to an ASR at the time it is defined in the LIMS. Each ASR has its own unique number.

   b. Sample Number - This number is assigned by the responsible Project Manager (or their designee) for each field sample collected for an ASR.

   c. QC Code - This two or three character alpha code is used to identify the nature of the sample for QC purposes. Field personnel will normally only use the following codes to identify field collected samples:

   __ = Field Sample (two underscore characters)
   FD = Field Duplicate
   FB = Field Blank
   FS = Field Spike
   FSD = Field Spike Duplicate

   Laboratory personnel will use the following codes to identify laboratory QC samples:

   MB = Method Blank
   LD = Laboratory Duplicate
   MS = Matrix Spike
   MSD = Matrix Spike Duplicate
   LCS = Laboratory Control Sample
   LFB = Laboratory Fortified Blank
   PE = Performance Evaluation sample
   PT = NELAC Performance Testing sample

3. The following examples are provided to illustrate some unique sample identifiers:

   26-1-__ - Field Sample number 1 for ASR Number 26
   26-1-FD - Field Duplicate of Field Sample above
   26-2-FB - Field Blank submitted for same ASR Number
   87-5-__ - Field Sample number 5 for ASR Number 87

87-5-MS - Matrix spike of Field Sample above
87-900-LCS - Lab Control Sample number 900 for ASR Number 87

4. Some quality control samples have meaning only when referenced to another sample (i.e. QC Codes of FD, FS, FSD, LD, MS, MSD). To facilitate the identification of the referenced sample, the LIMS has two fields for use with these QC samples: Ref Sample Number and Ref QC Code. Rules for determining the Sample Number, Ref Sample Number, and Ref QC Code for these QC samples are given below.

   a. The QC sample and the referenced sample (the sample that the QC sample is a spike or duplicate of) must have the same ASR Number and Matrix.

   b. Field QC samples (FD, FS, FSD) will be assigned the same Sample Number as the original Field Sample (__) that they are a duplicate or spike of. The Ref Sample Number, and Ref QC Code are automatically assigned by the LIMS and can not be edited by the user.

   c. Lab QC samples (LD, MS, MSD) that are a duplicate or spike of a Field Sample or Performance Testing sample (__, PT) will be assigned the same Sample Number as the original Field Sample or Performance Testing sample that they are a duplicate or spike of. By default, the Ref Sample Number will be set to the Sample Number and the Ref QC Code will be set to "__" by the LIMS. If the sample being spiked or duplicated is a Performance Testing sample, a Ref QC Code of "PT" will need to be manually entered into the LIMS. Note that it is not appropriate for a Field Sample and a Performance Testing sample to have the same Sample Number.

   d. Lab QC samples (LD, MS, MSD) that are a duplicate or spike of any other field collected sample (QC Code of FB, FD, FS, FSD) will be assigned a different Sample Number than the original sample that they are a duplicate or spike of. The Ref Sample Number and Ref QC Code will need to be manually entered into the LIMS. Although not a requirement, it is suggested that a Sample Number in the "800" range be used for the lab QC sample.

   e. MSD samples must have the same Sample Number, Ref Sample Number, and Ref QC Code as their associated MS sample. The MS sample must be defined in the LIMS before the MSD sample can be defined.

5. The following rules are provided for further clarification of the unique sample identifier assignment process:

a. Each sample collected of a specific media will have a unique sample identifier. For example, if two samples are collected at the same location and time, but are of two different media (e.g. air and solid, or water and tissue), the sample of each specific media will be considered a separate sample. Each sample will be assigned a separate sample number.

b. In-situ samples collected for instantaneous field determinations (e.g. pH, temperature, specific conductance, dissolved oxygen, residual chlorine) in connection with the collection of samples for submission to a laboratory for analysis will be identified by sample identification numbers. Results of field determinations are recorded on field sheets associated with the sample collected for laboratory analysis. The sample identification number of the sample used for the field determination will normally be the same as the sample identification number of the sample submitted for analysis.

c. Continuous samples do not require the assignment of sample identification numbers, but do require specific written documentation to record sampling locations, and times of sampling and readings. Since many continuous monitors provide strip charts and/or printouts of readings, this documentation should be kept to supplement other written documentation.

d. Even though samples for some analyses, such as those for volatile organics, are always collected in two or more containers, they are considered to be a single sample. Additionally, if multiple analyses are to be analyzed for (such as metals, pesticides and VOAs), separate containers will be needed for each analysis. These containers are collectively considered to be one sample and will have the same unique sample identifier.

e. Sample extracts are labeled by the person performing the extraction of the sample. The sample extract container is labeled by hand-copying the sample label's information onto a smaller sample extract label. The sample extract label must identify the extraction solvent. Transcription errors are prevented by double checking the sample extract label prior to affixing the sample extract label to the sample extract container. The sample extract label is then affixed to the sample extract container.

f. Digestates are labeled by the person performing the digestion of the sample. The sample digestate container is labeled by hand-copying the sample label's information onto a blank label. The sample digestate label must identify the requested analysis. Transcription errors are prevented by double checking the sample digestate label prior to affixing the sample

digestate label to the sample digestate container. The sample digestate label is then affixed to the sample digestate container.

g. As a general rule-of-thumb, Field Blanks that are associated with a group of samples will have their own Sample Number. Field Blanks that are associated with just one Field Sample (e.g. a separate Field Blank for each Field Sample) may have, but are not required to have, the same Sample Number as the Field Sample that it is associated with.

h. It is common practice for some laboratory QC samples (MB, LCS, LFB) to be assigned a Sample Number in the "900" range. This is not a requirement for these samples (any number may be used), however, it is a desirable practice as it helps avoid confusion by keeping these QC samples "numerically segregated" from Field Samples. For sampling events involving a large number of Field Samples, running into the 900 range, it may be desirable to number these QC samples in the 1500, 2000, or other appropriate range.

6. All samples submitted for analysis will have a sample label affixed to each sample container.

a. Sample labels currently in use are computer generated, therefore, minimal or no entries are required. Any entries made on the sample labels will be accomplished using indelible ink.

b. With the exception of volatile samples and samples packed inside a paint can for shipping, only one sample label is needed for each sample container. Since volatile and over-packed samples consist of more than one container, multiple labels are required so that each container (including the outside container) can be labeled.

NOTE: Since some of the computer-generated sample labels are susceptible to deterioration from water, clear plastic tape should be placed over these sample labels if they will come into contact with water (including ice) during storage, transport and/or shipment. Some computer-generated sample labels are water resistant; these labels will not require tape protection.

c. Each sample container must be uniquely identified by the sample label. Where there is only one container for an analysis (such as Metals in Water by ICP), the container is uniquely identified by the unique sample identifier (ASR Number, Sample Number, and QC Code) and the analysis abbreviation (such as Met W). Where there is more than one container for an analysis (such as VOCs in Water by GC/MS), the containers are

uniquely identified by the unique sample identifier (ASR Number, Sample Number, and QC Code), the analysis abbreviation (such as VOA W), and a sequential container number (1, 2, 3, etc.). "Specific" sample container labels generated by the laboratory's LIMS are uniquely identified as described above. When samples are received by the laboratory bearing LIMS "Generic" labels, labels generated by the sampler, or hand-made labels, the necessary additional information should be added to the label or a second label should be placed on the container to uniquely identify it. It is the responsibility of the laboratory person receiving the samples (RSCC or their alternate) to ensure that each container is uniquely identified.

F. SAMPLE DOCUMENTATION

1. A field sheet is used to document the field sample collection process and contains pertinent information relative to the sample collected. (Laboratory QC samples are documented on the sample prep and/or analysis log as described in SOP 2410.10, "Analytical Data Submission Package Contents and Review". This section deals primarily with field collected samples.)

2. A field sheet will be completed for each sample collected and will be the official document that provides a permanent record of each sample collected. Since this document is the essential written component required to establish the relationship between the sample collected and the analytical results obtained, it will be controlled and will become a part of the official file on a sampling event.

3. Field sheets can be generated by the laboratory's LIMS, or alternate forms may be used. A field sheet should contain, at a minimum, the following information:

   a. Unique Sample Identifier - This may be recorded as three separate pieces of information (ASR Number, Sample Number and QC Code) or written as one entry (separated by hyphens).

   b. Matrix Sampled - The RLAB matrix as defined in section C.7.a.

   c. Project Information - This should include such things as the Project Manager, Project ID and description, city, state and other pertinent information.

   d. Location/Description - This short description should identify, to the satisfaction of the Project Manager, where the sample was collected. This is typically done by describing or naming the sample collection location.

e. Sample Collection Date/Time - For time-composited samples, the start date and time and end date and time are required. For grab samples only the start date and time are needed. Times should be recorded in the 24-hour format.

f. Analyses - An unambiguous list of the required laboratory analyses.

g. Field Measurements - Recorded along with the measurement units.

h. Comments - As appropriate.

i. Sampler - The name of the person(s) collecting the sample.

4. The Project Manager is responsible for ensuring that all field sheets are properly and accurately completed, and are safeguarded until they are delivered to RLAB.

5. The original completed field sheets for each sampling activity will be delivered to RLAB along with the samples to be analyzed. They will be maintained in the RLAB analytical support file for the specific ASR.

6. All entries on the field sheets will be legible and completed in indelible ink. Corrections to entries on field sheets should be accomplished by drawing a single line through the entry to be corrected, entering the correction above or adjacent to the lined-through entry and dating and initialing the correction.

7. In addition to the field sheet, another essential component of sample documentation is chain-of-custody. SOP 2420.4 describes the procedures for chain-of-custody of field collected samples being delivered to RLAB. SOP 2420.2, "Storage and Security of Environmental Samples" describes chain-of-custody procedures for within-lab sample transfers of routine samples. For samples that are connected with a criminal investigation, SOP 2420.10, "RLAB Procedures for Custody and Tracking of Samples and Analytical Data Files to be used as Evidence in Criminal Investigations," describes chain-of-custody documentation procedures for within-lab sample transfers.

G. SAMPLE TRACKING

1. The LIMS database system is used for tracking the status of samples and sample analyses through the analytical process and for tracking and reporting the results of sample analysis. Numerous reports are available from the LIMS and provide a variety of information pertaining to the samples and sample analyses. SOP 2410.20 and the LIMS online help provides more information on this.

2. Information relating to the status of samples submitted for analysis and the status of sample analyses may be obtained by the Project Manager from the LIMS or the RLAB Data Coordinator.

3. It is recognized that changes frequently occur in the field which result in changes to planned sampling activities. Since the LIMS system is used for logging in samples upon receipt, tracking, and ultimately reporting the results, it is essential that Project Managers ensure the entries contained in LIMS for specific sampling activities are accurate and complete (especially any field data and measurements). Discrepancies relating to numbers and types of samples and parameters requested for analysis must be corrected at the time of sample receipt by RLAB in accordance with SOP 2420.1.

4. SOP 2420.2 describes, for routine samples, the procedures for tracking the location of samples and sample containers within the laboratory. For samples that are connected with a criminal investigation, SOP 2420.10 describes the procedures used for tracking the location of samples and sample containers within the laboratory.

5. Unless otherwise requested, environmental samples will be properly disposed of in accordance with SOP 2420.9, "Sample Disposal", upon completion of the analysis and finalization of the analytical results.

H. QUALITY ASSURANCE AND QUALITY CONTROL

It is incumbent on all parties involved with sample collection, analysis, and management that these procedures be followed. Conformance with these procedures shall be evaluated during scheduled audits of RLAB operations as described in SOP 2430.5, "Quality Control Spot Checks of Regional Laboratory Data Packages", and SOP 2430.6, "Periodic Internal Program Review of the Region 7 Laboratory".

I. REFERENCES

1. Region 7 SOP 2410.10, Analytical Data Submission Package Contents and Review
2. Region 7 SOP 2410.15, Estimating and Documenting Data Quality
3. Region 7 SOP 2410.20, R7LIMS Functions and Security
4. Region 7 SOP 2420.1, Sample Receipt and Log-in
5. Region 7 SOP 2420.2, Storage and Security of Environmental Samples
6. Region 7 SOP 2420.4, Field Chain-of-Custody for Environmental Samples
7. Region 7 SOP 2420.6, Sample Container Selection, Preservation and Holding Times
8. Region 7 SOP 2420.9, Sample Disposal
9. Region 7 SOP 2420.10, RLAB Procedures for Custody and Tracking of Samples and Analytical Data Files to be used as Evidence in Criminal Investigations
10. Region 7 SOP 2420.13, RLAB Procedures for Preparation of Field Sheets and Tags
11. Region 7 SOP 2430.5, Quality Control Spot Checks of Regional Laboratory Data Packages
12. Region 7 SOP 2430.6, Periodic Internal Program Review of the Region 7 Laboratory

STANDARD OPERATING PROCEDURE

No. 2420.6E

SAMPLE CONTAINER SELECTION, PRESERVATION AND HOLDING TIMES

March 14, 2006

By Larry Marchin
ENSV/RLAB/CATS

APPROVED:

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Recertified:

Reviewer					
Date					

Table of Contents

Attachments

A. PURPOSE AND APPLICABILITY

The purpose of this Standard Operating Procedure (SOP) is to provide guidance for the selection of the proper sample containers and intermediate sample collection containers or devices when collecting samples for specific constituents (parameters) or groups of constituents; and for determining sample preservation and the holding times of samples from the time of collection until analysis is performed.

The guidance contained herein is applicable to all personnel who collect environmental samples for analysis by the Environmental Services Division (ENSV), including EPA and contractor personnel.

B. DEFINITIONS

ASR	Analytical Services Request
BNA	Base-Neutral/Acid Extractable
BOD	Biochemical Oxygen Demand
CBOD	Carbonaceous Biochemical Oxygen Demand
CFR	Code of Federal Regulations
DBCP	1,2-Dibromo-3-chloropropane
DO	Dissolved Oxygen
EDB	Ethylene Dibromide
ENSV	Environmental Services Division
GFF	Glass Fiber Filter
HEM	Hexane Extractable Material
MS	Matrix Spike
MSD	Matrix Spike Duplicate
NPDES	National Pollutant Discharge Elimination System
PCB	Polychlorinated Biphenyl
PE	Performance Evaluation
PM	Project Manager
QC	Quality Control
RLAB	Regional Laboratory Branch
RSTC	Regional Science and Technology Center (facility where the RLAB is located)
RSCC	Regional Sample Control Coordinator
SOP	Standard Operating Procedure
SSR	Sampling Supplies and QC/PE Samples Request
SW	Solid Waste
TCLP	Toxicity Characteristic Leachate Procedure
TKN	Total Kjeldahl Nitrogen

TOX	Total Organic Halogens
TPH	Total Petroleum Hydrocarbons

C. **SAMPLE CONTAINERS**

1. The use of the proper sample container is extremely important to ensure the representativeness of the analytical data obtained, the sufficiency of the sample volume for analysis, and the non-interference or contamination of the sample resulting from the sample container material. When considering the sufficiency of the sample volume for analysis, any matrix spike and matrix spike duplicates (MS/MSD) must be accounted for, and the sampling volume should be adjusted as necessary (One sample for each analyte in each sampling event should at least be double the normal volume or larger volumes for all samples could be used. For example, since water samples for extractables are being collected in 1-gallon bottles, there should be sufficient volume to perform MS/MSD analysis on any sample in the batch.)

2. Special care will be taken to avoid any inadvertent contamination of sample containers prior to or during the sample collection process. Specifically:

   a. Sample containers shall be left sealed or containerized during storage and transport to the sampling location and until the time of actual sample collection. [Exception: Polyethylene cubitainers are received from the manufacturer with the screw caps not attached. The cubitainers are collapsed and nested. The caps are screwed on to the cubitainers after they are filled with sample.]

   b. Sample containers will not be rinsed with the media being sampled during the sample collection process unless specifically required for a given parameter or sampling process (e.g., collecting water samples for toxicity testing).

   Note: The specific SOPs on sample collection should be referred to for the appropriate collection procedure to be used.

3. The sample container selection process is governed by:

   a. The parameter or group of parameters to be analyzed; this includes the desired level of detection in many cases.

   b. The media or matrix to be sampled (i.e., air, solid, tissue, water or other).

c. The analytical method to be used for the analysis.

d. The laboratory (i.e., EPA Region 7 laboratory or a contract laboratory) to perform the analysis. The laboratory where samples will be analyzed is determined by the Laboratory Branch (RLAB) upon receipt of an Analytical Services Request (ASR) form.

4. Attachment 1 provides specific guidance for use in Region 7 in selecting the proper sample container by parameter, or parameter group, and the media being sampled. Additional guidance or requirements for acceptable materials of sample containers are contained, by the parameter to be analyzed, in the current 40 CFR Parts 136 and 141, and SW-846. Also, guidelines are normally found in the specific analytical methods and sampling procedures.

5. When the use of intermediate sample collection containers is necessary, guidance on recommended intermediate container materials may be found in Attachment 2.

D. SAMPLE PRESERVATION

1. The immediate on-site analysis of samples at the time of collection is, in most cases, neither possible nor practical. Therefore, methods have been established to maintain the integrity of the sample until analysis can be accomplished. Even when samples are preserved in an appropriate manner, they should be analyzed as soon as possible after collection. An integral part of preservation is the selection of the proper sample container, the pretreatment of a sample container (if necessary), and the holding time allowable prior to analysis.

2. The purpose of sample preservation is to 1) retard biological action; 2) retard hydrolysis of chemical compounds and complexes; 3) reduce volatility of constituents; and 4) reduce absorption effects. The preservation methods used are generally limited to pH control, chemical addition, refrigeration or freezing. As a rule, the refrigeration (or icing) of samples should be utilized to maintain the samples at a temperature of $4 \pm 2°C$ during sample collection (including the collection of time or flow-weighted composite samples), transport, and storage.

3. The current guidance for sample preservation for use in Region 7 is provided in Attachment 1. Although taken into consideration when preparing this guidance, additional specific guidelines and requirements for sample preservation may be found in regulations (e.g., 40 CFR Part 136), publications (e.g., SW-846) and applicable analytical methods.

4. The following guidance is provided for field personnel to use when preserving the types of samples indicated:

   a. Grab Samples: The applicable preservation method must be accomplished immediately upon sample collection.

   b. Manually Composited Water Samples: The applicable preservation must be added, in full, to the initial aliquot and thus be available for each subsequent aliquot.

   c. Automatically Composited Water Samples:

      (1) When collected for either a single parameter or a parameter group where the type and amount of preservative required are identical, the applicable preservative is added to each container receiving an aliquot, prior to compositing.

      (2) When collecting a composite sample that will later be split to create samples for a variety of individual parameters and each of these parameters requires different preservation methods, the samples collected for the composite should be iced to maintain a temperature of 4°C until the compositing and splitting can be completed. The appropriate preservative is then added at the time the composite is split into separate containers.

5. Samples of the following media will <u>not</u> be preserved with the addition of any chemical compound, but will be chilled to 4°C after collection and during transport and storage.

   a. Solids: soil, sediment, sludge

   b. Tissue (or freeze, -15 to -20°C)

   c. Other: non-aqueous solutions, product samples (liquid or solid), drum samples, wipe samples

6. The following parameters require special procedures:

   a. **Biochemical Oxygen Demand (BOD)/Carbonaceous Biochemical Oxygen Demand (CBOD).** Water samples of chlorinated effluents collected for analysis of this parameter must be labeled with the word "CHLORINATED" on the sample tag to alert laboratory personnel.

Chlorinated samples require different analytical procedures than unchlorinated samples for this parameter.

b. **Cyanide, Total and Amenable to Chlorination**. Water samples for these parameters should not be collected using automatic samplers, but should be collected manually either as a grab or a composite of several grab samples which are preserved at the time of collection. Since oxidizing agents such as chlorine decompose many cyanides, the sample must be treated to eliminate such agents, if they are present, at the time of collection. The presence of chlorine is determined by testing a drop of the sample with potassium iodide (KI)-starch test paper. A change in the color of the paper to blue indicates the need for treating the sample with a dechlorinization agent. This treatment is accomplished by adding ascorbic acid, a few crystals at a time followed by the subsequent testing of a drop of sample until no color is produced on the KI indicator paper. An additional 0.6 gram of ascorbic acid is then added for each liter of sample volume. Preservation of the sample is then accomplished by adding 2 mL of 10 N sodium hydroxide solution or 10 pellets of sodium hydroxide crystals per liter of sample (to pH $\geq$ 12) and by icing the sample to 4°C during transport and storage.

c. **Dissolved Oxygen (DO)**. Water samples for this parameter are collected only on a grab basis. When collecting a sample for this parameter, the sample bottle should be filled to overflowing to ensure that no air bubbles are entrapped in the bottle when the stopper is replaced. When immediate measurement is not possible on site utilizing the DO probe method, the sample will be "fixed" immediately upon collection by first adding 2 ml of manganous sulfate ($MnSO_4$) solution and then 2 ml of alkali-iodide-azide solution well below the surface of the liquid. The sample is mixed by inverting the bottle several times while holding the stopper in place and allowing to set until the floc has settled half way. Carefully remove the stopper and immediately add 2 ml of concentrated sulfuric acid (H_2SO_4) by allowing the acid to run down the neck of the bottle. Re-stopper and mix again and store at 10-20°C out of direct sunlight. Completion of the analysis for DO utilizing the Winkler titration method (Azide Modification) should be accomplished as soon as possible after collection and fixing, but not more than 8 hours after collection.

d. **Metals, Dissolved**. Water samples for this parameter must be filtered on site utilizing a 0.45μm membrane filter as soon as practical after collection and then acidified with 5 mL 1:1 HNO_3 per liter of sample.

e. **Microbiology (Total and Fecal Coliform; Fecal Streptococci).** Water samples for these parameters will be collected only on a grab basis. For chlorinated effluents, sodium thiosulfate (0.008% $Na_2S_2O_3$) is added to the sample. This dechlorinating agent is normally added during sample container preparation and is, therefore, normally present in the sample container. Care must be taken during sample collection to avoid overfilling or rinsing out the agent. In addition, care must be taken to avoid contamination of the sterile sample container prior to or during sample collection; i.e., leave cap on container until ready to collect sample and do not place fingers in container or on the inside of the cap, while collecting a sample. An air space should be left at the top of the container after the sample is collected. Ice to 4°C.

f. **Oil and Grease (Hexane Extractable Material, HEM)** Water samples for this parameter will be collected in one liter glass bottles on a grab basis only and acidified with 1:1 HCl to pH < 2 immediately after collection. The sample container should never be rinsed with the water or wastewater because these constituents tend to adhere to the sides of the container. Care should be taken to avoid contamination of the sample from fingers placed in the container or on the inside liner of the cap. In addition, enough air space should be allowed in the container to allow for the addition of the preservative.

g. **Organics, Volatiles**. Grab samples only are collected for these parameters. Each sample will consist of two (2) 40-mL vials. Generally, four (4) 40-mL vials per water sample will be collected for low detection level and drinking water samples.

(1) Drinking water samples containing residual chlorine must be treated with sodium thiosulfate or ascorbic acid (depending on the analytical method) at the time of collection.

(2) Wastewater samples containing residual chlorine must be treated with ascorbic acid (25 mg per 40 mL) at the time of collection. These dechlorinating agents must be placed in the vials prior to collecting the samples.

(3) When collecting water samples, fill the sample bottles to overflowing, but take care not to flush out the sodium thiosulfate or ascorbic acid, if present. No air bubbles should pass through the sample as the bottle is filled, or be trapped in the sample when the bottle is sealed. After collection, the pH of the sample is adjusted

to a pH < 2 by carefully adding one drop of 1:1 HCl for each 20 mL of sample volume. Seal the sample bottles teflon-face down, and shake vigorously for 1 minute. A proper seal can be checked by inverting the sample and lightly tapping the end on a solid surface. If air bubbles are present, open the vial, add additional sample, reseal, and recheck for air bubbles. Store samples out of direct sunlight.

h. **Phenols (Phenolics)**. Water samples for this parameter should not be collected by using automatic samplers, but should be collected as a grab or a manual composite of grabs and preserved with 2 mL H_2SO_4 to a pH < 2 at the time of collection. The samples should be iced to maintain them at 4°C during transport and storage.

E. SAMPLE HOLDING TIMES

1. The issue of holding times for samples is critical in the sample collection and analysis process, because the integrity of the samples can be affected depending on the parameter to be analyzed. Sample holding times are defined as the period of time between sample collection and initiation of sample analysis. In the case of timed composite samples, the holding time starts at the end of the compositing period (i.e., at the time the last portion of the composite sample is obtained).

2. Since holding times can affect the validity of the reported analytical results (especially in certain media programs and in enforcement actions), everyone involved in planning and executing sampling activities; planning and performing analyses; and reviewing analytical results must be cognizant of the implications of exceeding them during the process. In many instances, the holding times are required by specific regulations (e.g., 40 CFR Part 136 for wastewater samples under the NPDES program), while many others are recommended. Also, see Footnote 8 of Attachment 1.

3. Although many of the holding times contained in Attachment 1 were derived from regulatory requirements, the holding times should be considered as guidelines. When making decisions on the validity of analytical results based on holding times, personnel should consult appropriate regulations to determine if there are specific requirements for sample holding times.

F. OBTAINING SAMPLING SUPPLIES

1. When sampling supplies (e.g., sample containers, sample collection devices, preservatives, etc.) are needed, the project manager for the specific sampling

activity requests the necessary supplies on the Sampling Supplies and QC/PE Samples Request (SSR).

2. The requestor can pick up the supplies at the ENSV warehouse facility (3150 Dodge). The preservatives must be picked up at the RSTC (300 Minnesota Avenue). It is recommended that the requestor contact the Regional Sample Control Coordinator (RSCC) or designated back-up at the RLAB before going to either facility to ensure the supplies are ready for issuance.

G. REFERENCES

1. Code of Federal Regulations, Title 40 (40 CFR), Part 136.

2. Test Methods for Evaluating Solid Waste, Physical/Chemical Methods (SW-846), U.S. EPA.

3. Standard Methods for the Examination of Water and Wastewater, Joint Editorial Board, American Public Health Association, American Water Works Association and Water Environment Federation, Latest Edition.

Attachment 1

Guide for Sample Container Selection, Preservation and Holding Times

Parameter[1]	Sample Container[2]	Preservation	Holding Time[8]
I. **Air and Gaseous**			
Dioxins/Furans	Puff glass jar	Cool 4°C	365 days
Metals on High-Vol GFF	1-gal. resealable plastic bag	Ambient Temperature	
Ozone Precursors			
Ambient levels	6-L canister	None	60 days
Source levels	400 mL canister	None	60 days
Particulate Matter on High-Vol GFF	1-gal. resealable plastic bag	Ambient Temperature	
Pesticides/PCBs	Puff glass jar	Cool 4°C	7 days
Semivolatiles/BNA	Puff/XAD glass jar	Cool 4°C	14 days
Volatile Organics			
Ambient levels	6-L canister	None	60 days
Source levels	400 mL canister	None	60 days
GC/MS Scan	6-L canister	None	30 days
II. **Soil, Sediment and Solids**			
A. Non-Product Samples			
Asbestos	Plastic jar	None	None
Cyanide	8-oz. glass jar	Cool 4°C	28 days
Dioxin/Furans	8-oz. glass jar/sealable plastic bag in 1-qt. paint can	Cool 4°C	365 days
Explosives	8-oz. glass jar	Cool 4°C	14 days
Flashpoint	8-oz. glass jar	Cool 4°C	
GC/MS Scan, SemiVOA	8-oz. glass jar	Cool 4°C, store in dark	14 days
GC/MS Scan, VOA	2, 40mL VOA vials	Cool 4°C	14 days
Herbicides	8-oz. glass jar	Cool 4°C	14 days
Metals			
All metals except Cr^{+6} and Hg, collected separately	8-oz. glass jar	Cool 4°C	180 days
Mercury	8-oz. glass jar	Cool 4°C	180 days
Cr^{+6}	8-oz. glass jar	Cool 4°C	30 days
Methanol	8-oz. glass jar	Cool 4°C	14 days
Nutrients			
Nitrogen (NH_3, NO_2, TKN)	8-oz. glass jar	Cool 4°C	28 days
Phosphorous, total	8-oz. glass jar	Cool 4°C	
Oil and Grease	8-oz. glass jar	Cool 4°C	
pH	8-oz. glass jar	Cool 4°C	None
Phenolics (colorimetric)	8-oz. glass jar	Cool 4°C	28 days
Perchlorate	8-oz. glass jar	Cool 4°C	28 days
Pesticides/PCBs	8-oz. glass jar	Cool 4°C	14 days
Radioactivity	8-oz. glass jar	Cool 4°C	180 days

Parameter	Sample Container	Preservative	Holding Time
II. Soil, Sediment, and Solids (cont.)			
A. Non-Product Samples (cont.)			
Semivolatiles/BNA	8-oz. glass jar	Cool 4°C	14 days
Sulfate/Sulfide	8-oz. glass jar	Cool 4°C	28 days
Soil Toxicity Test	1-gal. ziplock bag	Cool 4°C	56 days
Total Organic Carbon	8-oz. glass jar	Cool 4°C	28 days
Total Petroleum Hydrocarbons (TPH)			
SemiVOA	8-oz. glass jar	Cool 4°C	14 days
VOA	2, 40-mL glass vials	Cool 4°C	14 days
Total Kjeldahl Nitrogen	8-oz. glass jar	Cool 4°C	None
Organic Parameters			
All except volatile organics	8-oz. glass jar	Cool 4°C	14 days
Volatile organics[3]	2, 40-mL glass vials	Cool 4°C or MeOH & Cool 4°C or $NaHSO_4$ & Cool 4°C	14 days
TCLP			
Metals, except Hg	8-oz. glass jar	Cool 4°C	180 days to extract, 180 days after extraction (required)
Mercury	8-oz. glass jar	Cool 4°C	28 days to extract, 28 days after extraction (required)
Volatile organics	2, 40-mL glass vials	Cool 4°C	14 days to extract, 14 days after extraction (required)
Semivolatile organics	8-oz. glass jar	Cool 4°C	14 days to extract, 7 days to extraction, 40 days after extraction (required)
Pesticides/Herbicides	8-oz. glass jar	Cool 4°C	14 days
B. Product Samples			
All parameters	8-oz. glass jar/sealable plastic bag in paint can	Cool 4°C	
III. Tissue			
Fish, collected for whole body/edible portion, all parameters	Double wrapped in heavy duty foil	Freeze	
Resectioned tissue, collected for:			
Metals	Double wrapped in heavy duty foil	Freeze	180 days
Semivolatiles	Double wrapped in heavy duty foil	Freeze	
Volatiles	Double wrapped in heavy duty foil	Freeze	
Foliage			
Herbicides/Pesticides	Double wrapped in heavy duty aluminum foil	Freeze	
Macroinvertibrates[6]		70% ethanol	6 months
Periphyton[6]			
Chlorophyll A		Freeze, store in dark	30 days
Enumeration		5% formalin, Cool 4°C, store in dark	6 months
Dioxins/Furans	Double wrapped in heavy duty foil	Freeze	365 days
Phytoplankton, collected for[6]			
Chlorophyll A		Cool 4°C, store in dark	14 days
Enumeration		5% formalin	6 months

Parameter	Sample Container	Preservative	Holding Time
IV. Aqueous Samples			
Chlorine Dioxide	1-L plastic cubitainer	Cool 4°C	None
Chlorophyll A	4-L plastic cubitainer	Cool 4°C, store in dark	14 days
Coliform, fecal	300-mL sterile plastic bottle	Cool 4°C, 0.008% $Na_2S_2O_3$	6 hours
Dioxins/Furans	1-L amber glass bottle	Cool 4°C	365 days
Dissolved Organic Carbon	1-L amber glass bottle	H_2SO_4 to pH<2 Cool 4°C	28 days
Explosives	128-oz. amber glass bottle	Cool 4°C	7 days to extract, 40 days after extraction
Herbicides	128-oz. amber glass bottle	Cool 4°C	7 days to extract, 40 days after extraction
Flashpoint	8-oz. glass jar	Cool 4°C	
Metals (except Hg and Cr^{+6})			
Total and acid soluble	1-L plastic cubitainer	HNO_3 to pH<2	6 months
Dissolved	1-L plastic cubitainer	Filter HNO_3 to pH<2	6 months
Chromium, hexavalent	1-L plastic cubitainer	Cool 4°C	24 hours
Mercury	1-L plastic cubitainer	HNO_3 to pH<2	28 days
Strontium	1-L plastic cubitainer	HNO_3 to pH<2	6 months
Acid, %	1-L plastic cubitainer	Cool 4°C	None
Alkalinity	1-L plastic cubitainer	Cool 4°C	14 days
BOD/CBOD	1-L plastic cubitainer	Cool 4°C	48 hours
COD	1-L plastic cubitainer	H_2SO_4 to pH<2 Cool 4°C	28 days
Chlorine (residual)	1-L plastic cubitainer	Cool 4°C	1 day
Conductivity	1-L plastic cubitainer	Cool 4°C	28 days
Cyanide (total and amenable To chlorine)	1-L plastic cubitainer	(Ascorbic acid), NaOH to pH>12, Cool 4°C	14 days
Halides (Br^-, Cl^-, F^-)	1-L plastic cubitainer	Cool 4°C	28 days
Haloacetic Acids/Dalapon	1-L amber glass bottle	Ammonium Chloride, Cool 4°C	14 days
Hardness	1-L plastic cubitainer	HNO_3 to pH<2, Cool 4°C	6 months
Inorganic Anions	1-L plastic cubitainer	EDA, Cool 4°C	14 days
Methane, Ethane, Ethene	2, 40-mL VOA vials	Cool 4°C	7 days
Methanol	1-L amber glass bottle	Cool 4°C	7 days
Nonfilterable Solids (NFS)	1-L plastic cubitainer	Cool 4°C	7 days
Oxygen, Dissolved (Winkler)	300-mL glass BOD bottle with Attached ground glass stopper	$MnSO_4$ + Alkali-Iodide-Azide, H_2SO_4	8 hours
Dissolved Oxygen, probe Method	1-L plastic cubitainer	Cool 4°C	1 day

Parameter	Sample Container	Preservative	Holding Time
IV. Aqueous Samples (cont.)			
pH	1-L plastic cubitainer	Cool 4°C	determine immediately
Perchlorate	1-L plastic cubitainer	Cool 4°C	28 days
Residue			
All but settleable	1-L plastic cubitainer	Cool 4°C	7 days
Settleable	1-L plastic cubitainer	Cool 4°C	48 hours
Sulfate	1-L plastic cubitainer	Cool 4°C	28 days
Sulfide	1-L plastic cubitainer	Zinc acetate + NaOH to pH>9, Cool 4°C	7 days
Total Dissolved Solids (TDS)	1-L plastic cubitainer	Cool 4°C	7 days
Total Kjeldahl Nitrogen	1-L plastic cubitainer	H_2SO_4 to pH<2.5, Cool 4°C	28 days
Total Solids	1-L plastic cubitainer	Cool 4°C	7 days
Turbidity	1-L plastic cubitainer	Cool 4°C	48 hours
Nutrients			
Nitrogen-Ammonia	1-L plastic cubitainer	H_2SO_4 to pH<2 , Cool 4°C	28 days
Nitrogen-Organic	1-L plastic cubitainer	H_2SO_4 to pH<2, Cool 4°C	28 days
Nitrate	1-L plastic cubitainer	Cool 4°C	48 hours
Nitrate-Nitrite	1-L plastic cubitainer	H_2SO_4 to pH<2, Cool 4°C	28 days
Nitrite	1-L plastic cubitainer	Cool 4°C	48 hours
Phosphorous (total)	1-L plastic cubitainer	H_2SO_4 to pH<2.5, Cool 4°C	28 days
Ortho-phosphate	4-oz. plastic bottle	Filter, Cool 4°C	48 hours
Phosphorous, (dissolved)	1-L plastic cubitainer	Filter, H_2SO_4 to pH<2, Cool 4°C	28 days
Carbamates[10]	60-mL screw cap vial	1.8 mL monochloroacetic acid buffer pH<3, Cool 4°C	14 days
Oil and Grease (HEM)	1-L glass jar	1:1 HCL to pH<2, Cool 4°C	28 days
Pesticides/PCBs	128-oz. amber glass bottle	Cool 4°C	7 days to extract, 40 days after extraction[9]
Phenolics	1-L glass jar	H_2SO_4 to pH<2, Cool 4°C	28 days
Radionuclides	1-L plastic cubitainer	HNO_3 to pH<2	6 months (water:alpha/beta) 5 days(gamma; DW:alpha/beta)
Semivolatile/BNA	128-oz. amber glass bottle	Cool 4°C, store in dark	7 days to extract, 40 days after extraction
GC/MS Scan (BNA)	128-oz. amber glass bottle	Cool 4°C, store in dark	7 days
Total Organic Carbon (TOC)	1-L amber glass bottle	H_2SO_4 to pH<2, Cool 4°C	28 days
Total Organic Halogens (TOX)	8-oz. amber glass bottle	Cool 4°C	7 days

Parameter	Sample Container	Preservative	Holding Time
IV. Aqueous Samples (cont.)			
Total Petroleum Hydrocarbons (TPH)			
SemiVOA	128-oz. amber glass bottle	Cool 4°C	7 days
VOA	2, 40-mL VOA vials	Cool 4°C	14 days
Toxicity Tests			
Acute	10-L (2.5-gal) plastic cubitainer	Cool 4°C	36 hours
Bioscreen (24 or 48 hr.)	4-L (1-gal.) plastic cubitainer or glass bottle	Cool 4°C	24 hours
Chronic	20-L (2.5/5-gal.) plastic cubitainer	Cool 4°C	36 hours
Triazine Herbicides	128-oz. amber glass bottle	Cool 4°C	14 days
Tritium	8-oz. glass jar	None	5 days
Volatile Organics[3, 5]			
Purgeable halocarbons	2, 40-mL glass vials	HCl to pH<2, Cool 4°C	14 days
Purgeable aromatic hydrocarbons	128-oz. amber glass bottle	HCl to pH<2, Cool 4°C	7 days
Routine Detection Level	2, 40-mL glass vials	HCl to pH<2, Cool 4°C	14 days
Low Detection Level	4, 40-mL glass vials	HCl to pH<2, Cool 4°C	14 days
EDB/DBCP	2, 40-mL glass vials	Sodium Thiosulfate Cool 4°C	14 days
GC/MS Scan (VOA)	2, 40-mL VOA vials	HCl to pH<2, Cool 4°C	14 days
V. Liquid, Non-Aqueous			
TCLP (> 5% solids)[4]			
Mercury	8-oz. glass jar	Cool 4°C	28 days to TCLP extract, 28 days after extraction
Metals, except Hg	8-oz. glass bottle	Cool 4°C	180 days
Volatile Organics	2, 40-mL glass vials	Cool 4°C	14 days to TCLP extract, 14 days after extraction
Semivolatile Organics	8-oz. glass jar	Cool 4°C	14 days to TCLP extract, 7 days to extraction, 40 days after extraction
Pesticides/Herbicides	128-oz. amber glass bottle	Cool 4°C	7 days
Organic Parameters			
All except volatile organics	8-oz glass jar	Cool 4°C	7 days to extract, 40 days after extraction
Volatile organics	8-oz glass jar	Cool 4°C	14 days
VI. Wipe Samples[7]			
Arsenic	8-oz. glass jar	Cool 4°C	
Cyanide	8-oz. glass jar	Cool 4°C	
Dioxin	8-oz. glass jar	Cool 4°C	365 days
Herbicides/Pesticides	8-oz. glass jar	Cool 4°C	14 days
Metals	8-oz. glass jar	Cool 4°C	180 days
Picric Acid	8-oz. glass jar	Cool 4°C	
Organic other parameters			
All except volatile organics	8-oz. glass jar	Cool 4°C	7 days to extract, 40 days after extraction
Volatile organics	8-oz. glass jar	Cool 4°C	

Parameter	Sample Container	Preservative	Holding Time
VI. Wipe Samples (cont.)			
TCLP (> 0.5% solids)			
Metals, except mercury	8-oz. glass jar	Cool 4°C	180 days to TCLP extract, 180 days after extraction

[1] Non-product and product sample definitions: A non-product sample is a sample which consists primarily of naturally occurring materials that may contain mechanically or chemically manufactured materials or substances as contaminants. A product sample is a sample which is known to consist primarily of a mechanically or chemically manufactured material that does not otherwise occur naturally in the immediate environment being sampled. A sample which cannot be identified as a non-product sample should be considered a product sample

[2] All glass containers require a Teflon-lined lid or cap.

[3] These parameters are always collected as replicates. The sample container consists of two or four 40-mL glass vials (VOA vials) and an activated carbon filled thimble contained in a 1-L plastic cubitainer.

[4] § 40 CFR Part 261 Appendix II, 2.1: For liquid wastes (i.e., those containing less than 0.5% dry solid material), the waste, after filtration through a 0.6 to 0.8 μm glass fiber filter, is defined as the TCLP extract.

[5] Sodium Thiosulfate ($Na_2S_2O_3$) or Ascorbic Acid ($C_6H_8O_6$) is utilized to de-chlorinate samples of chlorinated water or wastewater prior to pH adjustment. For drinking water samples, consult the applicable method to determine appropriate dechlorinating agent.

[6] Sample containers, preservation, and holding times vary. The information provided is Regional guidance. For compliance samples, consult appropriate references for complete preservation requirements.

[7] Each jar should contain a medical gauze pad.

[8] Generally, there are no required holding limits for air, soil, product and wipe media. (Exceptions: see Chapter 4 of SW-846). However, it is recommended that samples be analyzed within the holding time limits established in aqueous media for the specific analytes or analyte groups.

[9]Method 505 holding time 14 days (7 days for Heptachlor), preserve with Sodium Thiosulfate, 4° C, container 40-mL vial.

[10]Method 531.1 samples must be preserved to a pH<3 using monochloroacetic acid to minimize degradation of oxamyl, 3-hydroxycarbofuran, aldicarb sulfoxide, and carbaryl in neutral and basic waters. If residual chlorine is present add 80 mg of sodium thiosulfate per liter of sample to the sample bottle prior to collecting the sample.

Attachment 2

Guide for Selecting Intermediate Sample Container Material

Media Sampled/Parameter	Intermediate Sample Container Material
I. Soil, Sediment and Solids	
A. Non-Product and Product Samples	
All parameters except volatile organics	Glass, Aluminum
Volatile Organics[1]	Glass, Stainless Steel, Aluminum
II. Tissue	
A. Fish	
All parameters	Glass, Plastic
III. Liquids	
A. Aqueous Samples	
Chlorophyll A	Artificial substrate (glass slide)
Coliform, fecal[2]	Glass or Plastic
Dioxin/Furans	Glass, Stainless Steel (solvent rinsed)
Explosives	Glass
Metals:	
All except Cr^{+6}	Glass, Plastic, Automatic sampler equipped with Tygon intake tubing and glass or plastic compositing container.
Chromium, hexavalent	Glass, Plastic
Minerals and Dissolved Materials:	
Acid (%), Alkalinity, BOD, Chloride, Conductivity, Hardness, Residue, Sulfate, Turbidity	Glass, Plastic, Stainless steel, Automatic sampler equipped with Tygon (or Teflon) intake tubing, and glass or plastic compositing container
Chlorine	Glass, Plastic, Stainless steel
COD	Glass, Plastic, Stainless steel, Automatic sampler equipped with Tygon (or Teflon) intake tubing, and glass or plastic compositing container
Cyanide	Glass, Plastic, Stainless steel

Media Sampled/Parameter	Intermediate Sample Container Material
III. Liquids (cont.)	
A. Aqueous Samples (cont.)	
Fluoride	Plastic, Automatic sampler equipped with Tygon (or Teflon) intake tubing, and plastic compositing container
Oxygen, dissolved	Glass, Plastic, Stainless steel
pH, lab or field	Glass, Plastic, Stainless steel, Teflon
Sulfide	Glass, Plastic, Stainless steel
Nutrients (N & P)	Glass, Plastic, Stainless steel, Automatic sampler equipped with Tygon (or Teflon) intake tubing, and glass or plastic compositing container
Oil and Grease[2]	Glass
Pesticides/PCBs	Glass, Stainless steel, Automatic sampler equipped with Teflon intake tubing and glass compositing container (cleaned and solvent rinsed)
Phenols/Phenolics	Glass, Plastic, Stainless steel
Radionuclides	Glass, Plastic
Semivolatiles/BNA	Glass, Stainless steel, Automatic sampler equipped with Teflon intake tubing and glass compositing container (cleaned and solvent rinsed)
Total Organic Carbon	Automatic sampler equipped with Tygon (or Teflon) intake tubing, and glass or plastic compositing container
Total Organic Halogens	Glass, Plastic
Toxicity Tests: Acute Bioscreen Chronic	Automatic sampler equipped with Teflon or new Tygon tubing, and glass or Nalgene compositing container (see SOP No. 2334.6 for tubing and container cleaning)
Volatile Organics[1]	Glass, Stainless steel (not solvent rinsed)
B. Non-Aqueous Product Samples	
All parameters	Glass

[1]An intermediate sample collection device is not recommended for this parameter, but, if one is necessary, care must be taken to insure that the device has not been solvent rinsed.

[2]An intermediate container is not recommended for this parameter; therefore, every effort must be made to collect the sample directly into the sample container.

STANDARD OPERATING PROCEDURE

No. 2430.12E

REGIONAL LABORATORY QUALITY CONTROL POLICY

December 18, 2003

by

Harry Kimball

ENSV/RLAB

APPROVED:

Peer Reviewer — Date: 12/23/03

Manager, Regional Laboratory — Date: 12/24/03

Independent QA Reviewer — Date: 12/24/03

Recertified

Reviewer	HK	HK			
Date	11-19-04	1-5-07			

Table of Contents

Attachments

A. PURPOSE AND APPLICABILITY

The purpose of this Standard Operating Procedure (SOP) is to document the established Quality Control (QC) Policy for Region 7 Laboratory in-house analytical operations. This policy contains a series of uniform QC procedures to ensure that data are generated with sufficient and appropriate controls to meet client requirements for data quality. This SOP is intended to contain sufficient versatility and generality to cover changing client needs and program focus. The reader is referred to SOP 2410.10, "Analytical Data Submission Package Contents and Review" and SOP 2410.15, "Estimating and Documenting Data Quality," for additional details on the implementation of the policies described herein. It is recognized that some analyses cannot conform to all conditions in this policy. The QC requirements specified in particular regulations, reference methods, and/or RLAB Methods shall be followed.

The policies and procedures detailed in this SOP shall be utilized by Region 7 Laboratory personnel and in-house contractor personnel responsible for implementing, performing, reviewing, and/or reporting analyses. These policies and procedures shall be considered minimum requirements. Additional procedures may also be required by regulatory, method, contract, or SOP references. The QC policies and procedures required of contract laboratories [Contract Laboratory Program (CLP) and Regional Environmental Analysis Program (REAP) Contract] may be different than those listed below. Whenever possible, contract laboratories shall be required to follow the procedures and meet the standards listed below. However, where a contract Statement of Work (SOW) is incompatible with these procedures and standards, the QC requirements of the contract SOW will take precedence.

It is the goal of the Region 7 Laboratory to produce legally- and technically-defensible data of known and documented quality, which is useable for the purpose(s) to which it was intended. Every effort will be made to achieve this goal.

B. POLICY

The Region 7 analytical QC program will establish, maintain, and monitor data quality with internal QC checks. Internal QC checks shall be used to address three questions:

1. Has the laboratory's ability to produce data of acceptable quality been established and maintained?

2. Were laboratory operations "in control" (operating within acceptable QC limitations) during data generation?

3. What effect did the sample matrix have on the data being generated?

The first question is addressed by initial and on-going demonstrations of capability, establishment of method detection limits, and preventive maintenance procedures and records. The laboratory's overall abilities are assessed by the performance and documentation of these initial and on-going QC measures.

The second question is addressed by calibration checks, positive and negative method performance checks, and by following sample preservation and maximum holding-time protocols. Method calibration and positive performance checks are compared to established control limits. This information, in conjunction with negative performance checks (blanks), is used to assess analytical batch-level laboratory performance.

The third question is addressed by matrix-specific QC. Matrix-specific QC is based on the use of actual environmental samples for precision and bias determinations and relies on the analysis of matrix spike samples. Matrix spike information is used to assess the effect of the sample matrix on analytical data.

To meet the laboratory's quality objectives, the following steps will be taken:

1. All data generated within or for the Region 7 Laboratory will be subject to QC review.

2. Initial and ongoing demonstration of capability and determination of method detection limits shall be performed and documented for each analysis (RLAB Method) performed routinely in the laboratory.

3. One complete set of QC checks (as appropriate to each method) shall be run with each batch of field samples. A batch shall consist of no more than 20 field samples. (For the purposes of defining a batch, field samples include: "real" field samples, field duplicates, field blanks, field spikes, performance testing, and blind performance evaluation samples.) In general, a "complete set of QC checks" will include, depending on the method, at least these four analyses:

   - Laboratory control sample
   - Method blank
   - Matrix spike
   - Matrix spike duplicate

   The CLP SOWs specify in detail what QC checks will be run and when. Each Task Order issued under the REAP Contract shall specify what QC checks are to be run and how often.

4. For each analytical measurement performed routinely in the laboratory, QC information shall be stored in the laboratory information management system. From this historical database, control limits shall be established for each routine test measurement. Control limits shall be updated at least every two years in conjunction with the review of the corresponding RLAB Method. (See SOP 2410.15 for control limit calculation procedures.)

5. The Region 7 Laboratory shall report all in-house generated results, rounded to the number of significant figures specified in the RLAB Method (see SOP 2410.19, "Significant Figures (Digits)"), down to the Region 7 Reporting Limit (RL). RLAB Method RL's must be greater than the method detection limit (MDL) and are, typically, approximately three times the MDL. The CLP Statements of Work (SOW) specify "Contract Required Quantitation Limits (CRQLs)," down to which all CLP data will be reported. Each REAP Task Order SOW shall specify the limits to be reported (RLs or CRQLs), based on the project data quality objectives.

6. For conducting analyses in-house (not by an out-source contract laboratory), if any QC check fails to meet applicable control limits, corrective action is required for all associated data (e.g. the same prep and/or analysis batch). This includes a review of sample preparation and instrument performance data and/or a consultation with the supervisor to discuss data qualification and/or sample re-analysis. Possible courses of action may include, but are not limited to, dilution, re-analysis, method of standard additions, or use of a different method. It is possible that the outlier may be due to a challenging matrix or may be beyond the control of the laboratory, in which case no corrective action can be taken. The analytical supervisor is responsible for establishing and documenting the policy for common corrective actions. The analyst should consult the supervisor whenever there is any question about the appropriate corrective action for a given sample analysis. All corrective actions shall be fully documented in the back-up data file. When data quality exceptions are not corrected, associated data will be qualified (see SOP 2410.10 for data qualification guidelines).

   For conducting analyses under the CLP SOWs, the procedures for dealing with QC check anomalies are detailed in the SOWs and in SOPs 2430.2, 2430.3, and 2430.4. For conducting analyses under the REAP Contract, the REAP laboratory shall follow technically sound procedures for dealing with QC check anomalies and fully document all anomalies, corrective actions and their results in the data deliverable.

7. Any given procedure or activity may include additional QC which can be defined separately as the need arises.

C. DEFINITIONS

Analytical Services Request (ASR). An ASR is a document used by a Project Manager to request analytical services from RLAB. On the ASR, the Project Manager explicitly states the matrix of the samples to be collected, the analytical method to be applied to those samples, the analytes for which results are required, and any additional data quality objectives. Implicit in this request is that reported analytical results meet the requirements of this QC Policy.

Method Detection Limit (MDL). This term refers to the quantitative expression for the minimum concentration of a substance that an analytical method is capable of detecting and quantifying with 99% confidence that the concentration of the substance is greater than zero. The method detection limit is determined from the analysis of replicate samples in accordance with the procedures outlined in Title 40, Code of Federal Regulations (CFR), Part 136 Appendix B or as specified in the method.

Reporting Limit (RL). The Reporting Limit for an analyte is the concentration represented by the lowest level in the initial calibration curve where the analyte is detected, unless otherwise specified in the RLAB Method. RL's are, typically, approximately three times the method detection limit (MDL).

RLAB. The EPA Region 7 Laboratory.

RLAB Approved Data. Data that have been through the laboratory's data verification process (verified data). Any data that do not meet all prescribed requirements during the data verification process are qualified and accompanied by a narrative explaining the nature of the data quality exception. Data in the Data Transmittal sent by RLAB to the Project Manager are RLAB Approved Data.

RLAB Method. The RLAB Method is the Region 7 Laboratory document that describes an analytical procedure. Typically these are based on published EPA methods.

Validated Data. Data that have been reviewed by the Project Manager, or other end user, for conformance to the requirements of the specific use for which the data were generated. Typically, the first step in this process is for the Project Manager to obtain RLAB Approved Data from the Region 7 Laboratory.

Verified Data. Data that have been reviewed by RLAB for conformance to the requirements specified, implicitly and explicitly, in the analytical services request. These include the requirements set forth in this SOP (and SOPs referenced by this SOP), specific quality control requirements of the requested analysis (contained in the RLAB Method), and any additional requirements specified by the Project Manager in the ASR.

D. PERSONNEL RESPONSIBILITIES AND AUTHORITY

1. Laboratory Management

a. Members

The supervisors and managers who direct the analytical work of each laboratory group are directly responsible for ensuring that all employees reporting to them are complying with the Regional Laboratory QC Policy.

b. Responsibilities

Laboratory management is responsible for:

(1) Actively supporting the implementation of the Regional Laboratory QC Policy within the laboratory

(2) Maintaining accurate SOPs, SLOMs, and RLAB Methods and enforcing their use in the laboratory

(3) Maintaining a work environment that emphasizes the importance of data quality

(4) Providing management support to the Regional QA Manager

c. Authority

The managers and supervisors of the laboratory have the authority to accept or reject data based on well-defined QC criteria. In addition, managers and supervisors can accept data which fall outside of normal QC limits if, in their judgment, there are technical reasons which warrant the acceptance of the data. These circumstances must be well documented and any needed corrective action identified by the incident must be defined and initiated. The analyst shall generate this documentation and the supervisor shall approve the documentation. All such documentation shall

be maintained in the primary data file. The authority of the laboratory management comes directly from the ENSV Division Director.

2. Laboratory Personnel

a. Members

All laboratory personnel involved in the generation and reporting of data have a responsibility to understand and follow the Regional Laboratory QC Policy.

b. Responsibilities

Laboratory personnel are responsible for:

(1) Possessing a working knowledge of the Regional Laboratory QC Policy

(2) Ensuring that all work is generated in compliance with the Regional Laboratory QC Policy

(1) Performing all work according to written SOPs, SLOMs, and RLAB Methods

(2) Ensuring that all documentation related to their work is complete and accurate

(3) Providing management with immediate notification of quality problems

c. Authority

Laboratory personnel have the authority to accept or reject data based on compliance with well-defined QC acceptance criteria. The acceptance of data which fall outside QC criteria must be approved by laboratory management. The authority of the laboratory personnel flows from the Laboratory Program Managers.

3. In-House Contractors

The requirements discussed above as applicable to EPA employees shall also be required of all contractor employees using Regional Laboratory equipment and facilities.

E. SPECIFIC ELEMENTS OF THE LABORATORY QC POLICY

1. Initial and On-going Demonstration of Capability

a. Initial and on-going demonstration of capability is important for the documentation of the laboratory's ability to produce data of acceptable quality initially and on a continuing basis. This process is fully described in SOP 2410.11, "Analytical Proficiency Demonstration in the Region 7 Laboratory."

b. Contract laboratories conduct initial and ongoing QC as a part of obtaining the contracts, and in accordance with SOW and Task Order requirements. Such QC shall be checked as a part of the routine on-site evaluation process for contract laboratories.

2. Method Detection Limits

a. Method detection limits will be determined by the procedure specified in the method or by the 7 to 10 replicate procedure described in 40 CFR Part 136 Appendix B, "Definition and Procedure for the Determination of the Method Detection Limit."

b. The sample preparation, analysis, and calculation procedures used for field sample analysis will be employed in the determination of the method detection limit.

c. A method detection limit study will be performed initially when a new method is implemented and subsequently whenever there is a significant change in the method or instrumentation such that the detection limit may be affected.

3. Preventative Maintenance

Analytical instrumentation and equipment will be maintained by following established preventative maintenance procedures (per manufacturer's recommendations) and records of such maintenance shall be kept.

4. Calibration Data

a. Standard/Reagent Preparation

A critical element in the generation of quality data, is the purity/quality and traceability of the standard solutions and reagents used in the analytical operations. To ensure the highest purity possible, all primary reference standards and standard solutions are obtained from the National Institute of Standards and Technology (NIST), the EPA Repository, or other reliable commercial sources (e.g. NIST traceable).

Standard solutions are validated prior to use. Validation procedures can range from a check for chromatographic purity to verification of the concentration of the standard using a standard prepared at a different time or obtained from a different source and should be specified in the RLAB Method. Care is exercised in the proper storage and handling of standard solutions, and all containers are labeled as to compound, concentration, solvent, expiration date, initials of preparer and preparation date. Stock and working standards are checked regularly for expiration and signs of deterioration such as discoloration, formation of precipitates, or change of concentration. Supervisors are responsible for establishing policies for determining when standards must be replaced.

b. Instrument Calibration and Tuning

Instrumentation requires calibration and tuning to ensure that the analytical system is operating correctly and functioning at the proper sensitivity to meet established detection limits. Each instrument is tuned and calibrated with standard solutions appropriate to the type of instrument and the linear range established for the analytical method. The frequency of tuning, calibration, and the concentration of calibration standards is determined by the manufacturer's guidelines, the analytical method, or special program requirements and should be specified in the RLAB Method. Established calibration criteria must be met before analysis of samples, or data must be qualified. (See SOP 2410.10 for guidelines for data qualification.)

It is recognized, for multi-analyte analyses where the analytical standards are purchased as mixtures containing the analytes at equal concentrations, that the analytes may have widely differing responses. In these cases, a multi-point calibration curve may contain additional (e.g. 6 or more) calibration levels such that, for analytes with low response, the lower point(s) in the curve can be dropped/removed or, for analytes with high response, the upper point(s) in the curve can be dropped/removed in order to obtain an acceptable calibration for all of the analytes in the calibration mix. This is an acceptable practice as long as care it taken to note the reporting limit (lower end of the calibrated range) and upper end of the calibrated range for each analyte. However, it is not acceptable to drop/remove points in the middle of the curve.

5. Complete Set of QC Checks

For each batch of no more than 20 field samples, a set of laboratory QC samples (as appropriate for the method) will be analyzed. Typically this means that a prep and/or analysis batch will contain, in addition to field samples, the following four QC check samples.

- Laboratory control sample
- Method blank
- Matrix spike
- Matrix spike duplicate

Again, the QC required from contract laboratories shall be that specified in the SOWs and Task Orders. Whenever possible, contract laboratories shall be required to follow the procedures and meet the standards listed in this SOP. Data generated under the CLP Contract shall be evaluated in accordance with the SOWs and SOPs 2430.2, 2430.3, and 2430.4. Data generated under the REAP Contract shall be evaluated for adherence to the QA/QC requirements of the related Task Order SOW, which are based on the data quality objectives of the project.

a. Laboratory Control Sample (LCS)

An LCS is used to monitor the laboratory's batch-level performance of routine analytical methods. An LCS consists of a control matrix which has been spiked with a group of target compounds representative of the method analytes. The source of LCS spike solutions are kept as independent as possible from the calibration standards. A Performance Evaluation sample, obtained from an outside source, may be used for an

LCS. An LCS is analyzed with environmental samples to provide evidence that the laboratory is performing the analytical method within accepted QC guidelines.

For analyses with an extensive list of target analytes, not all analytes need be spiked. However, each LCS must include a representative mix of analytes and the laboratory shall ensure that all analytes in routine analyses are spiked within a two year period. For analyses having 1 to 10 analytes, spike all analytes. For analyses having 11 to 20 analytes, spike at least 10 or 80% whichever is greater. For analyses having more than 20 analytes, spike at least 16 analytes. Where the analysis contains multi-component analytes that interfere with accurate analysis of the mixture (e.g. technical chlordane, toxaphene, or PCBs), the LCS should contain those analytes that are most likely to be found in the samples and that don't interfere with one another.

Bias (percent recovery) data from the LCS are compared to control limits that have been established for each of the analytes monitored in the LCS. Control limits for bias are based on the historical average percent recovery of the LCS plus or minus three standard deviations (see SOP #2410.15 for details about statistical calculations and control limits). Decisions concerning laboratory performance of the method are based on QC data generated from an LCS.

In some situations control limits calculated by applying the above rules could result in an upper control limit (UCL) that is less than 105%, and/or a lower control limit (LCL) that is greater than 95% or lower than 10%. In cases where the calculation would result in the UCL being less than 105%, the UCL shall be established at 105%. In cases where the calculation would result in the LCL being greater than 95%, the LCL shall be established at 95%. In cases where the calculation would result in the LCL being less than 10%, the LCL shall be established at 10%.

Analytical data that are generated along with an LCS which falls within the established control limits are judged to be in control. Data generated along with an LCS which falls outside of the control limits are considered suspect and require corrective action or the associated data must be qualified (see SOP 2410.10). Corrective action shall include examination of instrument performance, sample preparation and analysis information, and a determination as to whether re-analysis is warranted. If the analyst has any doubt about the appropriate corrective action, the supervisor should be consulted.

An LCS has been established for each routine analytical method. Reagent water is used as the control matrix for the analysis of aqueous samples. The LCS compounds are spiked into reagent water and carried through the appropriate steps of the analysis. A universal blank matrix does not exist for solid samples. Therefore, at present, water is used as the blank matrix for inorganic analyses, and sodium sulfate is generally used for organic analyses. The LCS for solid samples consists of the LCS compounds spiked into the blank matrix and carried through the appropriate steps of the analysis.

b. Method Blank (MB)

Method blanks (reagent blanks, analytical blanks, or preparation blanks) are analyzed to assess the level of contamination which exists in the analytical system and which might lead to the reporting of elevated concentration levels or false positive data.

A method blank consists of reagents specific to the method which are carried through every aspect of the procedure, including preparation, cleanup, and analysis. Ideally, the concentration of an analyte in the method blank is below the method detection limit for that analyte. If it is not at least less than the reporting limit, corrective action shall be carried out. Corrective action shall consist of an investigation into, and elimination of, the source of the contamination (if at all possible), and re-analysis or qualification of the data.

When data must be qualified, the following "blank rule" shall be applied. No positive sample results will be reported without qualification for a field sample unless the concentration of the subject analyte in the sample exceeds 10 times the concentration in any associated method blank having that analyte at or above than the reporting limit. In instances where more than one method blank is associated with a given sample, qualification shall be based upon a comparison with the associated blank having the highest concentration of a contaminant. Sample results must NOT be corrected by subtracting any blank value. See SOP 2410.10 for further guidelines on data qualification.

It should be noted that the method blank analyses may not involve the same weights, volumes, or dilution factors as the associated samples. These factors must be taken into consideration when applying the "10 times" criteria, such that a comparison of the total amount of

contamination is actually made. It should also be noted that such qualified data may not meet the needs of the project. In such cases, corrective action and re-analysis shall be initiated if possible and practical.

c. Matrix Spike/Matrix Spike Duplicate (MS/MSD)

Matrix spike and spike duplicate results are used to assess the effects of a sample matrix on the analytical data. A minimum of one matrix spike/matrix spike duplicate set will be analyzed per batch of field samples. If a batch of field samples exhibits more than one apparent "sub-type" of matrix (e.g. different color, particle size, viscosity, etc.), more than one matrix spike/matrix spike duplicate set should be analyzed, one for each "sub-type" of matrix.

An MS is an aliquot from an environmental sample to which known concentrations of the analytes of interest have been added. The MS is taken through the entire analytical procedure and the percent recovery of the analyte results are calculated. MS percent recovery data are evaluated against control limits to assess the effect of the sample matrix on the bias of the analysis.

For analyses with an extensive list of target analytes, not all analytes need be spiked. However, each MS must include a representative mix of analytes and the laboratory shall ensure that all analytes in routine analyses are spiked within a two year period. For analyses having 1 to 10 analytes, spike all analytes. For analyses having 11 to 20 analytes, spike at least 10 or 80% whichever is greater. For analyses having more than 20 analytes, spike at least 16 analytes. Where the analysis contains multi-component analytes that interfere with accurate analysis of the mixture (e.g. technical chlordane, toxaphene, or PCBs), the MS should contain those analytes that are most likely to be found in the samples and that don't interfere with one another.

An MSD is a second aliquot of the same environmental sample used for the MS to which known concentrations (identical to those used for the MS) of the same analytes of interest are added. The MSD is taken through the entire analytical procedure along with the MS. In addition to the assessment of the sample matrix on the bias of the analysis, as described above, the measured results of the MSD are compared to those of the MS to determine the precision of the analysis. Precision results are expressed as Relative Percent Difference (RPD) between the MS and the MSD results, unless otherwise specified in the RLAB Method. Such data are

evaluated against control limits to assess the effect of the sample matrix on the precision of the analysis. It should be noted that for analyses where it is difficult to spike the MS and MSD at the same concentration level, evaluation of precision becomes problematic.

Control limits for bias are based on the historical average percent recovery of the MS and MSD plus or minus three standard deviations. Control limits for precision are based on the historical average relative percent difference plus three standard deviations. (See SOP #2410.15 for details about statistical calculations and control limits.)

In some situations control limits calculated by applying the above rules could result in an upper control limit (UCL) that is less than 105%, and/or a lower control limit (LCL) that is greater than 95% or lower than 10%, or a precision control limit (PCL) that is less than 5%. In cases where the calculation would result in the UCL being less than 105%, the UCL shall be established at 105%. In cases where the calculation would result in the LCL being greater than 95%, the LCL shall be established at 95%. In cases where the calculation would result in the LCL being less than 10%, the LCL shall be established at 10%. In cases where the calculation would result in the PCL being less than 5%, the PCL shall be established at 5%.

6. Additional QC Checks

a. Laboratory Fortified Blanks (LFB)

An LFB is similar to an LCS except that the spiking material is typically the same material as used in the calibration standard. Some methods specify the analysis of LFB samples in addition to, or instead of, an LCS. In general, these samples are treated in a fashion nearly identical to an LCS. (See SOPs 2410.10 and 2410.15.)

b. Laboratory Duplicates (LD)

Some analyses are not amenable to matrix spike analysis. For these analyses, precision is typically determined by the analysis of lab duplicates. Some methods specify the analysis of lab duplicates. In general, lab duplicate precision is treated in a fashion similar to MS/MSD precision. (See SOPs 2410.10 and 2410.15.)

c. Surrogates

Surrogates are compounds (normally organic) which should have similar chemical behavior to the analytes of interest, but which are not normally found in environmental samples. Surrogates are not routinely used in inorganic analyses. Surrogates are added to environmental samples to monitor the effect of the matrix on the bias of the analysis. Surrogate data can also be used to evaluate the effects of sample preparation on individual samples. Surrogate results are expressed in terms of percent recovery.

The laboratory routinely adds surrogates to samples requiring GC/MS (VOA, BNA) or GC (pesticide) analysis. The surrogate recoveries are evaluated against control limits to assess the effects of the matrix on analyte recovery. See SOP 2410.10 for information on surrogate recovery evaluation and SOP 2410.15 for information on surrogate recovery and control limit calculations.

d. Method of Standard Additions (MSA)

MSA is the practice of adding a series of known amounts of an analyte to aliquots of an environmental sample. The fortified samples are then analyzed and the recovery of the analytes calculated. MSA is generally used with metals and some conventional analyses to determine and compensate for the effect of the sample matrix on the bias of the analyses. MSA is required when the specific method, SOP, or regulation requires it, or when matrix specific QC checks indicate the need. Details will be contained in the RLAB Method where MSA is required.

e. Field Duplicates, Blanks, and Spikes

Field duplicates, field blanks, and field spikes are field QC samples submitted to the laboratory along with a batch of environmental samples. The results of field QC samples are used by the Project Manager in the data validation process. RLAB does not assess field QC sample results against control limits or qualify environmental sample data based on the results of field QC samples, but rather, reports field QC sample results the same as the results from environmental samples.

7. Sample Preservation and Holding Times

Samples must be properly collected and preserved and they must be analyzed within specified holding times in order for acceptable analytical results to be

obtained. Specific requirements should be listed in each RLAB Method. Also, see SOP 2420.6, "Sample Container Selection, Preservation and Holding Times."

F. SPECIFIC QC REQUIREMENTS

Specific QC requirements are contained in individual RLAB Methods and/or this SOP. However, Attachments #1, #2, and #3 are tables which summarize the QC required by EPA regulations or that are specified by the analytical methods required by those regulations which might in some cases require additional QC to that required by this SOP. These tables are not comprehensive, but they do include those analyses performed routinely by the Region 7 Laboratory for which there are specific methods required by the regulations. These attachments are included for ease of reference. Analysts must assure that all program required laboratory QC is performed for each analytical procedure.

These tables are based upon 30 basic categories of QC procedures. These categories are loosely defined to accommodate variations between methods and are spelled out in Attachment #4. Attachment #5 is a list of the acronyms used in these tables and their definitions.

A given QC procedure is considered required if, under any circumstances, a referenced method listed it as required. Not all QC which is listed as required is required each time the analysis is performed. For instance, MSA is required only if matrix spike recoveries indicate the need. Further, a given QC procedure is considered optional if it is mentioned in the method as desirable, but not mentioned as required under any circumstances.

The National Primary Drinking Water Regulations (NPDWR) require all monitoring to be conducted according to specified analytical methods, by certified laboratories. The Region 7 Laboratory does not actually perform many regulatory drinking water sample analyses. However, the ability and certification to perform such analyses is being maintained as a service to deal with eventualities. All drinking water analyses shall be performed according to appropriate regulatory requirements.

The National Pollution Discharge Elimination System (NPDES) regulations require all analyses in support of NPDES permits to be conducted in accordance with specified analytical methods. RLAB Methods have been written which conform to these regulatory requirements.

The Resource Conservation and Recovery Act (RCRA) regulations require the use of specified analytical methods in certain situations. The methods published in "Methods For The Evaluation Of Solid And Hazardous Wastes," SW-846, must be used when analyzing samples:

1. to be used in the de-listing process
2. relating to a trial burn
3. of free liquids
4. for waste characteristics testing

Otherwise, any scientifically sound analytical method may be used in support of those regulations. When analyzing samples which fall into the categories listed above, SW-846 methods and QC requirements shall be followed (technically sound and defensible methods are acceptable for all other program areas).

G. REFERENCES

1. Region 7 SOP 2410.10, Analytical Data Submission Package Contents and Review

2. Region 7 SOP 2410.11, Analytical Proficiency Demonstration in the Region 7 Laboratory

3. Region 7 SOP 2410.15, Estimating and Documenting Data Quality

4. Region 7 SOP 2410.19, Significant Figures (Digits)

5. Region 7 SOP 2420.6, Sample Container Selection, Preservation and Holding Times

6. Region 7 SOP 2430.2, Review of Data Deliverables Packages from Contract Laboratories (Format, Procedures and Content)

7. Region 7 SOP 2430.3, Contract Laboratory Program Data Review Functional Guidelines for Evaluating Organic (VOA, BNA, Pesticide/PCB) Analytical Data

8. Region 7 SOP 2430.4, Contract Laboratory Program Data Validation Functional Guidelines for Evaluating Inorganic Analytical Data

9. 40 CFR Part 136 Appendix B, Definition and Procedure for the Determination of the Method Detection Limit

QC Procedures

Required by The Drinking Water Methods

(December 2003)

(Additional Categories Beginning On Page 3)

Method Name/Number (Analytes)	1 Anal. Qual.	2 Samp. Coll. Pres. Hold.	3 Init. Demo. Of Abil.	4 Formal Q.C. Prog.	5 Meth. Det. Limit	6 Q.C. Chart & Stat.	7 Init. Cal.	8 Cont. Cal.	9 Inte-rnal Std.	10 M.S. Tune	11 Meth. Std. Add.	12 Surr-ogate Spike	13 Lab Spike	14 Mat-rix Spike	15 Q.C. Samp.
#110.1/Color[A]		*				*	*	*							
#120.1/Conductivity[D]		*						*							
#140.1/Odor[A]	*	*													
#150.1/pH[A,D]		*						*							
#160.1/Total Dissolved Solids (TDS)[A]		*													
#170.1/Temperature[D]		*					*								
#180.1/Turbidity[F]		*					*	*							
#200.7A/Metals by ICP[A,B,C,D] (Al[A],Ba[C],Ca[D],Cr[C],Cu[A,D],Fe[A],Mn[A],Ni[B], Ag[A],Na[C],Zn[A])		*	*		*	o	*	*			o		*	*	*
#200.9/Antimony (Sb) by GFAA[B]		*	*		*	o	*	*			o		*	*	o
#200.9/Arsenic (As) by GFAA[E]		*	*		*	o	*	*			o		*	*	o
#200.9/Beryllium (Be) by GFAA[B]		*	*		*	o	*	*			o		*	*	o
#200.9/Cadmium (Cd) by GFAA[C]		*	*		*	o	*	*			o		*	*	o
#200.9/Lead (Pb) by GFAA[D]		*	*		*	o	*	*			o		*	*	o
#245.1/Mercury (Hg) by ACV-AA[C]		*	*		*	o	*	*					*	*	o
#200.9/Selenium (Se) by GFAA[C]		*	*		*	o	*	*			o		*	*	o
#200.9/Thallium (Tl) by GFAA[B]		*	*		*	o	*	*			o		*	*	o
#310.1/Alkalinity by Pot. Tit.[D]		*						*							
#325.3/Chloride by Color. Tit.[A]	*	*													
#335.4/Cyanide by Color.[B]		*	*		*		*	*					*	*	*

[A] - Secondary Regulation, not enforceable.
[B] - Phase V Regulation, promulgated 7-17-92
[C] - Phase II Regulation, promulgated 1-30-91 & 7-1-91
[D] - Lead/Copper Rule, promulgated 6-7-91
[E] - NIPDWR, promulgated 12-75 & 7-76
[F] - Coliform & Surface Water Treatment Rules, promulgated 6-29-89
o - Optional
* - Required

Method Name/Number (Analytes)	1 Anal. Qual.	2 Samp. Coll. Pres. Hold.	3 Init. Demo. Of Abil.	4 Forml Q.C. Prog.	5 Meth. Det. Limit	6 Q.C. Chart & Stat.	7 Init. Cal.	8 Cont. Cal.	9 Inte-rnal Std.	10 M.S. Tune	11 Meth. Std. Add.	12 Surr-ogate Spike	13 Lab Spike	14 Mat-rix Spike	15 Q.C. Samp.
SM 4500-F C Fluoride by ISE[A]							*	*							
#353.2/Nitrate[C], Nitrite[C], and Nitrate+Nitrite[C] by ACR		*	*		*		*	*					*	*	*
#365.1/Ortho-Phosphate by Auto. Color.[D]							*	*							
#370.1/ Silica (Si) by Color.[D]							*	*							
#375.4/Sulfate by Auto.Turb.[A,B]							*	*							
SM16 408C/Free Chlorine by Amp. Tit.[F]															
SM16 512A/Foaming Agents by MBAS[A]							*	*							
#524.2/Volatile Organics by GC/MS		*	*		*	*	*	*	*	*		*	*	o	*
#504/EDB & DBCP by GC/ECD[C]		*	*	*	*	o	*						*		*
#507/Nitrogen & Phosphorus Pest. by GC/NPD[B,C]	*	*	*		*	*	*	*	o			*	*	*	*
#508/Chlorinated Pest. by GC/ECD[B,C]	*	*	*			o	*	*	o			*	*	*	*
#1613/Dioxin by GC/HRMS[B]	*	*	*	*		*	*	*	*	*		*	*	o	*
#515.1/Chlorinated Acid Herb. by GC/ECD[B,C]	*	*	*			*	*	*	o			*	*	*	*
#550.1/PAHs by HPLC[B]		*	*	*		*	*	*	o				*	*	*
Drinking Water Regulations		*			*										
Drinking Water Laboratory Certification Man.	*			*			*	*					*	*	*

- The Drinking Water Regulations also require laboratory certification, specific methods, acceptable performance evaluation (PE) study performance, and maximum detection limits.
- The Drinking Water Laboratory Certification Manual also requires chain-of-custody procedures, on-site audits, equipment and supplies specifications, records, and wavelength accuracy checks.

[A] - Secondary Regulation, not enforceable.
[B] - Phase V Regulation, promulgated 7-17-92
[C] - Phase II Regulation, promulgated 1-30-91 & 7-1-91
[D] - Lead/Copper Rule, promulgated 6-7-91
[E] - NIPDWR, promulgated 12-75 & 7-76
[F] - Coliform & Surface Water Treatment Rules, promulgated 6-29-89
o - Optional
* - Required

QC Procedures

Required by The Drinking Water Methods

(December 2003)

(Continued From Previous Pages)

Method Number/Name (Analytes)	16 Reag. Blank	17 Field Blank	18 Lab Dup.	19 Field Dup.	20 Qual-itat. Conf.	21 Anal. Break Down Check	22 Ser-ial Dil.	23 Inte-rfer. Check Samp.	24 Clean Up Val-idat.	25 G.C. Col. Perf. Check	26 Inst. Perf. Check	27 Flow Rate Check	28 Prev-entiv Maint.	29 Serv. Cont-ract	30 Bal. Check
#110.1/Color[A]	*														
#120.1/Conductivity[D]													*		
#140.1/Odor[A]															
#150.1/pH[A,D]													*		
#160.1/Total Dissolved Solids (TDS)[A]															
#170.1/Temperature[D]															
#180.1/Turbidity[F]	*														
#200.7A/Metals by ICP[A,B,C,D] (Al[A],Ba[C],Ca[D],Cr[C],Cu[A,D],Fe[A],Mn[A],Ni[B], Ag[A],Na[C],Zn[A])	*						o	*			*				
#200.9/Antimony (Sb) by GFAA[B]	*		o				o	*			*			o	o
#200.9/Arsenic (As) by GFAA[E]	*		o				o	*			*			o	o
#200.9/Beryllium (Be) by GFAA[B]	*		o				o	*			*			o	o
#200.9/Cadmium (Cd) By GFAA[C]	*		o				o	*			*			o	o
#200.9/Lead (Pb) by GFAA[D]	*		o				o	*			*			o	o
#245.1/Mercury (Hg) by ACV-AA[C]	*										*	*			
#200.9/Selenium (Se) by GFAA[C]	*		o				o	*			*			o	o
#200.9/Thallium (Tl) by GFAA[B]	*		o				o	*			*			o	o
#310.1/Alkalinity by Pot. Tit.[D]															
#325.3/Chloride by Color. Tit.[A]															
#335.4/Cyanide by Color.[B]	*										*				

[A] - Secondary Regulation, not enforceable.
[B] - Phase V Regulation, promulgated 7-17-92
[C] - Phase II Regulation, promulgated 1-30-91 & 7-1-91
[D] - Lead/Copper Rule, promulgated 6-7-91
[E] - NIPDWR, promulgated 12-75 & 7-76
[F] - Coliform & Surface Water Treatment Rules, promulgated 6-29-89
o - Optional
* - Required

Method Number/Name (Analytes)	16 Reag. Blank	17 Field Blank	18 Lab Dup.	19 Field Dup.	20 Qual-itat. Conf.	21 Anal. Break Down Check	22 Ser-ial Dil.	23 Inte-rfer. Check Samp.	24 Clean Up Val-idat.	25 G.C. Col. Perf. Check	26 Inst. Perf. Check	27 Flow Rate Check	28 Prev-entiv Maint.	29 Serv. Cont-ract	30 Bal. Check
SM 4500-F C Fluoride by ISE[A]	*														
#353.2/Nitrate[C], Nitrite[C], and Nitrate+Nitrite[C] by ACR	*										*				
#365.1/Ortho-Phosphate by Auto Color.[D]	*														
#370.1/Silica (Si) by Color.[D]	*														
#375.4/Sulfate by Auto Turb.[A,B]	*														
SM16 408C/Free Chlorine by Amp. Tit.[F]													*		
SM16 512A/Foaming Agents by MBAS[A]	*														
#524.2/Volatile Organics by GC/MS	*	o			*										
#504/EDB & DBCP by GC/ECD[C]	*	o		o	o						*				
#507/Nitrogen and Phosphorus Pest. by GC/NPD[B,C]	*	o	o	o	*					*	*				
#508/Chlorinated Pest. by GC/ECD[B,C]	*	o	o	o	*	*				*	*				
#1613/Dioxin by GC/HRMS[B]	*			o	*				*	*					
#515.1/Chlorinated Acid Herb. by GC/ECD[B,C]	*	o	o	o	*					*	*				
#550.1/PAHs by HPLC[B]	*	o	o	o	o				*						
Drinking Water Regulations															
Drinking Water Laboratory Certification Manual	*												*		*

[A] - Secondary Regulation, not enforceable.
[B] - Phase V Regulation, promulgated 7-17-92
[C] - Phase II Regulation, promulgated 1-30-91 & 7-1-91
[D] - Lead/Copper Rule, promulgated 6-7-91
[E] - NIPDWR, promulgated 12-75 & 7-76
[F] - Coliform & Surface Water Treatment Rules, promulgated 6-29-89
o - Optional
* - Required

QC Procedures

Required by The NPDES Wastewater Methods

(December 2003)

(Additional Categories beginning on Page 4)

Method Number/Name (Analytes)	1 Anal. Qual.	2 Samp. Coll. Pres. Hold.	3 Init. Demo. Of Abil.	4 Forml Q.C. Prog.	5 Meth. Det. Limit	6 Q.C. Chart & Stat.	7 Init. Cal.	8 Cont. Cal.	9 Inte-rnal Std.	10 M.S. Tune	11 Meth. Std. Add.	12 Surr-ogate Spike	13 Lab Spike	14 Mat-rix Spike	15 Q.C. Samp.
INORGANICS -															
#160.1[A]/SM16 #209B - TDS		*													
#160.2[A]/SM16 #209C - TSS		*													
#160.3[A]/SM16 #209A - Total Solids		*													
#200.7[A] - ICP Metals (Ag, Al, As, Ba, Cd, Co, Cr, Cu, Fe, Mn, Mo, Ni, Pb, Sb, Se, Tl, V, Zn, Ca, Mg, Na, K)		*	*		*	o	*	*			o		*	*	o
#200.9[A&B] - Antimony (Sb) by GFAA		*	*		*	o	*	*			o		*	*	o
#200.9[A&B] - Arsenic (As) by GFAA		*	*		*	o	*	*			o		*	*	o
#200.9[A&B] - Cadmium (Cd) by GFAA		*	*		*	o	*	*			o		*	*	o
#200.9[A&B] - Chromium (Cr) by GFAA		*	*		*	o	*	*			o		*	*	o
#200.9[A&B] - Lead (Pb) by GFAA		*	*		*	o	*	*			o		*	*	o
#245.1[A] - Mercury (Hg) by ACV-AA		*	*		*	o	*	*					*	*	o
#200.9[A&B] - Selenium (Se) by GFAA		*	*		*	o	*	*			o		*	*	o
#200.9[A&B] - Silver (Ag) by GFAA		*	*		*	o	*	*			o		*	*	o
#200.9[A&B] - Thallium (Tl) by GFAA		*	*		*	o	*	*			o		*	*	o
#335.3[A] - Cyanide, Total		*					*	*					*	*	o
#350.1[A] - Ammonia		*	*		*		*						*	*	*
#351.2[A] - TKN		*					*								
#353.2[A] - Nitrate + Nitrite		*					*								

[A] - "EPA Methods for Chemical Analysis of Water and Wastes"
[B] - The general AA methods section is also referenced.
[C] - SW-846 " Methods for the Evaluation of Solid and Hazardous Waste"
[D] - This is the Methyl Thymol Blue method.
[E] - Published in 40 CFR Part 136.

o - Optional
* - Required

Method Number/Name (Analytes)	1 Anal. Qual.	2 Samp. Coll. Pres. Hold.	3 Init. Demo. Of Abil.	4 Formal Q.C. Prog.	5 Meth. Det. Limit	6 Q.C. Chart & Stat.	7 Init. Cal.	8 Cont. Cal.	9 Inte-rnal Std.	10 M.S. Tune	11 Meth. Std. Add.	12 Surr-ogate Spike	13 Lab Spike	14 Mat-rix Spike	15 Q.C. Samp.
#365.1[A] - OrthoPhosphate		*	*		*		*	o					*	*	*
#365.4[A] - Total Phosphorus		*					*								
9036[C&D] - Sulfate		*					*								
#376.2[A] & SM16 #427C - Sulfide		*					*								
#410.1[A] - COD		*					*								
EPA 1664 - Oil and Grease		*	*		*								*	*	
#420.2[A] - Phenolics (4AAP)		*					*								
SM 2510 B - Spec. Conductivity							*	*							
SM16 #209D - Volatile Solids		*													
SM16 #214A - Turbidity		*					*	*							
SM14 #307B - Hexavalant Chromium		*					*								
SM16 #314A - Hardness (by calculation)	—	None	—												
SM16 #402 - Acidity		*													
#310.1[A] - Alkalinity		*													
SM 4500-Cl B - Chloride		*					*								
SM16 #412F - Cyanide Amenable to Chlor.		*					*								
SM 4500-F C - Fluoride		*					*	*							
SM16 #421B - Dissolved Oxygen		*													
EPA 150.1 - pH		*					*								
SM16 #505B - TOC		*					*								
SM 5210 B - BOD		*					*						*		
SM16 #512B - Surfactants (MBAS)							*								

[A] - "EPA Methods for Chemical Analysis of Water and Wastes"
[B] - The general AA methods section is also referenced.
[C] - SW-846 " Methods for the Evaluation of Solid and Hazardous Waste"
[D] - This is the Methyl Thymol Blue method.
[E] - Published in 40 CFR Part 136.

o - Optional
* - Required

Method Number/Name (Analytes)	1 Anal. Qual.	2 Samp. Coll. Pres. Hold.	3 Init. Demo. Of Abil.	4 Forml Q.C. Prog.	5 Meth. Det. Limit	6 Q.C. Chart & Stat.	7 Init. Cal.	8 Cont. Cal.	9 Inte-rnal Std.	10 M.S. Tune	11 Meth. Std. Add.	12 Surr-ogate Spike	13 Lab Spike	14 Mat-rix Spike	15 Q.C. Samp.
ORGANICS -															
#608[E] - Pesticides and PCBs	*	*	*	*	*	*	*	*	o				o	*	*
#624[E] - VOCs	*	*	*	*	*	*	*	*	*	*		*		*	*
#625[E] - SemiVolatiles	*	*	*	*	*	*	*	*	*	*		*		*	*
Regulations -		*			*										

[A] - "EPA Methods for Chemical Analysis of Water and Wastes"
[B] - The general AA methods section is also referenced.
[C] - SW-846 " Methods for the Evaluation of Solid and Hazardous Waste"
[D] - This is the Methyl Thymol Blue method.
[E] - Published in 40 CFR Part 136.

o - Optional
* - Required

QC Procedures

Required by The NPDES WasteWater Methods

(December 2003)

(Continued From Previous Pages)

Method Number/Name (Analytes)	16 Reag. Blank	17 Field Blank	18 Lab Dup./ Rep. Spike	19 Field Dup.	20 Qual-itat. Conf.	21 Anal. Break Down Check	22 Ser-ial Dil.	23 Inte-rfet. Check Samp.	24 Clean Up Val-idat.	25 G.C. Col. Perf. Check	26 Inst. Perf. Check	27 Flow Rate Check	28 Prev-entiv Maint.	29 Serv. Cont-ract	30 Bal. Check
INORGANICS -															
#160.1[A]/SM16 #209B - TDS															
#160.2[A]/SM16 #209C - TSS															
#160.3[A]/SM16 #209A - Total Solids															
#200.7[A] - ICP Metals (Ag, Al, As, Ba, Cd, Co, Cr, Cu, Fe, Mn, Mo, Ni, Pb, Sb, Se, Tl, V, Zn, Ca, Mg, Na, K)	*						o	*			*				
#200.9[A&B] - Antimony (Sb) by GFAA	*						o	*			*		*		
#200.9[A&B] - Arsenic (As) by GFAA	*						o	*			*		*		
#200.9[A&B] - Cadmium (Cd) by GFAA	*						o	*			*		*		
#200.9[A&B] - Chromium (Cr) by GFAA	*						o	*			*		*		
#200.9[A&B] - Lead (Pb) by GFAA	*						o	*			*		*		
#245.1[A] - Mercury (Hg) by ACV-AA	*											*	*		
#200.9[A&B] - Selenium (Se) by GFAA	*						o	*			*		*		
#200.9[A&B] - Silver (Ag) by GFAA	*						o	*			*		*		
#200.9[A&B] - Thallium (Tl) by GFAA	*						o	*			*		*		
#335.3[A] - Cyanide, Total	*														
#350.1[A] - Ammonia	*														
#351.2[A] - TKN	*														
#353.2[A] - Nitrate + Nitrite	*														

[A] - "EPA Methods for Chemical Analysis of Water and Wastes"
[B] - The general AA methods section is also referenced.
[C] - SW-846 " Methods for the Evaluation of Solid and Hazardous Waste"
[D] - This is the Methyl Thymol Blue method.
[E] - Published in 40 CFR Part 136.

o - Optional
* - Required

Method Number/Name (Analytes)	16 Reag. Blank	17 Field Blank	18 Lab Dup./ Rep. Spike	19 Field Dup.	20 Qual-itat. Conf.	21 Anal. Break Down Check	22 Ser-ial Dil.	23 Inte-rfer. Check Samp.	24 Clean Up Val-idat.	25 G.C. Col. Perf. Check	26 Inst. Perf. Check	27 Flow Rate Check	28 Prev-entiv Maint.	29 Serv. Cont-ract	30 Bal. Check
#365.1[A] - OrthoPhosphate	*														
#365.4[A] - Total Phosphorus	*														
9036[C&D] - Sulfate	*														
#376.2[A] & SM16 #427C - Sulfide	*														
#410.1[A] - COD	*														
EPA 1664 - Oil and Grease	*														
#420.2[A] - Phenolics (4AAP)	*														
SM 2510 B - Spec. Conductivity															
SM16 #209D - Volatile Solids															
SM16 #214A - Turbidity															
SM14 #307B - Hexavalant Chromium	*														
SM16 #314A - Hardness (by calculation)	---	None	---												
SM16 #402 - Acidity															
#310.1[A] - Alkalinity															
SM 4500-Cl B - Chloride															
SM16 #412F - Cyanide Amenable to Chlor.	*														
SM 4500-F C - Fluoride															
SM16 #421B - Dissolved Oxygen															
EPA 150.1 - pH															
SM16 #505B - TOC	*														
SM 5210 B - BOD	*														
SM16 #512B - Surfactants (MBAS)	*														

[A] - "EPA Methods for Chemical Analysis of Water and Wastes"
[B] - The general AA methods section is also referenced.
[C] - SW-846 " Methods for the Evaluation of Solid and Hazardous Waste"
[D] - This is the Methyl Thymol Blue method.
[E] - Published in 40 CFR Part 136.

o - Optional
* - Required

Method Number/Name (Analytes)	16 Reag. Blank	17 Field Blank	18 Lab Dup./ Rep. Spike	19 Field Dup.	20 Qual- itat. Conf.	21 Anal. Break Down Check	22 Ser- ial Dil.	23 Inte- rfer. Check Samp.	24 Clean Up Val- idat.	25 G.C. Col. Perf. Check	26 Inst. Perf. Check	27 Flow Rate Check	28 Prev- entiv Maint.	29 Serv. Cont- ract	30 Bal. Check
ORGANICS -															
#608[E] - Pesticides and PCBs	*		o	o	o				*						
#624[E] - VOCs	*		o	o											
#625[E] - SemiVolatiles	*		o	o						*					
Regulations -															

[A] - "EPA Methods for Chemical Analysis of Water and Wastes"
[B] - The general AA methods section is also referenced.
[C] - SW-846 " Methods for the Evaluation of Solid and Hazardous Waste"
[D] - This is the Methyl Thymol Blue method.
[E] - Published in 40 CFR Part 136.

o - Optional
* - Required

QC Procedures

Required by The RCRA Solid and Hazardous Waste Methods

(December 2003)

(Additional Categories On Page 2)

Method Number/Name (Analytes)	1 Anal. Qual.	2 Samp. Coll. Pres. Hold.	3 Init. Demo. Of Abil.	4 Forml Q.C. Prog.	5 Meth. Det. Limit	6 Q.C. Chart & Stat.	7 Init. Cal.	8 Cont. Cal.	9 Inte-rnal Std.	10 M.S. Tune	11 Meth. Std. Add.	12 Surr-ogate Spike	13 Lab Spike	14 Mat-rix Spike	15 Q.C. Samp
#1020[A]/Flash Point		*					*								
#1311[A]/TCLP	*	*		*	*	*								*	*
#6010B[A]/ICP Metals (Ag, Al, As, Ba, Cd, Co, Cr, Cu, Fe, Mn, Mo, Ni, Pb, Sb, Se, Ti[B], Tl, V, Zn, Ca, Mg, Na, K)	*	*		*	*	*	*	*			*		*	*	*
#7041[A&C]/Antimony (Sb) by GFAA	*	*		*	*	*	*	*			*		*	*	*
#7060[A&C]/Arsenic (As) by GFAA	*	*		*	*	*	*	*			*		*	*	*
#7131[A&C]/Cadmium (Cd) by GFAA	*	*		*	*	*	*	*			*		*	*	*
#7191[A&C]/Chromium (Cr) by GFAA	*	*		*	*	*	*	*			*		*	*	*
#7196[A]/Hexavalant Chromium (Cr^{+6})		*					*	*			*		*	*	
#7421[A&C]/Lead (Pb) by GFAA	*	*		*	*	*	*	*			*		*	*	*
#7471[A]/Mercury (Hg) by ACV-AA	*	*		*	*	*	*	*			*		*	*	*
#7740[A&C]/Selenium (Se) by GFAA	*	*		*	*	*	*	*			*		*	*	*
#7761[A&C]/Silver (Ag) by GFAA	*	*		*	*	*	*	*			*		*	*	*
#7841[A&C]/Thallium (Tl) by GFAA	*	*		*	*	*	*	*			*		*	*	*
#8081A[A&D]/Pesticides and #8082[A&D]/PCBs	*	*	*	*	*	*	*	*	o	*		*	*	*	*
#8151[A]/Herbicides	*	*		*		*	*	*	o		o	*		*	*
#8260[A]/Volatile Organics	*	*		*		*	*	*	*	*	o	*		*	*
#8270[A]/Semi-Volatile Organics	*	*		*		*	*	*	*	*	o	*		*	*
#8290[A]/Dioxin	*	*		*			*	*	*	*			*	o	o
#9010B[A]/Cyanide	*	*		*	*	*	*	*			*		*	*	*
Regulations -	----	None	----												

[A] - SW-846, "Methods for the Evaluation of Solid and Hazardous Waste"
[B] - This analyte is not listed in the SW-846 ICP method.
[C] - The general AA methods section, #7000, is also referenced.
[D] - Refers to other SW-846 Methods
[E] - Applies to 8081A only
o - Optional
* - Required

Attachment #3

QC Procedures

Required by The RCRA Solid and Hazardous Waste Methods

(December 2003)

(Continued From Previous Page)

Method Number/Name (Analytes)	16 Reag. Blank	17 Field Blank	18 Lab Dup./ Rep. Spike	19 Field Dup.	20 Qual-itat. Conf.	21 Anal. Break Down Check	22 Ser-ial Dil.	23 Inte-rfer. Check Samp.	24 Clean Up Val-idat.	25 G.C. Col. Perf. Check	26 Inst. Perf. Check	27 Flow Rate Check	28 Prev-entive Maint.	29 Serv. Cont-ract	30 Bal. Check
#1020[A]/Flash Point			*										*		
#1311[A]/TCLP	*		*										*		*
#1610B[A]/ICP Metals (Ag, Al, As, Ba, Cd, Co, Cr, Cu, Fe, Mn, Mo, Ni, Pb, Sb, Se, Ti[B], Tl, V, Zn, Ca, Mg, Na, K)	*		*				*	*					*		*
#7041[A&C]/Antimony (Sb) by GFAA	*		*				*	*					*		*
#7060[A&C]/Arsenic (As) by GFAA	*		*				*	*					*		*
#7131[A&C]/Cadmium (Cd) by GFAA	*		*				*	*					*		*
#7191[A&C]/Chromium (Cr) by GFAA	*		*				*	*					*		*
#7196[A]/Hexavalant Chromium (Cr^{+6})	*		*												
#7421[A&C]/Lead (Pb) by GFAA	*		*				*	*					*		*
#7471[A]/Mercury (Hg) by ACV-AA	*		*				*					*	*		*
#7740[A&C]/Selenium (Se) by GFAA	*		*				*	*					*		*
#7761[A&C]/Silver (Ag) by GFAA	*		*				*	*					*		*
#7841[A&C]/Thallium (Tl) by GFAA	*		*				*	*					*		*
#8081A[A&D]/Pesticides and #8082[A&D]/PCBs	*		*		o	* [E]			*				*		
#8151[A]/Herbicides	*	o	*		o				*						
#8260[A]/Volatile Organics	*	*	*		*						*				
#8270[A]/Semi-Volatile Organics	*	o	*		*	o				o	*				
#8290[A]/Dioxin		o	o						o	*					
#9010B[A]/Cyanide	*	*	*	*									*		*
Regulations -	----	None	----												

[A] - SW-846, "Methods for the Evaluation of Solid and Hazardous Waste"
[B] - This analyte is not listed in the SW-846 ICP method.
[C] - The general AA methods section, #7000, is also referenced.
[D] - Refers to other SW-846 Methods
[E] - Applies to 8081A only
o - Optional
* - Required

Attachment #3

Category Codes

The following categories represent requirements mandated in methods:

1 - Analyst Qualifications
2 - Sample Collection, Preservation and Holding Procedures
3 - Procedures for Initial Demonstration of Ability with the Method
4 - Formal Quality Assurance/Quality Control (QA/QC) Program
5 - Method Detection Limit (MDL) Determination
6 - Quality Control (QC) Charts and Statistics
7 - Initial Calibration Procedures
8 - Continuing Calibration Procedures
9 - Internal Standard Procedures
10 - Mass Spectrometer (MS) Tuning Procedures
11 - Method of Standard Additions (MSA)
12 - Surrogate Spikes (samples fortified with surrogate compounds)
13 - Laboratory Spikes (fortified blanks) (LFB or LCS)
14 - Matrix Spikes (fortified sample) (MS/MSD)
15 - Quality Control Samples (non-blind sample from an external source) (LCS or PE)
16 - Reagent Blanks (MB)
17 - Field Blank (blank sample taken to the field and shipped with the samples) (FB)
18 - Laboratory Duplicate (re-analysis of a sample split in the laboratory) (LD)
19 - Field Duplicate (two collocated samples or one sample split into two in the field) (FD)
20 - Qualitative Confirmation (re-analysis by another technique to confirm the analyte identity)
21 - Analyte Break-Down Check (to identify if any analytes are becoming other compounds during analysis)
22 - Serial Dilution (to identify and/or eliminate matrix interferences)
23 - Interference Check Samples (to identify ICP inter-element interferences)
24 - Clean-up Validation (to verify recovery of analytes from sample clean-up procedures)
25 - Gas Chromatographic Column Performance Check (to verify acceptable chromatography)
26 - Instrument Performance Check (to verify acceptable instrument performance during a given analytical run)
27 - Flow Rate Check
28 - Preventive Maintenance Procedures
29 - Service Contracts
30 - Balance Check (to verify the proper working of the analytical balance)

Acronyms

AA - Atomic Absorption
ACR - Automated Cadmium Reduction
ACV - Automated Cold Vapor
Amp. Tit. - Amperometric Titration
Auto. Color. - Automated Colorimetric
Auto. Turb. - Automated Turbidimetric

BOD - Biochemical Oxygen Demand

CFR - Code of Federal Regulations
Chlor. - Chlorination
COD - Chemical Oxygen Demand
Color. - Colorimetric
Color. Tit. - Colorimetric Titration
(cont.) - Continued from previous pages

DBCP - DiBromoChloroPropane

ECD - Electron Capture Detector
EDB - Ethylene DiBromide
ELCD - Electrolytic Conductivity Detector

GC - Gas Chromatography
GFAA - Graphite Furnace Atomic Absorption

Herb. - Herbicide
HRMS - High Resolution Mass Spectrometer (used as a GC detector)
HPLC - High Performance Liquid Chromatography

ICP - Inductively Coupled Plasma
ISE - Ion Selective Electrode

MBAS - Methylene Blue Active Substances
MDL - Method Detection Limit
MSA - Method of Standard Additions

NPD - Nitrogen Phosphorus Detector
NPDES - National Pollution Discharge Elimination System

PCBs - PolyChlorinated Biphenyls
Pest. - Pesticide
PID - Photo Ionization Detector
Pot. Tit. - Potentiometric Titration

QA - Quality Assurance
QC - Quality Control

RCRA - Resource Conservation Recovery Act

SM14 - Standard Methods for the Analysis of Water and Waste, 14th ed.
SM16 - Standard Methods for the Analysis of Water and Waste, 16th ed.
Spec. - Specific

TCLP - Toxicity Characteristic Leachate Procedure
TKN - Total Kjeldahl Nitrogen
TOC - Total Organic Carbon
Trmt. - Treatment

VOCs - Volatile Organic Compounds

4AAP - 4-Amino-AntiPyrine

STANDARD OPERATING PROCEDURE

No. 2440.5E

U.S. EPA REGION 7 LABORATORY

QUALITY ASSURANCE OPERATING PLAN

December 16, 2004

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ENSV/RLAB/CATS

APPROVED:

Peer Reviewer — Date: December 29, 2004

Laboratory Manager — Date: 17 December 2004

Independent QA Reviewer — Date: 12/29/04

Recertified

Reviewer					
Date					

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1.0 INTRODUCTION

The purpose of this Region 7 Laboratory (RLAB) Quality Assurance Operating Plan (QAOP) is to bring together in one umbrella document Quality Assurance (QA) and Quality Control (QC) requirements which affect the laboratory and which are found in numerous individual sources (Policies, Directives, Guidance Documents, SOPs, Practices), and are to be implemented within RLAB. The QAOP describes how RLAB is organized, how it functions, and how the QA/QC program is to be implemented within the organization. In many cases, this QAOP merely references the individual document(s) which govern in a given situation. In other cases, the QAOP itself describes the QA/QC requirements which are applicable. In most cases, the detailed QA/QC requirements found in the individual program documents take precedence and are the minimum standards which must be met. In those cases where it might not be clear which requirement should take precedence, the question should be brought to the attention of the RLAB Independent QA Reviewer who will provide guidance in consultation with other members of RLAB management, as necessary.

Several key Region 7 Standard Operating Procedures (SOPs) and Standard Laboratory Operating Methods (SLOMs) have been collected and are provided to staff members as a manual, entitled, *EPA Region 7 Laboratory Key Operational and Quality Control SOPs*. Staff members are required to read the manual and provide signed certification indicating that they have read and understand their responsibilities regarding ethics, quality workmanship, and performance of work in accordance with established methods and SOPs.

2.0 LABORATORY ACTIVITY DESCRIPTION

2.1 GENERAL

RLAB is part of the Region 7 Environmental Services Division (ENSV). RLAB provides technical and analytical support to other programs within ENSV and to the other Regional program divisions [Air, RCRA, Toxics Division (ARTD), Superfund Division (SUPR), Water, Wetlands, Pesticides Division (WWPD)]. Services provided by RLAB are primarily analytical in nature. Customers submit Analytical Service Requests (ASRs) to RLAB requesting analytical support. RLAB then manages the acquisition of these services for the customers. Analyses are performed in-house by either EPA or contract chemists, or acquired from outside contract laboratories through the contracting efforts of RLAB personnel.

Providing the above-described analytical services requires numerous support activities (such as data and sample management), a range of internal and external QA support (including preparation and review of QA/QC documents and SOPs, audit sample preparation, and contract laboratory data assessment), and other types of internal support including acquisition of equipment and supplies, training, safety, planning, supervision, and management.

In order to provide products which are consistently timely, of high quality, and produced at a reasonable cost, RLAB must acquire and maintain qualified technical and scientific personnel and equipment in accordance with the requirements of the analytical methods.

2.2 ACTIVITIES

RLAB technical and administrative operations must be conducted according to RLAB SOPs and specific directives. A complete listing of current RLAB SOPs may be obtained from the RLAB Program Office Manager, from the Data Integration and Support Operations (DISO) Program Office Manager or from the R7@Work intranet site. Operational protocols continually evolve as experience is gained requiring the modification of existing SOPs or the development of new ones. The generation and revision of procedural documents (SOPs, SLOMs, and RLAB Methods) by RLAB shall be performed according to SOP 1330.4, "Preparation of Standard Operating Procedures." RLAB technical and administrative personnel will be responsible for generation, review, approval and maintenance of analytical methods (RLAB Methods) in the Analytical Methods Manual (AMM) in a manner which will comply with SLOM 3000.2, "Maintenance of Region 7 Laboratory Analytical Methods Manual." All project plans and procedures must comply with the Quality Management Plan for Region 7.

Copies of all SOPs, SLOMs, and RLAB Methods) and required guidance documents to be used in performance of its duties shall be acquired and maintained by RLAB. The RLAB Independent QA Reviewer will be responsible for maintaining these documents on file and RLAB management will be responsible for managing RLAB work activities in compliance with the specified operating procedures.

Where practical, all routine facets of RLAB activities shall reference and follow developed SOPs, SLOMs, and RLAB Methods. Deviations from applicable guidance shall be documented in written reports associated with the activities in which the deviations occur.

Analytical support shall at a minimum meet all QA/QC criteria described in SOPs, SLOMs, and RLAB Methods for the performance of analytical activities. Established protocols and recommendations of the manufacturer will be followed for the regular maintenance of all analytical instruments so that routine sample loads will be accommodated in such a way that data of known and acceptable quality are generated in a timely manner, as described more fully below.

Contract Laboratory Program (CLP) data review, related data tracking and data reporting shall be conducted in accordance with SOPs 2430.2, "Review of Data Deliverables Packages from Contract Laboratories (Format, Procedures, and Content)," 2430.4, "Contract Laboratory Program Data Validation Functional Guidelines for Evaluating Inorganic Analytical Data," and 2410.7, "Inspection of CLP Data for Contract Compliance and Timely Payment: Acceptance, Rejection and Payment Recommendations."

Activities involving sample and data management shall be performed in accordance with all applicable SOPs (SOP series 2410 and 2420).

RLAB maintains a Laboratory Information Management System (LIMS) computer system. Data handling, management, and reporting activities generally will be performed using the LIMS system. To facilitate the electronic transfer of data, all software and hardware to be used in the performance of RLAB activities will be compatible with the LIMS.

QA/QC activities, both internal and external, shall be performed according to the applicable SOPs discussed below. All SOPs generated by RLAB shall conform to the requirements established in SOP 1330.4, "Preparation of Standard Operating Procedures."

As described in the Laboratory Chemical Hygiene Plan (CHP), the Laboratory Director is responsible for the health and safety of all employees during the performance of their duties. The Laboratory Director shall ensure employee compliance with established facility health and safety protocols.

All RLAB analytical work will be tracked through use of the internal LIMS tracking system. The tracking system will be used to report on the status of analytical project completion. The Program Managers will report weekly to the Laboratory Director on the status of the analytical work assigned to or controlled by their Programs.

RLAB will maintain a system of technical and administrative files sufficient to document all operations.

RLAB will maintain its QA/QC program in accordance with this QAOP. The purpose of this plan is to provide a coherent description of the practices and procedures utilized by RLAB to ensure that RLAB products are appropriate for meeting all project requirements and objectives. Consequently, this QA plan describes or references Regional SOPs which describe required QA/QC procedures and responsibilities to which all members of RLAB must conform.

3.0 QA/QC ORGANIZATION AND RESPONSIBILITIES

3.1 INTRODUCTION

Implementation of the RLAB QAOP requires that all RLAB staff be familiar with the goals and procedures described in the plan. The RLAB Laboratory Director, RLAB Program Managers, and RLAB Independent QA Reviewer are primarily responsible for managing the QA/QC program. However, implementation of QA/QC procedures are the responsibility of every RLAB staff member, and assigned duties have inherent in them the relevant requirements contained in this QAOP.

3.2 QA/QC RESPONSIBILITIES

RLAB staff members have a variety of duties and responsibilities. The following discussion describes RLAB staff duties and responsibilities related to the RLAB QA/QC program.

3.2.1 Laboratory Director

The Laboratory Director is responsible for establishing policies to ensure that the laboratory QA/QC program is in compliance with national EPA QA/QC directives and guidance, and with EPA Region 7 QA/QC policies and procedures and the laboratory ethics policy. The Laboratory Director is to perform the following QA/QC duties:

- Communicate the general laboratory QA policies to Program Managers and staff
- Ensure that the QA program receives sufficient priority within the laboratory
- Oversee the development and revision of the Region 7 RLAB QAOP
- Ensure that the Region 7 RLAB QAOP is implemented by all RLAB staff members
- Approve RLAB SOPs, SLOMs and RLAB methods

- Provide general advice to the Independent QA Reviewer concerning QA issues and concerns
- As required, participate in external systems audits of RLAB operations
- Enforce QA/QC requirements within the laboratory

3.2.2 Independent QA Reviewer

The Independent QA Reviewer is responsible for monitoring compliance of RLAB with national EPA QA/QC directives and guidance, and with EPA Region 7 RLAB QA/QC policy and procedures and the laboratory ethics policy. Staff assistance for the performance of QA/QC duties is provided on an as-needed basis from the RLAB staff Programs. The Independent QA Reviewer is to perform the following QA/QC duties:

- Maintain currency on all applicable Agency QA/QC requirements
- Develop and update the Region 7 RLAB QAOP and other QA/QC guidance documents
- Communicate QA/QC requirements to RLAB Program Managers and staff
- Approve RLAB Methods for inclusion in the RLAB Analytical Methods Manual
- Ensure that a complete set of Region 7-approved SOPs, SLOMs, and RLAB Methods is maintained and available to RLAB staff
- Regularly conduct internal systems audits of RLAB activities including periodic spot checks of analytical package products
- Help to identify the need for corrective actions and evaluate their effectiveness
- Participate in the development of corrective actions and maintain appropriate corrective action documentation
- Report to the Laboratory Director on the status of the implementation of the Region 7 RLAB QAOP
- Facilitate and participate in external performance and systems audits
- Maintain QA/QC record systems (i.e., QA/QC files, corrective action log)
- Review internal guidance documents (i.e., SOPs) and provide input to RLAB Program Managers

3.2.3 RLAB Program Managers

Program Managers are responsible for ensuring that the chemists, scientists, environmental specialists, and technicians produce products of appropriately documented and acceptable quality and that staff work is performed in compliance with the laboratory ethics policy. The Program Managers are to perform the following QA/QC duties:

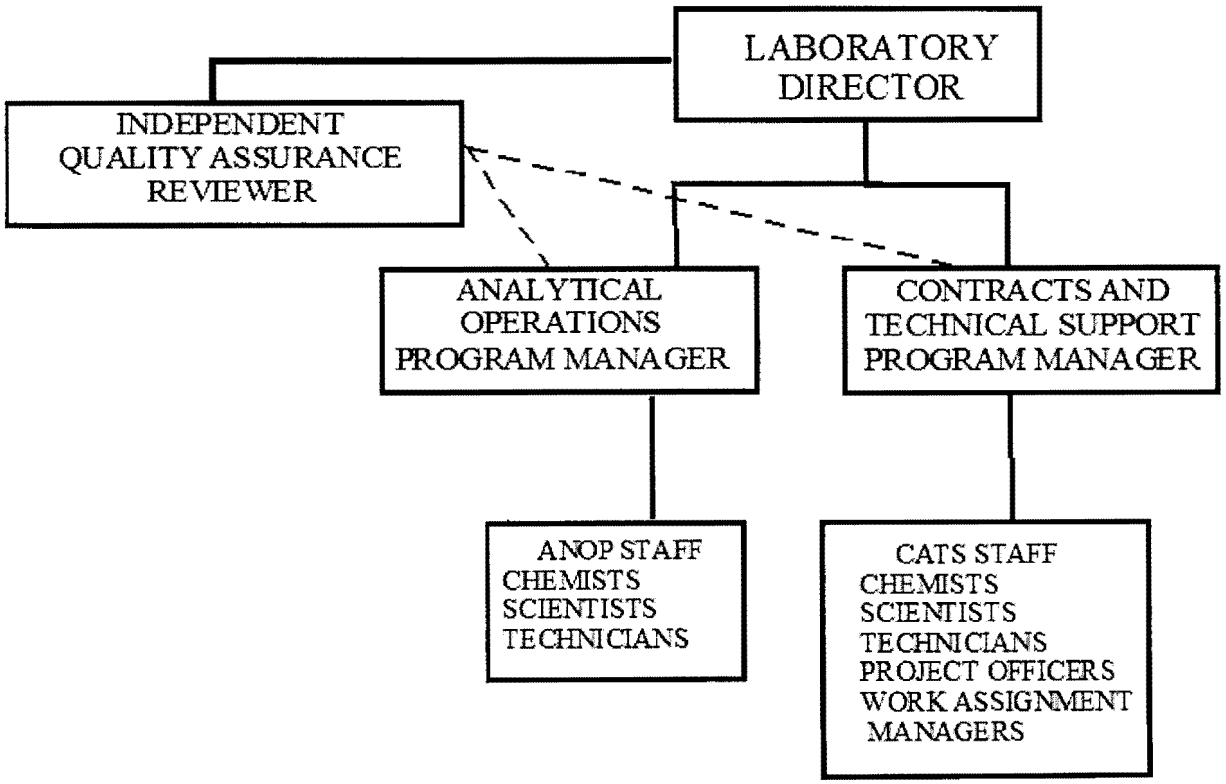
- Maintain complete SOPs, SLOMs, and RLAB Methods for all Program activities
- Develop accurate and complete RLAB SOPs, SLOMs, and RLAB Methods for routine Program activities not covered by ENSV/RLAB SOPs and assure that these documents are consistent with agency and laboratory QA/QC requirements
- The Program Managers will approve RLAB Methods for inclusion in the AMM
- Ensure that all routine work is performed according to established ENSV and RLAB SOPs, SLOMs, and RLAB Methods
- Ensure that work is performed according to any additional acceptance or performance criteria associated with a particular project
- Ensure that all staff members in the Program comply with ENSV and RLAB QC policy
- Initiate routine corrective actions, evaluate their effectiveness, and assure that appropriate documentation is maintained
- Report any quality-related issues or problems requiring formal corrective action to the RLAB Independent QA Reviewer, develop corrective action plans, provide memos documenting completed formal corrective actions to the RLAB Independent QA Reviewer
- Provide staff support on an as-needed basis to the Independent QA Reviewer for the QA/QC program

3.2.4 RLAB Chemists, Scientists, Specialists, and Technicians

The chemists, scientists, specialists, and technicians are responsible for producing products of appropriately documented and acceptable quality. They are to perform their work in compliance with the laboratory ethics policy. The chemists, scientists, specialists, and technicians are to perform the following QA/QC duties:

- Perform work according to established SOPs, SLOMs, and RLAB Methods and this QAOP
- Assist Program Managers in the development and revision of SOPs, SLOMs, and RLAB Methods as needed
- Understand and implement all applicable QA/QC policy (i.e., Region 7 RLAB QAOP, SOP 2430.12 "Regional Laboratory Quality Control Policy," and QC specified in individual RLAB Methods)
- Immediately notify Program Managers of any quality concerns or anomalies. Suggest and implement corrective actions for all quality issues that may be resolved at this level
- Maintain accurate and complete documentation related to work activities

REGION 7 LABORATORY HIERARCHY FOR QA/QC



4.0 QA OBJECTIVES FOR MEASUREMENT DATA

The QA/QC objectives for measurement data are to ensure that analytical data of known and acceptable quality are generated in the Regional and contract laboratories. Field and laboratory data will be used for site assessments and hazard determination, remedial investigations, engineering feasibility studies, community relations programs, and support of enforcement and cost recovery proceedings. Each of these uses may have specific QA/QC objectives. However, in general, the QA/QC objectives for analytical results will include:

Precision: The objective for precision is to equal or better the precision demonstrated for similar samples, and shall be within the established control limits for the methods according to Region 7 EPA criteria as defined in SOPs 2430.12, 2410.15, 2430.3, 2430.4 or the RLAB Method, as appropriate.

Bias: The objective for bias is to equal or better the bias demonstrated for these analytical methods on similar samples, and shall be within the established control limits for the methods according to Region 7 EPA criteria as defined in SOPs 2430.12, 2410.15, 2430.3, 2430.4 or RLAB Method, as appropriate (normally within three standard deviations of the mean).

Representativeness: Representativeness of a data set is a quality objective attributable to the selection of type and number of sub-samples to be taken from a sample and the analyses to be performed on the samples taken so that the data generated will adequately represent the complete sample. Concerns such as proper techniques for isolating a portion of sample to be analyzed from the quantity of samples taken from the field, and isolation of samples from subsequent contamination are critical. Representativeness is evaluated through an examination of duplicate data.

Comparability: To determine whether laboratory data are comparable, the analytical methodologies implemented at the laboratories are evaluated for uniformity and conformity. Standard analytical methodologies facilitate comparability. RLAB performs sample analysis according to methodologies specified by EPA methods and ENSV/RLAB SOPs.

Completeness: To evaluate the completeness of the data, a determination must be made of whether the required samples and analyses produce an adequate database to meet the data quality objectives. This determination is made by the project manager.

Timeliness: To evaluate the timeliness of analyses, the actual holding times of samples are evaluated against holding time requirements which may be required for technical or legal defensibility of the data.

Sensitivity: To evaluate the sensitivity of the analyses, the method detection limits obtained through detection limit studies may be compared to the method detection limits required for particular analyses. Additionally, calibration results and the results obtained for low-level check standards can be used to gauge sensitivity.

The objective of the Regional Laboratory is to provide superior support to the Regional EPA programs in sample analyses and analytical-related efforts. To achieve this objective, all data generated by RLAB must meet or exceed Region 7 QA objectives and QC requirements. Additionally, all data generated must satisfy any site-specific data quality objectives that are communicated by the project manager.

5.0 PROCEDURAL DOCUMENTS

5.1 STANDARD OPERATING PROCEDURES (SOPs)

Region 7 SOPs are to be reviewed systematically on a two-year cycle, and subsequently revised, recertified without changes or archived, as described in SOP 1330.4, "Preparation of Standard Operating Procedures." Additionally, a procedural document may be recertified with cosmetic or non-substantial changes. Cosmetic changes are those resulting from electronic reformatting, such as font changes or porting from WordPerfect to Word or Adobe PDF (note that this may entail changes to page numbers in the table of contents). Non-substantial changes are those resulting from correction of spelling, punctuation, and typographical errors. Minor restructuring or rewording of sentences for the purpose of improving clarity and/or correcting errors may, at the discretion of the author, be considered as non-substantial changes as long as the changes do not alter policy or procedures. As a general rule-of-thumb, a document may be recertified with cosmetic or non-substantial changes if the recertified and original documents are equivalent in substance and equally valid to use.

RLAB procedures for complying with Region 7 review/recertification requirements, as identified above, are as follows. On a quarterly basis, a report is generated by the RLAB Independent Quality Assurance Reviewer which identifies all the SOPs which will expire within the next three months or have been identified as in need of review. The RLAB Program Managers contact the staff member responsible for any identified SOP to ensure that the identified SOP is reviewed, and subsequently revised, recertified or archived. If being recertified, the author shall initial and date the recertification block on the cover page of the original official document.

5.2 RLAB METHODS AND STANDARD LABORATORY OPERATING METHODS (SLOMs)

RLAB Methods are used to provide standard procedures for performing laboratory analyses. Standard Laboratory Operating Methods (SLOMs) are used to provide standard procedures for performing laboratory operational routines, such as glassware cleaning or management of the RLAB Analytical Methods Manual. The term RLAB Analytical Methods Manual refers to the collection of all RLAB Methods and SLOMs which are approved for laboratory use.

When RLAB Methods and SLOMs are newly written or revised they are routed to the RLAB Laboratory Director, RLAB Independent Quality Assurance Reviewer and the Analytical Operations (ANOP) and/or the Contract and Technical Support (CATS) Program Manager for approval. Upon approval, they are distributed for use throughout the facility.

RLAB Methods and SLOMs will be reviewed every two years. Upon review of these documents, determination will be made as to whether the documents should be revised, archived, or recertified.

On a quarterly basis, a report is generated by the RLAB Independent Quality Assurance Reviewer which identifies all the RLAB Methods and SLOMs which will expire within the next three months or have been identified as in need of review. The ANOP and/or CATS Program Manager contacts staff members responsible for the identified RLAB Method or SLOM to ensure that the identified RLAB Method or SLOM is reviewed, and revised, recertified or archived. If being recertified, the author and Program Manager shall initial and date the recertification block on the cover page of the original official document.

6.0 SAMPLING ACTIVITIES

The Regional Laboratory may be involved occasionally in providing technical advice related to sampling activities, but generally samples analyzed by RLAB will be collected by others.

The specific methods and techniques to be utilized in sampling activities are contained in site-specific sampling plans and/or QA Project Plans (QAPPs). Each sampling plan or QAPP describes the procedures to be used for acquiring samples that best represent the environmental matrix of concern. SOPs (SOP series 2334 or 4230) and standard EPA publications shall be used in the development of any sampling plans or QAPPs for which RLAB is responsible or for which RLAB provides technical advice. The following

guidance should be followed in these situations.

6.1 SAMPLING PROCEDURES

The site-specific sampling plan or QAPP should include, as appropriate, locations, design, drilling, development techniques for monitoring wells, and sampling equipment required for each matrix. QA/QC requirements and documentation applicable to sampling procedures are to be discussed in the sampling plan regardless of which specific procedures have been or will be employed.

6.2 SAMPLE VOLUME

The volumes of samples taken are to be sufficient for the determination of all analytical parameters of interest. Such volumes will be determined during the planning stages of the field activities. All applicable SOPs and related documents should be referenced; additionally, analysts may be consulted if necessary. Appropriate references shall be included in the sampling plan or QAPP. Volumes should be sufficient to provide the necessary laboratory QC.

6.3 BLANKS

Depending on the site conditions and the objectives of the work assignment, field blanks, trip blanks and/or laboratory blanks may be utilized. The analysis of field blanks will be used to indicate whether contamination occurred during sampling activities. Trip blanks will be used to indicate whether sample contamination occurred during transport to the laboratory. The methods for collecting field blanks, the frequencies of inclusion of field and trip blanks, and the analyses required for field and trip blanks will be described in the site-specific field sampling plan or QAPP. Laboratory blanks will be used to evaluate the occurrence of sample contamination within the laboratory as described in SOP 2430.12.

6.4 DUPLICATES/MATRIX SPIKE DUPLICATES

Selected samples of each matrix (i.e., water, soil, sediment) will be duplicated or spiked with known amounts of analytes to provide an indication of precision and bias for the methods used. The frequency of these samples will be described in the site-specific sampling plan or QAPP, and will provide for collection of samples adequate to conform to SOP 2430.12. Duplicate or split samples may also be analyzed by another laboratory to evaluate the degree of variance between the laboratories.

6.5 SAMPLE PRESERVATION AND HOLDING TIMES

The integrity of samples must be maintained from the time of collection until analyses are performed. Therefore, the samples should be preserved prior to transport or storage to prevent or retard the degradation of the samples. Preservation techniques and sample holding times shall be in accordance with US EPA protocols for specific analytes, and shall be described in the site-specific sampling plan or QAPP. See SOPs of the 2420 series for additional information.

7.0 SAMPLE CUSTODY

The history of each sample is documented from the time of collection through all transfers of custody until received by the analytical laboratory. SOPs of the 2420 series shall be followed for sample documentation and management. Internal laboratory records will document custody of samples through final disposition.

7.1 SAMPLE CUSTODY PROCEDURES

Since samples are physical evidence and it is essential that control of evidence be maintained, sample identification and chain-of-custody procedures are to be followed carefully. Samples to be analyzed shall be handled according to SOPs of the 2420 series. Sample custody in the field and in transit shall be performed according to the procedures developed for the specific sampling program. Samples required to be analyzed by RLAB personnel in the Region 7 Laboratory shall be handled according to the guidelines described below and according to all applicable SOPs, including SOPs 2420.4 and 2420.5.

7.2 LABORATORY CUSTODY PROCEDURES

The Regional Sample Control Coordinator (RSCC) is the designated sample custodian to accept custody of the shipped samples and verify that the information on the sample labels matches the information recorded on the chain-of-custody forms. Pertinent information such as shipment, pickup, courier, etc., shall be recorded on the chain-of-custody forms. The RSCC will enter the sample label data into the sample tracking system of the laboratory. This system will use the sample label number or assign a unique laboratory number to each sample label. The RSCC will ensure that all samples are transferred to the proper analyst or are stored in the secured sample storage area. The RSCC shall follow SOPs in the 2420 series related to laboratory sample management.

Stricter policies may be enacted as necessary to respond to situations which

demand greater security. For example, the Laboratory Director, Program Managers, or the Independent QA Reviewer may designate a sample custodian who is responsible for controlling access to samples of extraordinary legal or proprietary sensitivity. Samples of this nature are usually kept in a locked refrigerator inside the secured sample storage area where routine samples are stored. Generally, only the designated sample custodian and a backup are provided with keys to the locked refrigerator.

When sample analyses and necessary QA/QC checks have been completed in the laboratory, the unused portion of the sample and the sample container must be prepared for proper disposal as described in Section 7.3 below. The RLAB Sample Disposal Coordinator will take custody of the samples at this stage and make the necessary arrangements for proper disposal. All data sheets, chain-of-custody and laboratory records shall be retained as part of the permanent documentation. Samples received by the laboratory will be retained until analyses and QA/QC checks are completed in accordance with the practices of the Region 7 Laboratory.

When analyses are being performed in support of criminal investigations, SOP 2420.10 entitled "Laboratory Custody and Tracking of Samples and Analytical Data Files to be used as Evidence in Criminal Investigations," must be followed.

7.3 SAMPLE DISPOSAL

Sample disposal activities shall be performed in accordance with SOP 2420.9 "Sample Disposal," policies, and technical direction, including the RLAB Chemical Hygiene Plan (CHP) and applicable federal, state, and local regulations. Sample disposal records are maintained primarily by the RLAB Sample Disposal Coordinator.

7.4 HAZARDOUS WASTE TRANSFER

Hazardous laboratory waste transfer activities shall be performed in accordance with applicable SOP 2440.6, "Transfer and Storage of Laboratory-Generated Wastes," the RLAB CHP and applicable federal, state, and local regulations. Records are maintained primarily by the Region 7 Laboratory Environmental Compliance Officer.

7.5 SAMPLE SHIPPING

Sample shipping duties shall be performed according to applicable SOPs including SOP 2420.7 "RLAB Procedures for Sample Shipping to Contract Laboratories," and Department of Transportation (DOT) regulations. Tasks

related to shipping shall be coordinated by the RSCC to ensure maintenance of sample integrity (proper preservation, avoidance of breakage, etc.) and custody throughout the shipping process. The RSCC, or designee, is to be notified immediately if missing, damaged, broken, or mislabeled samples are encountered.

8.0 CALIBRATION PROCEDURES

8.1 SCOPE

This section describes the requirements for control, calibration, adjustment, and maintenance of analytical measuring and testing devices that may be used by RLAB personnel for performing tests. Calibration activities shall be performed according to all applicable SOPs, methodologies, and guidance. Devices shall be calibrated and adjusted at specified, predetermined intervals using equipment and material having known, valid, relationships to National Bureau of Standards (NBS) or other recognized standards. Documentation of calibration activities will be maintained in log books kept at the instrument or in data files centrally located as appropriate per analytical group.

8.2 GENERAL OPERATIONS

RLAB personnel are responsible for insuring that the following guidelines are implemented for all equipment:

- The measuring and testing devices used will be of the proper range, type, and accuracy for the test being performed.
- Prior to use, all measuring and testing devices shall be calibrated in accordance with Region 7 policies and SOPs. Documentation of calibrations will be maintained according to applicable policies and SOPs.
- Appropriate methods will be employed to assure proper handling, storage, and care of the test equipment in order to maintain its required accuracy.

Routine calibration standards will be used by the analytical laboratory to demonstrate that the performance of an instrument does not cause unnecessary error in the analysis. This calibration will indicate instrument stability and sensitivity. The methods for verification and documentation of instrument conditions prior to and during testing shall be detailed in specific laboratory SOPs/SLOMs/RLAB Methods.

8.3 LABORATORY PRACTICES

The calibration procedures and required frequency of calibration are described in various SLOMS and RLAB Methods which reflect the individual method requirements. These procedures are to be followed closely.

Test equipment found to be out of calibration shall be recalibrated according to the requirements of this section or the applicable SLOM or RLAB Method. When test equipment is found to be out of calibration or damaged, an evaluation shall be made to determine the validity of test results generated since the last calibration check. When it is necessary to assure the acceptability of suspect test results, the originally required tests shall be repeated using properly calibrated equipment. Test equipment consistently found to be out of calibration shall be reported to the appropriate Program Manager.

Inspection and test reports shall include identification of the test equipment used to perform the inspection and/or tests.

8.4 LINEARITY

Test equipment will be calibrated according to method specifications, using all applicable procedures and equations as provided in each individual method.

In the event that calibration procedures are not defined adequately by the method, linear calibration through linear regression will be used. In the event that linear regression equations are not provided by the method, the equations provided in the following section may be used; otherwise, the analyst will document the equations used. In the event that linearity acceptance criteria are not defined by the method, a correlation coefficient of 0.995 or better will be considered to be an indicator of acceptable linearity.

8.5 LINEAR REGRESSION

Linear regression uses the method of least squares to determine the best linear equation describing a given set of data points. Calibration curves are often constructed using linear regression. Concentrations of sample analyte may be determined by comparing sample analyte measurements to the calibration curve.

Sub-quantities to be used in regression equations:

$$S_{xx} = \sum \left(x_i - \bar{x} \right)^2$$

$$S_{yy} = \sum (y_i - \bar{y})^2$$

$$S_{xy} = \sum (x_i - \bar{x})(y_i - \bar{y})$$

Regression equations:

Slope:

$$m = S_{xy} / S_{xx}$$

Intercept:

$$b = \bar{y} - m\bar{x}$$

Correlation Coefficient:

$$r = \frac{S_{xy}}{\sqrt{(S_{xx}) \cdot (S_{yy})}}$$

8.6 TRACEABILITY OF STANDARD MATERIALS

Detailed records are to be maintained for reagent and standard preparation. These records shall indicate traceability to purchased stocks or neat compounds, reference to the method of preparation, date of preparation, expiration date and preparer's initials. Lot numbers of standards and reagents are to be documented by the laboratory personnel.

Reagent preparation can be recorded in several ways depending on the type of reagent and its length of use. Information documenting acids, bases, organic solvents, and other similar types of materials which are used unprepared must be recorded on the sample preparation sheets. Large quantities of these transient materials are used in sample preparation. Traceability will be easier if the records are included with the sample preparation information. Reagents which are prepared and used for longer periods of time, such as those for automated analysis, COD, BOD, mercury, and others, must be documented in laboratory log books for reagent preparation.

In summary, records must be kept for all standards and reagents, and those records must include the following information:

1) traceability to purchased stocks or neat compounds,

2) reference to the method of preparation,
3) date of preparation,
4) expiration date, and
5) preparer's initials.

9.0 ANALYTICAL PROCEDURES

9.1 BACKGROUND

Analyses shall be performed in substantial conformance with approved SOPs and the RLAB Analytical Methods Manual. SOPs and the Methods Manual will implement the methods required by the regulations for the programs being supported. The SOPs and Methods Manual generally contain detailed sections describing procedures to be followed.

In certain situations, it might not be practical to develop an SOP or Method Manual procedure for each non-routine analytical project. In these situations, the Program Manager will specify the method to be followed. In these non-routine situations, the method used will be carefully documented in the analytical back-up file associated with the analytical activity.

In other situations, when adequate methods are not available, the Program Manager may task RLAB staff (or contractors) to develop an appropriate analytical method. The methods specified or developed for each project must be approved by the Program Manager prior to implementation. All methods developed by RLAB, whether for routine or non-routine use, shall describe in detail the exact procedures and materials required to analyze the samples. The following items are generally included in the documentation of analytical procedures:

- Medium of application (i.e., water, soil, air)
- Principle of method
- Sample size requirements
- Detection limits
- Interferences and corrective measures
- Apparatus and reagents (including instrumental parameters)
- Calibration procedure
- Sample preparation (i.e., extraction, digestion)
- Diagrams or tables that describe the method
- Step-by-step analytical procedure
- Details of calculation

- QC requirements (i.e., blanks, spikes, duplicates)
- Report requirements
- Safety
- Environmental Compliance/Pollution Prevention
- References

Data will be included, if appropriate, to support the limitations and the applicability of the method.

RLAB staff shall use specified SLOMs and RLAB Methods as written except when otherwise approved by the Program Manager. All changes and modifications to standard procedures will be documented in the file associated with the analytical activity.

9.2 SPECIFIC ANALYTICAL CHEMICAL PROCEDURES

As much as is practical, the RLAB Program Managers and Independent QA Reviewer shall ensure that routine detailed analytical and QC procedures are described in SOPs or the Methods Manual. Laboratory procedures will be performed according to the SOPs and the Methods Manual. As necessary, the RLAB Program Managers and/or the Independent QA Reviewer may direct RLAB or task contractor staff, respectively, to develop SOPs or methods for procedures not described by current SOPs. The RLAB Methods Manual lists analytical methods approved and documented for use in the Region 7 Laboratory. Analytical procedures which are followed, but which are not included in the Methods Manual, shall be carefully and specifically documented in the file associated with the analytical activity.

10.0 DATA REDUCTION, VERIFICATION, AND REPORTING

10.1 DATA REDUCTION

Data reduction includes those activities involving conversion of raw data to reportable units, transfer of data between recording media, and computation of summary statistics, standard errors, confidence intervals, tests of hypotheses relative to the parameters, and model validation. Statistically acceptable data analysis procedures shall be implemented for all data reduction steps. RLAB's automated data reduction activities will substantially conform to the recommendations contained in EPA OIRM Document 2185, August 1995, "Good Automated Laboratory Practices" (GALP).

Data are initially collected, converted to standard reporting units, and recorded in standard formats by the analysts. The analysts conduct preliminary data analyses using a variety of methods and procedures. Because many analytical instruments are microprocessor-controlled, some of the analyses can be performed directly in the operating or output mode of the instruments. Those instruments interfaced to stand-alone computers or microprocessors often permit data analysis programs to be written and modified. These programs can produce data formats specifically suited to end user requirements. In all cases, whether the data reduction procedures are performed by the analysts or by computer, the data reduction procedures must be thoroughly documented.

Data requiring manual recording or integration must be reduced to a format appropriate for submission. During all stages and aspects of data processing, the data are to be double checked for translation or transcription errors. The appropriate analytical Program Manager, or other qualified individual not involved in the analysis, will perform a review of the data for acceptability prior to submission of the data.

Data packages submitted by RLAB analysts shall substantially conform to SOP 2410.10 "Analytical Data Submission Package Contents and Review."

The background data produced for internal records and not reported as part of the analytical data could include: laboratory worksheets, laboratory notebooks, sample tracking system forms, instrument logs, standards records, maintenance records, calibration records, and associated QC. These records shall be kept in official files and shall be available for inspection during audits and during data verification steps.

Documentation of data transfer should indicate each transfer step used in processing data. During QA/QC audits and routine data review, this documentation will be used to trace a data set from stored raw data to the final data package. The independent data review of each data package will also include random checks for transfer accuracy and completeness. Independent reviews are performed by Program Managers or designated peers. This aspect of QC will be emphasized in the training of RLAB staff for performing peer reviews.

Data reduction frequently includes computation of analytical results and summary statistics from raw instrument data. Computation may be made of standard errors, confidence intervals, tests of hypotheses relative to the parameters, and model validation. The equations and typical calculation sequence which should be followed to reduce the data to the acceptable format is normally established by the analytical method. When standard methods are modified with the approval of the Program Manager, data reduction techniques will be documented in the primary

data back-up file.

10.2 DATA VERIFICATION AND SPOT CHECKS

Data review and verification is the process by which data are determined to be of acceptable or unacceptable quality based on a set of predefined criteria. These criteria depend upon the type(s) of data involved and the purpose for which data are collected. The data review and verification process is described in SOPs 2410.10, 2430.12, and 2430.2.

In addition to the data review described in the above SOPs, it is the RLAB policy to periodically perform internal quantitative and qualitative evaluations (QC Spot Checks) of analytical data packages. The process for these spot checks is described in SOP 2430.5.

Review of CLP and RECAP data packages shall be performed according to SOPs, Delivery Orders, Statements of Work, and any other applicable guidance. The data review SOPs for CLP are based on the National Functional Guidelines for Data Review. When functional guidelines or an SOP are not available for a particular type of data, the appropriate analytical method shall be used as part of the review.

10.3 DATA REPORTING

Submitted data must conform to ENSV format directives. Data shall be submitted in the form of LIMS reports, indicating that the data are present in the RLAB data system after analysis, review, and approval. The Contractor and Technical Support Section is responsible for ensuring that all data supplied to LIMS conform to RLAB format directives.

10.4 MANUAL INTEGRATION OF DATA

While software generally handles peak quantitation correctly in the great majority of cases, from time to time an analyst may need to adjust the area of a peak to correct for incorrect baseline placement by the software. For example in GC, in cases where peaks are inadequately resolved or a peak shoulder is present, manual adjustment of the data may be justified. While analytical SOPs may address some of the technical items relating to this topic, there are general policy and procedure issues regarding manual integration of data, which are addressed below and in SLOM 3000.6.

10.4.1 Improper Manual Integration

Since improper manual integration may not only raise issues regarding the quality of the resulting data but can also result in potential charges of data fraud, it is imperative that employees fully understand when manual integration of data is improper, and avoid performing manual integrations in such cases or in such a manner. A discussion of this subject will be included in the training/orientation of all analysts before they are certified to generate data independently on an instrument.

Adding area to a QC compound peak ("mountain ranging"), or removing area from under a peak ("peak shaving") for the sole reason of enabling the result to meet control limits is unethical and will be dealt with appropriately. However, it is recognized that errors in the data handling software can affect a QC peak as easily as any other peak. In such cases it is imperative to avoid the appearance of improper data manipulation. Such cases should be discussed with the Program Manager and documented in the project file.

10.4.2 Documentation of Manual Integration

Consistent with generally recognized good laboratory practice, manually integrated data will be documented in the following manner. 1). A "before" printout ("snapshot") of the data prior to manual integration will be printed for the project file, and labeled as such. (This inclusion allows the original result to be used, if at a later time it is decided that manual integration should not have been employed, or was improperly performed.) 2). An "after" printout of the data will likewise be included in the project file, labeled as such. 3). The "after" printout will include the initials of the analyst and the Program Manager, the date, and a brief statement of the reason the manual integration was performed. "Reason Codes" may be used on the raw data, with a key included in the data package. A discussion (as appropriate) should also be included in the documentation for the activity.

10.4.3 Role of the Independent QA Reviewer

The Independent QA Reviewer will ensure manually integrated data are examined as part of routine data audits, to ensure compliance with this section. An SOP providing additional guidance and technical details (e.g., examples of chromatograms) on this subject may be developed as appropriate. The Independent QA Reviewer may require sign-off sheets for analysts to certify that they have been appropriately trained in this area.

11.0 DOCUMENTATION

11.1 GENERAL

It shall be the responsibility of each RLAB staff member to maintain records in accordance with the requirements of this section.

The RLAB Document Control Program assures that all project documents issued or generated by the RLAB staff shall be accountable upon completion of each activity. Document control is required for work related to all RLAB tasks. All documents used or generated by RLAB staff in the execution of an analytical activity are accountable upon completion of that activity. The following documents are included; other documents should also be included as appropriate.

- Chain-of-custody record
- SOPs
- Raw data sheets, graphs, chart recordings, etc.
- External correspondence
- Report notes, calculations
- Data review checklists, work sheets
- References
- Sample inventory, check-out logs
- Final reports

SOP 2410.2, "Analytical Data Files," describes how RLAB data files are to be maintained. SOP 2410.10, "Analytical Data Submission Package Contents and Review," describes the contents of the packages necessary for documenting the submitted data.

11.2 LABORATORY NOTEBOOKS

Data forms or check sheets are preferable to laboratory notebooks for most of the tasks performed by RLAB staff. However, for some projects or laboratory activities laboratory notebooks may be desirable or required. When notebooks are used, the following procedures should be followed.

Notebooks may be assigned to either individuals or to laboratory operational areas, depending on the application and need. Laboratory notebooks which are assigned to a given operational area or activity rather than to an individual may be kept in the assigned area, however, the location where they are kept must be

readily accessible and known to all personnel who use the notebook. Additionally, the Program Manager will keep a log of all active notebooks to include their title and location. Individual analyst notebooks may be kept in the possession of the analyst using the notebook, but must be turned in to the Program Manager, Independent QA Reviewer, or designee upon request, transfer, or termination of employment.

The notebooks should be bound and the pages should be numbered consecutively. If the page numbers are not preprinted, the numbers should be written with permanent ink. Pages should be designated in the front of the notebook for a Table of Contents. Entries in the Table of Contents should be made as work progresses.

A title that describes the type of information to be recorded in the notebook should be clearly written on the front page. All personnel who make an entry into the notebook should write their full name followed by written initials on the front page below the title.

Data should be entered directly into the notebook and not transcribed from notes. Information should be recorded in sufficient detail that a knowledgeable co-worker could continue the work by reading the notebook. Dates, RLAB Methods, critical steps, and deviations from the methods should always be recorded.

All entries should be made in permanent ink to ensure that the entries remain legible and are not easily altered. Pencils or nonpermanent felt tip pens should not be used. Entries should be dated and initialed or signed. If several entries are made on the same day by the same person, it is sufficient to provide a date at the top of the entries and a signature or initials after the entries. Space should be provided at the bottom of each page for a Program Manager's, or designee's, signature and date to indicate that the entries have been reviewed.

Entries must never be written over to correct errors. Errors must be corrected by crossing out with a single stroke so that the error can be clearly read. The correction must be written clearly, initialed, and dated. Corrections must not be made by erasure or using correction fluid.

If a data-gathering activity is not completed, a reason should be recorded. If any data are deleted at later stages, a reason for the deletion should be recorded in the notebook. Unused spaces between entries should be crossed out.

Data printouts may be used in a notebook instead of hand-recorded data if the printouts are permanent (i.e., will not fade over time). Photocopies of printouts on thermal paper are preferred; however, each such copy must have an original

signature in ink. Printouts should be taped or glued, not stapled, onto notebook pages. Taped- or glued-in printouts should be signed so that the signature is partially on the notebook page and partially on the printout.

11.3 DATA FORMS AND CHECK SHEETS

Standard data forms are strongly recommended for repetitive manual data recording. They provide a consistent format for pertinent data and ensure that data are properly labeled and identified. In the same manner, check sheets serve as a systematic reminder of all items that need to be observed or serviced for a particular operation. By initialing each item as it is observed or serviced, the check sheet also serves as documentation that all tasks have been completed.

As with laboratory notebooks, all entries should be made directly onto the form in permanent ink. Errors must not be written over (cross them out with a single stroke), and each form should have space for the date and the data recorder's initials. Data sheets may be bound and pages numbered prior to data entry.

Check sheets should be designed in a manner that requires each item on the sheet to be checked or recorded before the sheet is completed. For items that would not be checked or recorded each time a check is used, the check sheet design should make it obvious when the item should and should not be recorded, or a separate check sheet should be used. More useful data are acquired when the check sheet design requires the recorder to enter a physical observation rather than a check.

11.4 INSTRUMENT PRINTOUTS AND ELECTRONIC MEDIA

At a minimum, the following should be included in each printout or electronically stored data set.

- Date: the date the task was performed
- Operator: the person operating the instrument or computer
- Identification: a description of the information being recorded
- Information: the information itself, or an explanation of why it is not recorded
- Correction Factors due to non-routine sample amounts, dilutions, amount injected/analyzed, etc.

The following items may also be essential, depending upon the information being recorded.

- Instrument ID: type, model number, serial number, or other information that describes the type of instrument being used
- Study ID: a description of the study for which the information is being recorded
- Time: the time of day the task was performed
- Units of Measure: the units that are used for each type of measurement that is recorded
- Coding: an explanation of any special codes that are recorded

Printouts should use media that will not fade over time, or should be photocopied onto a medium that will not fade, and should be signed in permanent ink.

Raw data that are manually entered into a computer from an original document should be checked for accuracy against a computer printout of the raw data. It should be noted on the printout that the raw data were checked, and the printout should be dated and signed.

12.0 INTERNAL QUALITY CONTROL

12.1 ANALYTICAL QC SAMPLES

Unless otherwise specified by the Program Manager or the individual RLAB Method, every batch of samples analyzed shall contain a method blank, a laboratory control sample, a matrix spike, and a matrix spike duplicate, as described in SOP 2430.12, "Regional Laboratory Quality Control Policy," and policies concerning analytical data quality. If any of the QC elements described above fails, then the analyst shall notify the Program Manager and corrective action shall proceed as described in SOP 2430.12 and in this QA Operating Plan.

The Program Manager may approve data that does not conform to the documented QC specs provided that the data is usable for its intended purpose. This must be documented in full.

12.2 PEER REVIEW

All data packages shall be peer reviewed in accordance with SOP 2410.10 to ensure that the data are free from transcription and calculation errors. The peer reviewer shall indicate approval of all analytical work including the resultant documentation by signing or initialing and dating the appropriate form or space provided. Differences between the peer reviewer(s) and work performer(s) shall be discussed and resolved. If agreement cannot be reached, the differences shall

be brought to the attention of succeeding higher levels of management until resolution is achieved.

The following acceptance criteria shall be considered if pertinent to the specific activity:

- Conformance with applicable SOPs, standards, technical guidance, and instructions
- Equipment utilized meets specifications
- Equipment utilized was referenced and calibrated as required
- Appropriate forms, logs, or formats were utilized
- Completeness of documentation generated during performance:
  - All blank titled spaces of forms have been considered
  - Total presentation is legible and reproducible
  - Data entries, calculations, and results are interpretable
  - Plots, charts, data summaries, graphs, etc. are precise, and parameters are clearly defined as per Section 12.6
  - Both input data and reported results have been accurately transcribed and are properly referenced

Other acceptance criteria which describe performance and documentation may also be incorporated as necessary.

There should be documentation present that all analytical activities have been checked and verified. A check of documentation shall be performed at the completion of the task.

12.3 SYSTEMS ASSESSMENTS

Periodic internal systems assessments shall be performed as described in SOP 2430.6. These assessments will be coordinated by the Independent QA Reviewer. Internal assessments will be performed according to the criteria described in Section 14.1 of this document. Additional criteria may be included as necessary to document compliance to all applicable QA/QC guidelines and requirements and to ensure that a high level of quality is maintained for all RLAB products.

12.4 PERFORMANCE EVALUATION/PERFORMANCE TEST (PE/PT) SAMPLE PROGRAM

It is the policy of the Region 7 ENSV, where practical, to incorporate PE samples into analytical activities managed by ENSV regardless of where the analyses are

performed. The PE sample program shall be conducted in conformance with SOP 2430.7, "Performance Evaluation Sample Program Guidance."

The highest levels of QA/QC are necessary in the preparation of QC or PE samples so that the Agency can be confident that any problems uncovered are due to the analytical system being checked rather than the preparation process. SOPs for the management and operation of the PE sample program shall be rigorously followed. All steps in the preparation of QC or PE samples will be documented through the use of preparation sheets which are archived in binders. Shelf lives of reagents and prepared audit materials shall be watched to identify questionable materials. For most water PE samples, characterization is not necessary. Where applicable, the control limits established by the PE provider in preparing the concentrates used in generating the PE are utilized in evaluation of the resulting data. For non-aqueous PE materials, the lot of PE materials shall be characterized and the resulting data statistically evaluated in accordance with SOP series 2430 before such materials are released for use.

The Laboratory shall participate in Performance Testing (PT) studies, as described in SOP 2430.11, with the necessary frequency to obtain and maintain NELAC Accreditation for the list of analyses determined by RLAB Management. The Independent QA Reviewer oversees the RLAB PT program in coordination with the Program Managers.

12.5 TRAINING

Proper training of new staff is one means of ensuring high quality performance by the staff. Program Managers are responsible for providing training and assuring their staff have adequate and current skills. Each analyst is responsible for developing their skills to the best of their abilities, and keeping current with progress in their field of expertise. As appropriate, the analyst and Program Manager may identify training opportunities to keep analyst(s) skills current. It is the responsibility of the Program Manager to negotiate such opportunities with the Laboratory Director on behalf of the analytical staff, and the responsibility of the Laboratory Director to obtain funding to allow such training from EPA management. The Independent QA Reviewer will notify the Program Managers and Laboratory Director when lack of training is impacting data quality. The Independent QA Reviewer will implement a program certifying analyst training and documenting this training with sign-off forms signed by the analyst and Program Manager. These forms will be maintained in a separate training file, subject to audit.

12.6 ASSIGNMENT TRACKING

In order to ensure the timeliness of products and in order for management to effectively allocate resources to allow consistent achievement of quality objectives, all RLAB analytical work will be tracked through use of internal computer tracking systems ("Backlog Reports" in the LIMS). The tracking systems will be used to report on the status of analytical project completion. The Program Managers will report weekly to the Laboratory Director on the status of the analytical work assigned to or controlled by their Programs.

12.7 SOFTWARE QUALITY ASSURANCE

All RLAB computers which are connected to the ENSV LAN will be automatically checked for viruses through the virus software which is resident on the LAN. A reasonable effort should be made to periodically check for viruses on other RLAB computers not connected to the LAN. If a user suspects a computer virus is present on a computer, the unit is to be shut down immediately and a note placed on the unit that it is not to be used due to possible infection with a computer virus. The Program Manager and Independent QA Reviewer should be notified immediately.

Only authorized software shall be placed on RLAB computers. Unauthorized software specifically includes: software or copies of software which are owned by employees, "pirated" software and unauthorized replicates of software which RLAB is licensed to use. RLAB will use only software, versions of software, and number of copies of software for which RLAB is licensed. Generally, software may not be transferred between EPA computers; exceptions would be transfers which are properly approved (transfers are subject to all applicable copyright laws). All computers assigned for RLAB use are subject to unannounced checks to evaluate compliance with these directives.

13.0 PERFORMANCE AND SYSTEMS AUDITS

13.1 SYSTEMS AUDITS

Systems audits of RLAB operations shall be conducted in compliance with the EPA Region 7 Quality Management Plan. The Independent QA Reviewer shall conduct annual internal program reviews of RLAB operations as described in SOP 2430.6. In addition, the Independent QA Reviewer will periodically perform QC spot checks of selected data packages as described in SOP 2430.5. Other RLAB personnel may be tasked to assist the Independent QA Reviewer in the performance of these reviews or other audits of RLAB systems. The reviews will

verify generally that approved procedures are in place and being used, and that corrective actions are being performed by laboratory personnel in a timely and responsive manner.

Audit reports summarizing findings will be written and submitted to the Laboratory Director.

13.2 MANAGEMENT SYSTEM EVALUATION

The RLAB Management Team will, at least annually, perform an evaluation of RLAB operations and the quality system. The evaluation will include extensive discussions between the Laboratory Director and the RLAB management team. Laboratory operations will be assessed in terms of anticipated workload (type, volume, and timing of analyses required to meet customer's needs within a one- to five-year time frame) and capability for meeting workload requirements. After analysis of the anticipated workload is completed, an assessment of the laboratory's capabilities for meeting workload requirements will be conducted. Specific items to be evaluated include the following:

- Personnel - evaluate expertise, level of experience, and staff size.
- Equipment - evaluate current equipment, applicability of SOP 2440.2 (Planning for Regional Laboratory Capital Equipment Procurement), and need for maintenance contracts
- Supplies - evaluate use of credit card and blanket purchases for routine and emergency purchases
- Facility - evaluate space and usage

In addition to the items listed above, RLAB management will consider any conflicting requirements, including overall laboratory capacity and other non-analytical priorities, as part of the laboratory operations evaluation.

The annual management evaluation of the quality system will evaluate its continuing suitability and effectiveness, and should identify any necessary changes or improvements. The evaluation will take into account reports from managerial and supervisory personnel, the outcome of recent internal audits, assessments by external bodies, the results of inter-laboratory comparisons or proficiency tests, any changes in the volume and type of work undertaken, feedback from clients, corrective actions, and other relevant factors. The evaluation should also assure that RLAB policies and procedures for SOP review and development are being followed.

The evaluation findings and the management team's recommendations will be documented in a report to the Laboratory Director.

13.3 PERFORMANCE AUDITS

RLAB shall participate in performance audits as described in the 1350 series of the SOPs.

13.4 EXTERNAL AUDITS

RLAB shall participate in external audits, as appropriate, from such sources as NELAC, KDHE, NERL, Management System Review (HQ or QAO) and others.

13.5 RESOLUTION OF DISCREPANCIES

If there are discrepancies, deficiencies, or indeterminate results arising from the QA/QC audits, review of analytical packages, or audits performed by the Independent QA Reviewer, the Program Manager shall implement appropriate corrective actions and the Independent QA Reviewer will ensure that such corrective actions are completed prior to closing the audit file. If resolution cannot be reached, the problems shall be brought to the attention of the Laboratory Director or higher management to attain resolution.

The Program Manager, Independent QA Reviewer and Laboratory Director or delegated representatives shall evaluate the problems, provide solutions, and verify implementation of solutions prior to allowing the activity to resume.

14.0 PREVENTIVE MAINTENANCE

14.1 OBJECTIVE

The objective of the preventive maintenance program is to avoid generating environmental measurements that could lead to inappropriate remedial responses or impede an enforcement action. Instrument maintenance, both routine and preventive, shall be performed as defined in SOPs or as otherwise determined to be necessary. Preventative maintenance allows equipment problems to be rectified before they become serious and also brings attention to those areas of the instrument susceptible to degradation from aging, toxic/corrosive attack, and clogging due to environmental factors.

14.2 ANALYTICAL EQUIPMENT

Analytical equipment shall be kept in good working order. Procedures for preventive maintenance are contained in each instrument operations manual and/or in SLOMS and RLAB Methods for the specific operation being performed. The maintenance procedures for equipment shall be approved for use by the Program Manager. Records of equipment maintenance shall be logged as required by the maintenance procedures.

Program Managers are responsible for ensuring that supplies, spare parts, and expendables for proper instrument maintenance are available for instruments used by their Program, and that analysts are aware of the location of these items. Program Managers will ensure that analysts are properly trained in applicable preventive maintenance procedures as part of the analysts' training on a given instrument.

Each analyst is responsible for ensuring the proper operating condition of an instrument before performing analyses, and for stopping analyses if the instrument's performance deteriorates during analysis to the point that quality data cannot be generated. Analysts are responsible for performing preventive maintenance as indicated during their training and per the applicable SLOM, RLAB Method, or manufacturer's manual. Analysts are responsible for promptly alerting the Program Manager when supplies, spare parts, and expandable items need to be obtained or reordered, or a service call by an instrument repair technician is required.

14.3 SUPPORT EQUIPMENT

Support equipment for preventive maintenance purposes should be periodically inspected by the analysts in order to maintain the performance standards necessary for proper and efficient execution of all tasks and responsibilities. At a minimum, maintenance in accordance with the equipment operator's manual will be performed.

15.0 SPECIFIC ROUTINE PROCEDURES USED TO ASSESS DATA QUALITY

15.1 LABORATORY ANALYSIS

Decisions regarding the precision, bias, and completeness of data are made in accordance with the appropriate SOP (normally 2430.12), or EPA directives. The procedures for making these assessments shall be as prescribed in the RLAB Method and/or SOP 2410.10.

RLAB staff will monitor QC data to ensure that they are within the established control limits for the methods. Data accuracy and precision will be assessed for each sample group using samples spiked at a known level. Control limits shall be established as specified in SOP 2430.12.

15.2 PROCEDURE VALIDATION AND PROFICIENCY DEMONSTRATION

The requirement for proficiency demonstrations that are to be performed by RLAB in general and by each analyst are as described in SOP 2410.11.

When RLAB Methods are developed, the data necessary to characterize the method or to demonstrate quality parameters shall be included. These data will include calculations of the standard deviation, percent recovery, and coefficient of variation which will be used in determining the MDL, precision, and bias for each method.

16.0 CORRECTIVE ACTION

16.1 ROUTINE CORRECTIVE ACTION

Routine corrective action is performed in cases of abnormal instrument performance, isolated instances of QC outliers, or errors due to calculation or transcription. However, at times, if modification of standard practices or procedures is required to correct a problem, formal corrective action should be initiated. All routine corrective actions must be performed according to the applicable SOPs, methodologies, or directives.

Program Managers are responsible for quality within their Programs and, consequently, are required to notify the Independent QA Reviewer about quality-related issues as they arise. For example, if outliers or errors of any particular type (QC sample, transcription error, calculation error, etc.) are occurring at an abnormal frequency, the Independent QA Reviewer should be notified and a joint investigation should be performed by the Program Manager and Independent QA Reviewer to determine if formal corrective action should be taken.

16.2 FORMAL CORRECTIVE ACTION

Formal corrective action may be initiated due to a variety of conditions (PT results, low scores on customer evaluations, systems audits results, inter-laboratory comparisons, special outside request, or failure to adhere to this QAOP). Additionally, problems which are not resolved through routine

corrective actions as described above must be resolved through the formal corrective action process.

After examination of the area of concern, the Laboratory Director, the Independent QA Reviewer, and the Program Manager will agree upon a corrective action plan to be implemented and an implementation date by which the corrective action should be completed. After the implementation date, the Independent QA Reviewer, in coordination with the Program Manager, will perform a systems assessment of the area to verify that the corrective action has been properly implemented and is adequate to correct the problem. Corrective actions are closed when the effectiveness of the corrective action has been validated by the Independent QA Reviewer through the assessment process.

16.3 DOCUMENTATION

For formal corrective actions, the Independent QA Reviewer will maintain a corrective action log which contains a brief description of the corrective action, assignment of responsibility for the corrective action, and the dates when the corrective action was opened, due, and closed. The Independent QA Reviewer will also prepare either a corrective action form or a memo which documents the problem and indicates the nature of the corrective action to be taken. The Program Manager should submit a memo to the Laboratory Director and the Independent QA Reviewer that documents the completion of any formal corrective action. Copies of corrective action memos, corrective action forms, and completed pages from the corrective action log will be maintained in the centrally located QA/QC file. Quarterly progress reports will detail all problems and subsequent resolutions.

Routine corrective actions, as defined above, are to be documented in the routine flow of daily activity as follows. Corrective actions due to isolated instances of instrumental QC outliers are to be documented in the instrument run logs. Other routine corrective actions should be documented through "memos to file" from Program Managers or staff involved which are retained with the data packages or, if appropriate, by notation on the data quality assessment record which is provided with each data package submittal. The Program Manager should provide copies of any QA/QC-related correspondence, which are either submitted to or received from others outside RLAB, to the Independent QA Reviewer.

All documentation described above will be subject to review in systems audits.

17.0 QUALITY ASSURANCE REPORTS TO MANAGEMENT

QA/QC status reports will be generated on a quarterly basis and provided to the Laboratory Director. The Independent QA Reviewer is responsible for generating quarterly reports which include significant QA/QC actions such as corrective action requirements and resolutions.

The Independent QA Reviewer's reporting responsibilities also include the annual update of the QA Operating Plan and documentation of systems audits, performance audits, and corrective actions.

18.0 LABORATORY ETHICS POLICY

Since the integrity and quality of our work is paramount in maintaining our customers' confidence and ensuring the analytical data that we report are of known quality and defensible, we have an inherent responsibility to perform and document our work consistent with the highest principles of ethical behavior and scientific practices. As a result of an identified need for a uniform policy statement among the 10 regional laboratories, the regional laboratory directors and/or branch chiefs developed and agreed upon a statement for all of us to adopt. The following is the specific statement which we are adopting as the Region 7 Laboratory ethics statement and is the foundation for performing our mission functions:

> **It shall be the policy of the Region 7 Laboratory to conduct all business with integrity and in an ethical manner. It is a basic and expected responsibility of each staff member and each manager to hold to the highest ethical standard of professional conduct in the performance of all duties and to adhere to EPA's *Principles of Scientific Integrity*, dated November 24, 1999.**

EPA's Principles of Scientific Integrity which is dated November 24, 1999, and cited above, was reaffirmed by then EPA Administer Christine Whitman on July 10, 2002. The entire document is being incorporated into this plan and is quoted in its entirety as follows:

> "It is essential that EPA's scientific and technical activities be of the highest quality and credibility if EPA is to carry out its responsibilities to protect human health and the environment. Honesty and integrity in its activities and decision-making processes are vital if the American public is to have trust and confidence in EPA's decisions. EPA adheres to these Principles of Scientific Integrity.
>
> EPA employees, whatever their grade, job or duties, must:

- Ensure their work is of the highest integrity – this means that the work must be performed objectively and without predetermined outcomes using the most appropriate techniques. Employees are responsible and accountable for the integrity and validity of their own work. Fabrication or falsification of work results are direct assaults on the integrity of EPA and will not be tolerated.
- Represent their own work fairly and accurately. When representing the work of others, employees must seek to understand the results and the implications of this work and also represent it fairly and accurately.
- Represent and acknowledge the intellectual contributions of others in representing their work to the public or in published writings such as journal writings or technical reports. To do otherwise is plagiarism. Employees should also refrain from taking credit for work with which they were not materially involved.
- Avoid financial conflicts of interest and ensure impartiality in the performance of their duties by respecting and adhering to the principles of ethical conduct and implementing standards contained in Standards of Ethical Conduct for Employees of the Executive Branch and in supplemental agency regulations.
- Be cognizant of and understand the specific, programmatic statutes that guide the employee's work.
- Accept the affirmative responsibility to report any breach of these principles.
- Welcome differing views and opinions on scientific and technical matters as a legitimate and necessary part of the process to provide the best possible information to regulatory and policy decision-makers.

Adherence by all EPA employees to these principles will assure the American people that they can have confidence and trust in EPA's work and in its decisions."

18.1 ETHICS TRAINING AND CERTIFICATIONS

The Program Manager of a new employee will ensure that the employee has received orientation in laboratory ethics, and both the new employee and the Program Manager will sign a certification page documenting completion of this training. The Independent QA Reviewer will conduct periodic (at least annually) group refresher training sessions in laboratory ethics, after which attending employees will update their certification forms. Current ethics certification forms will be maintained on file for all employees by the Independent QA Reviewer or designee.

18.2 INCIDENT MANAGEMENT

The Independent QA Reviewer will discuss with an employee any ethical issue

reported, to determine whether the issue affects data quality/integrity, or is a strictly interpersonal ethical issue (e.g., a victim of character defamation). All conversations regarding incident management will be treated as strictly confidential. Correspondence regarding an incident shall be treated as confidential, although available for inspection by auditing bodies (as determined by the appropriate level of management).

- If the incident may affect data quality or integrity, the Independent QA Reviewer is authorized to investigate the matter on behalf of the Laboratory Director like any other data quality problem. If a problem is found, it will be investigated and reported per Section 16 of this document (Corrective Actions).
- If the incident appears related to personnel matters only, the issue will be reported to the Program Manager (or Laboratory Director, if it involves the Program Manager) for handling through existing mechanisms addressing personnel matters.

Environmental Protection Agency

300 Minnesota Ave.
Kansas City, KS 66101

MEMORANDUM

DATE: April 5, 2006

SUBJECT: Addendum to Standard Operating Procedure No. 2440.5E, "U.S. EPA Region 7 Laboratory Quality Assurance Operating Plan"

FROM: Harold Brown, Ph.D., Independent QA Reviewer

THRU: Joe Arello, ANOP Manager, ENSV/RLAB/ANOP
Dale Bates, RLAB Manager, ENSV/RLAB

TO: SOP Files: SOP 2440.5E

The purpose of this addendum is to incorporate a requirement for timely notification to the responsible Regional Project Manager of irregularities or other potential problems that may affect data usability are discovered after the data has been formally transmitted.

When SOP 2440.5E is updated, the following section will be added:

16.3 TIMELY NOTIFICATION TO DATA USERS

If a condition that requires a corrective action is identified, all affected data will be evaluated to determine the impact on data quality. For data that has already been transmitted, notification of the impact on data quality and usability will be made to the project manager within three working days after the data is evaluated and the impact determined.

[The current section 16.3 will be changed to section 16.4.]

STANDARD OPERATING PROCEDURE

4220.03A

Protocols for the Region 7 Lead-Contaminated Residential Yard Soil Cleanup Actions Procedures and Sequencing

June 8, 2007

Mark Doolan
SUPR/SPEB

APPROVED:

OSC/Peer Review — Date 7/23/07

Risk Assessor Peer Review — Date 04/10/08

Chief, Special Emphasis Remedial Branch — Date 5/7/08

Chief, Emergency Response & Removal North Branch — Date 5/8/08

Chief, Emergency Response & Removal South Branch — Date 5/7/08

Independent QA Reviewer — Date 05/21/2008
dew 5/15/08

Recertified:

Branch/Name					
Date					

Table of Contents

A. PURPOSE AND APPLICABILITY

The purpose of this Standard Operating Procedure (SOP) is to describe the recommended uniform procedures for sampling activities, and the sequencing of cleanup response actions for residential property yard soils at lead-contaminated sites. The procedures in this SOP should only be applied to response actions after agency decision making processes as outlined it the National Contingency Plan (NCP) regulation at 40 CFR Part 300 have been followed. Prior to the initiation of any yard soil cleanup response actions, the sampling activities described in Section E.1 should be fully considered, scoped, and planned to enable appropriate decisions and sequencing for subsequent cleanup options described in Section E.3.

The procedures contained herein are applicable to all personnel of the Superfund Division (SUPR) and their contractors who collect samples and perform residential yard soil cleanup response actions. This SOP supplements and is integral to the objectives of site-specific sampling, which have to be established in a Quality Assurance Project Plan prior to initiation of sampling. This SOP also supplements and is integral to cleanup response actions, which have to be determined in accordance with the NCP.

This SOP applies, but is not limited to, residential properties affected by active and inactive lead mines, mills, and smelters and their associated activities.

B. DEFINITIONS

Residential properties: As defined in the Superfund Lead-Contaminated Residential Sites Handbook (Handbook), residential properties are areas with high accessibility to sensitive populations, including but not limited to properties containing single and multi-family dwellings, apartment complexes, vacant lots in residential areas, and other High Child Impact Areas (HCIAs).

High Child Impact Areas (HCIAs): Areas where large numbers of young children congregate including day-care centers, schools, community centers, playgrounds, parks, green ways, and any other area where children may be exposed to site-related contaminated media.

X-Ray Fluorescence (XRF) spectrometer: An instrument used to resolve radiation into spectra to determine measurements. The XRF is used to analyze soils for metals contamination as described in the Instruction Manual for the XRF spectrometer. The XRF may be used to analyze paint chips for metals contamination.

Integrated Exposure Uptake Biokinetic Model (IEUBK): Predicts blood-lead concentrations (PbBs) for an individual child, or group of similarly exposed children (6 months to 7 years old), who are exposed to lead in the environment.

C. PERSONNEL QUALIFICATIONS

All field personnel are to take the 40-hour health and safety training course (as per 29 CFR 1910.120(b) (4)), and regular refresher courses prior to engaging in any field collection activities.

D. SUMMARY OF SAMPLING METHODS

1. Soil

Personnel conducting the soil sampling activities should review, and follow the EPA's SOP 4230.19A, Soil Sampling at Lead-Contaminated Residential Sites, July 3, 2007. Soil samples may be collected using a variety of methods and equipment. Collection is dependent on the depth of the desired sample, the type of sample required (disturbed vs. undisturbed), and the soil type. Surface soils may be easily sampled using a spoon, spade, trowel, and scoop. The site-specific soil sampling protocols will be prepared in accordance with the QAPP for the site.

2. Interior Dust

Personnel conducting the field interior dust sampling activities should review and follow EPA's SOP 4230.18A, Interior Dust Sampling at Lead-Contaminated Residential Sites, June 8, 2007.

The amount of lead in interior dust samples can be expressed as a loading or as a concentration. Loading is the weight of lead per area sampled and the typical units are micrograms per square foot ($\mu g/ft^2$). Concentration is the weight of lead per weight of the sample, and is typically reported as $\mu g/g$. Vacuum dust collection is able to generate both loading and concentration results. Both loading and concentration data results are essential for residential site investigations. Interior dust concentration data is required for lead risk assessment using the Integrated Exposure Uptake Biokinetic (IEUBK) model.

At each residential property where interior dust will be sampled, it is anticipated that three samples will be collected. Since each residence will have a different floor plan and furniture arrangement, it will not be possible to predetermine the exact sample location. Interior dust samples will be collected by wipe sampling, and/or by the use of a high-volume cyclonic vacuum.

Wipe samples are collected from smooth surfaces to indicate surficial contamination; a sample location is measured and marked off. While wearing a new pair of surgical gloves, a sterile gauze pad is opened, and soaked with solvent. The solvent used is dependent on the surface being sampled. The pad is then stroked firmly over the sample surface, first vertically, then horizontally, to ensure complete coverage. The pad is then transferred to the sample container. All wipe samples will be submitted for laboratory analysis of arsenic, barium, cadmium, and lead according to EPA Method 6010B.

Contaminant levels will be expressed in $\mu g/ft^2$, which will represent the contaminant loading at each sampling location.

Interior dust samples from floors will be collected in accordance with Paragraph 11.11 and 11.2 of ASTM D5438-05 contained in Attachment A (with the modification of using a sieve size of 250 μm). Samples will be submitted for laboratory analysis of arsenic, barium, cadmium, and lead according to EPA Method 6020. The contaminant levels will be expressed in mg/kg, which will represent the total concentration of each contaminant, and $\mu g/ft^2$, which will represent the contaminant loading at each sampling location.

3. Lead-Based Paint

Lead-based paint (LBP) screening should be conducted on the interior and exterior of homes, and properties, where dust samples are collected. The purpose of the paint screening is to determine whether lead is present in the paint on the home at a level that may create an exposure risk. It is not intended to be as comprehensive as a LBP inspection or a lead hazard screen as defined in the EPA regulations at 40 CFR 745.227. Paint on the exterior and interior of the home selected for testing should be analyzed with an X-Ray Fluorescence (XRF) Spectrometer to determine the presence of lead in the paint.

E. PROCEDURAL STEPS

1. Initial Site Investigation and Removal Assessment Sampling Requirements

Prior to initiating yard soil cleanup actions, the sampling activities described in this section should be fully considered, scoped, and planned to enable appropriate decisions and sequencing for subsequent cleanup action. The information collected during the initial sampling phase should be sufficient to enable the program to make removal action decisions on a property-by-property basis, and for the Risk Assessor to conduct an assessment and determine a risk-based cleanup level using the Integrated Exposure Uptake Biokinetic (IEUBK) Model. Additionally, sufficient sampling should be conducted in each individual residential property, so that no other additional sampling would be required prior to cleanup actions. All sampling should be conducted in accordance with the "Superfund Lead-Contaminated Residential Sites Handbook, OWSER 9258.7-50, and May 2003," and as further defined in this SOP and the other generic lead sampling and SOPs and QAPPs.

Yard Soil: Once the agency has been made the decision that a site-response action may be required, yard sampling should be planned, and conducted as expeditiously as possible to identify the total number of affected residential properties. This should include sampling to support the data needs for potential listing of the site on the NPL, and the full scope of the response action to include all residential properties where samples of yard soils show lead concentrations exceeding EPA's soil screening level of 400 parts per million (ppm). If economically feasible, each and every individual residential property within the site or area of potential impact should be sampled.

Sampling should begin at the source, progress outward until an outer boundary of the contamination is identified, and each residential property within the boundary has been sampled. At large sites where funding does not allow the sampling of every property within the 400 ppm boundary during the initial investigations, the extent of lead contamination exceeding EPA's time-critical removal action level of 1,200 ppm should, at a minimum, be defined. Every property within the 1,200 ppm boundary should be sampled along with a sufficient number of properties to determine the general location of the 400 ppm boundary.

Fine Soil Fraction Analysis: Soil samples collected for concentration input parameters in the IEUBK model (EPA 2000) will be sieved prior to analysis. Several studies indicate that the particle size fraction of soil and dust that sticks to the hands, and incidentally ingested by young children, is the fine fraction less than 250 microns (μm). It is generally expected that the fine fraction will contain a higher concentration of lead or be "enriched" as compared to the total soil fraction or bulk sample. Thus, EPA guidance indicates that the soil and dust lead concentration from the fine fraction (< 250 μm) should be used as the concentration input for the IEUBK model (EPA 2000). Rather than sieving every sample with a 250 μm, or #60 sieve, at least 20%, or a minimum of 30 surface soil samples should be analyzed for lead concentration in both the fine and total soil fraction. A statistical analysis of the data will be conducted to determine the site-specific relationship between the concentrations in the two fractions, as discussed in lead risk assessment guidance (EPA 2000). For large sites where sampling will be conducted over several years, additional samples should be collected over time to further refine this relationship. It also is important to collect soil samples with a broad range of lead concentrations. The results of the statistical analysis must be used to establish a site-specific, "adjusted" cleanup goal when using total soil fraction, or bulk sample data for decision-making.

Interior Lead-Based Paint/Interior Dust: The lead content of interior lead-based paint, lead loading, and concentration of interior dust should be determined at a statistically significant number of residential properties, generally 20 percent. This assessment is required for both use in the risk assessment, and for determining cleanup requirements.

Exterior Lead-Based Paint: Lead concentrations in exterior paint should be analyzed, and the general condition of exterior surfaces should be assessed to determine the potential for recontamination of the clean soil placed in the yards during the cleanup. Measurement of the lead concentration in exterior paint should be made at each residential property where yard soil samples are obtained.

High Child Impact Areas: Daycares, school properties, playgrounds, parks, and green spaces are all areas where large numbers of young children concentrate. All of these types of facilities and areas should be included in the sampling plan and sampled during the early stages of the investigations at a site because these locations

may present a health risk to large numbers of children. These areas should be considered the same as residential properties for all sampling and response activities.

Bioavailability Testing: Soil samples collected for use in the IEUBK model (EPA 2000) will be analyzed for lead bioavailability. A site-specific determination of lead bioavailability in soil is critical to accurately predict blood lead concentrations and derive residential yard soil cleanup levels using the IEUBK model. EPA has determined that a specific *in vitro* bioaccessibility method (IVBA) is an appropriate methodology for predicting site-specific lead relative bioavailability in soil (EPA 2007c). Thus, surface soil samples should be collected, and analyzed using the IVBA method protocol described in (EPA 2007b). A sufficient number of soil samples should be collected from across the site, and analyzed to characterize the variability that might be associated with differences in soil characteristics, lead mineralogy, varying lead concentrations, proximity to sources of lead contamination, or other factors.

Toxicity Characteristic Leaching Procedure (TCLP): Soil samples should be collected and analyzed using the TCLP under RCRA, Method SW846 1311. TCLP analysis of yard soil samples from a representative number of residential properties will determine disposal and/or pre-disposal treatment requirements. Sufficient samples must be collected at varying concentration levels to confidently determine the level where the soils will likely fail TCLP for lead.

Speciation Testing: If warranted, and after consultation with Regional Counsel, when considering enforcement options, and/or if there is doubt concerning the lead source, a soil-lead speciation study should be conducted using micro-probe analytical methods or other appropriate methods. Generally, no fewer than 30 soil samples should be collected throughout the site for speciation testing, unless the site is very small, and conditions are homogeneous. Consideration should also be given to an interior dust-lead speciation study when collecting data for risk assessment using the IEUBK model.

Residential Water Wells: All private residential drinking water wells located within the suspected boundaries of the site should be sampled to determine the contribution of lead and other metals to overall risk.

2. Sampling Procedures

2.1 Soil Sampling Procedures

All soil-sampling procedures should follow the guidance presented in the Superfund Lead-Contaminated Residential Sites Handbook (Handbook). Residential yards should be divided into quadrants based on the size of the yard, and a separate drip zone adjacent to the foundation of the house. Composite samples comprised of five aliquots or more should be collected from each quadrant, and a separate drip zone sample comprised of a minimum of four aliquots. The guidance from the Handbook is summarized below.

Yard Soil Samples: Residential lots with a total surface area less than 5,000 square feet will be divided into two quadrants; front yard and back yard. Aliquots comprising of the front and back yard composites should be equally spaced within the respective portion of the yard, and should be collected outside of the drip zone and away from influences of any other painted surfaces. Composites should consist of aliquots collected from the same depth interval.

For residential lots with a total surface area greater than 5,000 square feet, the property should be divided into four quadrants of roughly equal surface area. The two quadrants in the front yard should encompass one-half of the side yard; likewise for the two quadrants in the back yard. One five-aliquot composite should be obtained from each quadrant.

Any properties over one acre in size, including very large residential yards, vacant lots, schools, or parks, should be divided into 1/4 acre sections. One five aliquot composite sample should be collected from each section. The aliquots should be equally spaced, and collected away from influences of the drip zone, and any other painted surfaces. Composite samples must be comprised of aliquots from the same depth interval.

Drip Zones: A four-aliquot composite sample should be collected from the drip zone of each residential property, regardless of lot size. The composite sample should consist of a minimum of four aliquots collected between 6 and 30 inches from the exterior walls of the house. Each aliquot should generally be collected from the midpoint of each side of the house. Collection of additional aliquots should be considered if other factors exist, such as bare spots, distinct differences in the house exterior, and areas where runoff collects such as in downspout discharge areas.

Play Areas, Gardens, and Driveways: Distinct play areas and vegetable gardens, if present, should be sampled separately as discrete areas of the yard. Samples should also be collected in other locations depending upon the potential for exposure, such as under porches or crawl spaces and gravel driveways.

Depth of Sampling: In order to determine the depth of lead contamination throughout the site, and to provide data for selecting appropriate cleanup depths, samples should be collected at a depth from a representative number of homes throughout the site. Generally, 20 to 30 homes, equally spaced across the site, should be sampled to a depth of 24 inches. Individual composites should be collected from each discrete quadrant at a depth of 0-1 inch; 0-6 inches; 6-12 inches; 12-18 inches, and 18-24 inches. Samples collected from any portion within the 0 to 1 inch depth interval will be used to determine whether a property qualifies for cleanup. The appropriate number of samples will be determined based on a number of factors, such as the source of lead contamination, known heterogeneity across the site, etc.

Confirmatory Samples: Most field sampling for metals in residential properties is conducted using field portable X-Ray fluorescence (XRF) instruments. Laboratory confirmation samples should be collected at a rate of no less than five percent during the sampling event, and submitted to the laboratory for wet chemistry analysis to confirm the results of the XRF. The XRF determined values should be considered valid if the laboratory analytical results of the same sample are within plus or minus 30 percent of the XRF results. For decision-making purposes where both an XRF, and laboratory result was obtained for a sample, and both show concentrations near the action level; it is EPA Region 7 Superfund Management policy that the higher value of the two lead concentrations will be used. For example, if the XRF recorded a result of 390 ppm lead for a sample from a yard quadrant, and the laboratory recorded 425 ppm for the same sample, the XRF sample result will be considered valid but the quadrant will be assigned a lead value of 425 ppm.

Sample Collection: Once the yard has been divided into quadrants, a five-aliquot composite sample should be collected from each quadrant. The aliquot should be collected from the same depth interval with a disposable or stainless steel spoon or trowel. The soil from each aliquot should be dried, sieved with a No. 10 sieve (2 mm), and homogenized. For those soil samples that are collected for risk assessment purposes, the sample will also be processed through a No. 60 sieve (250 µm) to obtain the fine fraction. Then three XRF measurements should be obtained from the fine fraction sample. The three readings should be averaged to obtain the metals concentration for that quadrant. All three readings should be within 10 percent of each other. If any of the three readings falls outside the 10 percent range, the sample must be re-mixed, and the procedure repeated until the criterion is achieved. If a laboratory confirmation sample is to be obtained from the quadrant, a portion of the sample should be placed into the XRF specimen cup after the three readings have been obtained and averaged. The XRF instrument will then be used to analyze the specimen cup, and the reading will be recorded. The specimen cup should then be submitted to laboratory for wet chemistry analysis. Comparison of the XRF reading and laboratory analysis of the specimen cup should be made to determine the relative percent difference. Detailed maps or sketches of sampled yards and properties should be developed to include GPS coordinates of boundaries and structures, and depictions of quadrant sampling results.

2.2 Dust Sampling Procedures

The amount of lead in dust samples can be expressed as a loading or as a concentration. Loading is the weight of lead per area sampled, and the typical units are $\mu g/ft^2$. Concentration is the weight of lead per weight of sample and is typically reported as µg/g. Vacuum dust collection is able to generate both loading and concentration results. Both loading and concentration is required for residential site investigations while interior dust concentration is required for risk assessment using the IEUBK model. In general, no less than three dust samples should be collected from each residence

scheduled for assessment. The general sample area with a description of sample location criteria is presented below. Dust samples from floors should be collected in accordance with Paragraph 11.1, and 11.2 of ASTM D5438-05 (with the modification of using a sieve size of 250 μm).

Entry Way: A vacuum sample will be collected from the most frequently used entryway to the residence. The sample location should be collected just inside the door. If there is an option between a hard floor surface and a carpeted floor surface, the hard floor surface area should be chosen over the carpeted surface due to the potential for better sample collection. The sample will then be collected using the appropriate high-volume cyclonic vacuum method for the floor type.

Floor: A sample of floor dust should be collected from the most commonly used room in the residence other than a bedroom. The room, (other than the bedroom) where children living in the home spend the most time on the floor should be chosen for the sample collection. Children are defined as less than 7 years old or 84 months. If no children live at the residence, the room where residents spend the most time will be chosen. Sample location should be based on the floor type in the room. Hard floor surface should be given preference over carpeted areas in the room. A sample location that is not in the main walking pathway of the room, and is large enough to accommodate the sampling requirements, will be chosen as the sample location.

Bedroom: A sample should be collected from one bedroom in the residence. If there are children living at the residence, the youngest child's bedroom should be selected for sampling. If there are no children living at the residence, the bedroom where the most time is spent should be selected. If a child's room is selected, regardless of floor type, the sample location should be chosen based on where the child's play area is in the room, or where they spend the most time on the floor. If an adult bedroom is selected, the sample should be collected based on floor type and a hard floor surface should be given preference over a carpeted floor.

2.3 Lead-Based Paint Assessment

Lead-based paint (LBP) screening should be conducted on the interior and exterior of homes, and properties where dust samples are collected. The purpose of the LBP screening is to determine whether LBP is present in the home at a level that may create a potential health risk. It is not intended to be as comprehensive as a LBP inspection ,or a lead hazard screen as defined in the EPA regulations at 40 CFR 745.227.

LBP readings should be taken using an XRF instrument capable of providing data in micrograms per square centimeter ($\mu g/cm^2$) and capable of analyzing lead to less than 1 $\mu g/cm^2$. The following procedures will be followed during the LBP screening assessments.

An initial visual inspection should be conducted of the exterior walls of the home, and the interior painted surfaces in rooms where dust samples are collected to assess

whether significant deteriorating paint is present. If significant deteriorating painted surfaces are observed on the exterior walls of the residence, each of the exterior walls of the residence will be analyzed for LBP. If significant deteriorating painted surfaces are observed in the interior rooms where the dust samples are collected, each of the four walls in the room and a minimum of two windowsills should be analyzed.

The XRF readings should be taken at the location of the deteriorating painted surfaces. If deteriorating painted surfaces are not observed on the exterior walls of the residence, each of the four walls of the residence will be analyzed for LBP using a XRF instrument. If deteriorating paint is not observed in the rooms where dust samples are collected, XRF readings will be taken from each of the four walls and a minimum of two windowsills. The sampling team will document the general description of the interior walls and windowsills in the rooms where XRF readings are taken.

3. Residential Yard Soil Cleanup Guidance

The residential yard soil cleanup procedures in the following sections of this SOP should only be applied to response actions after agency decision making processes as outlined in the National Contingency Plan (NCP) regulation at 40 CFR Part 300 have been followed.

3.1 Initial Actions/Time-Critical Removal

The following actions should be initiated as soon as the information listed above is gathered, and an unacceptable risk has been determined either through a risk assessment, or the identification of a significant number of properties that exceed EPA's time-critical removal action level of 1,200 ppm lead. Several of these actions do not involve soil cleanup, but are necessary to rapidly begin to control exposure.

Blood-Lead Monitoring: The project manager should immediately coordinate with the local or county health department(s) to initiate, or enhance blood-lead monitoring of young children with the goal of 100% participation. Funding should be provided, if needed, by the department to begin, and/or enhance the program. Agreements between the health departments and the Agency must be executed to allow the release of information to EPA personnel on children identified with elevated blood-lead concentration for inclusion in cleanup actions.

Health Education: Coordination with the local/county health department, and existing citizens groups to initiate health education programs in the community should begin in a feasible period, and early in the project. Funding will likely need to be provided to these entities to conduct the program, and can usually be combined with funding provided for blood-lead monitoring.

High Efficiency Particulate Air (HEPA) Vacuums: If the interior dust sampling results indicate there is a significant health risk from interior dust, a HEPA vacuum loan program should be initiated at the site. This program is usually part of the

health education program funded through the local health department. Establishing this program early in the project can aid in significantly reducing the health risk at the site pending completion of the cleanup actions for sources and contaminated soils.

Soil Disposal: The project manager must identify soil disposal options and establish a yard soil disposal facility prior to any excavation activities. Off-site soil disposal other than in a permitted RCRA Subtitle C and/or D landfill will require that a RCRA Remedial Action Plan is prepared, and issued by the RCRA program. Consideration should be given to innovative on-site disposal methods, such as deep fill for construction projects, capping of mining wastes, or other uses where exposure of young children is mitigated.

Yard Soil Removal: If the yard sampling results indicate that a manageable number of contaminated residential properties exists on the site and they can be cleaned up within the removal budget as established by the Action Memorandum, then the cleanup of all residential yards where lead samples exceed 400 ppm (or a site-specific action level) should be completed during implementation of the removal action. At large sites where the excavation of contaminated soils from all residential properties with lead samples exceeding 400 ppm may not be possible within the removal budget, a time-critical removal action should be initiated to address: (1) all residential properties where soil-lead samples exceed 1,200 ppm; and, (2) all residential properties where soil-lead samples exceed 400 ppm where a child resides with an identified high blood-lead level (i.e., > 10 µg/dL).

High Child Impact Areas (HCIA): All soil exceeding 400 ppm lead, or the site-specific action level, should be excavated for each HCIA within the site. Early priority for cleanup should be given to HCIAs, especially day care facilities, in the response activities because these areas can affect the largest number of young children at the site.

Exterior Lead-Based Paint: For residential properties where soil excavation is planned, exterior lead-based paint with the potential to recontaminate the yard after cleanup should be controlled prior to the soil excavation. Data collected from several sites across the United States indicate that, in as little as two to three years, exterior lead-based paint has recontaminated yard soils that were cleaned up, thereby, recreating a significant health risk to young children living in the homes.

3.2 Subsequent Actions/Non-Time-Critical or Remedial

Long-term cleanup actions, either non-time critical removals at non-NPL sites or remedial actions at NPL sites should be initiated immediately at the conclusion of the time-critical actions. These cleanup actions will include the engineering activities described below.

Additional Yard Soil Sampling: At large sites where removal budgets were insufficient to characterize every property within the site, the remaining uncharacterized properties must be sampled until all yards potentially exceeding the site-specific actions level are identified.

Yard Soil Removal: Cleanup of all residences above the site-specific action level should continue. Remediation of all day care facilities, parks, schools, and other green spaces where soil-lead exceeds the action level not previously addressed during the removal actions should be given the highest priority.

Exterior Lead-Based Paint: Exterior lead-based paint with the potential to recontaminate the remediated residential yards should be controlled prior to the soil excavation at each residence requiring remediation.

Interior Dust: Interior dust attributed to exterior sources should be addressed after the yard soil cleanup at each residence. Once the soils have been cleaned up, a thorough one-time cleaning of all interior surfaces or other measures should be considered to mitigate the remaining health risk caused by dust in the home.

Source Control: Any sources of lead contamination that could potentially recontaminate residential yards should be controlled simultaneously, or shortly after the yard soil cleanup actions have been completed.

4. Soil Excavation Criteria

The following describes the requirements and procedures for excavation of yard soil from residential properties and HCIAs.

Depth of Excavation: Generally, one foot of clean soil between the underlying contaminated soil and the yard surface is considered protective of young children playing in the yard under current conditions. Therefore, excavation of one foot of soil is normally all that is required from a residential yard. Excavation depth should, however be based on the contaminant concentration data generated during the sampling event. For example, if the sampling results indicated the contamination is generally restricted to the upper six inches of soil, only a six-inch excavation would be required. Conversely, if the depth sampling indicates that the soil contamination exceeding the action level extends to 18 inches, consideration should be given to excavating to 18 inches to remove all contamination. In any case, the excavation should generally not be deeper than 24 inches in the yard area.

Vegetable garden areas must be treated differently than the general yard area. Studies have shown that garden vegetables will uptake metals (primarily lead and cadmium) through roots to a depth of up to 24 inches. Therefore, existing gardens where soils exceed 400 ppm lead, should be excavated to a depth of at least 24 inches, or until the soil concentration is less than 400 ppm lead, and/or equals the site-specific cleanup value. In communities where vegetable gardens are extremely

prevalent, consideration must be given to excavating the general yard soil to 24 inches, or less than 400 ppm, or to the site-specific cleanup value.

Bottom of Excavation Barriers: A site-specific determination will be made regarding whether a barrier, such as heavy plastic construction or snow fencing, will be placed at the bottom of the excavation. This decision will be based on a number of factors, including, but not limited, to the depth of excavation, lead concentration remaining at depth, the extent of remaining contamination, future land use, and the use of institutional controls. Sampling must be conducted in the bottom of the excavation to determine the need for barrier installation prior to backfilling with clean soil. However, XRF sampling results alone are sufficient to make this decision. No laboratory confirmation samples need to be collected from the bottom of the excavation (assuming sufficient confirmation samples already exist to show the XRF accurately measures site metals concentrations).

Drip Zone: The drip zone soil in yards of older homes where exterior lead-based paint was applied generally contains contaminated soils with significantly higher lead concentrations than the rest of the yard. It is not uncommon for parts of the yard to have samples that are below the site-specific action level for the site while the drip zone soil samples greatly exceed the action level. Therefore, to ensure the overall protectiveness of the remedy for each residential property addressed during the cleanup action, all portions of the drip zone with samples that exceed the action level should be excavated and removed. For example, the quadrant sampling data for a yard might show that only one quadrant in the back yard of the property exceeds the action level, but that the drip zone exceeds the action level around the entire perimeter of the house. In this case, soil must be excavated from the backyard quadrant and the entire drip zone surrounding the house.

5. Source Control

Any sources that could potentially recontaminate residential property yard soils within a short time-frame should be remediated prior to conducting the yard soil cleanup actions. For example, typical sources that would require cleanup are mine waste piles located within the vicinity of residential properties where yards could receive run off, wind-blown dust, or smelter emissions containing high concentrations of metals, that when deposited on yards through fallout would recontaminate the yard within a relatively short period of time. These recontamination sources (or others like them) must be brought under control prior to remediation of residential yards.

F. RECORDS MANAGEMENT

Documentation of environmental data collection and analysis procedures (i.e., laboratory documentation, field log book, photo documentation, chain-of-custody) should be completed and managed using the requirements specified in the Generic Quality Assurance Project Plan for Region 7's Superfund Lead-Contaminated Sites. Consideration should be give during the scoping and planning of sampling events to the

development of a database to record sampling data, location information and maps, and site activities.

G. QUALITY ASSURANCE AND QUALITY CONTROL

The Superfund Division Director in EPA Region 7 has the responsibility for oversight and assessment of this Regional protocol. This responsibility can be delegated to the Superfund Deputy Division Director and to Branch Managers within the Superfund Division.

H. REFERENCES

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STANDARD OPERATING PROCEDURE

4230.19A

Soil Sampling at Lead-Contaminated Residential Sites

July 3, 2007

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SUPR/EFLR

APPROVED:

RPM Peer Review	Date 7-25-07
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Date					

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A. PURPOSE AND APPLICABILITY

The purpose of this Standard Operating Procedure (SOP) is to describe the procedures for the collection of representative surface soil samples at lead-contaminated residential sites as described in the Superfund Lead-Contaminated Residential Sites Handbook (Handbook, 2003). The sampling depths are specific to investigations for this type of site. Analysis of soil samples may determine whether concentrations of specific pollutants (e.g., lead, barium, cadmium, cobalt, copper, mercury, nickel and zinc) exceed established action levels, or if the concentrations of pollutants present a risk to public health, welfare, or the environment.

These are standard (i.e., typically applicable) operating procedures which may be varied or changed as required, dependent upon site conditions, equipment limitations or limitations imposed by the procedure. In all instances, the actual procedures used should be documented and described in an appropriate site report.

Mention of trade names or commercial products does not constitute U.S. Environmental Protection Agency (EPA) endorsement or recommendation for use.

B. SUMMARY OF METHOD

Soil samples may be collected using a variety of methods and equipment depending on the depth of the desired sample, the type of sample required (disturbed vs. undisturbed), and the soil type. Surface soils may be easily sampled using a spade, trowel, and scoop.

The major category of sites where sampling will be performed includes, but is not limited to active/former lead mining, milling and smelter sites, areas impacted by mining, milling, and smelter activities, mining depositories, transportation routes from mining, milling and smelter sites and the use of mining wastes in public and residential areas.

C. DEFINITIONS

Residential properties: As defined in the Handbook, residential properties are any areas with high accessibility to sensitive populations, and include properties containing single- and multi-family dwellings, apartment complexes, vacant lots in residential areas, schools, day-care centers, community centers, playgrounds, parks, green ways, and any other areas where children may be exposed to site-related contaminated media.

X-Ray Fluorescence (XRF) spectrometer: An instrument used to resolve radiation into spectra to determine measurements. Will be used to analyze soils for metals contamination as described in the Instruction Manual for the XRF spectrometer.

Integrated Exposure Uptake Biokinetic Model (IEUBK) – Predicts blood-lead concentrations (PbBs) for an individual child, or group of similarly exposed children (six months to seven 7 years old), who are exposed to lead in the environment.

D. HEALTH AND SAFETY WARNINGS

Proper health and safety procedures must be observed during the investigation at all times. The Occupational Safety and Health Administration (OSHA) regulation for Hazardous Waste Operations and Emergency Response (HAZWOPER), specified in 29 CFR 1910.120(b)(4), requires a site-specific Health and Safety Plan (HASP) for each site where workers are engaged in handling/operations involving hazardous waste. In compliance with this regulation, all responding Region 7 personnel and their designated representatives are covered by a site-specific HASP developed to address the health and safety hazards, physical and chemical, which may be encountered at each site. The HASP also identifies procedures for protecting employees while on the site.

E. CAUTIONS

This section is not applicable to this SOP.

F. INTERFERENCES

This section is not applicable to this SOP.

G. PERSONNEL QUALIFICATIONS

All field personnel are required to take the 40-hour health and safety training course (as per 29 CFR 1910.120(b)(4)) and regular refresher courses prior to engaging in any field data collection activities.

H. EQUIPMENT AND SUPPLIES

Equipment and supplies used in the field to perform surface soil sampling may include but are not limited to:

- Maps/plot plan
- Safety equipment, as specified in the site-specific Health and Safety Plan
- Survey equipment or global positioning system (GPS) to locate sampling points
- Tape measure
- Survey stakes or flags
- Camera and film
- Stainless steel, plastic, or other appropriate homogenization bucket, bowl or pan
- Appropriate size sample containers
- Ziplock plastic bags
- Logbook
- Labels
- Chain of Custody records and custody seals

- Field data sheets and sample labels
- Decontamination supplies/equipment
- Canvas or plastic sheet
- Spade or shovel
- Spatula
- Scoop
- Plastic or stainless steel spoons
- Trowel(s)
- Continuous flight (screw) auger
- Bucket auger
- Post hole auger
- Extension rods
- T-handle
- Sampling trier
- Thin wall tube sampler
- Split spoons
- Vehimeyer soil sampler outfit
  - Tubes
  - Points
  - Drive head
  - Drop hammer
  - Puller jack and grip
- Shaker sieve #10
- Shaker sieve (initially 250 micron #60 for risk assessment)
- X-Ray Fluorescence (XRF) spectrometer

I. PROCEDURAL STEPS

Soil screening activities will be conducted in accordance with the guidelines established in the Handbook.

1. PREPARATION

- Determine the extent of the sampling effort, the sampling methods to be employed, and the types and amounts of equipment and supplies required.
- Obtain necessary sampling and monitoring equipment.
- Decontaminate or pre-clean equipment, and ensure that it is in working order.
- Prepare schedules and coordinate with staff, client, and regulatory agencies, if appropriate.
- Perform a general site survey prior to site entry in accordance with the site specific Health and Safety Plan.
- Use stakes, flagging, or buoys to identify and mark all sampling locations.

Specific site factors, including extent and nature of contaminant, should be considered when selecting sample location.

2. SAMPLING STRATEGY

The Handbook provides the sampling strategy when sampling residential properties. The sampling strategy is specific to the following categories:

- Residential yards;
- Drip zones;
- Play areas, gardens, and driveways;
- Potable water, lead-based paint, and interior dust; and
- Backfill and waste soil.

Soil sampling will be conducted in accordance with the guidelines established in the Handbook.

3. SAMPLING METHOD

3.1 Sample Collection

The Handbook describes the sampling depth when sampling residential properties. The following has been taken from this document.

Composite samples should consist of discrete aliquots of equal amounts of soil. The soil from each aliquot should be collected into one clean container, such as a stainless steel bowl or plastic bag, and thoroughly mixed. After mixing, the sample can then be analyzed by XRF spectrometer or sent to the laboratory. Remaining sample volume can then be disposed in the general location from where it was collected, or archived, depending on the requirements of the project. In some, cases material other than grass and/or soil will be encountered at a sample location, e.g., wood chips and sand are often found in recreation areas of day-care and school playgrounds. Samples of the soil below the cover material should be collected.

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

This method can be used in most soil types but is limited to sampling at or near the ground surface. Accurate, representative samples can be collected with this procedure depending on the care and precision demonstrated by the sample team member. A flat, pointed mason trowel to cut a block of the desired soil is helpful when undisturbed samples are required. Tools plated with chrome or

other materials should not be used. Plating is particularly common with garden implements such as potting trowels.

3.2 Sample Depth

The Handbook describes the sampling depth when sampling residential properties. Collection of samples from specified depth intervals serves two primary purposes: risk assessment and remedial decision-making. The following has been taken from this document.

3.2.1 Surface Soil Sampling For Risk Assessment Decision Making

With respect to risk assessment, the top inch of soil best represents current exposure to contaminants and is the source of data typically used in the IEUBK model to represent exposure from soil. This sampling should be done at all properties and will be used to determine whether a property exceeds the cleanup criteria and qualifies for response actions.

A five-point composite surface soil samples should be collected from any portion within the 0- to 1-inch depth interval for human health risk assessment purposes. The samples should be collected using the procedure described in Section 3.1. If a measuring device is not used to determine the 1-inch depth, then the spoon or sampling device should sample the upper portion of the 0- to 1-inch interval to avoid going below the 1-inch depth.

3.2.2 Soil Sampling for Cleanup Decisions

The sampling design discussed below is based on the assumption that a minimum of 12-inch soil cover is adequate.

Initial sampling for lead contamination in residential soils should also be conducted to a depth of at least 18 inches, but does not need to exceed 24 inches to define the vertical extent of contamination for cleanup purposes. Composite samples should be collected at 6 inch depth intervals, i.e., 0-6 inches, 6-12 inches, 12-18 inches, and 18-24 inches. Additional sampling may be required at lead sites when contamination is associated with coarse-grained material. Stone-sized material, such as tailings and crushed battery casings, will, over time, migrate upward through the soil via freeze/thaw effects. At such sites, composite sampling should be conducted at 6-inch intervals to the approximate maximum frost depth. In all cases, composites should consist of aliquots collected from the same depth interval.

In site-specific situations, deeper sampling may be conducted to determine the total vertical extent of contamination for groundwater issues or institutional controls (ICs), and to determine if complete removal of contaminated soil is possible. Depth sampling should be conducted until the vertical extent of contamination has been adequately defined, but does not need to be conducted on every property.

3.3 Sample Preparation

The Handbook describes the sampling preparation when sampling residential properties. The following has been taken from this document.

Composite samples should consist of discrete aliquots of equal amounts of soil. The soil from each aliquot should be collected into one clean container, such as a stainless steel bowl or plastic bag, and thoroughly mixed.

Samples collected from all depth intervals should be dried, sieved with a No. 10 sieve (2 mm), and homogenized. Samples should not be ground prior to sieving, as this changes the physical structure of the soil and may bias the analytical results.

For those soil samples that are collected for risk assessment purposes, the sample will also be processed through a No. 60 sieve (250 μm) to obtain the fine fraction. The EPA Technical Review Workgroup (TRW) and American Society for Testing and Materials (ASTM) have issued guidance on sieving (ASTM, 1998; EPA, 2000). To reduce sampling costs, it may be desirable to develop a correlation between sieved and unsieved data, to eliminate the need to sieve all samples. The correlation can be used to predict sieved results from unsieved samples. The EPA TRW guidance addresses appropriate sieve size (No. 60) and a method for predicting the concentration in the fine fraction using concentrations measured in unsieved samples. A portion of each homogenized sample from each sampling area will be screened for lead using XRF spectrometer or submitted for laboratory analysis.

3.4 Sample Analysis

The Handbook describes the sampling analysis when sampling residential properties. The 4220.03A SOP should also be consulted for decision making for using the XRF spectrometer.

J. DATA AND RECORDS MANAGEMENT

Documentation of environmental data collection and analysis procedures (i.e. laboratory documentation, field logbook, photo documentation, chain-of-custody) should be completed and managed using the requirements specified in the Generic Quality Assurance Project Plan for Region 7's Superfund Lead-Contaminated Sites.

K. QUALITY ASSURANCE AND QUALITY CONTROL

There are no specific quality assurance (QA) activities which apply to the implementation of these procedures. However, the following QA procedures apply:

1. All data must be documented on field data sheets or within site logbooks.

2. The XRF spectrometer is not calibrated. Accuracy checks are performed using certified prepared standards daily. Record these accuracy checks in the field logbook. The following information is recorded.

- Equipment identification (name) and control number.
- Date of accuracy check.
- Activity performed on instrument.
- Adjustments made and accuracy of equipment before and following accuracy check (where applicable).
- Record of equipment failure.
- Identification of person performing accuracy.

L. REFERENCES

American Society for Testing and Materials (ASTM). 1998. *Standard Test Method for Particle-Size Analysis of Soils*. D 422-63.

U.S. Environmental Protection Agency (EPA). 2000. *Short Sheet: TRW Recommendations for Sampling and Analysis of Soil at Lead (Pb) Sites*. April. OSWER Publication 9285.7-38. U.S. Environmental Protection Agency. Office of Solid Waste and Emergency Response. EPA Publication EPA/540-F-00-010.

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U.S. Department of Housing and Urban Development (HUD). 1995. *Guidelines for the Evaluation and Control of Lead-Based Paint Hazards in Housing*. June.

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METHOD 5035

CLOSED-SYSTEM PURGE-AND-TRAP AND EXTRACTION FOR VOLATILE ORGANICS IN SOIL AND WASTE SAMPLES

1.0 SCOPE AND APPLICATION

1.1 This method describes a closed-system purge-and-trap process for the analysis of volatile organic compounds (VOCs) in solid materials (e.g., soils, sediments, and solid waste). While the method is designed for use on samples containing low levels of VOCs, procedures are also provided for collecting and preparing solid samples containing high concentrations of VOCs and for oily wastes. For these high concentration and oily materials, sample collection and preparation are performed using the procedures described here, and sample introduction is performed using the aqueous purge-and-trap procedure in Method 5030. These procedures may be used in conjunction with any appropriate determinative gas chromatographic procedure, including, but not limited to, Methods 8015, 8021, and 8260.

1.2 The low soil method utilizes a hermetically-sealed sample vial, the seal of which is never broken from the time of sampling to the time of analysis. Since the sample is never exposed to the atmosphere after sampling, the losses of VOCs during sample transport, handling, and analysis are negligible. The applicable concentration range of the low soil method is dependent on the determinative method, matrix, and compound. However, it will generally fall in the 0.5 to 200 µg/kg range.

1.3 Procedures are included for preparing high concentration samples for purging by Method 5030. High concentration samples are those containing VOC levels of >200 µg/kg.

1.4 Procedures are also included for addressing oily wastes that are soluble in a water-miscible solvent. These samples are also purged using Method 5030..

1.5 Method 5035 can be used for most volatile organic compounds that have boiling points below 200°C and that are insoluble or slightly soluble in water. Volatile, water-soluble compounds can be included in this analytical technique. However, quantitation limits (by GC or GC/MS) are approximately ten times higher because of poor purging efficiency.

1.6 Method 5035, in conjunction with Method 8015 (GC/FID), may be used for the analysis of the aliphatic hydrocarbon fraction in the light ends of total petroleum hydrocarbons, e.g., gasoline. For the aromatic fraction (BTEX), use Method 5035 and Method 8021 (GC/PID). A total determinative analysis of gasoline fractions may be obtained using Method 8021 in series with Method 8015.

1.7 As with any preparative method for volatiles, samples should be screened to avoid contamination of the purge-and-trap system by samples that contain very high concentrations of purgeable material above the calibration range of the low concentration method. In addition, because the sealed sample container cannot be opened to remove a sample aliquot without compromising the integrity of the sample, multiple sample aliquots should be collected to allow for screening and reanalysis.

1.8 The closed-system purge-and-trap equipment employed for low concentration samples is not appropriate for soil samples preserved in the field with methanol. Such samples should be analyzed using Method 5030 (see the note in Sec. 6.2.2).

1.9 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 Low concentration soil method - generally applicable to and soils and other solid samples with VOC concentrations in the range of 0.5 to 200 µg/kg.

Volatile organic compounds (VOCs) are determined by collecting an approximately 5-g sample, weighed in the field at the time of collection, and placing it in a pre-weighed vial with a septum-sealed screw-cap (see Sec. 4) that already contains a stirring bar and a sodium bisulfate preservative solution. The vial is sealed and shipped to a laboratory or appropriate analysis site. The entire vial is then placed, unopened, into the instrument carousel. Immediately before analysis, organic-free reagent water, surrogates, and internal standards (if applicable) are automatically added without opening the sample vial. The vial containing the sample is heated to 40°C and the volatiles purged into an appropriate trap using an inert gas combined with agitation of the sample. Purged components travel via a transfer line to a trap. When purging is complete, the trap is heated and backflushed with helium to desorb the trapped sample components into a gas chromatograph for analysis by an appropriate determinative method.

2.2 High concentration soil method - generally applicable to soils and other solid samples with VOC concentrations greater than 200 µg/kg.

The sample introduction technique in Sec. 2.1 is not applicable to all samples, particularly those containing high concentrations (generally greater than 200 µg/kg) of VOCs which may overload either the volatile trapping material or exceed the working range of the determinative instrument system (e.g., GC/MS, GC/FID, GC/EC, etc.). In such instances, this method describes two sample collection options and the corresponding sample purging procedures.

2.2.1 The first option is to collect a bulk sample in a vial or other suitable container without the use of the preservative solution described in Sec. 2.1. A portion of that sample is removed from the container in the laboratory and is dispersed in a water-miscible solvent to dissolve the volatile organic constituents. An aliquot of the solution is added to 5 mL of reagent water in a purge tube. Surrogates and internal standards (if applicable) are added to the solution, then purged using Method 5030, and analyzed by an appropriate determinative method. Because the procedure involves opening the vial and removing a portion of the soil, some volatile constituents may be lost during handling.

2.2.2 The second option is to collect an approximately 5-g sample in a pre-weighed vial with a septum-sealed screw-cap (see Sec 4) that contains 5 mL of a water-miscible organic solvent (e.g., methanol). At the time of analysis, surrogates are added to the vial, then an aliquot of the solvent is removed from the vial, purged using Method 5030 and analyzed by an appropriate determinative method.

2.3 High concentration oily waste method - generally applicable to oily samples with VOC concentrations greater than 200 µg/kg that can be diluted in a water-miscible solvent.

Samples that are comprised of oils or samples that contain significant amounts of oil present additional analytical challenges. This procedure is generally appropriate for such samples when they are soluble in a water-miscible solvent.

2.3.1 After demonstrating that a test aliquot of the sample is soluble in methanol or polyethylene glycol (PEG), a separate aliquot of the sample is spiked with surrogates and diluted in the appropriate solvent. An aliquot of the solution is added to 5 mL of reagent water in a purge tube, taking care to ensure that a floating layer of oil is not present in the purge tube. Internal standards (if applicable) are added to the solution which is then purged using Method 5030 and analyzed by an appropriate determinative method.

2.3.2 Samples that contain oily materials that are not soluble in water-miscible solvents must be prepared according to Method 3585.

3.0 INTERFERENCES

3.1 Impurities in the purge gas and from organic compounds out-gassing from the plumbing ahead of the trap account for the majority of contamination problems. The analytical system must be demonstrated to be free from contamination under the conditions of the analysis by running method blanks. The use of non-polytetrafluoroethylene (non-PTFE) plastic coating, non-PTFE thread sealants, or flow controllers with rubber components in the purging device must be avoided, since such materials out-gas organic compounds which will be concentrated in the trap during the purge operation. These compounds will result in interferences or false positives in the determinative step.

3.2 Samples can be contaminated by diffusion of volatile organics (particularly methylene chloride and fluorocarbons) through the septum seal of the sample vial during shipment and storage. A trip blank prepared from organic-free reagent water and carried through sampling and handling protocols serves as a check on such contamination.

3.3 Contamination by carryover can occur whenever high-concentration and low-concentration samples are analyzed in sequence. Where practical, samples with unusually high concentrations of analytes should be followed by an analysis of organic-free reagent water to check for cross-contamination. If the target compounds present in an unusually concentrated sample are also found to be present in the subsequent samples, the analyst must demonstrate that the compounds are not due to carryover. Conversely, if those target compounds are not present in the subsequent sample, then the analysis of organic-free reagent water is not necessary.

3.4 The laboratory where volatile analysis is performed should be completely free of solvents. Special precautions must be taken to determine methylene chloride. The analytical and sample storage area should be isolated from all atmospheric sources of methylene chloride, otherwise random background levels will result. Since methylene chloride will permeate through PTFE tubing, all GC carrier gas lines and purge gas plumbing should be constructed of stainless steel or copper tubing. Laboratory workers' clothing previously exposed to methylene chloride fumes during common liquid/liquid extraction procedures can contribute to sample contamination. The presence of other organic solvents in the laboratory where volatile organics are analyzed will also lead to random background levels and the same precautions must be taken.

4.0 APPARATUS AND MATERIALS

4.1 Sample Containers

The specific sample containers required will depend on the purge-and-trap system to be employed (see Sec. 4.2). Several systems are commercially available. Some systems employ 40-mL clear vials with a special frit and equipped with two PTFE-faced silicone septa. Other

systems permit the use of any good quality glass vial that is large enough to contain at least 5 g of soil or solid material and at least 10 mL of water and that can be sealed with a screw-cap containing a PTFE-faced silicone septum. Consult the purge-and-trap system manufacturer's instructions regarding the suitable specific vials, septa, caps, and mechanical agitation devices.

4.2 Purge-and-Trap System

The purge-and-trap system consists of a unit that automatically adds water, surrogates, and internal standards (if applicable) to a vial containing the sample, purges the VOCs using an inert gas stream while agitating the contents of the vial, and also traps the released VOCs for subsequent desorption into the gas chromatograph. Such systems are commercially available from several sources and shall meet the following specifications.

4.2.1 The purging device should be capable of accepting a vial sufficiently large to contain a 5-g soil sample plus a magnetic stirring bar and 10 mL of water. The device must be capable of heating a soil vial to 40°C and holding it at that temperature while the inert purge gas is allowed to pass through the sample. The device should also be capable of introducing at least 5 mL of organic-free reagent water into the sample vial while trapping the displaced headspace vapors. It must also be capable of agitating the sealed sample during purging, (e.g., using a magnetic stirring bar added to the vial prior to sample collection, sonication, or other means). The analytes being purged must be quantitatively transferred to an absorber trap. The trap must be capable of transferring the absorbed VOCs to the gas chromatograph (see 4.2.2).

NOTE: The equipment used to develop this method was a Dynatech PTA-30 W/S Autosampler. This device was subsequently sold to Varian, and is now available as the Archon Purge and Trap Autosampler. See the Disclaimer at the front of this manual for guidance on the use of alternative equipment.

4.2.2 A variety of traps and trapping materials may be employed with this method. The choice of trapping material may depend on the analytes of interest. Whichever trap is employed, it must demonstrate sufficient adsorption and desorption characteristics to meet the quantitation limits of all the target analytes for a given project and the QC requirements in Method 8000 and the determinative method. The most difficult analytes are generally the gases, especially dichlorodifluoromethane. The trap must be capable of desorbing the late eluting target analytes.

NOTE: Check the responses of the brominated compounds when using alternative charcoal traps (especially Vocarb 4000), as some degradation has been noted when higher desorption temperatures (especially above 240 - 250°C) are employed. 2-Chloroethyl vinyl ether is degraded on Vocarb 4000 but performs adequately when Vocarb 3000 is used. The primary criterion, as stated above, is that all target analytes meet the sensitivity requirements for a given project.

4.2.2.1 The trap used to develop this method was 25 cm long, with an inside diameter of 0.105 inches, and was packed with Carbopack/Carbosieve (Supelco, Inc.).

4.2.2.2 The standard trap used in other EPA purge-and-trap methods is also acceptable. That trap is 25 cm long and has an inside diameter of at least 0.105 in. Starting from the inlet, the trap contains the equal amounts of the adsorbents listed below. It is recommended that 1.0 cm of methyl silicone-coated packing (35/60 mesh, Davison, grade 15 or equivalent) be inserted at the inlet to extend the life of the trap. If

the analysis of dichlorodifluoromethane or other fluorocarbons of similar volatility is not required, then the charcoal can be eliminated and the polymer increased to fill 2/3 of the trap. If only compounds boiling above 35°C are to be analyzed, both the silica gel and charcoal can be eliminated and the polymer increased to fill the entire trap.

4.2.2.2.1 2,6-Diphenylene oxide polymer - 60/80 mesh, chromatographic grade (Tenax GC or equivalent).

4.2.2.2.2 Methyl silicone packing - OV-1 (3%) on Chromosorb-W, 60/80 mesh or equivalent.

4.2.2.2.3 Coconut charcoal - Prepare from Barnebey Cheney, CA-580-26, or equivalent, by crushing through 26 mesh screen.

4.2.2.3 Trapping materials other than those listed above also may be employed, provided that they meet the specifications in Sec. 4.2.3, below.

4.2.3 The desorber for the trap must be capable of rapidly heating the trap to the temperature recommended by the trap material manufacturer, prior to the beginning of the flow of desorption gas. Several commercial desorbers (purge-and-trap units) are available.

4.3 Syringe and Syringe Valves

4.3.1 25-mL glass hypodermic syringes with Luer-Lok (or equivalent) tip (other sizes are acceptable depending on sample volume used).

4.3.2 2-way syringe valves with Luer ends.

4.3.3 25-µL micro syringe with a 2 inch x 0.006 inch ID, 22° bevel needle (Hamilton #702N or equivalent).

4.3.4 Micro syringes - 10-, 100-µL.

4.3.5 Syringes - 0.5-, 1.0-, and 5-mL, gas-tight with shut-off valve.

4.4 Miscellaneous

4.4.1 Glass vials

4.4.1.1 60-mL, septum-sealed, to collect samples for screening, dry weight determination.

4.4.1.2 40-mL, screw-cap, PTFE lined, septum-sealed. Examine each vial prior to use to ensure that the vial has a flat, uniform sealing surface.

4.4.2 Top-loading balance - Capable of accurately weighing to 0.01 g.

4.4.3 Glass scintillation vials - 20-mL, with screw-caps and PTFE liners, or glass culture tubes with screw-caps and PTFE liners, for dilution of oily waste samples.

4.4.4 Volumetric flasks - Class A, 10-mL and 100-mL, with ground-glass stoppers.

4.4.5 2-mL glass vials, for GC autosampler - Used for oily waste samples extracted with methanol or PEG.

4.4.6 Spatula, stainless steel - narrow enough to fit into a sample vial.

4.4.7 Disposable Pasteur pipettes.

4.4.8 Magnetic stirring bars - PTFE- or glass-coated, of the appropriate size to fit the sample vials. Consult manufacturer's recommendation for specific stirring bars. Stirring bars may be reused, provided that they are thoroughly cleaned between uses. Consult the manufacturers of the purging device and the stirring bars for suggested cleaning procedures.

4.5 Field Sampling Equipment

4.5.1 Purge-and-Trap Soil Sampler - Model 3780PT (Associated Design and Manufacturing Company, 814 North Henry Street, Alexandria, VA 22314), or equivalent.

4.5.2 EnCore™ sampler - (En Chem, Inc., 1795 Industrial Drive, Green Bay, WI 54302), or equivalent.

4.5.3 Alternatively, disposable plastic syringes with a barrel smaller than the neck of the soil vial may be used to collect the sample. The syringe end of the barrel is cut off prior to sampling. One syringe is needed for each sample aliquot to be collected.

4.5.4 Portable balance - For field use, capable of weighing to 0.01 g.

4.5.5 Balance weights - Balances employed in the field should be checked against an appropriate reference weight at least once daily, prior to weighing any samples, or as described in the sampling plan. The specific weights used will depend on the total weight of the sample container, sample, stirring bar, reagent water added, cap, and septum.

5.0 REAGENTS

5.1 Organic-free reagent water - All references to water in this method refer to organic-free reagent water, as defined in Chapter One.

5.2 Methanol, CH_3OH - purge-and-trap quality or equivalent. Store away from other solvents.

5.3 Polyethylene glycol (PEG), $H(OCH_2CH_2)_nOH$ - free of interferences at the detection limit of the target analytes.

5.4 Low concentration sample preservative

5.4.1 Sodium bisulfate, $NaHSO_4$ - ACS reagent grade or equivalent.

5.4.2 The preservative should be added to the vial prior to shipment to the field, and must be present in the vial prior to adding the sample.

5.5 See the determinative method and Method 5000 for guidance on internal standards and surrogates to be employed in this procedure.

6.0 SAMPLE COLLECTION, PRESERVATION, AND HANDLING

Refer to the introductory material in this chapter, Organic Analytes, Sec. 4.1, for general sample collection information. The low concentration portion of this method employs sample vials that are filled and weighed in the field and never opened during the analytical process. As a result, sampling personnel should be equipped with a portable balance capable of weighing to 0.01 g.

6.1 Preparation of sample vials

The specific preparation procedures for sample vials depend on the expected concentration range of the sample, with separate preparation procedures for low concentration soil samples and high concentration soil and solid waste samples. Sample vials should be prepared in a fixed laboratory or other controlled environment, sealed, and shipped to the field location. Gloves should be worn during the preparation steps.

6.1.1 Low concentration soil samples

The following steps apply to the preparation of vials used in the collection of low concentration soil samples to be analyzed by the closed-system purge-and-trap equipment described in Method 5035.

6.1.1.1 Add a clean magnetic stirring bar to each clean vial. If the purge-and-trap device (Sec. 4.2) employs a means of stirring the sample other than a magnetic stirrer (e.g., sonication or other mechanical means), then the stir bar is omitted.

6.1.1.2 Add preservative to each vial. The preservative is added to each vial prior to shipping the vial to the field. Add approximately 1 g of sodium bisulfate to each vial. If samples markedly smaller or larger than 5 g are to be collected, adjust the amount of preservative added to correspond to approximately 0.2 g of preservative for each 1 g of sample. Enough sodium bisulfate should be present to ensure a sample pH of ≤ 2.

6.1.1.3 Add 5 mL of organic-free reagent water to each vial. The water and the preservative will form an acid solution that will reduce or eliminate the majority of the biological activity in the sample, thereby preventing biodegradation of the volatile target analytes.

6.1.1.4 Seal the vial with the screw-cap and septum seal. If the double-ended, fritted, vials are used, seal both ends as recommended by the manufacturer.

6.1.1.5 Affix a label to each vial. This eliminates the need to label the vials in the field and assures that the tare weight of the vial includes the label. (The weight of any markings added to the label in the field is negligible).

6.1.1.6 Weigh the prepared vial to the nearest 0.01 g, record the tare weight, and write it on the label.

6.1.1.7 Because volatile organics will partition into the headspace of the vial from the aqueous solution and will be lost when the vial is opened, surrogates, matrix spikes, and internal standards (if applicable) should only be added to the vials after the sample has been added to the vial. These standards should be introduced back in the

laboratory, either manually by puncturing the septum with a small-gauge needle or automatically by the sample introduction system, just prior to analysis.

6.1.2 High concentration soil samples collected without a preservative

When high concentration samples are collected without a preservative, a variety of sample containers may be employed, including 60-mL glass vials with septum seals (see Sec. 4.4).

6.1.3 High concentration soil samples collected and preserved in the field

The following steps apply to the preparation of vials used in the collection of high concentration soil samples to be preserved in the field with methanol and analyzed by the aqueous purge-and-trap equipment described in Method 5030.

6.1.3.1 Add 10 mL of methanol to each vial.

6.1.3.2 Seal the vial with the screw-cap and septum seal.

6.1.3.3 Affix a label to each vial. This eliminates the need to label the vials in the field and assures that the tare weight of the vial includes the label. (The weight of any markings added to the label in the field is negligible).

6.1.3.4 Weigh the prepared vial to the nearest 0.01 g, record the tare weight, and write it on the label.

NOTE: Vials containing methanol should be weighed a second time on the day that they are to be used. Vials found to have lost methanol (reduction in weight of >0.01 g) should not be used for sample collection.

6.1.3.5 Surrogates, internal standards and matrix spikes (if applicable) should be added to the sample after it is returned to the laboratory and prior to analysis.

6.1.4 Oily waste samples

When oily waste samples are known to be soluble in methanol or PEG, sample vials may be prepared as described in Sec. 6.1.3, using the appropriate solvent. However, when the solubility of the waste is unknown, the sample should be collected without the use of a preservative, in a vial such as that described in Sec. 6.1.2.

6.2 Sample collection

Collect the sample according to the procedures outlined in the sampling plan. As with any sampling procedure for volatiles, care must be taken to minimize the disturbance of the sample in order to minimize the loss of the volatile components. Several techniques may be used to transfer a sample to the relatively narrow opening of the low concentration soil vial. These include devices such as the EnCore™ sampler, the Purge-and-Trap Soil Sampler ™, and a cut plastic syringe. Always wear gloves whenever handling the tared sample vials.

6.2.1 Low concentration soil samples

6.2.1.1 Using an appropriate sample collection device, collect approximately 5 g of sample as soon as possible after the surface of the soil or other solid material has been exposed to the atmosphere: generally within a few minutes at most. Carefully wipe the exterior of the sample collection device with a clean cloth or towel.

6.2.1.2 Using the sample collection device, add about 5 g (2 - 3 cm) of soil to the sample vial containing the preservative solution. Quickly brush any soil off the vial threads and immediately seal the vial with the septum and screw-cap. Store samples on ice at 4°C.

NOTE: Soil samples that contain carbonate minerals (either from natural sources or applied as an amendment) may effervesce upon contact with the acidic preservative solution in the low concentration sample vial. If the amount of gas generated is very small (i.e., several mL), any loss of volatiles as a result of such effervescence may be minimal if the vial is sealed quickly. However, if larger amounts of gas are generated, not only may the sample lose a significant amount of analyte, but the gas pressure may shatter the vial if the sample vial is sealed. Therefore, when samples are known or suspected to contain high levels of carbonates, a test sample should be collected, added to a vial, and checked for effervescence. If a rapid or vigorous reaction occurs, discard the sample and collect low concentration samples in vials that do not contain the preservative solution.

6.2.1.3 When practical, use a portable balance to weigh the sealed vial containing the sample to ensure that 5.0 ± 0.5 g of sample were added. The balance should be calibrated in the field using an appropriate weight for the sample containers employed (Sec. 4.5.5). Record the weight of the sealed vial containing the sample to the nearest 0.01 g.

6.2.1.4 Alternatively, collect several trial samples with plastic syringes. Weigh each trial sample and note the length of the soil column in the syringe. Use these data to determine the length of soil in the syringe that corresponds to 5.0 ± 0.5 g. Discard each trial sample.

6.2.1.5 As with the collection of aqueous samples for volatiles, collect at least two replicate samples. This will allow the laboratory an additional sample for reanalysis. The second sample should be taken from the same soil stratum or the same section of the solid waste being sampled, and within close proximity to the location from which the original sample was collected.

6.2.1.6 In addition, since the soil vial cannot be opened without compromising the integrity of the sample, at least one additional aliquot of sample must be collected for screening, dry weight determination, and high concentration analysis (if necessary). This third aliquot may be collected in a 60-mL glass vial or a third 40-mL soil sample vial. However, this third vial must *not* contain the sample preservative solution, as an aliquot will be used to determine dry weight. If high concentration samples are collected in vials containing methanol, then two additional aliquots should be collected, one for high concentration analysis collected in a vial containing methanol, and another for the dry weight determination in a vial without either methanol or the low concentration aqueous preservative solution.

6.2.1.7 If samples are known or expected to contain target analytes over a wide range of concentrations, thereby requiring the analyses of multiple sample aliquots, it may be advisable and practical to take an additional sample aliquot in a low concentration soil vial containing the preservative, but collecting only 1-2 g instead of the 5 g collected in Sec. 6.2.1.1. This aliquot may be used for those analytes that exceed the instrument calibration range in the 5-g analysis.

6.2.1.8 The EnCore™ sampler has not been thoroughly evaluated by EPA as a sample storage device. While preliminary results indicate that storage in the EnCore™ device may be appropriate for up to 48 hours, samples collected in this device should be transferred to the soil sample vials as soon as possible, or analyzed within 48 hours.

6.2.1.9 The collection of low concentration soil samples in vials that contain methanol is not appropriate for samples analyzed with the closed-system purge-and-trap equipment described in this method (see Sec. 6.2.2).

6.2.2 High concentration soil samples preserved in the field

The collection of soil samples in vials that contain methanol has been suggested by some as a combined preservation and extraction procedure. However, this procedure is not appropriate for use with the low concentration soil procedure described in this method.

NOTE: The use of methanol preservation has not been formally evaluated by EPA and analysts must be aware of two potential problems. First, the use of methanol as a preservative and extraction solvent introduces a significant dilution factor that will raise the method quantitation limit beyond the operating range of the low concentration direct purge-and-trap procedure (0.5-200 µg/kg). The exact dilution factor will depend on the masses of solvent and sample, but generally exceeds 1000, and may make it difficult to demonstrate compliance with regulatory limits or action levels for some analytes. Because the analytes of interest are volatile, the methanol extract cannot be concentrated to overcome the dilution problem. Thus, for samples of unknown composition, it may still be necessary to collect an aliquot for analysis by this closed-system procedure and another aliquot preserved in methanol and analyzed by other procedures. The second problem is that the addition of methanol to the sample is likely to cause the sample to fail the ignitability characteristic, thereby making the unused sample volume a hazardous waste.

6.2.2.1 When samples are known to contain volatiles at concentrations high enough that the dilution factor will not preclude obtaining results within the calibration range of the appropriate determinative method, a sample may be collected and immediately placed in a sample vial containing purge-and-trap grade methanol.

6.2.2.2 Using an appropriate sample collection device, collect approximately 5 g of sample as soon as possible after the surface of the soil or other solid material has been exposed to the atmosphere: generally within a few minutes at most. Carefully wipe the exterior of the sample collection device with a clean cloth or towel.

6.2.2.3 Using the sample collection device, add about 5 g (2 - 3 cm) of soil to the vial containing 10 mL of methanol. Quickly brush any soil off the vial threads and immediately seal the vial with the septum and screw-cap. Store samples on ice at 4°C.

6.2.2.4 When practical, use a portable balance to weigh the sealed vial containing the sample to ensure that 5.0 ± 0.5 g of sample were added. The balance should be calibrated in the field using an appropriate weight for the sample containers employed (Sec. 4.5.5). Record the weight of the sealed vial containing the sample to the nearest 0.01 g.

6.2.2.5 Alternatively, collect several trial samples with plastic syringes. Weigh each trial sample and note the length of the soil column in the syringe. Use these data to determine the length of soil in the syringe that corresponds to 5.0 ± 0.5 g. Discard each trial sample.

6.2.2.6 Other sample weights and volumes of methanol may be employed, provided that the analyst can demonstrate that the sensitivity of the overall analytical procedure is appropriate for the intended application.

6.2.2.7 The collection of at least one additional sample aliquot is required for the determination of the dry weight, as described in Sec. 6.2.1.6. Samples collected in methanol should be shipped as described in Sec. 6.3, and must be clearly labeled as containing methanol, so that the samples are not analyzed using the closed-system purge-and-trap equipment described in this procedure.

6.2.3 High concentration soil sample not preserved in the field

The collection of high concentration soil samples that are not preserved in the field generally follows similar procedures as for the other types of samples described in Secs. 6.2.1 and 6.2.2, with the obvious exception that the sample vials contain neither the aqueous preservative solution nor methanol. However, when field preservation is not employed, it is better to collect a larger volume sample, filling the sample container as full as practical in order to minimize the headspace. Such collection procedures generally do not require the collection of a separate aliquot for dry weight determination, but it may be advisable to collect a second sample aliquot for screening purposes, in order to minimize the loss of volatiles in either aliquot.

6.2.4 Oily waste samples

The collection procedures for oily samples depend on knowledge of the waste and its solubility in methanol or other solvents.

6.2.4.1 When an oily waste is known to be soluble in methanol or PEG, the sample may be collected in a vial containing such a solvent (see Sec. 6.1.4), using procedures similar to those described in Sec. 6.2.2.

6.2.4.2 When the solubility of the oily waste is not known, the sample should either be collected in a vial without a preservative, as described in Sec. 6.2.3, or the solubility of a trial sample should be tested in the field, using a vial containing solvent. If the trial sample is soluble in the solvent, then collect the oily waste sample as described in Sec. 6.2.2. Otherwise, collect an unpreserved sample as described in Sec. 6.2.3.

6.3 Sample handling and shipment

All samples for volatiles analysis should be cooled to approximately 4°C, packed in appropriate containers, and shipped to the laboratory on ice, as described in the sampling plan.

6.4 Sample storage

6.4.1 Once in the laboratory, store samples at 4°C until analysis. The sample storage area should be free of organic solvent vapors.

6.4.2 All samples should be analyzed as soon as practical, and within the designated holding time from collection. Samples not analyzed within the designated holding time must be noted and the data are considered minimum values.

6.4.3 When the low concentration samples are strongly alkaline or highly calcareous in nature, the sodium bisulfate preservative solution may not be strong enough to reduce the pH of the soil/water solution to below 2. Therefore, when low concentration soils to be sampled are known or suspected to be strongly alkaline or highly calcareous, additional steps may be required to preserve the samples. Such steps include: addition of larger amounts of the sodium bisulfate preservative to non-calcareous samples, storage of low concentration samples at -10°C (taking care not to fill the vials so full that the expansion of the water in the vial breaks the vial), or significantly reducing the maximum holding time for low concentration soil samples. Whichever steps are employed, they should be clearly described in the sampling and QA project plans and distributed to both the field and laboratory personnel. See Sec. 6.2.1.2 for additional information.

7.0 PROCEDURE

This section describes procedures for sample screening, the low concentration soil method, the high concentration soil method, and the procedure for oily waste samples. High concentration samples are to be introduced into the GC system using Method 5030. Oily waste samples are to be introduced into the GC system using Method 5030 if they are soluble in a water-miscible solvent, or using Method 3585 if they are not.

7.1 Sample screening

7.1.1 It is highly recommended that all samples be screened prior to the purge-and-trap GC or GC/MS analysis. Samples may contain higher than expected quantities of purgeable organics that will contaminate the purge-and-trap system**,** thereby requiring extensive cleanup and instrument maintenance. The screening data are used to determine which is the appropriate sample preparation procedure for the particular sample, the low concentration closed-system direct purge-and-trap method (Sec. 7.2), the high concentration (methanol extraction) method (Sec. 7.3), or the nonaqueous liquid (oily waste) methanol or PEG dilution procedure (Sec. 7.4).

7.1.2 The analyst may employ any appropriate screening technique. Two suggested screening techniques employing SW-846 methods are:

7.1.2.1 Automated headspace (Method 5021) using a gas chromatograph (GC) equipped with a photoionization detector (PID) and an electrolytic conductivity detector (HECD) in series, or,

7.1.2.2 Extraction of the sample with hexadecane (Method 3820) and analysis of the extract on a GC equipped with a FID and/or an ECD.

7.1.3 The analyst may inject a calibration standard containing the analytes of interest at a concentration equivalent to the upper limit of the calibration range of the low concentration soil method. The results from this standard may be used to determine when the screening results approach the upper limit of the low concentration soil method. There are no linearity or other performance criteria associated with the injection of such a standard, and other approaches may be employed to estimate sample concentrations.

7.1.4 Use the low concentration closed-system purge-and-trap method (Sec. 7.2) if the estimated concentration from the screening procedure falls within the calibration range of the selected determinative method. If the concentration exceeds the calibration range of the low concentration soil method, then use either the high concentration soil method (Sec. 7.3), or the oily waste method (Sec. 7.4).

7.2 Low concentration soil method (Approximate concentration range of 0.5 to 200 µg/kg - the concentration range is dependent upon the determinative method and the sensitivity of each analyte.)

7.2.1 Initial calibration

Prior to using this introduction technique for any GC or GC/MS method, the system must be calibrated. General calibration procedures are discussed in Method 8000, while the determinative methods and Method 5000 provide specific information on calibration and preparation of standards. Normally, external standard calibration is preferred for the GC methods (non-MS detection) because of possible interference problems with internal standards. If interferences are not a problem, or when a GC/MS method is used, internal standard calibration may be employed.

7.2.1.1 Assemble a purge-and-trap device that meets the specification in Sec. 4.2 and that is connected to a gas chromatograph or a gas chromatograph/mass spectrometer system.

7.2.1.2 Before initial use, a Carbopack/Carbosieve trap should be conditioned overnight at 245°C by backflushing with an inert gas flow of at least 20 mL/minute. If other trapping materials are substituted for the Carbopack/Carbosieve, follow the manufacturers recommendations for conditioning. Vent the trap effluent to the hood, not to the analytical column. Prior to daily use, the trap should be conditioned for 10 minutes at 245°C with backflushing. The trap may be vented to the analytical column during daily conditioning; however, the column must be run through the temperature program prior to analysis of samples.

7.2.1.3 If the standard trap in Sec. 4.2.2.2 is employed, prior to initial use, the trap should be conditioned overnight at 180°C by backflushing with an inert gas flow of at least 20 mL/min, or according to the manufacturer's recommendations. Vent the trap effluent to the hood, not to the analytical column. Prior to daily use, the trap should be conditioned for 10 min at 180°C with backflushing. The trap may be vented to the analytical column during daily conditioning; however, the column must be run through the temperature program prior to analysis of samples.

7.2.1.4 Establish the purge-and-trap instrument operating conditions. Adjust the instrument to inject 5 mL of water, to heat the sample to 40°C, and to hold the sample at 40°C for 1.5 minutes before commencing the purge process, or as recommended by the instrument manufacturer.

7.2.1.5 Prepare a minimum of five initial calibration standards containing all the analytes of interest and surrogates, as described in Method 8000, and following the instrument manufacturer's instructions. The calibration standards are prepared in organic-free reagent water. The volume of organic-free reagent water used for calibration must be the same volume used for sample analysis (normally 5 mL added to the vial before shipping it to the field plus the organic-free reagent water added by the instrument). The calibration standards should also contain approximately the same amount of the sodium bisulfate preservative as the sample (e.g., ~1 g), as the presence of the preservative will affect the purging efficiencies of the analytes. The internal standard solution must be added automatically, by the instrument, in the same fashion as used for the samples. Place the soil vial containing the solution in the instrument carousel. In order to calibrate the surrogates using standards at five concentrations, it may be necessary to disable the automatic addition of surrogates to each vial containing a calibration standard (consult the manufacturer's instructions). Prior to purging, heat the sample vial to 40°C for 1.5 minutes, or as recommended by the manufacturer.

7.2.1.6 Carry out the purge-and-trap procedure as outlined in Secs. 7.2.3. to 7.2.5.

7.2.1.7 Calculate calibration factors (CF) or response factors (RF) for each analyte of interest using the procedures described in Method 8000. Calculate the average CF (external standards) or RF (internal standards) for each compound, as described in Method 8000. Evaluate the linearity of the calibration data, or choose another calibration model, as described in Method 8000 and the specific determinative method.

7.2.1.8 For GC/MS analysis, a system performance check must be made before this calibration curve is used (see Method 8260). If the purge-and-trap procedure is used with Method 8021, evaluate the response for the following four compounds: chloromethane; 1,1-dichloroethane; bromoform; and 1,1,2,2-tetrachloroethane. They are used to check for proper purge flow and to check for degradation caused by contaminated lines or active sites in the system.

7.2.1.8.1 Chloromethane is the most likely compound to be lost if the purge flow is too fast.

7.2.1.8.2 Bromoform is one of the compounds most likely to be purged very poorly if the purge flow is too slow. Cold spots and/or active sites in the transfer lines may adversely affect response.

7.2.1.8.3 Tetrachloroethane and 1,1-dichloroethane are degraded by contaminated transfer lines in purge-and-trap systems and/or active sites in trapping materials.

7.2.1.9 When analyzing for very late eluting compounds with Method 8021 (i.e., hexachlorobutadiene, 1,2,3-trichlorobenzene, etc.), cross-contamination and memory effects from a high concentration sample or even the standard are a common problem.

Extra rinsing of the purge chamber after analysis normally corrects this. The newer purge-and-trap systems often overcome this problem with better bakeout of the system following the purge-and-trap process. Also, the charcoal traps retain less moisture and decrease the problem.

7.2.2 Calibration verification

Refer to Method 8000 for details on calibration verification. A single standard near the mid-point of calibration range is used for verification. This standard should also contain approximately 1 g of sodium bisulfate.

7.2.3 Sample purge-and-trap

This method is designed for a 5-g sample size, but smaller sample sizes may be used. Consult the instrument manufacturer's instructions regarding larger sample sizes, in order to avoid clogging of the purging apparatus. The soil vial is hermetically sealed at the sampling site, and MUST remain so in order to guarantee the integrity of the sample. Gloves must be worn when handling the sample vial since the vial has been tared. If any soil is noted on the exterior of the vial or cap, it must be carefully removed prior to weighing. Weigh the vial and contents to the nearest 0.01 g, even if the sample weight was determined in the field, and record this weight. This second weighing provides a check on the field sampling procedures and provides additional assurance that the reported sample weight is accurate. Data users should be advised on significant discrepancies between the field and laboratory weights.

7.2.3.1 Remove the sample vial from storage and allow it to warm to room temperature. Shake the vial gently, to ensure that the contents move freely and that stirring will be effective. Place the sample vial in the instrument carousel according to the manufacturer's instructions.

7.2.3.2 Without disturbing the hermetic seal on the sample vial, add 5 mL of organic-free reagent water, the internal standards, and the surrogate compounds. This is carried out using the automated sampler. Other volumes of organic-free reagent water may be used, however, it is imperative that all samples, blanks, and calibration standards have exactly the same final volume of organic-free reagent water. Prior to purging, heat the sample vial to 40°C for 1.5 minutes, or as described by the manufacturer.

7.2.3.3 For the sample selected for matrix spiking, add the matrix spiking solution described in Sec. 5.0 of Method 5000, either manually, or automatically, following the manufacturer's instructions. The concentration of the spiking solution and the amount added should be established as described in Sec. 8.0 of Method 8000.

7.2.3.4 Purge the sample with helium or another inert gas at a flow rate of up to 40 mL/minute (the flow rate may vary from 20 to 40 mL/min, depending on the target analyte group) for 11 minutes while the sample is being agitated with the magnetic stirring bar or other mechanical means. The purged analytes are allowed to flow out of the vial through a glass-lined transfer line to a trap packed with suitable sorbent materials.

7.2.4 Sample Desorption

7.2.4.1 Non-cryogenic interface - After the 11 minute purge, place the purge-and-trap system in the desorb mode and preheat the trap to 245°C without a flow

of desorption gas. Start the flow of desorption gas at 10 mL/minute for about four minutes (1.5 min is normally adequate for analytes in Method 8015). Begin the temperature program of the gas chromatograph and start data acquisition.

7.2.4.2 Cryogenic interface - After the 11 minute purge, place the purge-and-trap system in the desorb mode, make sure that the cryogenic interface is at -150°C or lower, and rapidly heat the trap to 245°C while backflushing with an inert gas at 4 mL/minute for about 5 minutes (1.5 min is normally adequate for analytes in Methods 8015). At the end of the 5-minute desorption cycle, rapidly heat the cryogenic trap to 250°C. Begin the temperature program of the gas chromatograph and start the data acquisition.

7.2.5 Trap Reconditioning

After desorbing the sample for 4 minutes, recondition the trap by returning the purge-and-trap system to the purge mode. Maintain the trap temperature at 245°C (or other temperature recommended by the manufacturer of the trap packing materials). After approximately 10 minutes, turn off the trap heater and halt the purge flow through the trap. When the trap is cool, the next sample can be analyzed.

7.2.6 Data Interpretation

Perform qualitative and quantitative analysis following the guidance given in the determinative method and Method 8000. If the concentration of any target analyte exceeds the calibration range of the instrument, it will be necessary to reanalyze the sample by the high concentration method. Such reanalyses need only address those analytes for which the concentration exceeded the calibration range of the low concentration method. Alternatively, if a sample aliquot of 1-2 g was also collected (see Sec. 6.2.1.7), it may be practical to analyze that aliquot for the analytes that exceeded the instrument calibration range in the 5-g analysis. If results are to be reported on a dry weight basis, proceed to Sec. 7.5

7.3 High concentration method for soil samples with concentrations generally greater than 200 µg/kg.

The high concentration method for soil is based on a solvent extraction. A solid sample is either extracted or diluted, depending on sample solubility in a water-miscible solvent. An aliquot of the extract is added to organic-free reagent water containing surrogates and, if applicable, internal and matrix spiking standards, purged according to Method 5030, and analyzed by an appropriate determinative method. Wastes that are insoluble in methanol (i.e., petroleum and coke wastes) are diluted with hexadecane (see Sec. 7.3.8).

The specific sample preparation steps depend on whether or not the sample was preserved in the field. Samples that were <u>not</u> preserved in the field are prepared using the steps below, beginning at Sec. 7.3.1. If solvent preservation was employed in the field, then the preparation begins with Sec. 7.3.4.

7.3.1 When the high concentration sample is <u>not</u> preserved in the field, the sample consists of the entire contents of the sample container. Do not discard any supernatant liquids. Whenever practical, mix the contents of the sample container by shaking or other mechanical means without opening the vial. When shaking is not practical, quickly mix the contents of the vial with a narrow metal spatula and immediately reseal the vial.

7.3.2 If the sample is from an unknown source, perform a solubility test before proceeding. Remove several grams of material from the sample container. Quickly reseal the container to minimize the loss of volatiles. Weigh 1-g aliquots of the sample into several test tubes or other suitable containers. Add 10 mL of methanol to the first tube, 10 mL of PEG to the second, and 10 mL of hexadecane to the third. Swirl the sample and determine if it is soluble in the solvent. Once the solubility has been evaluated, discard these test solutions. If the sample is soluble in either methanol or PEG, proceed with Sec. 7.3.3. If the sample is only soluble in hexadecane, proceed with Sec. 7.3.8.

7.3.3 For soil and solid waste samples that are soluble in methanol, add 9.0 mL of methanol and 1.0 mL of the surrogate spiking solution to a tared 20-mL vial. Using a top-loading balance, weigh 5 g (wet weight) of sample into the vial. Quickly cap the vial and reweigh the vial. Record the weight to 0.1 g. Shake the vial for 2 min. If the sample was not soluble in methanol, but was soluble in PEG, employ the same procedure described above, but use 9.0 mL of PEG in place of the methanol. Proceed with Sec. 7.3.5.

NOTE: The steps in Secs. 7.3.1, 7.3.2, and 7.3.3 must be performed rapidly and without interruption to avoid loss of volatile organics. These steps must be performed in a laboratory free from solvent fumes.

7.3.4 For soil and solid waste samples that were collected in methanol or PEG (see Sec. 6.2.2), weigh the vial to 0.1 g as a check on the weight recorded in the field, add the surrogate spiking solution to the vial by injecting it through the septum, shake for 2 min, as described above, and proceed with Sec. 7.3.5.

7.3.5 Pipet approximately 1 mL of the extract from either Sec. 7.3.3 or 7.3.4 into a GC vial for storage, using a disposable pipet, and seal the vial. The remainder of the extract may be discarded. Add approximately 1 mL of methanol or PEG to a separate GC vial for use as the method blank for each set of samples extracted with the same solvent.

7.3.6 The extracts must be stored at 4°C in the dark, prior to analysis. Add an appropriate aliquot of the extract (see Table 2) to 5.0 mL of organic-free reagent water and analyze by Method 5030 in conjunction with the appropriate determinative method. Proceed to Sec. 7.0 in Method 5030 and follow the procedure for purging high concentration samples.

7.3.7 If results are to be reported on a dry weight basis, determine the dry weight of a separate aliquot of the sample, using the procedure in Sec. 7.5, after the sample extract has been transferred to a GC vial and the vial sealed.

7.3.8 For solids that are not soluble in methanol or PEG (including those samples consisting primarily of petroleum or coking waste) dilute or extract the sample with hexadecane using the procedures in Sec. 7.0 of Method 3585.

7.4 High concentration method for oily waste samples

This procedure for the analysis of oily waste samples involves the dilution of the sample in methanol or PEG. However, care must be taken to avoid introducing any of the floating oil layer into the instrument. A portion of the diluted sample is then added to 5.0 mL of organic-free reagent water, purged according to Method 5030, and analyzed using an appropriate determinative method.

For oily samples that are not soluble in methanol or PEG (including those samples consisting primarily of petroleum or coking waste), dilute or extract with hexadecane using the procedures in Sec. 7.0 of Method 3585.

The specific sample preparation steps depend on whether or not the sample was preserved in the field. Samples that were not preserved in the field are prepared using the steps below, beginning at Sec. 7.4.1. If methanol preservation was employed in the field, then the preparation begins with Sec. 7.4.3.

7.4.1 If the waste was not preserved in the field and it is soluble in methanol or PEG, weigh 1 g (wet weight) of the sample into a tared 10-mL volumetric flask, a tared scintillation vial, or a tared culture tube. If a vial or tube is used instead of a volumetric flask, it must be calibrated prior to use. This operation must be performed prior to opening the sample vial and weighing out the aliquot for analysis.

7.4.1.1 To calibrate the vessel, pipet 10.0 mL of methanol or PEG into the vial or tube and mark the bottom of the meniscus.

7.4.1.2 Discard this solvent, and proceed with weighing out the 1-g sample aliquot.

7.4.2 Quickly add 1.0 mL of surrogate spiking solution to the flask, vial, or tube, and dilute to 10.0 mL with the appropriate solvent (methanol or PEG). Swirl the vial to mix the contents and then shake vigorously for 2 minutes.

7.4.3 If the sample was collected in the field in a vial containing methanol or PEG, weigh the vial to 0.1 g as a check on the weight recorded in the field, add the surrogate spiking solution to the vial by injecting it through the septum. Swirl the vial to mix the contents and then shake vigorously for 2 minutes and proceed with Sec. 7.4.4.

7.4.4 Regardless of how the sample was collected, the target analytes are extracted into the solvent along with the majority of the oily waste (i.e., some of the oil may still be floating on the surface). If oil is floating on the surface, transfer 1 to 2 mL of the extract to a clean GC vial using a Pasteur pipet. Ensure that no oil is transferred to the vial.

7.4.5 Add 10 - 50 µL of the methanol extract to 5 mL of organic-free reagent water for purge-and-trap analysis, using Method 5030.

7.4.6 Prepare a matrix spike sample by adding 10 - 50 µL of the matrix spike standard dissolved in methanol to a 1-g aliquot of the oily waste. Shake the vial to disperse the matrix spike solution throughout the oil. Then add 10 mL of extraction solvent and proceed with the extraction and analysis, as described in Secs. 7.4.2 - 7.4.5. Calculate the recovery of the spiked analytes as described in Method 8000. If the recovery is not within the acceptance limits for the application, use the hexadecane dilution technique in Sec. 7.0 of Method 3585.

7.5 Determination of % Dry Weight

If results are to be reported on a dry weight basis, it is necessary to determine the dry weight of the sample.

NOTE: It is highly recommended that the dry weight determination only be made after the analyst has determined that no sample aliquots will be taken from the 60-mL vial for high

concentration analysis. This is to minimize loss of volatiles and to avoid sample contamination from the laboratory atmosphere. There is no holding time associated with the dry weight determination. Thus, this determination can be made any time prior to reporting the sample results, as long as the vial containing the additional sample has remained sealed and properly stored.

7.5.1 Weigh 5-10 g of the sample from the 60-mL VOA vial into a tared crucible.

7.5.2 Dry this aliquot overnight at 105°C. Allow to cool in a desiccator before weighing. Calculate the % dry weight as follows:

$$\% \text{ dry weight} = \frac{\text{g of dry sample}}{\text{g of sample}} \times 100$$

WARNING: The drying oven should be contained in a hood or vented. Significant laboratory contamination may result from a heavily contaminated hazardous waste sample.

8.0 QUALITY CONTROL

8.1 Refer to Chapter One for specific quality control procedures and Method 5000 for sample preparation QC procedures.

8.2 Before processing any samples, the analyst should demonstrate through the analysis of an organic-free reagent water method blank that all glassware and reagents are interference free. Each time a set of samples is extracted, or there is a change in reagents, a method blank should be processed as a safeguard against chronic laboratory contamination. The blank samples should be carried through all stages of the sample preparation and measurement.

8.3 Initial Demonstration of Proficiency - Each laboratory must demonstrate initial proficiency with each sample preparation and determinative method combination it utilizes, by generating data of acceptable accuracy and precision for target analytes in a clean matrix. The laboratory must also repeat this demonstration whenever new staff are trained or significant changes in instrumentation are made. See Sec. 8.0 of Methods 5000 and 8000 for information on how to accomplish this demonstration.

8.4 Sample Quality Control for Preparation and Analysis - See Sec. 8.0 in Method 5000 and Method 8000 for procedures to follow to demonstrate acceptable continuing performance on each set of samples to be analyzed. These include the method blank, either a matrix spike/matrix spike duplicate or a matrix spike and duplicate sample analysis, a laboratory control sample (LCS), and the addition of surrogates to each sample and QC sample.

8.5 It is recommended that the laboratory adopt additional quality assurance practices for use with this method. The specific practices that are most productive depend upon the needs of the laboratory and the nature of the samples. Whenever possible, the laboratory should analyze standard reference materials and participate in relevant performance evaluation studies.

9.0 METHOD PERFORMANCE

9.1 Single laboratory accuracy and precision data were obtained for the method analytes in three soil matrices, sand, a soil collected 10 feet below the surface of a hazardous landfill, called the

C-Horizon, and a surface garden soil. Each sample was fortified with the analytes at a concentration of 20 ng/5 g, which is equivalent to 4 µg/kg. These data are listed in tables found in Method 8260.

9.2 Single laboratory accuracy and precision data were obtained for certain method analytes when extracting oily liquid using methanol as the extraction solvent. The data are presented in a table in Method 8260. The compounds were spiked into three portions of an oily liquid (taken from a waste site) following the procedure for matrix spiking described in Sec. 7.4. This represents a worst case set of data based on recovery data from many sources of oily liquid.

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TABLE 1

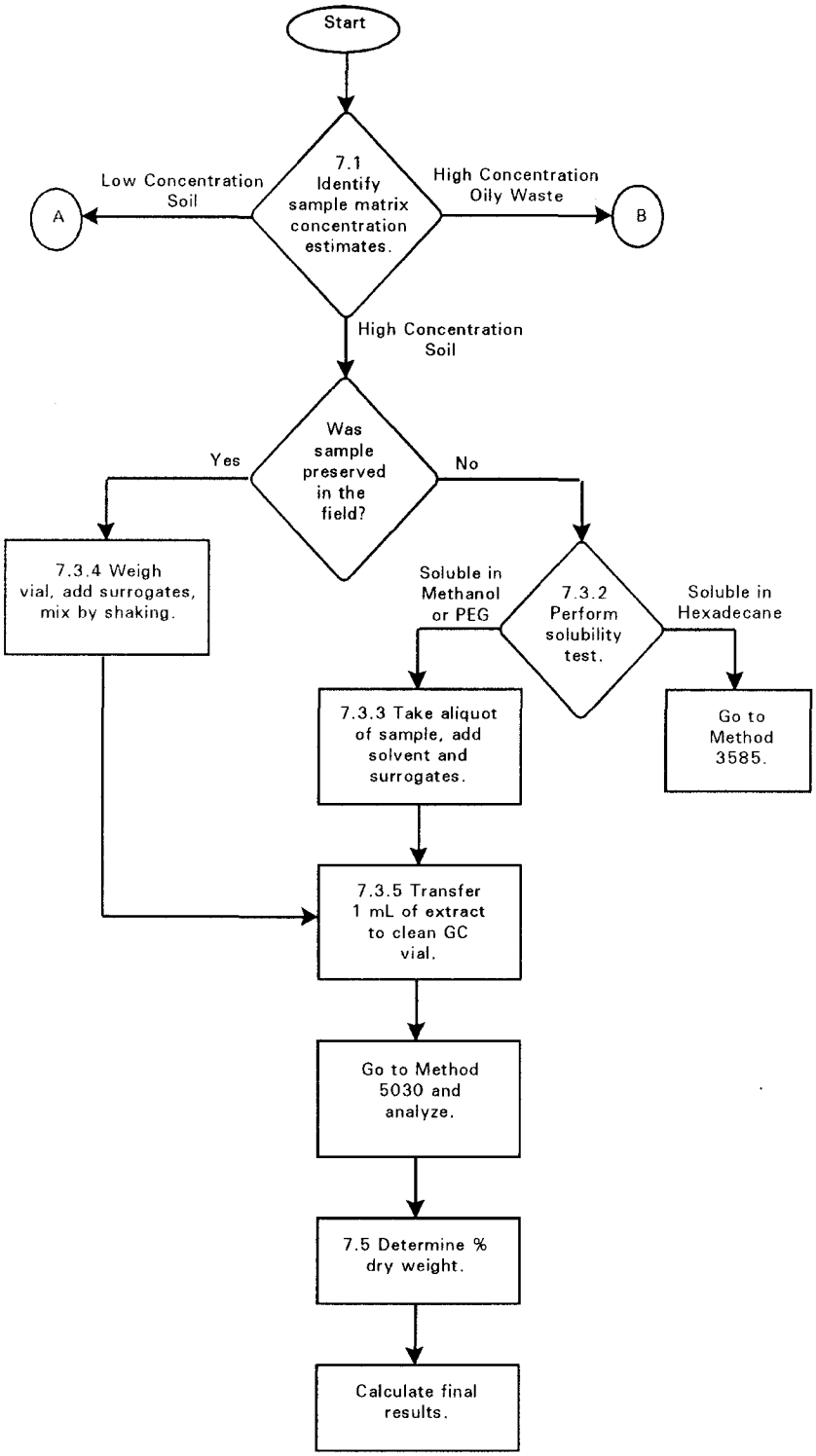
QUANTITY OF METHANOL EXTRACT REQUIRED FOR ANALYSIS OF HIGH CONCENTRATION SOILS/SEDIMENTS

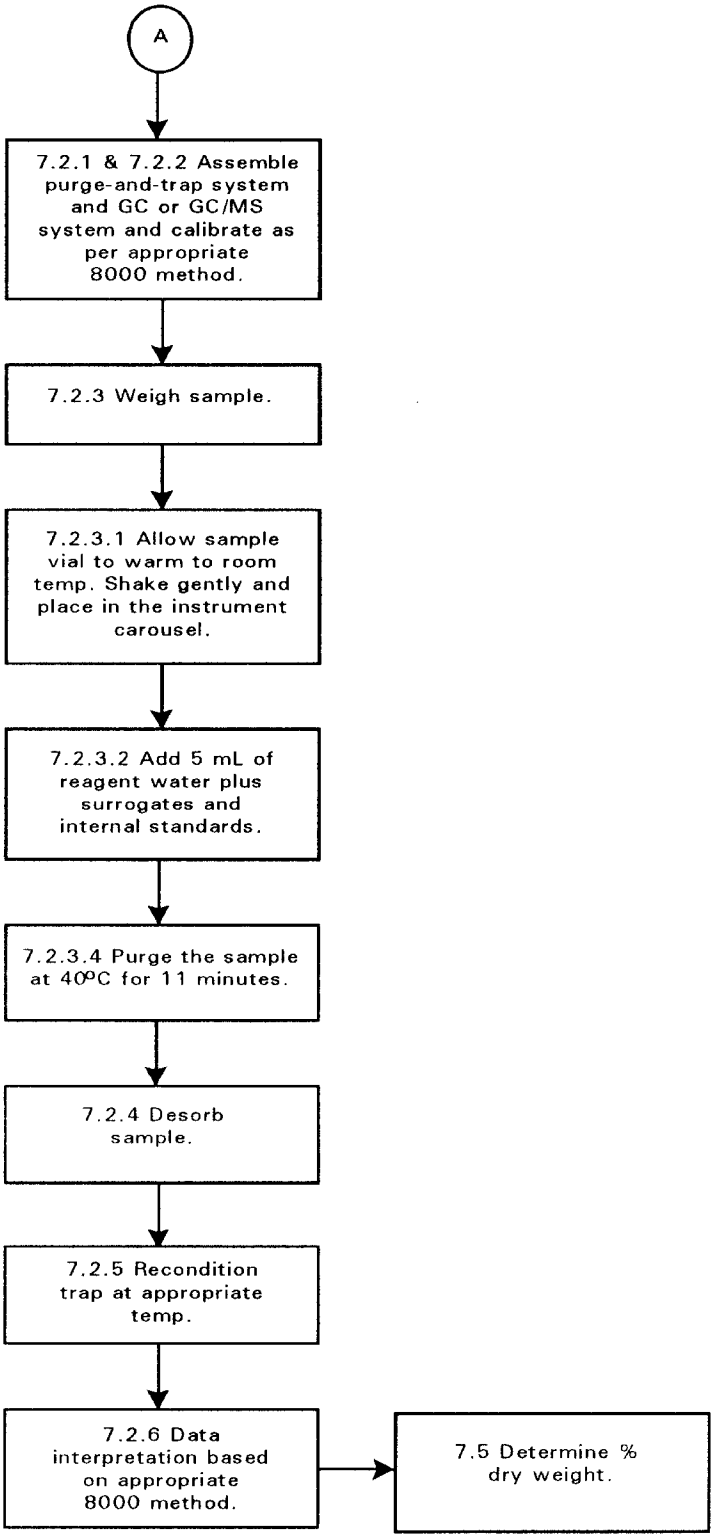
Approximate Concentration Range	Volume of Methanol Extract[a]
500 - 10,000 µg/kg	100 µL
1,000 - 20,000 µg/kg	50 µL
5,000 - 100,000 µg/kg	10 µL
25,000 - 500,000 µg/kg	100 µL of 1/50 dilution[b]

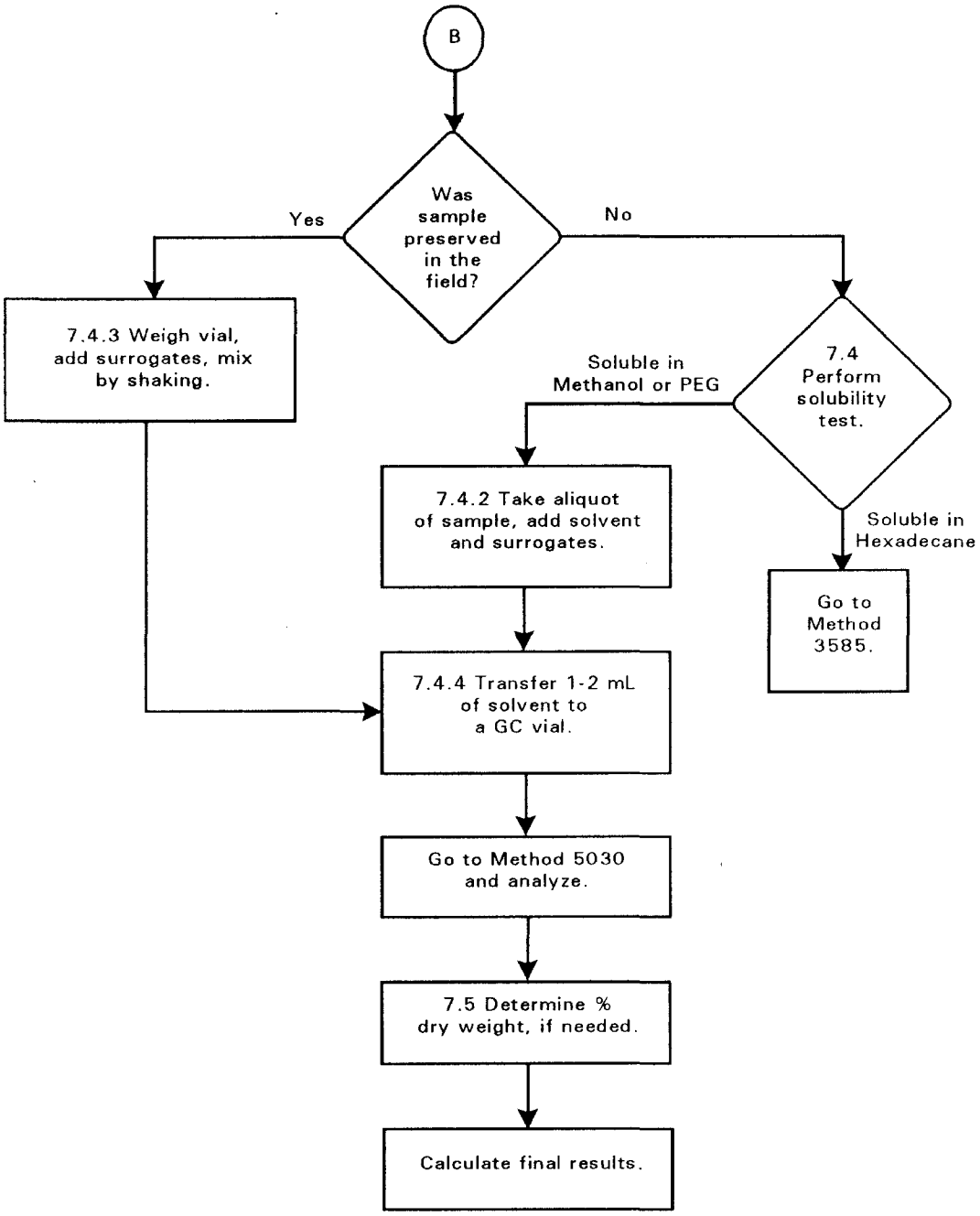
Calculate appropriate dilution factor for concentrations exceeding those in this table.

[a] The volume of methanol added to 5 mL of water being purged should be kept constant. Therefore, add to the 5-mL syringe whatever volume of methanol is necessary to maintain a total volume of 100 µL of methanol.

[b] Dilute an aliquot of the methanol extract and then take 100 µL for analysis.







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

1.0 SCOPE AND APPLICATION

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

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

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

2.0 SUMMARY OF METHOD

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

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

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

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

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

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

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

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

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

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

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

3.0 DEFINITIONS

3.1 FPXRF: Field portable x-ray fluorescence.

3.2 MCA: Multichannel analyzer for measuring pulse amplitude.

3.3 SSCS: Site specific calibration standard.

3.4 FP: Fundamental parameter.

3.5 ROI: Region of interest.

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

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

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

4.0 INTERFERENCES

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

5.0 SAFETY

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

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

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

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

6.0 EQUIPMENT AND SUPPLIES

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

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

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

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

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

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

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

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

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

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

6.2 Spare battery chargers.

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

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

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

6.6 Containers: glass or plastic to store samples.

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

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

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

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

7.0 REAGENTS AND STANDARDS

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

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

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

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

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

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

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

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

8.0 SAMPLE COLLECTION, PRESERVATION, AND STORAGE

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

9.0 QUALITY CONTROL

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

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

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

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

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

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

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

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

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

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

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

The equation for calculating RSD is as follows:

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

where:

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

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

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

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

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

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

10.0 CALIBRATION AND STANDARDIZATION

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

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

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

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

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

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

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

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

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

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

The equation to calibrate %D is as follows:

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

where:

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

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

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

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

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

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

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

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

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

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

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

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

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

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

11.0 PROCEDURE

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

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

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

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

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

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

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

12.0 DATA ANALYSIS AND CALCULATIONS

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

13.0 METHOD PERFORMANCE

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

14.0 POLLUTION PREVENTION

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

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

15.0 WASTE MANAGEMENT

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

16.0 REFERENCES

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

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

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

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

17.0 TABLES, DIAGRAMS, FLOWCHARTS, AND VALIDATION DATA

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

TABLE 1
INTERFERENCE FREE DETECTION LIMITS

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

Source: References 1, 2, and 3

TABLE 2
SUMMARY OF RADIOISOTOPE SOURCE CHARACTERISTICS

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

Source: Reference 1, 2, and 3

TABLE 3
SUMMARY OF X-RAY TUBE SOURCE CHARACTERISTICS

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

Source: Reference 4

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

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

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

Source: Reference 4

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

NR Not reported.

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

TABLE 5
PRECISION

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

Source: Reference 4

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

TABLE 6
PRECISION AS AFFECTED BY SAMPLE PREPARATION

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

Source: Reference 4

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

TABLE 7
ACCURACY

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

Source: Reference 4

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

TABLE 8
ACCURACY FOR TN 9000[a]

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

Source: Reference 4

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

TABLE 9
REGRESSION PARAMETERS FOR COMPARABILITY[1]

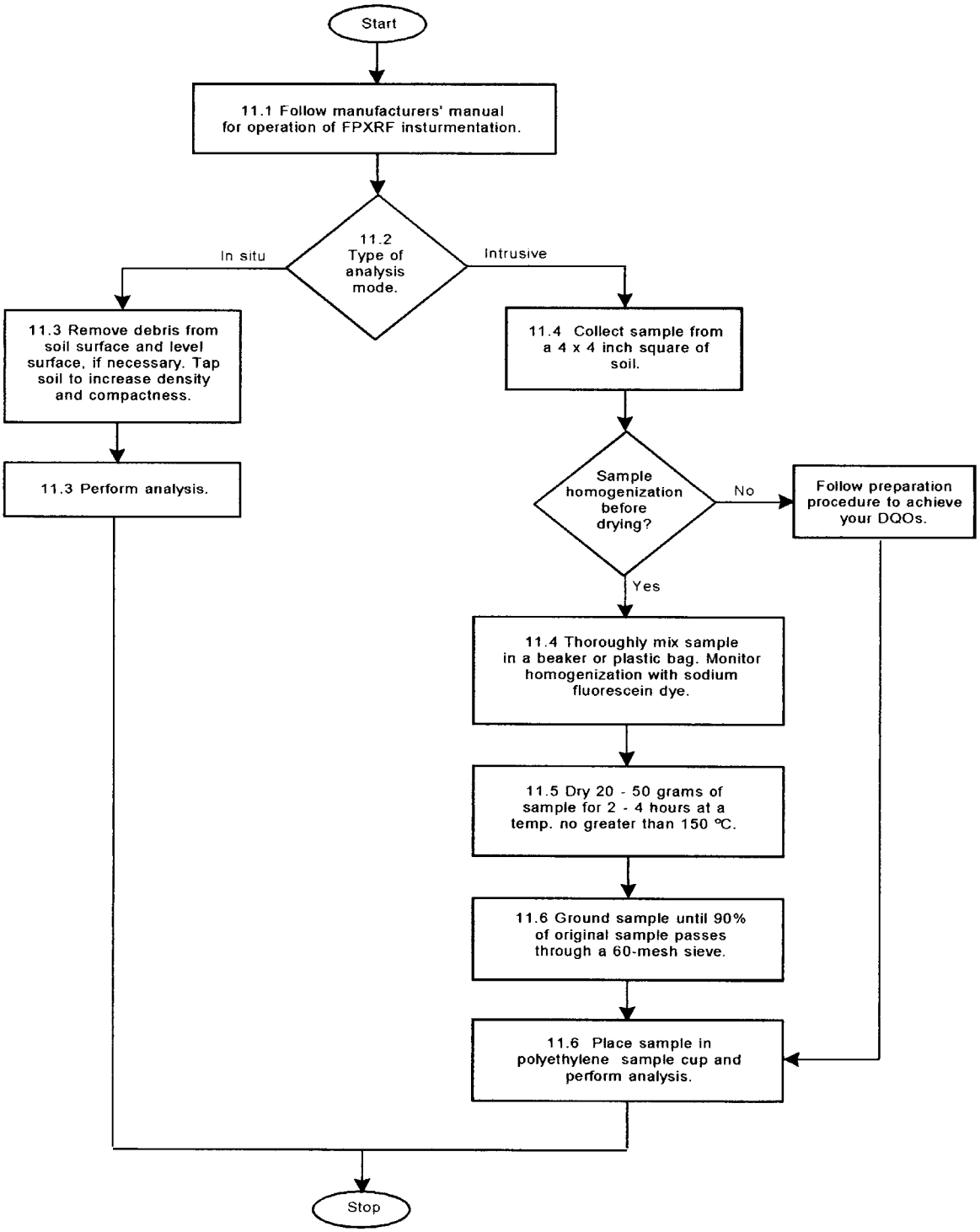
	Arsenic				Barium				Copper			
	n	r^2	Int.	Slope	n	r^2	Int.	Slope	n	r^2	Int.	Slope
All Data	824	0.94	1.62	0.94	1255	0.71	60.3	0.54	984	0.93	2.19	0.93
Soil 1	368	0.96	1.41	0.95	393	0.05	42.6	0.11	385	0.94	1.26	0.99
Soil 2	453	0.94	1.51	0.96	462	0.56	30.2	0.66	463	0.92	2.09	0.95
Soil 3	—	—	—	—	400	0.85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

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

Source: Reference 4

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

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



EPA United States Environmental Protection Agency
Office of Emergency and Remedial Response
OSWER 9285.7-50
August 2003

Superfund Lead-Contaminated Residential Sites Handbook

Final: August 2003

Prepared by the

Environmental Protection Agency
Lead Sites Workgroup (LSW)

NOTICE

This document has been reviewed in accordance with U.S. EPA policy and is approved for publication. Mention of trade names or commercial products does not constitute endorsement or recommendation.

DISCLAIMER

This document provides guidance to EPA Regions concerning how the Agency intends to exercise its discretion in implementing one aspect of the CERCLA remedy selection process. The guidance is designed to implement national policy on these issues.

Some of the statutory provisions described in this document contain legally binding requirements. However, this document does not substitute for those provisions or regulations, nor is it a regulation itself. Thus, it cannot impose legally-binding requirements on EPA, states, or the regulated community, and may not apply to a particular situation based upon the circumstances. Any decisions regarding a particular remedy selection will be made based on the statute and regulations, and EPA decision makers retain the discretion to adopt approaches on a case-by-case basis that differ from this guidance where appropriate.

Interested parties are free to raise questions and objections about the substance of this guidance and the appropriateness of the application of this guidance to a particular situation, and the Agency welcomes public input on this document at any time. EPA may change this guidance in the future.

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CONTENTS

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ACRONYMS

ARARs	Applicable or Relevant and Appropriate Requirements
ASTM	American Society for Testing and Materials
ASTSWMO	Association of State and Territorial Solid Waste Management Officials
ATSDR	Agency for Toxic Substances and Disease Registry
BMPs	Best Management Practices
BRAC	Base Realignment and Closure
CAGs	Community Advisory Groups
CERCLA	Comprehensive Environmental Response, Compensation, and Liability Act
CIC/CIS	Community Involvement Coordinator/ Specialist
DOD	Department of Defense
FOSL	Finding of Suitability to Lease
FOST`	Finding of Suitability to Transfer
FP-XRF	Field-Portable X-Ray Fluorescence
HUD	Department of Housing and Urban Development
IC	Institutional Control
LBP	Lead-Based Paint
IEUBK	Integrated Exposure Uptake Biokinetic Model for Lead in Children
LSCG	Lead Sites Consultation Group
MCL	Maximum Contaminant Level
NCP	National Contingency Plan
NLLAP	National Lead Laboratory Accreditation Program
NTCRA	Non-Time-Critical Removal Action
OSWER	EPA Office of Solid Waste and Emergency Response
PRG	Preliminary Remediation Goal
PRP	Potentially Responsible Party
RCRA	Resource Conservation and Recovery Act
SEP	Supplemental Environmental Project
TAG	Technical Assistance Grant
TCLP	Toxicity Characteristic Leaching Procedure
TCRA	Time-Critical Removal Action
TITLE X	Title X of the Housing and Community Development Act of 1992, 42 U.S.C. 4822
TRW	EPA Technical Review Workgroup
TSCA	Toxic Substances Control Act
UAO	Unilateral Administrative Order

1.0 INTRODUCTION

This Superfund Lead-Contaminated Residential Sites Handbook (subsequently called the Handbook) has been developed by the U.S. Environmental Protection Agency (EPA) to promote a nationally consistent decision-making process for assessing and managing risks associated with lead-contaminated residential sites across the country.

The primary audience for this risk management document is Superfund project managers working on the characterization and cleanup of lead-contaminated residential sites; however, Resource Conservation and Recovery Act (RCRA) project managers may also find it useful. This information was developed primarily for EPA staff, but may prove useful to others working on lead-contaminated residential sites, including states, other federal agencies, tribes, local governments, public interest groups, and private industry. While this Handbook is not intended to apply to lead-contaminated commercial or industrial properties, other non-residential areas, or sites with ecological risks, some of the concepts may be useful for such properties. Addressing lead-contaminated properties at federal facilities requires a different approach, and this Handbook provides a special section (Section 8) on addressing this universe of sites.

Generally, Comprehensive Environmental Response, Compensation, and Liability Act (CERCLA) response actions are undertaken to address a release or threat of release of a hazardous substance such as lead into the environment. Lead contamination found inside homes may be caused by deteriorating lead-based paint (LBP), plumbing, or other sources not resulting from a release into the environment, and therefore may be more appropriately addressed by authorities and programs other than CERCLA (see Appendix A and Section 6.6 of this Handbook). However, it may be appropriate to use CERCLA authorities to conduct sampling and site characterization activities to determine the source of the lead contamination and to differentiate between various site-related sources.

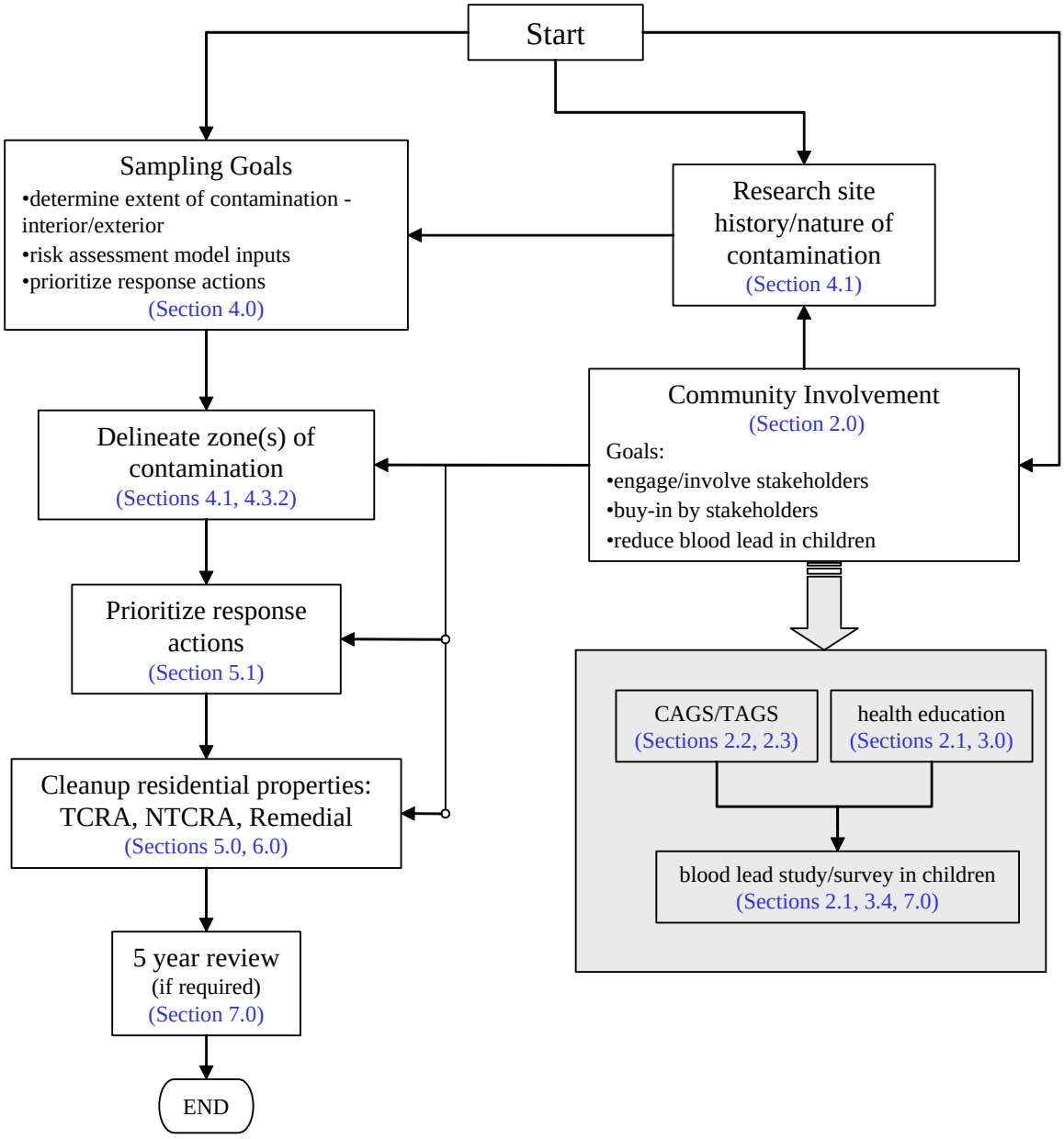
The Handbook lays out only the minimum considerations for addressing lead-contaminated residential sites and encourages users to refer to appropriate agency guidance and/or policy to conduct more stringent investigation and clean-up activities on a site-specific basis, if necessary. In addition, the site manager should determine the applicable and relevant or appropriate requirements (ARARs), including state laws and regulations, that apply to the site. It should also be noted that this Handbook does not, outside the federal facilities universe, apply to lead-contaminated residential sites addressed under Title X (HUD, 1992) procedures.

Lead site characterization and clean-up procedures are unique owing to the ubiquitous nature of lead exposures and the reliance on blood lead concentrations to describe lead exposure and toxicity. Lead

risks are characterized by predicting blood lead levels with computer models and guidance developed by EPA, which are available on the internet: http://www.epa.gov/superfund/programs/lead/products.htm. Major improvements in the removal of lead from gasoline, paint, and food packaging have significantly reduced the incidence of severe lead poisoning. The results of this progress mean that most environmental sources of lead exposure are more likely to cause subtle adverse health effects, primarily behavioral and learning impairments.

An overview to the clean-up process is provided as Figure 1-1. Section numbers are provided in the figure to help the reader locate information within this document.

Figure 1-1. An Overview to the Clean-up Process



1.1 BACKGROUND

Elevated blood lead concentrations in young children in the United States are still prevalent in many areas. Major sources of lead contamination historically included mining and milling sites, primary and secondary smelters, battery manufacturing and recycling facilities, pesticide formulators, pesticide use in orchards, and paint manufacturers (prior to 1978). Many of the source facilities are located near residential areas or have had residential areas develop around them. Fugitive emissions from the facilities have resulted in soil contamination in the yards of residences, which in turn can cause high blood lead levels in children.

Although numerous sites of this type exist, EPA has remediated, or overseen the remediation of, many of these sites and surrounding residences. Many different clean-up methods have been implemented with varying degrees of success. This document is based on the lessons learned from EPA's experience in remediating residential lead sites. It is intended to promote consistency in the characterization and cleanup of lead-contaminated residential sites, while retaining the flexibility needed to respond to different sites and communities to ensure success of the remedy and provide long-term protection of human health. The document also provides guidance on addressing lead sources and media that the Superfund does not usually remediate, such as LBP and lead plumbing. It is anticipated that this information will be periodically updated as we strive to improve our ability to respond to environmental lead hazards.

1.2 GENERAL DISCUSSION ON CERCLA'S APPLICABILITY TO LEAD SITES

This section provides a general discussion of the sections of CERCLA that address lead-contaminated sites. A description of Title X and EPA's Toxic Substances Control Act (TSCA) IV Lead Program is provided in Appendix A. The Title X discussion is provided for informational purposes and is primarily applicable to federal facilities. Section 4.2.5 also provides useful information for LBP and dust sampling.

1.2.1 Background

Historically, the CERCLA has been used as a tool to implement clean-up activities at a large number of sites across the country. CERCLA authorities have been used for cleanups ranging from the removal of drums of hazardous substances from long-abandoned sites, to major privately funded remedial actions at sites on the National Priorities List (NPL).

CERCLA may apply any time there is a release or threatened release of: (1) a hazardous substance into the environment, or (2) a pollutant or contaminant "which may present an imminent and substantial endangerment to the public health or welfare" (EPA, 2000a). The term "release" is defined broadly in the statute and includes discharging or leaking of substances into the environment. This also includes the abandonment of closed containers containing hazardous substances, pollutants, or contaminants.

The definition of hazardous substance is extremely broad, and is defined in CERCLA Section 101(14). A comprehensive list of these substances is provided in 40 CFR 302.4. In addition to general listings for “lead”, “lead and compounds”, and “lead compounds,” the regulation lists fourteen other subcategories of lead.

Additionally, CERCLA is not media-specific. Thus, it may address releases to air, surface water, groundwater, and soils. This multi-media aspect of CERCLA makes it possible to conduct environmental assessments and design clean-up projects that address site contaminants in a comprehensive way.

The Agency has pursued a number of CERCLA response actions involving lead-contaminated soil using the abatement authority under Section 106 (which also requires a showing of imminent and substantial endangerment). CERCLA covers almost every constituent found at mining and mineral processing (primary lead and other metals smelters) sites. Exceptions include petroleum (that is not mixed with a hazardous substance) and, in some cases, responses to releases of a naturally occurring substance in its unaltered form. It should be noted, however, that the latter exception does not include any of the releases typically dealt with at mining sites, such as acid mine drainage, waste rock, or any ore exposed to the elements by man.

1.2.2 Response Authorities

CERCLA's main strength is its response authorities. EPA can either use the Superfund to perform response (removal or remedial) activities (Section 104) or require private parties to perform such activities (Section 106). CERCLA gives EPA the flexibility to clean up sites based upon site-specific circumstances. EPA's clean-up decisions generally are based upon both risk assessment and consideration of ARARs. As long as the jurisdictional prerequisites have been met, CERCLA gives EPA the ability to perform virtually any clean-up activity necessary to protect public health and the environment.

There are potential limitations in CERCLA which may be relevant to lead-contaminated sites. For example, Section 104(a)(3) limits EPA’s ability to respond to releases within residential structures as follows:

> "Limitations on Response. The President (EPA) shall not provide for removal or remedial action under this section in response to a release or threat of release . . . from products which are part of the structure of , and result in exposure within, residential buildings or business or community structures . . . "

The above cited section of CERCLA generally limits EPA's authority to respond to LBP inside a structure or house as written in Section 6.6.1 of this Handbook. However as noted in Section 6.6.1 of the Handbook, EPA has the authority to conduct response actions addressing soils contaminated by a release of lead-contaminated paint chips from the exterior of homes to prevent recontamination of soils that have been remediated. In addition, Section 104(a)(4) provides an exception to the limitations in Section 104(a)(3).

CERCLA provides EPA with the authority to perform "removal" and "remedial" actions. Assessments generally are considered "removal" actions and evaluate contaminants of concern, exposure pathways, and potential receptors. The assessment process includes the review of available information, as well as sampling, to obtain other necessary information. The process is broad in its application and is a powerful tool in evaluating environmental risks posed by a site. Removal actions can be performed on mining and mineral processing (primary lead and other metals smelters) sites, and other sites with lead releases to the environment, of any size. Removal actions are subject to limits on time (12 months) and money ($2,000,000) under the statute; however, these limits are subject to exceptions.

Remedial actions are typically long-term responses performed at those sites placed on the NPL. Remedial actions also may be performed at non-NPL sites, through administrative orders on consent (AOCs) or consent decrees, if they are privately financed. Remedial actions are not subject to the time or dollar limitations imposed on removal actions, but require a more detailed and formal decision process.

1.2.3 Applicable or Relevant and Appropriate Requirements (ARARs)

Under Section 121(d) of CERCLA, remedial actions must comply with substantive provisions of federal environmental laws and more stringent, timely identified state environmental or facility siting laws. Removal actions should comply with ARARs to the extent practicable. "Applicable" requirements are those federal or state laws or regulations that specifically address a hazardous substance, pollutant, contaminant, remedial action, location, or other circumstance found at a CERCLA site. "Relevant and appropriate" requirements are not "applicable," but address problems or situations similar enough to those at the CERCLA site that their use is well suited to the site.

State requirements are not considered ARARs unless they are identified in a timely manner and are more stringent than federal requirements. The recently published TSCA §403 Soil Hazard Rule, which establishes a soil-lead hazard of 400 ppm for bare soil in play areas and 1,200 ppm for bare soil in non-play areas of the yard, should not be treated as an ARAR. As recognized in the TSCA §403 Rule, lead contamination at levels equal to or exceeding the 400 ppm and 1,200 ppm standards may pose serious health risks based upon a site-specific evaluation and may warrant timely response actions. However, the soil-lead hazard levels under the TSCA §403 Rule should not be used to modify approaches to addressing brownfields, NPL sites, state Superfund sites, federal CERCLA removal actions and CERCLA non-NPL facilities.

EPA has published a manual outlining potential federal ARARs that may be requirements at Superfund sites. Published in two parts, the manual is entitled *CERCLA Compliance with Other Laws Manua*l, Part I, August 1988, and Part II, August 1989, and is available at EPA libraries (EPA, 1988).

1.3 DEFINITION AND PURPOSE

Residential properties are defined in the Handbook as any area with high accessibility to sensitive populations, and includes properties containing single- and multi-family dwellings, apartment complexes, vacant lots in residential areas, schools, day-care centers, community centers, playgrounds, parks, green ways, and any other areas where children may be exposed to site-related contaminated media (EPA, 1996a, 1997a, 1998a). This document defines sensitive populations as young children (those under 7 years of age, who are most vulnerable to lead poisoning) and pregnant women. Focus is put on children less than 7 years old because blood lead levels typically peak in this age range (EPA, 1986, 1990a; CDC, 1991). Unfortunately, this age range is also when children are most vulnerable to adverse cognitive effects of lead (Rodder, 1995). Pregnant women are included due to the effects of lead on the fetus (Gayer, 1990; Graziano et al., 1990; Carbone et al., 1998). Other EPA guidance (EPA, 1995a, 2001b) and local zoning regulations should also be consulted prior to determining which properties will be treated as residential.

Lead-contaminated residential sites are defined, for the purposes of this document, as sites where lead is the primary contaminant of concern in residential soils. Generally, lead-contaminated sites contain other metals of concern, such as cadmium and arsenic. This document, while addressing primarily lead contamination, may also be appropriate for use in the remediation of sites contaminated by other metals. In all cases, looking at the site history (type of lead site, depositional environment for the lead contamination, fill activities, previous epidemiological studies, etc.) is important in the use of the Handbook. Typically, the types of sites addressed by the Handbook are sites where the lead contamination has resulted primarily from primary or secondary lead smelting, battery cracking, or

mining and milling operations. Lead paint and dust, along with other sources of lead and other toxic metals, may also be present at these sites.

The Handbook is primarily based on a compilation of the Superfund program knowledge and experiences, as well as existing technical and scientific literature addressing lead-contaminated residential sites. The Handbook has undergone broad review by the Agency for Toxic Substances and Disease Registry (ATSDR), the Association of State and Territorial Solid Waste Management Officials (ASTSWMO), and national and regional EPA offices. Because the Handbook is written for use by CERCLA program staff, there are frequent references to guidance or other documents developed under the Superfund auspices. The Handbook does not supersede or modify any existing EPA guidance or policy. This guidance does not suggest that CERCLA authorities are to be applied at all lead-contaminated residential sites. Rather, these references are provided to the reader as resources to be considered in developing site characterization and clean-up strategies under whatever regulatory or non-regulatory approach is appropriate at a particular site. However, the NCP should be followed and other applicable guidance consulted when addressing lead-contaminated residential sites under CERCLA. The Handbook does not address ecological risks from lead and lead sites.

2.0 COMMUNITY INVOLVEMENT

The sustainability of a residential clean-up project in many ways is contingent upon support from affected residents, elected officials, local public health agencies, municipal and public works staff, state government personnel, and other stakeholders. Few sites impact more citizens of a community than large residential clean-up projects, with many projects exceeding a thousand homes and several thousand residents. If the residents recognize the risks posed to their community and feel involved in the decision-making process, they are more likely to accept the need for cleanup. House-to-house personal interaction with residents can be useful to learn their concerns (or lack of concerns) and can also be an effective part of educating the public regarding risks posed by the site. The project manager should issue bulletins and/or fact sheets to help keep the community informed of site activities and should consider establishing a toll free number for residents to contact her/him with questions about the site. Likewise, without the support of local governments, portions, if not all, of the selected remedy may be more difficult to implement. Many remedies rely in part on health education and institutional controls (ICs) as part of the actions taken to protect human health, both of which may rely on the active participation of local governments and health departments. The following sub-sections provide information on involving the community.

2.1 EDUCATION ACTIVITIES

This section discusses how to involve the local health departments and community in the education activities and the overall benefits and limitations of health education. Section 3 addresses health education activities in detail.

Several studies have shown that a significant short-term reduction in blood lead concentrations can be achieved through the education of the public on the dangers of lead exposure and on methods they can take to limit their exposure (Kimbrough et al., 1994; Hilts et al., 1998; Schultz et al., 1999). However, EPA does not consider health education, as the only action, to be an effective, permanent remedy for Superfund sites (Appendix B). Often, in-home education activities have been combined with regular house cleaning. One key to begin reduction of elevated blood lead concentrations in children is to initiate health education activities, and where appropriate, blood lead screening, as early as possible in the process. These activities should be started as soon as elevated blood lead levels or elevated soil levels are detected at a site. Education should be sustained throughout the project. If residual contamination, such as encapsulated wastes, LBP, or other such potential sources are left on site after completion of the remedy, then education activities should be sustained in perpetuity.

Generally, EPA does not directly conduct the majority of education activities. One of the responsibilities of the project manager is to educate the community on the risks of lead exposure and to coordinate with various health agencies in establishing lead education programs. These programs are often implemented by local health districts that, in turn, typically coordinate with schools and other community groups working with families and children. Initial tasks include educating the community regarding their lead exposure and associated health risks. Typically, a significant amount of effort will be required to explain the rationale and procedures of the EPA risk assessment method for lead, using the Integrated Exposure Uptake Biokinetic Model (IEUBK), and the need to collect data to estimate site-specific values for model parameters. It is advisable to obtain input on exposure parameters specific to the community (e.g., how often they frequent locations that are not residential). Community input into the risk assessment is not relevant to those parameters that require site-specific studies to generate empirical data (e.g., an animal feeding study to determine bioavailability). Often, local health officials will be unfamiliar with EPA's risk assessment process and will benefit from education along with the general public. The need for community education is heightened by the subtle nature of the low-dose adverse health effects of lead, which cannot be diagnosed in an individual because the scientific basis for cognitive impairments caused by low to moderate exposures relies on carefully controlled comparisons of large numbers of children exhibiting a range of blood lead levels (NRC, 1993; Needleman and Bellinger, 2001). Once the public and local health officials are made aware of the potential risks presented by the site, specific programs, discussed in detail in Section 3 (Health Education), can be implemented. Education and clean-up activities should be easier to implement, more effective, and more widely accepted by the community when the citizens understand the risks and believe that the community is at risk.

Integrated Exposure Uptake Biokinetic Model (IEUBK) – Predicts blood-lead concentrations (PbBs) for an individual child, or group of similarly exposed children (6 months to 7 years old), who are exposed to lead in the environment. More information is available from the Technical Review Workgroup for Lead (TRW) web site: http://www.epa.gov/superfund/programs/lead/ieubk.htm

2.2 COMMUNITY ADVISORY GROUPS

Community Advisory Groups (CAGs) can be invaluable in assuring the success of the project (EPA, 1995b). A supporting and active CAG, comprised of a wide cross section of the community, has been demonstrated on several projects to greatly contribute to the success of meeting the remedial goal. Establishing an open dialogue with the CAG

Community Advisory Group (CAG) – Members of the community make up a CAG, which serves as the focal point for the exchange of information among the local community, EPA, the state regulatory agency, and other pertinent federal agencies involved in cleanup of the Superfund site. Additional information is available online: http://www.epa.gov/superfund/tools/cag/index.htm

and understanding and addressing its concerns, leads to increased satisfaction in the community at the completion of the project. Concurrent with the establishment of health education activities, formation of citizens groups should be encouraged at the very onset of the project. Delay in forming the groups until significant progress has occurred may lead to mistrust by the community, as well as delay or loss of the valuable contributions they can make in assisting EPA.

Citizens groups should be representative of the community. Examples include residents, workers, and business owners from affected neighborhoods, as well as minority leaders, realtors, bankers or lending institution officers, school board members, health officials, elected officials, city public works staff, local environmental group members, and other groups in the community. Additionally, the project manager should coordinate with other federal and state agencies to attend citizen group meetings. Relevant agencies may include the ATSDR, HUD, and state health and environmental departments.

Citizens groups can create a feeling of ownership that facilitates the long-term success of the remedy. They can contribute significantly to education activities in numerous ways. A few examples of the successful programs and activities accomplished by citizens groups at sites include: general education and awareness of the segment of the community they individually represent; creating site-specific education material such as coloring/story books; hosting health fairs; creating health education programs for local school districts; establishing lead poisoning prevention merit badges for girl and boy scout organizations; developing instructional videos; and establishing pre- and post-natal education programs at local hospitals.

2.3 EPA'S TECHNICAL ASSISTANCE GRANT PROGRAM

EPA provides assistance grants to communities to help citizens understand site-related information. By regulation, EPA must inform communities about the availability of Technical Assistance Grants (TAGs) and assist them in applying for these grants (EPA, 1992). EPA also informs citizens about obtaining assistance through other programs such as the university-based Technical Outreach Services for Communities program and the Department of Defense's Technical Assistance for Public Participation (TAPP) program.

Under the TAG program, initial grants of up to $50,000 are available to qualified groups affected by a response action. Additional funding is available for unusually large or complex sites. A group applying for a TAG need not be incorporated as a non-profit organization at the time it submits its application, but must incorporate as a non-profit organization before EPA can award the grant.

The group must contribute 20 percent of the total project costs to be supported by the TAG grant. This requirement can be met in a number of ways, including with cash, donated supplies, and volunteered services. TAG groups must prepare a budget and work plan for using the funds. There may be only one TAG award per NPL site. If more than one group applies for the same TAG, they are encouraged to form a coalition to apply for the grant.

TAGs are used to hire a technical advisor, who is an independent expert who can review site-related documents, interpret them, and explain technical or health-related information to community members. A TAG advisor will often make site visits to gain a better understanding of the clean-up activities. A technical advisor can also help communicate the community's concerns to EPA. TAG funds may not be used to generate new data (e.g., to conduct additional sampling) or for lawsuits or other legal actions. For further information on TAGs, see the recently revised TAG regulation (EPA, 2000b), which is available from the EPA TAG web site.

2.4 INFORMATIONAL MEETINGS

As important as the health education activities and the establishment of citizens groups are, the project manager should consider holding frequent public meetings to inform the community of current and planned EPA activities and to collect feedback and concerns from citizens. If a CAG has been formed at the site, meetings with the group should be frequent and open to the general public. It is recommended that in the early phases of the project, information sessions should be held at least monthly. Once the community becomes aware of the site risks, current site activities, and becomes relatively involved in the process, the frequency of the meetings can be reduced. However, it is recommended that public informational meetings, separate from the citizens task force meetings, be conducted at least once every six months. This frequency can help ensure that the public stays informed of site progress and has an opportunity to provide meaningful input to the process.

In addition to the meetings pursuant to CERCLA (e.g., prior to release of the Record of Decision) meetings are helpful at the following points in the process: (1) before sampling is conducted, to explain the reason that lead contamination is suspected, how residents can reduce exposure as a safety precaution while awaiting sampling results, and the overall goals of the project (e.g., if the goal of the project is to reduce exposure by remediating only surface soils and therefore the sampling is designed to evaluate only surface soils, the issue of ICs for any contaminated soils remaining at depth should be discussed with the property owners early in the process); (2) after sampling is conducted, to explain results, reiterate how residents can reduce exposure (if results show elevated levels), explain plans and the schedule for conducting remediation, discuss plans for re-landscaping the property, and discuss what sort of ICs may be appropriate; and (3) after remediation is completed, to explain what was done, provide documentation

of the results of the remediation, discuss any problems with the landscaping, and discuss any applicable ICs.

2.5 COMMUNITY INVOLVEMENT SPECIALIST/COORDINATOR

When the site is large and cleanup is expected to last several years, consideration should be given to housing a full time community involvement specialist/coordinator (CIS/CIC) at the site. The roles of the CIS/CIC are (1) to coordinate community involvement activities, and (2) to be readily accessible to the public to provide information and answer questions concerning site activities. The CIS/CIC should be intimately familiar with all activities at the site, as well as the documented health risks, and should maintain an office with business hours convenient to the public. Additionally, the CIS/CIC can use information gained from their constant contact with the local community to brief project staff on issues important to the successful remediation of the site.

> Community Involvement Specialist/ Coordinator - is the primary point of contact for a community and a Community Advisory Group (CAG), if one was formed for the site. He or she answers questions and provides other assistance directly as well as sees that a CAG's concerns and other issues are transmitted to other Regional Office staff who can help.

3.0 HEALTH EDUCATION

Health education provides information to the public about the risks associated with exposure to contamination and, in turn, how to reduce the exposures. Health education may be considered one of many tools the project manager can use at lead-contaminated sites to reduce exposure to humans.

3.1 APPROPRIATE USES FOR HEALTH EDUCATION

Health education is an informational device and this type of instrument is largely unenforceable. Furthermore, health education has not been demonstrated to be effective over the longer term. Health education may be effective when combined with other measures as an overall remedy for a site. Health education is not a stand-alone remedy. EPA's policy is that health education is only appropriate as a supplemental component of the permanent, health protective remedy selected at a contaminated lead site.

For these reasons, EPA advocates that health education be layered or implemented in series with ICs and engineered remedies. Layering means using different types of ICs and engineered remedies at the same time to enhance the protectiveness of the remedy. Using ICs in series is the use of ICs at different points in the investigation and remediation process to ensure the short- and long-term protection of human health and the environment.

3.2 PLANNING FOR HEALTH EDUCATION

Generally, the specific goals of the health education program should be described in a site-specific decision document. A plan that clearly defines the goals and how they should be achieved is also more likely to succeed. Health education at large lead sites may have a performance period of several years and cost hundreds of thousands of dollars. For these large projects, a clearly defined health education program is even more important.

An early step in any health education planning process includes conducting a community profile and assessing the educational needs of the community. A comprehensive health education program for a typical large lead site would normally attempt to focus on reaching the general public, with special emphasis on schools and other groups involved with young children. Also, it is important to coordinate with city, county, and other local governmental entities. The most important target population, though, is parents, particularly young parents, and parents with a child whose blood lead tested high. Other means of targeted education may include those homes with children that have high dust lead concentrations or lead loadings, which have been shown to be highly predictive of homes where a child is likely to have an elevated blood lead level during the summer peak (EPA, 1996b; von Lindern and Spalinger, 2001).

The response plan should describe what actions and activities are necessary to reach the community-at-large and the targeted groups. It is very important to consider that there are costs associated with the development, implementation, and follow up of health education and that these factors should be thoroughly understood and estimated. Other key points to consider are that the responsibilities for conducting this work should be clear and agreements should be made in writing in the planning stages of site response process.

3.3 EVALUATION OF HEALTH EDUCATION ACTIVITIES

It is important to monitor the effectiveness of health education projects that have been implemented at lead-contaminated sites. Many sites may include health education activities as a major component of the remedy, especially in the early phases of the cleanup. Failure to establish the education part of the remedy may trigger reconsideration and imposition of additional requirements, or more extensive and costly clean-up efforts.

The project manager should monitor the organization(s) performing the educational activities for proper implementation of the health education program and assess the effectiveness of the program. Project managers should ensure that the objectives of the program are being met to protect children's health. If health education is included as part of the final remedy, it should be carefully scrutinized during the Five-Year Review process.

3.4 AGENCY FOR TOXIC SUBSTANCES AND DISEASE REGISTRY (ATSDR) INVOLVEMENT

Health education is often implemented through grants from ATSDR to its partners in state health departments or directly through agreements with local health departments. When health education is specified as a major part of EPA's clean-up activities, strong consideration should be given to establishing an interagency agreement with ATSDR to assist in funding the required activities. ATSDR as a federal health agency is well positioned in terms of health education resources to administer such grants. ATSDR can provide expertise not only with the CAGs but also with public health assessments, health consultations, and health surveillance. An emphasis should be placed on developing the collaborative partnerships between EPA, ATSDR, and other federal, state, and local health departments for health education activities at contaminated lead sites.

Health education at lead sites is often accompanied with blood lead screening. Centers for Disease Control and Prevention (CDC) has issued guidelines for increasing intensity of health intervention activities based on blood lead test results (CDC, 1991). Increased collaboration among the involved

agencies is important to properly implement a health education/blood lead screening project. Additionally, ATSDR and many state and local health departments have ongoing lead screening and health education programs. Information from targeted screening is valuable for (1) targeting follow-up education to individual families with children identified with elevated blood lead levels; (2) determining the areal and demographic extent of the problem; and (3) effectively evaluating the impact of health education.

3.5 OUTREACH

EPA has had success in health education activities at several sites because the programs were tailored specifically for the site by the site team (i.e., project manager, toxicologist, on-scene coordinator, CIS/CIC, etc.). These programs have included significant amounts of outreach activities in the communities. The success of any health education program generally can be attributed to the amount of community outreach that is conducted at the site. As discussed in Section 2, the outreach can consist of a wide variety of activities. A few examples include the following: site specific coloring books distributed to the parents of young children, scouting merit badges on lead-poisoning prevention, school curriculums developed to inform student of the hazards of lead and good hygiene, health and environmental fairs conducted in the community, and blood lead testing events held at community celebrations. Consultation with local health officials and community groups can provide numerous ideas for outreach, which can be incorporated into specific programs to best meet the needs of the community. Typically, the local health officials should lead the outreach efforts. Funding should be provided by EPA when other funds, such as from ATSDR, are unavailable to support the outreach activities.

4.0 SITE CHARACTERIZATION

EPA has reviewed various sampling designs historically employed at lead-contaminated residential sites and assessed the ability of these sampling designs to meet risk assessment needs and support the development of clean-up levels. Over a 20-year period, several large area lead sites (e.g., Bunker Hill, Shoshone County, Idaho; Joplin, Missouri; NL Industries/Taracorp-Granite City, Illinois; Tar Creek, Ottawa County, Oklahoma) have used a variety of sampling techniques to characterize residential properties. Additionally, many different approaches to applying selected clean-up levels have been taken. As stated, this document was developed to promote consistent procedures, criteria and goals in the investigation and clean-up activities at Superfund lead-contaminated residential sites. However, a level of flexibility is needed to best respond to different site conditions, communities, and uncertainties.

The overall goals of the sampling effort are to estimate an average soil lead concentration for risk assessment purposes and to provide information to determine the scope of any required clean-up actions. This information can also be used for public education and intervention. The sampling designs discussed in this section are intended to provide, within one sampling effort, the necessary data for all phases of a clean-up project so that residents are not inconvenienced by repeated sampling of the same property. Project managers should carefully choose the sampling points needed to estimate the average lead concentration in a cost-effective manner. Some uncertainty is acceptable to reduce the overall cost of sampling at large lead sites. The selection of sample locations within areas with potential for exposure has been the subject of recent articles which describe methods to manage decision uncertainty by balancing sampling and clean-up costs (Englund & Heravi, 1994; Crumbling et al., 2001). Table C-1 (Appendix C) lists contacts within the agency who can provide assistance in various aspects of sample planning and design, and also lists software that may be used for sample planning and decision support.

Section 4.0 discusses: (1) delineating the contamination zones; (2) residential property sampling locations; (3) sampling method; (4) sampling requirements for backfill material and excavated soil for off-site disposal.

4.1 CONTAMINANT ZONE DELINEATION

Historical information on site operations and use is crucial for the design of sampling plans that are intended to delineate contaminant zone(s), and for the interpretation of data generated from the sampling effort. In addition to gathering data on the nature of the source of contamination, information should be gathered to identify areas where soils may have been moved or where fill or topsoil may have been placed. Guidance on how to gather historical site data is available (EPA, 2001f, 2001g). Sites that have been contaminated primarily by airborne-derived lead, such as smelter areas, can initially be sampled in a

grid pattern. This will usually allow concentration contours to be defined across the community and to establish the extent of horizontal contamination for cleanup and costing purposes. If grid sampling is used for initial characterization to define the horizontal extent of contamination, follow-up sampling of each yard located within the identified clean-up zone should be used to characterize each individual property for clean-up requirements. For other sites where the variability is expected to be higher, such as mining sites with discrete individual tailings piles located throughout the area, delineating the contaminant zones by establishing concentration contours will be more uncertain and consideration should be given to sampling every home in the potentially affected area, moving laterally away from the source until clean areas of the community have been identified.

Delineating the zone of contamination generally amounts to distinguishing soil with "background" lead concentration from soil that has been impacted by site-related activities. There are basically two types of background: naturally occurring and anthropogenic (see insert for definitions) (EPA, 1989, 1995c, 2002). EPA guidance defines background for inorganics as *"…the concentration of inorganics found in soils or sediments surrounding a waste site, but which are not influenced by site activities or releases"* (EPA, 1995c). Natural background concentrations of lead vary widely with the local geology, and can be as high as 250 ppm or more in mining areas (SRC, 1999). Local background concentrations, which include natural and non-site-related anthropogenic sources (e.g., historic automobile emissions) can be substantially higher. Background samples should be collected from areas near the site that are not influenced by site contamination, but that have the same basic characteristics (e.g., soil type, land use).

Types of Background

naturally occurring: ambient concentrations of lead present in the environment that have not been influenced by humans

anthropogenic: lead concentrations that are present in the environment due to human-made, non-site sources (e.g., automobile exhaust)

Statistical approaches to delineating contaminant zones are useful for some sites. In these cases, the project manager should consult with a statistician to design an efficient sampling plan. The Agency is developing guidance on characterizing background chemicals in soil that includes statistical methods for delineating contaminated areas (EPA, 2001i). Geostatistics is widely recognized for offering graphical methods that are ideally suited for delineating contaminant zones (Gilbert and Simpson, 1983; Flatman and Yfantis, 1984; Journel, 1984; Englund and Heravi, 1994; Goovaerts, 1997). Geostatistics also provides powerful methods for detecting contaminated areas from background when sample locations have not been randomly selected (e.g., Quimby, 1986; Borgman and Quimby, 1996), for sampling plan design (e.g., Flatman and Yfantis, 1984; Borgman et al., 1996), and for aiding in the design of remedial

responses (e.g., Ryti, 1993). For smaller sites, rigorous statistical analyses may be unnecessary because site-related and non-site-related contamination clearly differ. For these sites, the sampling plan should focus on establishing a reliable representation of the extent (in two or three dimensions) of a contaminated area (EPA, 1989) .

4.2 RESIDENTIAL PROPERTIES

For the purposes of this document, a residential property includes properties that contain single and multi-family dwellings, apartment complexes, vacant lots in residential areas, schools, day-care centers, playgrounds, parks, and green ways (EPA, 1996a, 1997a). In all cases, historical site information (type of lead site, fill activities, previous epidemiological studies, etc.) is important in the application of this Handbook.

Rationale for collecting yard soil samples and water samples on a residential property is provided in Table 4-1. The collection of other types of media are important to determine overall risk, however CERCLA has limited authority to address these media (e.g., interior paint, dust, and potable water).

4.2.1 Sampling Access

Prior to conducting any sampling or clean-up activities at a residential property, access must be obtained from the property owner; access obtained from tenants or renters is not sufficient. It is essential to begin access procurement as early as possible in the remedial process to avoid potentially lengthy delays. It is recommended that access be obtained by going door-to-door. If residents are not home, a blank access agreement with instructions for signature and submission to EPA, along with relevant contact information should be left at the residence (but not in the mailbox). Examples of access agreements are presented in Appendix D, pages D-2 and D-3. If possible, access for remediation should be obtained at the same time access for sampling is sought. Examples of combined sampling/remediation access agreements are included on pages D-4 and D-5 of Appendix D. Combining sampling and clean-up access will avoid potentially lengthy delays. Additionally, access should be obtained for any interior dust sampling and/or cleaning that will be performed at the residence (Section 6.6.2). Sample access agreements for dust cleanup are presented in Appendix E.

Table 4-1.
Rationale for Sampling Residential Properties

Sample Location	Rationale for Sample Collection
Residential yard soils	Residential soil may present a direct exposure pathway to persons working, playing, or conducting other recreational activities on the property. Soil samples should be collected and quantitatively analyzed to estimate lead concentrations. Residential soils may also present an indirect exposure pathway via house dust exposure (see below).
Gravel driveways	Fine-grained driveway material may present a direct exposure pathway to persons working or engaged in recreational activities on driveways. Soil samples should be collected and quantitatively analyzed to estimate lead concentrations. Gravel driveways with elevated soil concentrations may also contribute to the transport of contaminants throughout the community.
Drip zones and soils below roof gutter downspouts	Rooftops may collect fine-grained sediments that contain high concentrations of lead. In yard areas where downspouts discharge during a storm event, the fine-grained material washed from a roof may accumulate and result in a localized increase in soil lead concentrations. Soil samples should be collected and quantitatively analyzed to estimate lead concentrations. Drip zone areas may also contain LBP influences and are important to characterize for health intervention purposes, as drip zones are often used as play areas.
Soils in play areas	Play area soils may present a direct exposure pathway to children under the age of seven. Soil samples should be collected and quantitatively analyzed to estimate lead concentrations.
Garden soils	Garden soils may present a direct exposure pathway to persons who actively maintain a garden. Soil samples should be collected and quantitatively analyzed to estimate lead concentrations.
Interior lead dust	Lead in household dust may be a significant contributor to elevated blood lead levels, especially in younger children. Dust samples should be collected and quantitatively analyzed to estimate lead concentrations. Lead-contaminated interior dust can be derived from multiple sources; dust mat samples and speciation can be used to identify lead sources.
Lead-based paint	Deteriorating LBP may contribute lead to household dust, which can be a significant source of lead exposure, particularly for young children. If elevated concentrations of lead are found in interior dust, samples of interior paint should be collected and quantitatively analyzed to estimate lead concentrations. Exterior LBP may contribute to the recontamination of remediated properties. Samples of exterior LBP should be collected and quantitatively analyzed to estimate lead concentrations.
First run and purged tap water	Groundwater and surface water near the site may contain elevated lead concentrations. Some residences located within the site may use local groundwater or nearby surface water as a source of drinking, cooking, bathing, or irrigation water. The water may represent a direct exposure or ingestion pathway. Samples of both water standing in the pipes (first run sample) and water discharged after the system has been flushed (purged sample) should be collected and quantitatively analyzed to estimate lead concentrations. These results can also be used to help determine if the drinking water is contaminated with site-related contamination (exceedance in purged), or to determine if there is lead in the home's plumbing (exceedance in first run), or both, which may be used for remediation or intervention purposes, respectively.
Crawl Spaces	Crawl space sampling is recommended if the crawl space is accessible to children or pets. At some sites (e.g., Bunker Hill) this has been found to be a significant pathway (IDHW, 2000; TerraGraphics, 2000). Even when spaces are too small for children, pets have been found to access these spaces and move significant amounts of fine dust containing elevated lead levels into the child's bedroom (e.g., where a pet may sleep on the child's bed at night). Information on concentrations of lead beneath the structure may be used to document the need to preclude access or take other remedial measures.
Other areas	During field work, other potential sources of lead contamination may be identified. If the sources appear to represent a potential exposure pathway to occupants of a residence, sampling may be recommended. Other areas should be evaluated on a case-by-case basis and could include sediment, surface water, or secondary play areas. If deemed appropriate, samples should be collected and quantitatively analyzed to estimate lead concentrations.

4.2.2 Residential Yards

It is recommended that when sampling residential lots with a total surface area less than 5,000 square feet (a typical urban lot size), five-point composite samples should, at a minimum, be collected from each of the following locations: the front yard, the back yard, and the side yard (if the size of the latter is substantial). The front, back, and side (if needed) yard composites should be equally spaced within the respective portion of the yard, and should be outside of the drip zone and away from influences of any other painted surfaces (Figures 4-1a and 4-1b). Composites should consist of aliquots collected from the same depth interval.

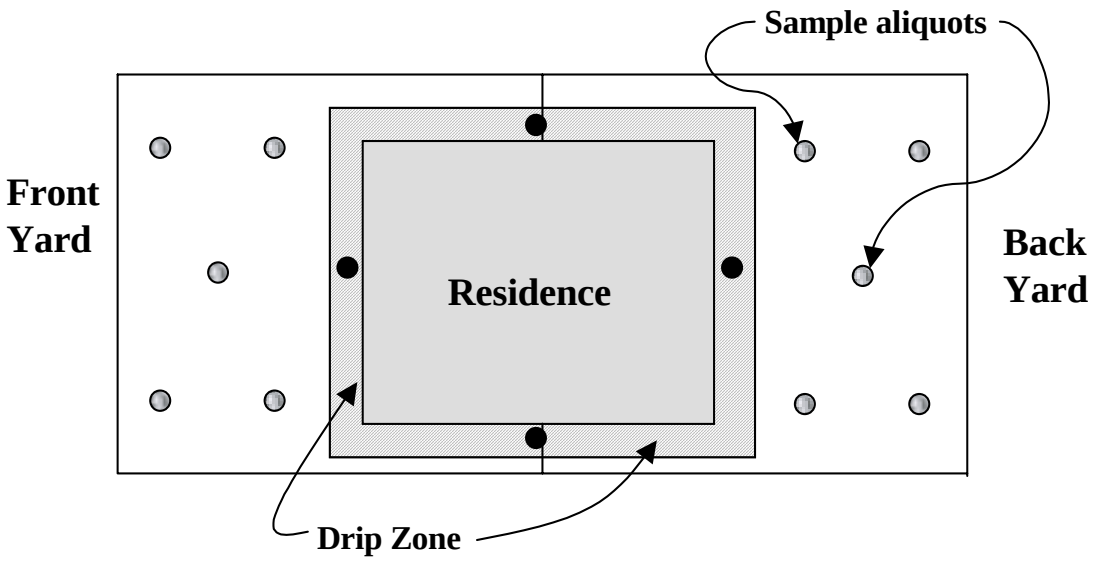


Figure 4-1a. Recommended minimum soil sampling in yards less than or equal to 5,000 square feet with small side yard. Five point composite samples should be collected from each of the front and back yards. Four point composites should be collected from the drip zone; each aliquot should generally be collected from the midpoint along each side of the residence. Aliquots for a single composite sample should be collected from the same depth interval. Soil samples should also be collected from distinct play areas and gardens if they are present, as well as unpaved driveways and minimal use areas such as areas under porches and crawl spaces. The locations of the aliquots should be equally spaced within the area of the yard the composite is collected from. The figure illustrates one possible arrangement of the sample aliquots. Please refer to Section 4.2.2 for further explanation.

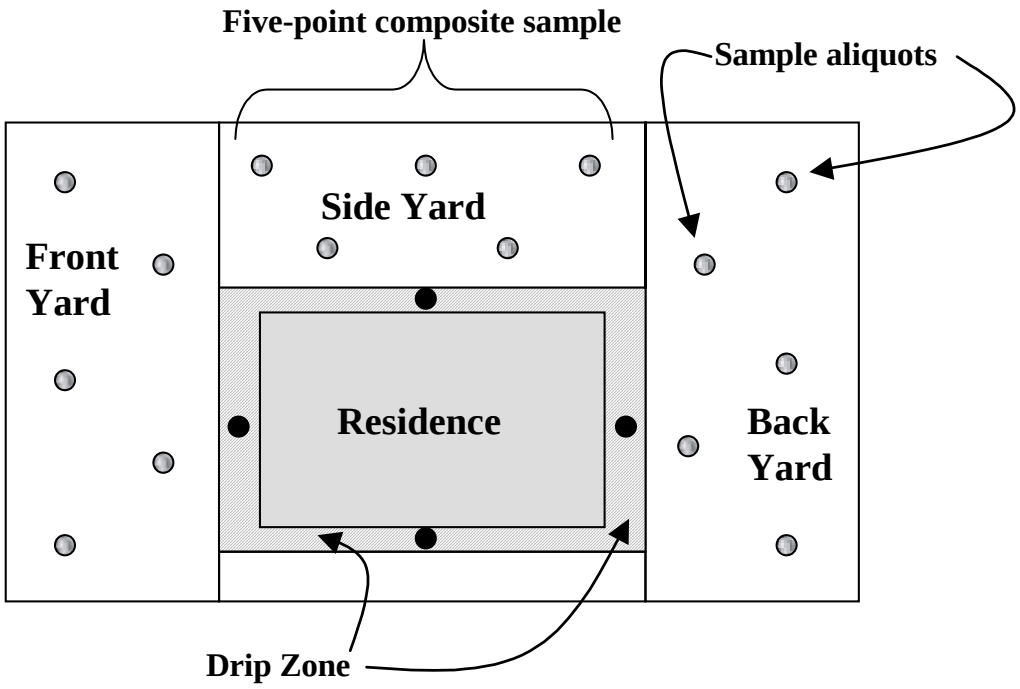


Figure 4-1b. Recommended minimum soil sampling in yards less than or equal to 5,000 square feet with substantial side yard. Five point composite samples should be collected from each of the front, back, and side yards, along with other areas as described in Figure 4-1a. The locations of the aliquots should be equally spaced within the area of the yard the composite is collected from. The figure illustrates one possible arrangement of the sample aliquots Aliquots for a single composite sample should be collected from the same depth interval. Please refer to Section 4.2.2 for further explanation.

For residential lots with a total surface area greater than 5,000 square feet, it is advisable that the property be divided into four quadrants of roughly equal surface area. The two quadrants in the front yard should encompass one half of the side yard; likewise for the two quadrants in the back yard. One five-point composite of aliquots collected at equal spacing and from the same depth interval should be obtained from each quadrant. Each aliquot should be collected away from influences of the drip zone and any other painted surfaces (Figure 4-2).

Properties over one acre in size should be divided into 1/4 acre sections. One five-point composite sample should be collected from each section. For large properties, consideration should be given to whether elevated concentrations trigger partial removal of soils or access restriction (see Section 6.5).

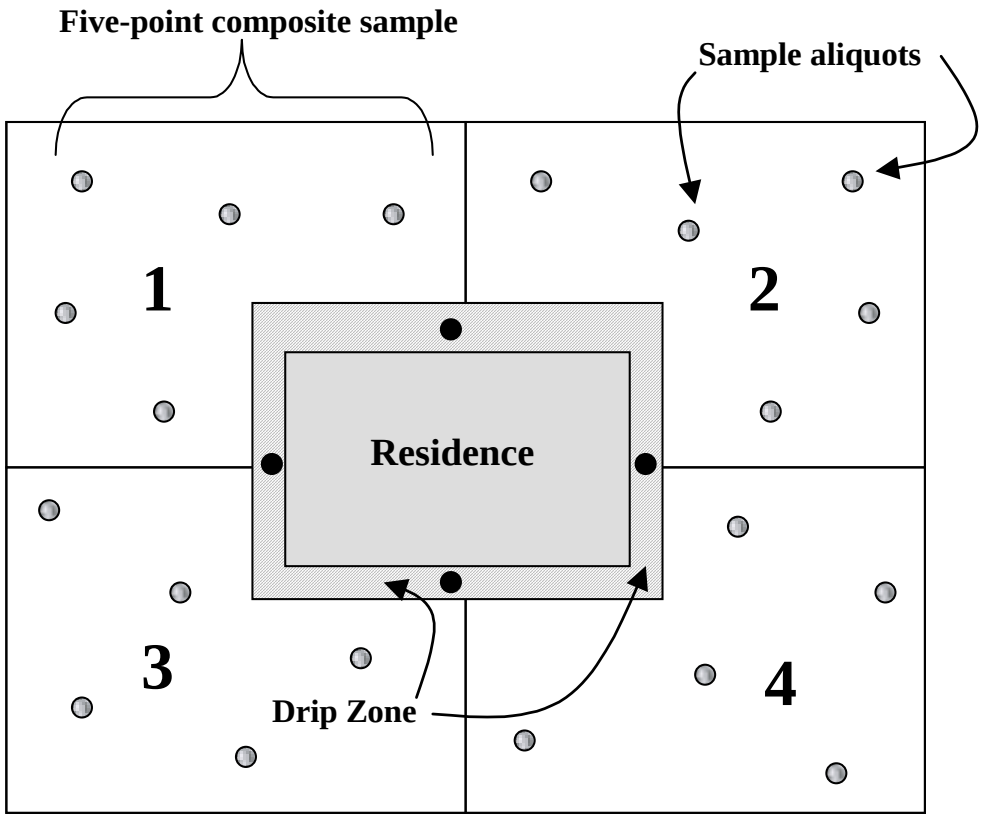


Figure 4-2. Recommended minimum soil sampling in yards greater than 5,000 square feet. Five point composite samples should be collected from each of the four quadrants as indicated above. The locations of the aliquots should be equally spaced within each of the quadrants. The figure illustrates one possible arrangement of the sample aliquots. Four point composites should be collected from the drip zone; each aliquot should generally be collected from the midpoint along each side of the residence. Aliquots for a single composite sample should be collected from the same depth interval. Additional samples should be collected from distinct play areas and gardens if they are present, as well as unpaved driveways and minimal use areas such as areas under porches and crawl spaces. Please refer to Section 4.2.2 for further explanation.

4.2.3 Drip Zones

Lead-contaminated soils are frequently found within the drip zone of houses. It is recommended that a four-point composite sample be collected from the drip zone of each residential property (Figures 4-1a, 4-1b, and 4-2). The composite sample (taken from any size lot) should consist of a minimum of four aliquots collected between 6 and 30 inches from the exterior walls of the house. Each aliquot should generally be collected from the midpoint of each side of the house. Collection of additional aliquots should be considered if other factors exist, such as bare spots, distinct differences in the house exterior, and areas where runoff collects. Rooftops may collect fine-grained sediments that contain high concentrations of lead. In yard areas where downspouts discharge during a storm event, the fine-grained material washed from a roof may accumulate and result in a localized increase in soil lead

concentrations. Samples of the soil from the downspout discharge area should also be sampled if present.

4.2.4 Play Areas, Gardens, and Driveways

Distinct play areas and gardens, if present, should generally be sampled separately as discrete areas of the yard. At some sites, collection of a right-of-way/easement composite may also be appropriate, such as residential areas with unpaved streets and alleys. Paved surfaces such as asphalt/concrete driveways, patios, alleys, and parking lots should, in most cases, not be sampled. Samples should also be collected in other locations depending upon the potential for exposure or recontamination, for example, under porches and crawl spaces and areas with incomplete barriers such as gravel driveways.

4.2.5 Potable Water, Lead-Based Paint and Interior Dust

Drinking water supply samples should be collected to determine if exposure to lead in drinking water is occurring. First-run and purged samples of potable water should be collected to differentiate site-related sources of lead from lead derived from plumbing that is located within the residence. CERCLA authority for remedial action may be limited with regard to lead derived from plumbing that is located within the residence.

Deteriorating LBP may contribute lead to household dust. If elevated concentrations of lead are found in interior dust, samples of interior paint should be collected. Exterior LBP may contribute to the recontamination of remediated properties (Section 6.7). Samples of exterior LBP should be collected and analyzed to estimate lead concentrations. Lead in household dust may be a significant contributor to elevated blood lead levels, especially in younger children. Lead-contaminated interior dust can be derived from multiple sources; dust mat samples and speciation can be used to identify lead sources. Dust samples should be collected and analyzed to estimate its potential contribution to lead exposure. Guidance on LBP and dust sampling is available from HUD (HUD, 1995).

4.2.6 Backfill and Waste Soil

Backfill soil should be sampled to ensure that uncontaminated material is being placed on the site. The list of analytes and the frequency of sampling should be based on site-specific factors including the location of the source for the backfill material relative to potential sources of contamination, the geology of the borrow area, and the heterogeneity of the material. For example, on the Bunker Hill Superfund Site, four-point composite samples were collected for each 200 yd^3 of soil (TerraGraphics, 1997a). Gravel for driveway backfill was also sampled every 200 yd^3 (TerraGraphics, 1997b). Samples of excavated soil should be analyzed by the toxicity characteristic leaching procedure (TCLP) method to

determine the appropriate method of disposal. The frequency required for TCLP sampling should be based on the heterogeneity of the lead and other contaminant(s), if any, on the site.

4.3 SAMPLING METHOD AND ANALYSIS

4.3.1 Sample Collection

Composite samples should consist of discrete aliquots of equal amounts of soil. The soil from each aliquot should be collected into one clean container, such as a stainless steel bowl or plastic bag, and thoroughly mixed. After mixing, the sample can then be analyzed by X-Ray Fluorescence (XRF) (see Section 4.3.4) or sent to the laboratory. Remaining sample volume can then be disposed in the general location from where it was collected, or archived, depending on the requirements of the project. In some cases, material other than grass and/or soil will be encountered at a sample location, e.g., wood chips and sand are often found in recreational areas of day-care and school playgrounds. Samples of the soil below the cover material should be collected.

The use of a dynamic sampling and analysis strategy should be considered (EPA, 2001d). A dynamic sampling and analysis strategy takes full advantage of the real-time that data field analytical methods provide, which can limit the sampling effort and minimize cost (EPA, 2001d). This document suggests the use of field portable X-Ray Fluorescence (FP-XRF) analysis.

4.3.2 Sample Depth

The following sampling design is based on the assumption that removal of surficial contaminated soils and placement of a cover of clean soil will be protective of human health and the environment (see Section 4.0). Furthermore, the sampling design outlined below is based on the assumption that a minimum of 12 inch soil cover is adequate.

Initial sampling for lead contamination in residential soils should be conducted to a depth of at least 18 inches, but does not need to exceed 24 inches to define the vertical extent of contamination for clean-up purposes. Composite samples should be collected at 6 inch depth intervals, i.e., 0–6 inches, 6–12 inches, 12–18 inches, and 18–24 inches. Additional sampling may be required at lead sites in cold weather regions when contamination is associated with coarse grained material. Stone-sized material, such as tailings and crushed battery casings, will, over time, migrate upward through the soil via freeze/thaw effects. At such sites, composite sampling should be conducted at 6 inch intervals to the approximate maximum frost depth for the region. In all cases, composites should consist of aliquots collected from the same depth interval.

In site-specific situations, deeper sampling may be conducted to determine the total vertical extent of contamination for groundwater issues or ICs, and to determine if complete removal of contaminated soil is possible. Depth sampling should be conducted until the vertical extent of contamination has been adequately defined, but does not need to be conducted on every property.

In addition to the composite samples collected to define the vertical extent of contamination, five-point composite surface soil samples should be collected from 0 to 1 inch for human health risk assessment purposes (EPA, 1989, 1996c). The samples should be collected using the procedure described in Section 4.3.1. These surface soil samples should be collected from every property within the identified zone of contamination; however, after collecting a statistically valid number of both 0–1" and 1–6" samples, the project manager may want to compare both sample horizons (e.g., paired-sample t-test; Wilcoxon Rank Sum test) (Gilbert, 1987; Snedecor and Cochran, 1989) to determine if the 0–1" depth can be eliminated (i.e., sample from 0–6"), to further decrease sampling costs. This may be particularly useful at mine waste sites where contamination often extends to depth or at sites where lead-contaminated soil has been used as fill material; in such cases, the lead concentration may increase with depth. Conversely, the 0–1" horizon may be far more contaminated than the 1–6" at smelter sites, making individual horizon sampling crucial to remedial decision-making.

Collection of samples from specified depth intervals serves two primary purposes: risk assessment and remedial decision-making. With respect to risk assessment, the top inch of soil best represents current exposure to contaminants (EPA, 1989, 1996c) and is the source of data used in the IEUBK model to represent exposure from soil. The various depth intervals are used in remedial decision-making to determine if a residential yard requires cleanup by evaluating if any of the horizons exceed the site-specific action level. The lower soil horizons represent possible future exposures, such as homeowner projects, children’s play areas, and other home activities that periodically go beneath the top inch of vegetation/soil (EPA, 1989). All soil horizons should be used for clean-up decision-making. The 6 inch depth intervals recommended in this document are based on the performance that may be reasonably expected of operators of small equipment working in relatively small spaces around homes. Specifically, a “bobcat” is most efficiently used for soil removal on a property if the soil is removed in 6 inch intervals, rather than in smaller increments, which would be far more difficult to achieve in a consistent or cost-effective manner. This approach has been developed to ensure a residential yard is cleaned up if it poses an immediate or long-term risk to human health in a manner that relates the sampling methodology closely to reasonable and cost-effective construction equipment performance.

A secondary goal of the sample collection effort is to facilitate the implementation of ICs for sites where contamination at depth is left in place.

4.3.3 Sample Preparation

Residential soil lead samples should represent the exposure potential of young children who are most vulnerable to adverse effects of exposure. Children inadvertently ingest lead in soil and dust that adheres to their hands (Succop et al., 1998). The smaller particles are more representative of this type of exposure (Duggan et al., 1985; Kissel et al., 1996; Mielke et al., 1997). Additionally, smaller particles are preferentially brought into the home. Sieving is conducted to better represent the soil fraction that is ingested by the typical child. Sieving has also been used in soil ingestion and bioavailability studies (Calabrese et al., 1996; Casteel et al., 1997; Stanek et al., 1999). Samples collected from all depth intervals should be sieved. Samples should not be ground prior to sieving, as this changes the physical structure of the soil and may bias the analytical results. To reduce sampling costs, it may be desirable to develop a correlation between sieved and unsieved data, to eliminate the need to sieve all samples. The correlation can be used to predict sieved results from unsieved samples. The EPA Technical Review Workgroup (TRW) and American Society for Testing and Materials (ASTM) have issued guidance on sieving (ASTM, 1998; EPA, 2000c). The EPA TRW guidance addresses appropriate sieve size (No. 60) and a method for predicting the concentration in the fine fraction using concentrations measured in unsieved samples.

Technical Review Workgroup (TRW) – The TRW is an interoffice workgroup that consists of key scientific experts from various EPA regions, labs, and headquarters that supports and promotes consistent application of the best science in the field of lead (Pb) risk assessment at contaminated sites nationwide.

The presence of paint chips in a soil sample can represent a large proportion of the total lead concentration that is measured. On this issue, the Handbook directs the reader to existing HUD guidance, which states “If paint chips are present in the soil, they should be included as part of the sample. However, there should be no special attempt to over-sample paint chips. The laboratory should be instructed to disaggregate (‘break up’) paint chips by forcing them through a sieve in the laboratory. Although paint chips should not be oversampled, they should not be excluded from the soil sample, since they are part of the soil matrix.” (HUD, 1995). The TRW website should be checked periodically for additional sampling guidance.

4.3.4 Sample Analysis

EPA’s experience in sample analyses at large residential contamination sites (with several thousand homes on a site) shows that both FP-XRF or fixed-site laboratory analyses (acid digestion/Inductively Coupled Spectroscopy) provide reliable information (EPA, 1996d, 1998b, 2001c, 2001d; Crumbling et al., 2001). The objective of using a FP-XRF is to predict Contract Laboratory Program (CLP) values with

less expensive real-time data. A sufficient amount of data should be collected to develop a site-specific relationship (i.e., correlation) between FP-XRF and CLP lab data.

The comparison should consider sample preparation (drying and sieving) and analytical methods. Typically, a large number of laboratory confirmation samples should be analyzed at the beginning of the project to estimate the correlation between the FP-XRF and the CLP results and the FP-XRF precision and accuracy. Additional confirmatory samples should then be analyzed at key decision points when the FP-XRF results are close to action levels or when the reliability of the FP-XRF unit is in question (EPA, 2001d). For example, initial sample analyses using an FP-XRF instrument could include 20 percent laboratory confirmatory samples to assess the accuracy and precision of the FP-XRF. Once the accuracy and precision of the FP-XRF results have been determined (and assuming they satisfy the requirements of the project), the number of laboratory confirmatory samples could be reduced (e.g., to 5 percent). Additional information on analyzing soil (and other media) in the field with FP-XRF is available on the EPA web site: http://www.epa.gov/superfund/programs/dfa/ (EPA, 2001e).

Proper calibration of the FP-XRF unit is important to obtaining reliable results (EPA, 1996d). Correlation between the FP-XRF and laboratory analyses is best achieved with small sample volume. Laboratory confirmatory samples should be collected in the specimen cup available from the FP-XRF manufacturer. The sample is first analyzed with the FP-XRF and then sent to the laboratory for wet chemistry analysis. Soil moisture can introduce error in FP-XRF results to varying degrees, depending on the instrument being used (EPA, 1996d). The correlation between the FP-XRF measurements on dried and undried samples should be estimated. The correlation analysis should then be used to establish a cutoff or ‘soil moisture ceiling’. The ‘soil moisture ceiling’ represents the maximum moisture content at which useful results (i.e., of sufficient precision and accuracy) can be obtained with the FP-XRF. Field portable instruments capable of measuring moisture content are available and should be used to compare sample moisture content to the ‘soil moisture ceiling’. Samples with moisture contents greater than the ‘soil moisture ceiling’ should be dried prior to analysis with the FP-XRF.

5.0 CLEAN-UP LEVEL SELECTION

Generally, the approach to human health risk assessment for lead differs from that of other metals and contaminants. Typically, risks from lead exposures are estimated from long-term exposures, although elevated blood lead concentrations also result from short-term exposures (CDC, 1991). EPA has developed the IEUBK model to predict blood lead (PbB) concentrations in children exposed to lead. The model considers several different media through which children can be exposed to lead.

EPA and the CDC have determined that childhood PbB concentrations at or above 10 micrograms of lead per deciliter of blood (: g Pb/dL) present risks to children's health (CDC, 1991). Accordingly, EPA seeks to limit the risk that children will have Pb concentrations above 10 : g Pb/dL. The IEUBK model predicts the geometric mean PbB for a child exposed to lead in various media (or a group of similarly exposed children). The model also calculates the probability that the child's PbB exceeds 10 : g Pb/dL (P_{10}). Preliminary remediation goals (PRGs) generally are determined with the model by adjusting the soil concentration term until the P_{10} is below 5%. Final clean-up level selection for Superfund sites generally is based on the IEUBK model results and the nine criteria analysis per the National Contingency Plan (NCP) (EPA, 1990b), which includes an analysis of ARARs. More information on the IEUBK model is available from the EPA TRW web site.

Typically at large lead sites, early actions taken to mitigate the identified site risks consist of time-critical removal actions (TCRAs), most often taken as an interim action. These actions are usually followed by long-term remedial actions. The following sections describe the different approaches that should be used for prioritizing response actions and selecting clean-up levels for both early (interim) and long-term (permanent) response actions.

5.1 PRIORITIZING RESPONSE ACTIONS

For early, interim actions, a tiered approach should be used for prioritizing clean-up actions. A tiered-response approach is recommended when sufficient resources are not available to fully address lead risks. The size and complexity of many lead sites often requires implementation of response actions over an extended period of time; therefore, it is often necessary to implement interim clean-up actions to manage short-term health risk concerns while response actions to address long-term risk are planned and implemented. Early removal actions at residential lead sites should contribute to the performance of the long-term permanent remedy.

The tiered approach is depicted in Figure 5-1. Figure 5-1 is a flowchart that provides a roadmap of the recommended clean-up process for lead-contaminated residential sites. An overview to the clean-up process is provided in Figure 1-1. The first page of Figure 5-1 provides a more detailed overview; the subsequent pages provide additional details of the process.

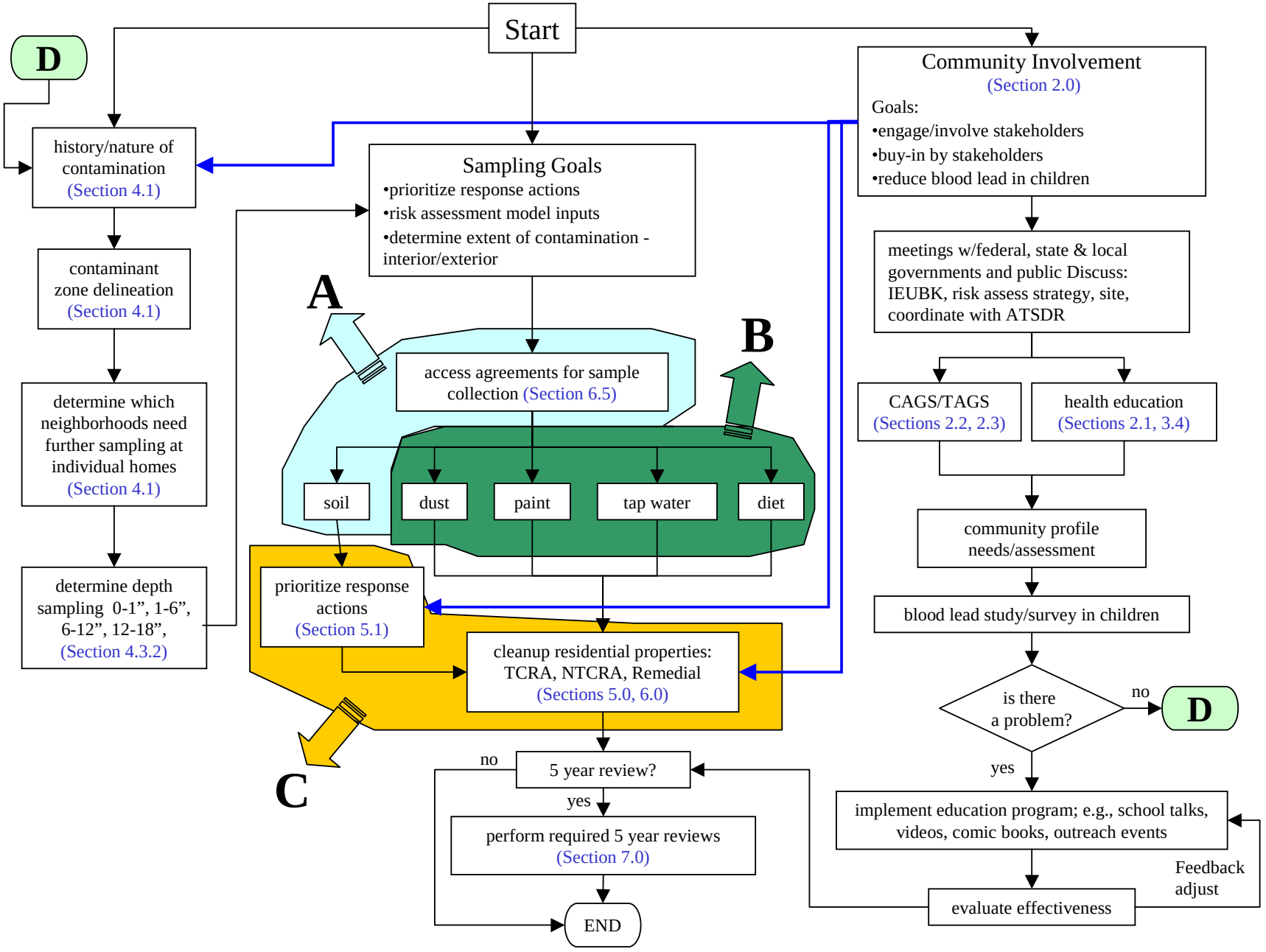


Figure 5-1. Recommended clean-up process for lead-contaminated residential sites. Refer to Figure 1-1 for an overview of the process. The shaded portions of the figure, labeled A-C, are expanded on the second through the fourth pages of the flowchart.

Figure 5-1. (continued)

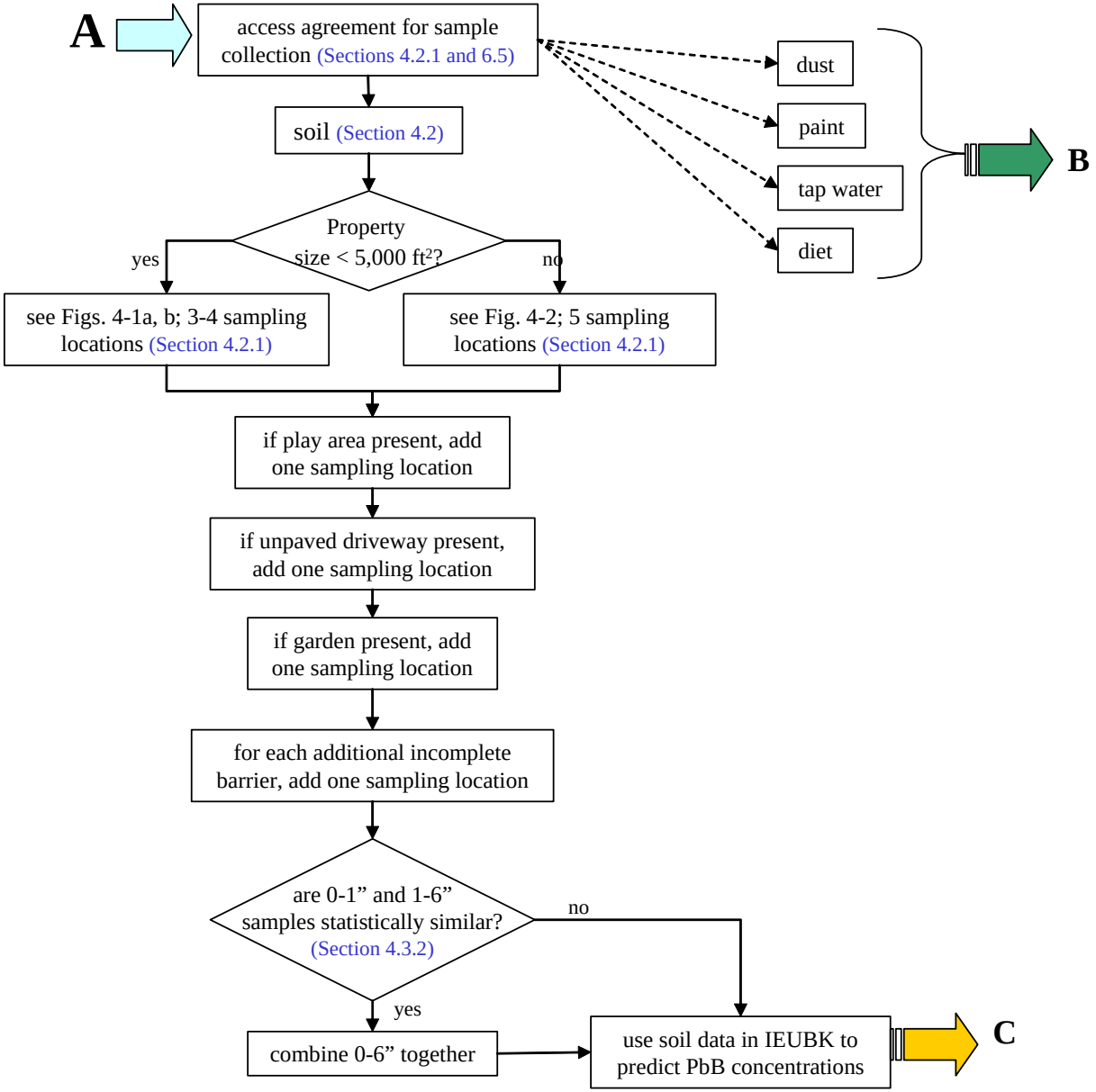


Figure 5-1. (continued)

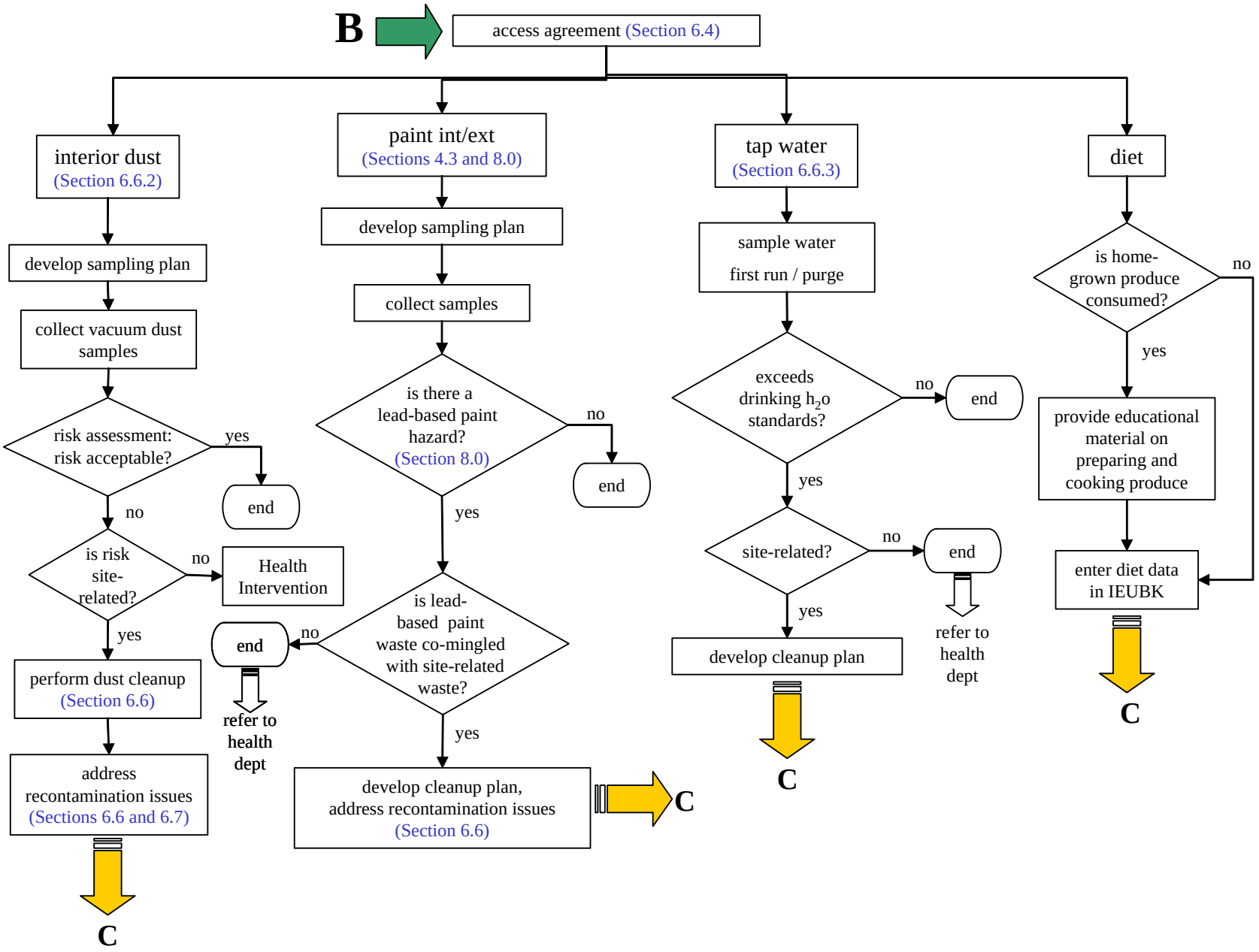
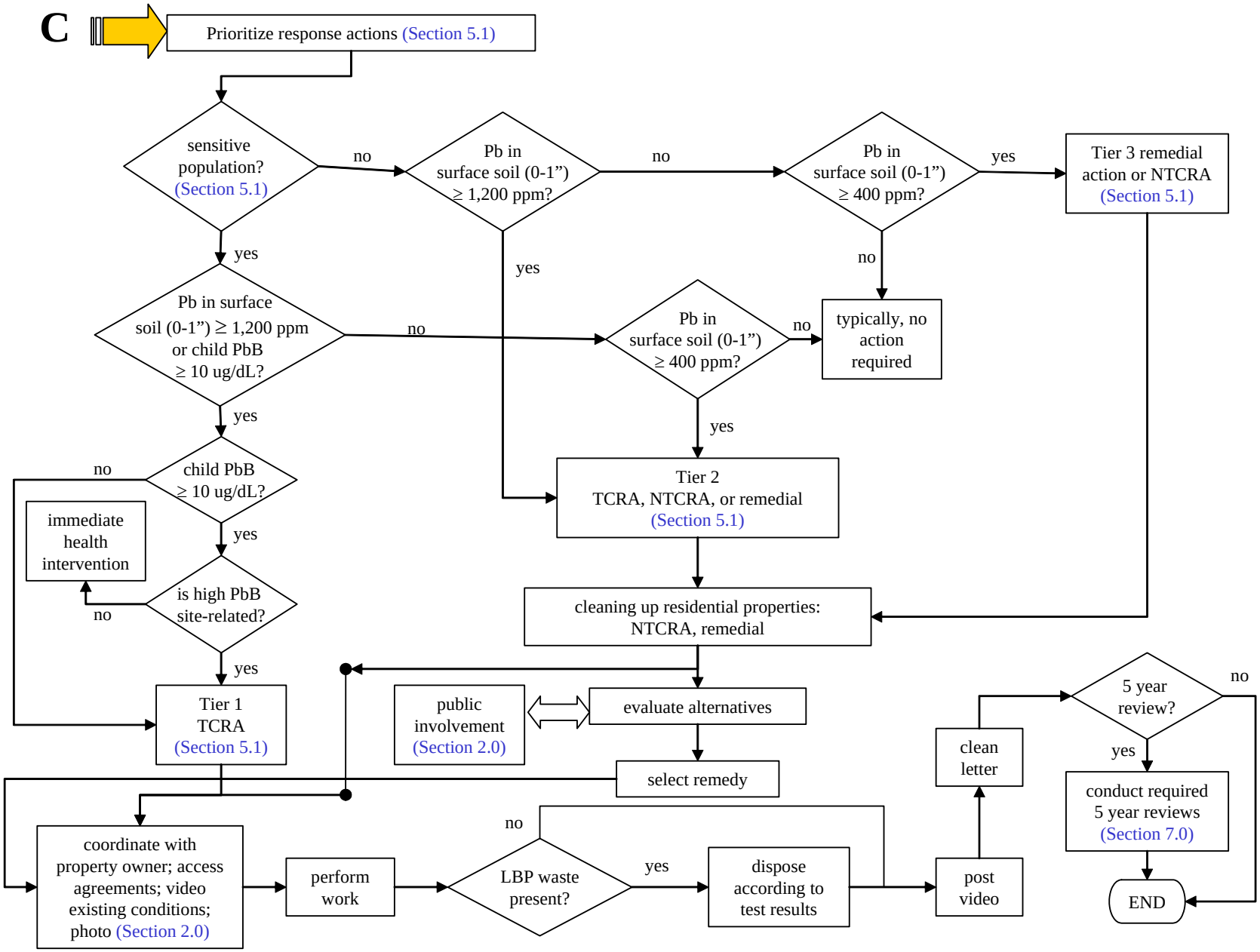


Figure 5-1. (continued)



The concentrations that are used to define tiers should not be confused with clean-up numbers, which are based on the PRG determined with the IEUBK model and an analysis that includes the nine criteria listed in the NCP (EPA, 1990b). The 1,200 ppm concentration is not an action level for TCRAs, but is intended to provide an alternative to running the IEUBK model if the project manager believes the site poses an urgent threat (EPA, 1997b, 1997c). Certainly, a TCRA could be justified above or below this concentration depending on the conditions at the site. The tiers, for the purposes of this guidance, are defined below (see also Figure 5-1). (Please note the Agency is considering developing new guidance for removal actions.)

- Tier 1 properties have both sensitive populations (children up to 7 years old or pregnant women) and soil concentrations in the surface soils (0–1" depth) at or above 1,200 ppm (EPA, 1997b, 1997c). Also, Tier 1 sites can be identified based upon a demonstration of children's blood lead levels at or above 10 µg/dL. Generally, TCRAs would be taken at Tier 1 properties.

- Tier 2 properties have either sensitive populations and soil lead concentrations in surface soils between 400 ppm and 1,200 ppm, or no sensitive populations and surface soil lead concentrations above 1,200 ppm, but not both. Tier 2 properties can be addressed through TCRAs, or non-time-critical removal actions (NTCRAs), or long-term remedial actions.

- Tier 3 properties have surface soil concentrations below 1,200 ppm, but above 400 ppm, and no sensitive populations present. Tier 3 sites would typically be addressed through long-term remedial actions or NTCRAs.

Tier 1 should be the highest priority for immediate action and Tier 3 should be the lowest priority for immediate action. Residential properties can move into a different tier if conditions change (e.g., small children or pregnant women move into a house). A typical residential lead site will contain a combination of properties that fit into different tiers. The project manager should use judgement to determine whether or not to perform a complete cleanup of contaminated residential properties (as defined in Section 1.3).

As discussed below, remedial actions for residential lead sites should use the IEUBK model. The IEUBK model should be used to assess risks posed by contaminated soils and to determine PRGs for soils at residential lead sites. In order to facilitate TCRAs, a demonstration of elevated blood lead levels or elevated soil-lead levels at or above 1,200 ppm will usually be sufficient. If elevated blood lead levels are the basis for concern, occupational contributions of lead, elevated lead levels in drinking water, lead from LBP, and lead dust in the homes of children or adults with elevated blood lead should be investigated first because these sources of lead can be significant (Appendix B). At this stage, consultation with Regional

risk assessors and public health officials (such as ATSDR) to better understand health impacts is encouraged.

The Agency plans on publishing a future lead removal directive which includes further information on site-tier approaches.

5.2 LONG-TERM REMEDIAL ACTION

The 1994 Office of Solid Waste and Emergency Response (OSWER) Directive 9355.4-12 states OSWER's risk reduction goal for residential lead sites: "... generally, OSWER will attempt to limit exposure to soil lead levels such that a typical (or hypothetical) child or group of similarly exposed children would have an estimated risk of no more than 5% exceeding the 10 ⁻g lead/dL blood lead level." (P_{10}<5%) (EPA, 1994b). It is important to note that this recommendation (i.e., P_{10}<5%) is meant to apply to a single residential property or another discrete exposure area, not on an area- or community-wide basis (i.e., 5 children out of every 100 actually exceed 10 ⁻g/dL). It is also important to note that selecting a soil lead concentration in this manner will not guarantee that a given child will not exceed a blood lead level of 10 ⁻g/dL. Many factors other than soil concentration cause variance in blood lead levels: pica behavior, or other sources of lead not included in the exposure unit, such as paint, diet, etc. (e.g., this could include soil at a camping site or other remote site frequented by the child).

The 1998 OSWER Directive 9200.4-27P ('Clarification') (EPA, 1998a) recommends that the IEUBK Model be used as the primary tool to generate risk-based soil clean-up levels at lead sites for current and future residential use (Appendix B). Additionally, the 1998 Clarification states that response actions can be taken using IEUBK predictions alone, and that blood lead studies, while providing useful information, should not be used for establishing long-term remedial or non-time-critical removal clean-up levels at lead sites. Regarding exposure units at residential lead sites, the 1998 Clarification states: "... it is recommended that risk assessments conducted at lead-contaminated residential sites use the individual residence as the primary exposure unit of concern" (EPA, 1998a; Appendix B). This document clarifies the definition of exposure unit provided in the 1998 Clarification. In addition to the individual residence, accessible site-related lead sources outside the residential setting should also be evaluated to understand how these other potential exposures contribute to the overall risk to children. When the evaluation indicates a significant contribution to risk, clean-up measures should be determined for those areas.

Empirical blood lead data occasionally deviates significantly from IEUBK Model predictions. This can be due to numerous factors, including the implementation of lead exposure-reduction and health education programs, and uncertainties in the exposure parameters of the Model as well as uncertainties in the blood lead data (Mushak, 1998). Regarding this issue, the 1998 Clarification states: "Where actual

blood lead data varies significantly from IEUBK Model predictions, the model parameters should not automatically be changed. In such a case, the issue should be raised to the TRW to further identify the source of those differences" (Appendix B). Basically, model inputs should be changed only when defensible, site-specific information that is specifically applicable to the parameters is collected. Moreover, these changes should also ensure that model outputs are protective of future residents. Examples of such information are dust lead concentration, drinking water concentration, bioavailability data (e.g., *in vivo* pig studies), and soil-to-dust ratio. The predictive capacity of the IEUBK Model depends upon the representativeness of the inputs. Section 4 discusses the collection of the data used to estimate some of these inputs.

In summary, there is no national clean-up standard for lead in residential soil on a Superfund site; however, there is a consistent process by which residential soil lead clean-up levels are selected. One step is to gather site-specific data as recommended in Section 4 of this Handbook and review other guidance on the use of the IEUBK Model (EPA, 1994b; TRW web site: http://www.epa.gov/superfund/programs/lead/ieubk.htm). Risk assessors (and other data users) should be consulted early to assist with data collection and planning (EPA, 2000d). Another step is to get assistance from the regional risk assessor(s) to run the IEUBK Model with applicable site-specific inputs. Running the model should allow the determination of a site-specific PRG that corresponds to a P_{10} for a typical child, or group of similarly exposed children, that is no more than 5%. Another step is to select a site-specific residential soil lead clean-up level that is based on the model-derived soil lead PRG and an analysis of the nine criteria consistent with the NCP (Superfund sites only) (EPA, 1990b). If the proposed clean-up level is outside of the range of 400 ppm to 1,200 ppm lead, then the draft decision document for the site is sent to the Lead Sites Consultation Group (LSCG) for review (EPA, 1997b).

Lead Sites Consultation Group (LSCG) – The Lead Sites Consultation Group (LSCG) was created in 1997 to promote national consistency in decision-making at lead sites across the country (EPA, 1997b). The main purpose of the group is to review key response decisions at lead sites. The LSCG is comprised of senior management representatives from the Waste Management Divisions in all 10 EPA regions along with senior representatives from the Office of Emergency and Remedial Response in EPA headquarters.

The LSCG is supported by EPA's Technical Review Workgroup for Lead (TRW) and the national Lead Sites Workgroup (LSW). According to Agency policy, there are three triggers that cause the review of lead-related proposed plans by the LSCG (EPA, 1997b):

1) Residential contaminated lead sites with proposed cleanup levels outside a 400 to 1,200 ppm soil-lead level;
2) Sites that envision actions to address non-soil lead-contaminated media;
3) Routine LSW deliberations that identify a unique or precedent setting site issue(s).

6.0 APPLICATION OF CLEAN-UP NUMBERS/REMEDIATION

The following section provides a detailed discussion of recommended minimum considerations to remediate residential soil and other sources of lead in residential settings. The guidelines stated below apply to early/interim actions and long-term remedial actions. However, due to statutory funding limitations that apply to time-critical removal actions, site-specific determinations regarding yard size limitations, and whether to clean up empty lots and other sources of lead (paint, dust, tap water), should be made by the project manager on a site-by-site basis.

6.1 MINIMUM EXCAVATION DEPTH/SOIL COVER THICKNESS

Based on Agency experience, it is strongly recommended that a minimum of twelve (12) inches of clean soil be used to establish an adequate barrier from contaminated soil in a residential yard for the protection of human health. Cover soil can either be placed after excavation as backfill or placed on top of the contaminated yard soil. The rationale for establishing a minimum cover thickness of 12 inches is that the top 12 inches of soil in a residential yard can be considered to be available for direct human contact. With the exception of gardening, the typical activities of children and adults in residential properties do not extend below a 12-inch depth. Thus, placement of a barrier of at least 12 inches of clean soil will generally prevent direct human contact and exposure to contaminated soil left at depth.

Removal of lead-contaminated soil to depths greater than 12 inches should be considered at sites in cold regions with non-soil lead-contamination sources, such as tailings and crushed battery casings, and whenever it is cost-effective. The additional response cost should be compared to future IC and monitoring costs associated with leaving the material in place. Full vertical removal of residential soil has many advantages, such as reducing or avoiding the costs of maintaining the soil cover, the placement of subsurface barriers/markers, and obtaining environmental easements. Full removal of contaminated soil also satisfies EPA's preference for permanent remedies and normally allows the remediated yard to return to unrestricted use.

Twenty-four (24) inches of clean soil cover is generally considered to be adequate for gardening areas; however, site specific conditions that may require more soil cover (e.g., presence of burrowing animals) should be considered. A 24-inch barrier normally is necessary to prevent contact of contaminated soil at depth with plant roots, root vegetables, and clean soil that is mixed via deep rototilling. Raised garden beds may be built to obtain 24 inches of clean soil, and may be more cost effective than excavating to 24 inches in depth, e.g., excavate 12 inches of contaminated soil, then add 24 inches of soil to create a 12" raised bed.

6.2 SOIL CLEAN-UP OPTIONS

Currently, there are only two remedial actions that generally are considered to be protective, long-term (not interim) remedial actions at residential properties: (1) excavation of contaminated soil followed by the placement of a soil cover barrier and (2) placement of a soil cover barrier without any excavation of contaminated soils. Excavation followed by the placement of a soil cover is the preferred method and is strongly recommended at sites with relatively shallow contamination, such as many smelter sites. In most cases, excavation and placement of a soil cover should be performed whenever the specific conditions of a site do not preclude it. For example, it may not be feasible to fully excavate a very large site cost-effectively, therefore capping, also considered to be protective, may be more appropriate. The advantage of the preferred method is that it is a permanent remedy in terms of removal of lead from areas where children may be exposed.

Several treatment technologies are currently under development to reduce the bioavailability of soil lead, but have not yet been proven to be protective in the long-term. These include amending the soil with phosphorus or high iron biosolids composts. Preliminary results have shown phosphate treatment to reduce the bioavailability of lead in soil by as much as 50 percent. This would mean that soil with lead concentrations in the range between clean-up levels calculated with the pre- and post-treatment bioavailability values could be treated instead of removed (e.g., if the IEUBK model-derived clean-up number using the pre-treatment bioavailability were 400 ppm lead, and the calculated post-treatment clean-up level were 800 ppm lead, then the yards with lead concentrations between 400 ppm and 800 ppm could be treated rather than excavated or capped).

Over time, the efficacy of the phosphorous treatments appears to increase. This is consistent with what is predicted using thermodynamics. To date, the treatability studies have been monitored for 3–5 years. Additional monitoring will be necessary to assure the long-term stability of the observed reduction in bioavailability.

Some other existing technologies for soil remediation that are not currently considered acceptable for residential lead cleanups are rototilling, phytoremediation, and interim controls, such as mulching, seeding, and sodding (without prior removal of contaminated soil). Rototilling is not considered a permanent, protective remedy in that no lead removal occurs, and adequate mixing of soil is difficult, if not impossible, to achieve; additionally, rototilling may increase the volume of soil, which ultimately requires remediation. Mulch, sod, or other vegetative covers are generally not considered permanent, protective remedies in that no lead removal occurs, and there is no guarantee that grass, mulch, or other vegetative cover will be maintained in good condition over time.

Additionally, land use changes that may occur within a yard, such as starting a garden or putting in a swing set, are not precluded in any way by mulch, sod, or other vegetative cover. Lastly, phytoremediation is not currently an appropriate technology for residential lead cleanups due to several factors: (1) the lead concentrations at many residential sites are not within the optimal performance range for the plants; (2) the plants may concentrate lower level lead contamination and present an increased disposal cost if the plants fail the TCLP test, but the unremediated yard soil does not fail; (3) the length of time required for remediation; (4) the potential conflicts with local regulations pertaining to yard maintenance; and (5) the depth of remediation achieved may be inadequate.

6.3 INTERPRETING SAMPLING RESULTS

Based upon the results of the sampling efforts (Section 4.0), this section describes the implementation of two clean-up options: (1) excavation and backfill (and placement of a visible barrier if applicable); or (2) soil cover placement (and placement of a visible barrier if applicable). The options should be performed as described below (see also Figure 6-1). The goal should be to remove all contaminated soil or provide a minimum 12" clean soil barrier. The following describes the implementation of option 1:

- If the 0–1" horizon exceeds the clean-up level, a 6 or 12" excavation is recommended, depending on the 6–12" sample horizon results;
- If the 1–6" or 0–6" horizon exceeds the clean-up level, a 6 or 12" excavation is recommended, depending on the 6–12" sample horizon results;
- If the 6–12" horizon exceeds the clean-up level, a 12" excavation is recommended. A visual barrier is required if the 12–18" horizon exceeds the clean-up level;
- If the 0–1, 0–6 or 1–6" horizons exceed the clean-up level and the 6–12" horizon does not exceed the clean-up level, a 6" excavation is recommended; a visual barrier is not needed.

	Depth	Soil Concentration Exceed Action Level?							
	0-1”	Yes	Yes	Yes	Yes	No	No	No	No
	1-6” (or 0-6”)	Yes	Yes	No	No	No	Yes	No	Yes
Remedial Action Options	6-12”	Yes	No	Yes	No	No	No	Yes	Yes
Option 1: Excavation (& Backfill)	Depth of excavation	12”	6”	12”	6”	No action	6”	12”	12”
Option 2: Capping	Soil cover thickness	12”	12”	12”	12”	No action	12”	6”	12”

Figure 6-1. Interpreting Sampling Results. The figure suggests remedial actions based on the results of composite soil samples collected for each of the depth intervals shown. The figure includes two remedial action options: (1) excavation followed by backfilling, and (2) placement of a clean soil cover without removal of soil that exceeds the action level. To use the figure, find the column of the table that agrees with the soil sample results for your site, then read down the table to determine the depth of soil to remove (option 1: excavation remedies) or the thickness of the soil cover recommended (option 2: capping remedies). For example, the heavy border around the third column of the table corresponds to a situation where the average lead concentration in the 0–1" and 1–6" depth intervals exceed the action level, but the 6–12" interval does not. In this example, it is recommended to remove the top 6" of contaminated soil and replace it with clean soil, or to place a 12" clean soil cover (cap). The goal is to provide a minimum 12” barrier of clean soil when the underlying soil exceeds the action level. Please refer to Section 6.3 for further explanation.

The following describes the implementation of option 2:

- If the 0–1" horizon exceeds the clean-up level, a 12" soil cover and visual barrier should be used;
- If the 0–6" or 1–6" horizon exceeds the clean-up level, a 12" soil cover and visual barrier should be used;
- If the 6–12" horizon exceeds the clean-up level (but not the 0–1", 1–6", or 0–6" intervals), a 6" soil cover should be used;
- If only the 12–18" horizon exceeds the clean-up level, no capping is needed.

The decision to perform soil cleanup to depths greater than 12 inches should be considered on a site-by-site basis. Some advantages to full vertical soil cleanup are listed in Section 6.1. However, there are many sites where lead contamination is located at depth. Full vertical soil cleanup may not be cost-effective and/or feasible at such sites. The depth of excavation and soil cover thickness is an important factor to be considered during the analysis of the nine criteria per the NCP (for Superfund sites) (EPA, 1990b). Potential for freeze/thaw upward migration, groundwater contamination, and the cost, extent, and effectiveness of ICs are some of the factors to be considered in this analysis.

Sampling results obtained for residential lots may indicate that only a portion of the lot contains soil that exceeds the selected clean-up level. For properties less than 5,000 square feet, the spatial scale for the remedial decision should be one-half of the yard. For properties greater than 5,000 square feet, the property should be divided into four quadrants and a remedial decision should be made for each quadrant. It is usually protective to excavate only the portion(s) of the lot that exceed the clean-up level (Figures 6-2a and 6-2b). However, removal of the sod layer and resodding/reseeding the unexcavated portion(s) of the lot is strongly recommended to promote consistency in the vegetative cover of the yard for homeowner satisfaction. When interpreting sampling results for a property, the sampling results of surrounding properties should also be considered to lessen the probability of mislabeling the property as being below the clean-up level, when it is actually above, and to avoid “patchwork clean-up” patterns, which are prone to recontamination.

If the only portion of the yard that exceeds the selected clean-up level is the drip zone, the exterior paint should be checked for lead content. If the drip zone contamination does not appear to be paint-related, the drip zone should generally be cleaned up. If the drip zone contamination appears to be solely paint-related, EPA should promote the remediation of the exterior LBP by local health agencies, other local government agencies, state health agencies, and/or the homeowner. At a minimum, the resident should be notified and informed of the disclosure requirements (Appendix A). Consideration should be given to also notifying the relevant local government agencies and informing them about available remedies, such as HUD grants.

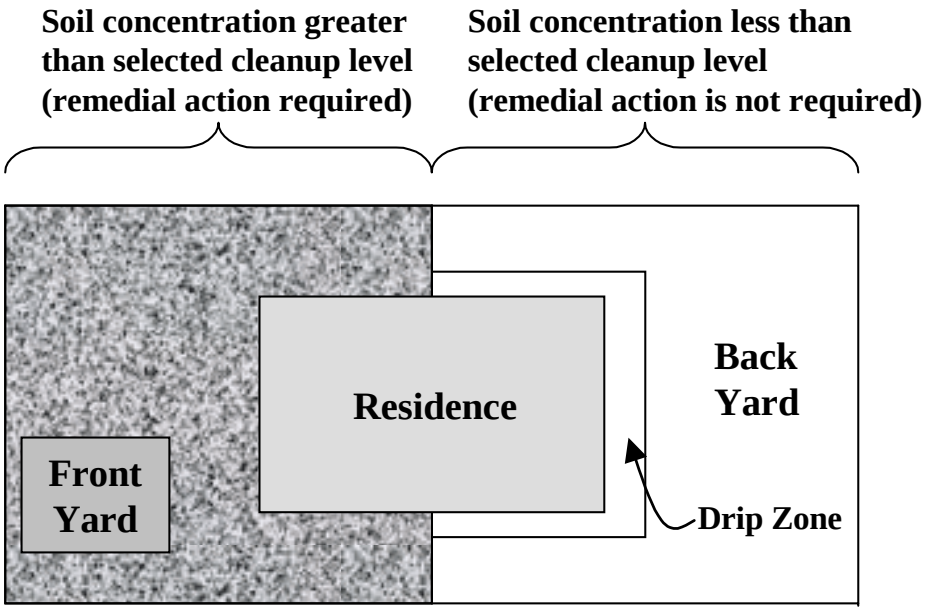


Figure 6-2a. Partial cleanup of residential lot less than or equal to 5,000 square feet in size. In this example, the lead concentration measured in the front yard exceeds the selected clean-up level while the concentration measured in the backyard does not. Cleanup may be limited to the front yard although it is recommended that the sod layer in the entire lot be removed to promote consistency in the vegetative cover on the property for homeowner satisfaction. The entire drip zone should be cleaned up if the average lead concentration exceeds the clean-up level. For example, in the above figure, the drip zone in the back yard (as well as the front yard) should be cleaned up if the average concentration in the drip zone exceeds the clean-up level. Please refer to Section 6.3 for further explanation.

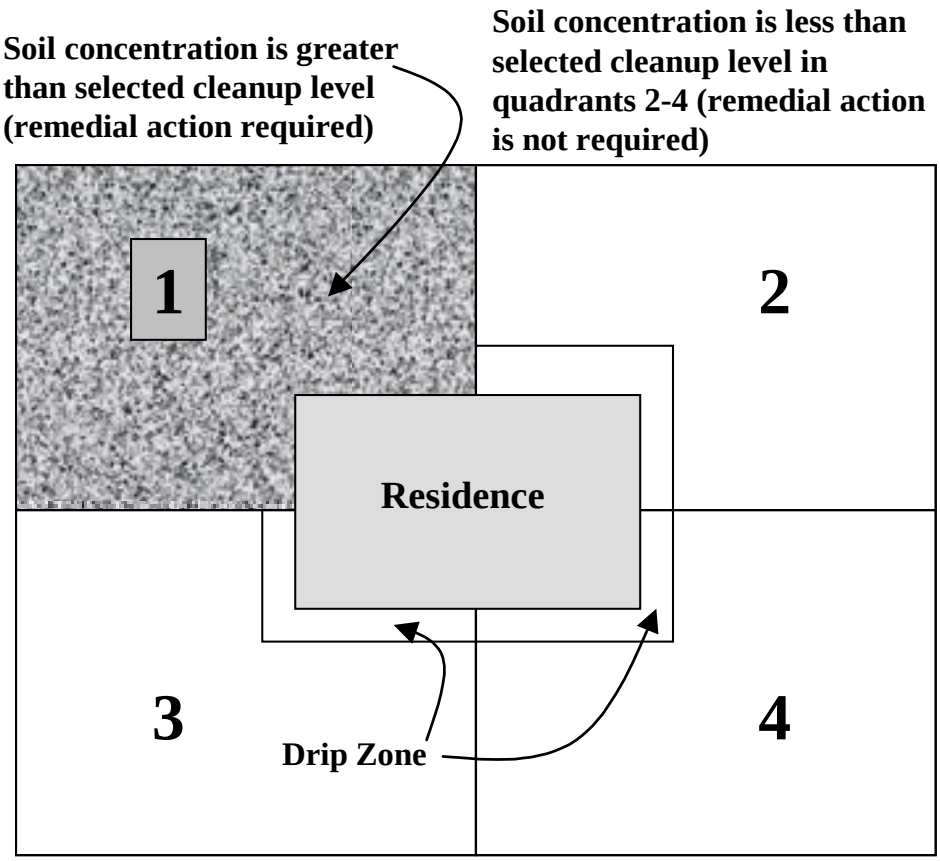


Figure 6-2b. Partial cleanup of residential lot greater than 5,000 square feet in size. In this example, the lead concentration measured in quadrant 1 exceeds the selected clean-up level while the concentration measured in quadrants 2–4 do not. Cleanup may be limited to quadrant 1 although it is recommended that the sod layer in the entire lot be removed to promote consistency in the vegetative cover on the property for homeowner satisfaction. The entire drip zone should be cleaned up if the average lead concentration exceeds the clean-up level. For example, in the above figure, the drip zone in quadrants 2–4 (as well as quadrant 1) should be cleaned up if the average concentration in the drip zone exceeds the clean-up level. Please refer to Section 6.3 for further explanation.

6.4 OTHER CLEANUP CONSIDERATIONS

The area remediated on a single property normally should not exceed one acre. This limitation is based on three factors: (1) typical lot sizes in residential areas throughout the country generally do not exceed one acre; (2) the portion of a property where the majority of exposure to contaminated soil occurs generally does not exceed one acre; and (3) EPA should generally not excavate/cover with soil the entirety of very large yards due to cost-effectiveness considerations.

The goal for cleanup of a yard that exceeds one acre is to excavate or cap the portion of the yard that is in frequent use and continue to limit exposure in the unremediated portion of the yard. To this end, it is recommended that the unremediated portion of such a yard be fenced to clearly delineate the remediated and unremediated areas and to limit the potential for off-site migration of contaminants (e.g., vehicle tracking). Exceptions to this general approach may include areas outside the one-acre area that are used for recreation and gardening, areas with the potential for residential development, and areas in close proximity to other residential areas. As stated in Section 6.5, any unremediated areas of a property should be documented on the clean-up documentation letter for such property, and consideration should be given to implementing ICs for those areas.

If contaminated soil is not removed to the full depth of contamination (i.e., where soil concentration is greater than clean-up level) on a property, a permanent barrier/marker that is permeable, easily visible and not prone to frost heave, should be placed to separate the clean fill from the contamination. This applies to both incomplete vertical excavation with placement of a soil cover and placement of a soil cover without excavating contaminated soil. Selection of an appropriate permanent barrier/marker should be based on the type of contamination left in place, the chemical/physical characteristics of the soil (e.g., pH), the potential for upward migration of the contamination, and/or the types of ICs developed for the site. Examples of suitable barriers/markers include snow fencing (usually orange), a clean, crushed limestone layer, and geofabric.

Empty lots that are zoned residential and contain soils with lead concentrations greater than the clean-up level should be cleaned up when in close proximity to other residential lots. Examples of this are lots between two houses and lots that are near occupied lots. A site-specific determination should be made for these situations. Also, unpaved lots used for vehicle parking should be sampled, and cleaned up if necessary, or access restrictions put in place to prevent recontamination (e.g., vehicle tracking of contaminants) even if no current direct exposure exists. However, it is not the intent of EPA to clean up tracts of remote, undeveloped, lead-contaminated land that may be developed into residential lots in the future. This clean-up responsibility should be borne by the land developer. Institutional controls should

be developed to ensure safe development in these areas, since under CERCLA developers could be held liable for improper cleanup.

6.4.1 Background Lead Concentrations

Many of the “Lead Sites” on the NPL are located in areas with high natural background lead concentration. Often this problem is exacerbated by the presence of high background concentrations of lead in various media (such as soil and groundwater) from anthropogenic sources such as automobile emissions, mining, and smelting (the latter two sources would be considered ‘background’ if they are not associated with the site). It should be noted that CERCLA 104 (a)(3) limits the Agency from taking response actions to address "... naturally occurring substance in its unaltered form, or altered solely through naturally occurring processes or phenomena, from a location where it is naturally found” (EPA, 2000a). Generally, under CERCLA, clean-up levels are not set below natural or anthropogenic background concentrations (EPA, 1996c, 1997d, 2002). Cleanup below natural or anthropogenic background concentrations is normally not performed because it is not cost-effective, it is technically infeasible and there is a high likelihood of recontamination by surrounding areas that have not been remediated (EPA, 2002).

Public education about ubiquitous risks should be incorporated early in the process to help the community understand that Superfund actions are designed to address risks from specific releases to the environment (EPA, 2002). In situations like these, it may be appropriate to examine land uses that limit exposures through implementation of ICs. For more information on this approach, please refer to the 1998 Clarification to the Revised Interim Soil Lead Guidance for CERCLA Sites and RCRA Corrective Action Facilities (Appendix B). Site-specific factors should determine what range of alternatives and what clean-up levels will achieve a protective remedy satisfying the nine criteria specified in the NCP.

Remedial decisions often involve a comprehensive response coordinated with other responsible authorities, such as a local public health district, state departments of environmental protection, housing agencies, and private parties. An effort should be made to identify other programs or regulations that may have the authority and capability of addressing risks associated with high natural or anthropogenic background (EPA, 2002). Additional guidance is available for developing a risk management-based response strategy that is protective of human health and the environment (EPA, 1988).

6.5 YARD CLEANUP SPECIFICS

It is important to define the limits of the properties that will be remediated. The use of property lines rather than temporary features, such as fence lines, to delineate boundaries is recommended. The use of temporary features may result in partial cleanup of some properties.

Whether remediation consists of excavation and placement of soil cover or just the placement of a soil cover, consultation with the property owners is important to the development and implementation of response actions and may necessitate property-specific deviations to the guidelines listed in this section. Flexibility is essential to a successful residential lead clean-up program. Some residents may want to pay for upgrades during the cleanup of their yard, such as paving a driveway after excavation, or to have some yard features removed, such as taking out a damaged patio. Within reasonable limits, such requests should be entertained on a yard-by-yard basis. Granting such requests can greatly contribute to building public trust and satisfaction with the clean-up program. All additional costs associated with special requests and considerations must be borne by the homeowner.

Prior to cleanup of a residential yard, access from the property owner should be obtained; access obtained from tenants or renters is not sufficient. It is recommended that access be obtained by going door-to-door. If residents are not home, a blank access agreement with instructions for signature and submission to EPA, along with relevant contact information should be left at the residence (but not in the mailbox). An example access agreement form is presented on page D-6 of Appendix D. As stated in Section 4.2.1, it is suggested that access for remediation be obtained at the time access for sampling is sought. Examples of combined sampling/remediation access agreements are presented on pages D-4 and D-5. An example of a dust cleanup access agreement form is presented on page E-2 of Appendix E. Many residents may refuse access for dust cleanup while granting access for yard-soil cleanup. Combining dust access agreements with other access agreements is not recommended.

Prior to initiating clean-up activity, the condition of each property should be documented and recorded on videotape. 'Clean-up activity' includes any disturbance of the property, including the removal of debris and dilapidated structures that may be required prior to initiating the excavation of contaminated soil. An example of a property inspection form is provided in Appendix F. EPA should enter into a written agreement with the resident regarding any special requests or considerations in cleaning up the yard, e.g., replacing concrete walkway with brick. All additional costs associated with special requests and considerations must be borne by the homeowner. Any contaminated yard areas that will not be cleaned up, special resident concerns, and any deviations from strict soil excavation or capping should be noted on this agreement.

Other possibilities for cleanup-related agreements include sod/lawn watering agreements. A sod-watering agreement basically allows for payment to residents for watering the sod that is placed by the remediation contractor. A payment is made before watering is required to cover the water bill and some of the time involved. A second payment is made if, at the end of one month, the sod is in good condition. A similar agreement should be established for maintaining lawns that have been initiated by hydroseeding. This can be a useful incentive program that can also save money. The contract with the remediation contractor should require the contractor to establish vegetation on each property, restore the pre-construction drainage patterns on each property, and perform repairs for damages to the property.

Relocation of residents during yard soil remediation is rarely needed and is generally not recommended (EPA, 1999b). (Guidance is available online at: http://www.epa.gov/oerrpage/superfund/tools/topics/relocation/index.htm.)

Specific safety issues during residential yard cleanup, including ingress and egress to the home, should be coordinated with the property owner/residents and spelled out in the Health and Safety Plan.

Incomplete barriers (such as rock or gravel) or minimal use areas (such as areas under porches), which exceed the applicable clean-up level, should be cleaned up to the extent practical. Although removal is preferred, if it is not feasible to clean up the area, a barrier, which effectively limits access, should be constructed. For example, for areas underneath porches, typically the preferred barrier would be shot-crete (sprayed concrete that can easily be placed in tight or confined areas). It may be preferable to place asphalt rather than gravel on heavily-trafficked roads or driveways, especially those that experience severe erosion.

In all cases, every attempt should be made to clean up the entire yard (subject to cost limitations discussed below), however, any residential yard areas without permanent barriers that the resident requests to leave unremediated, such as gardens or patios, should be sampled separately to determine if the selected clean-up level is exceeded. If the clean-up level is exceeded and the owner refuses to allow cleanup of that portion of the yard, then the clean-up documentation letter issued to the owner should note the unremediated area.

The steps of a typical soil cleanup are shown in the text box below.

Steps of a Typical Soil Response Action

Step 1 (Access Agreement) - Collect access agreement(s) from each owner and/or tenant before any work is conducted.

Step 2 (Initial Survey) - Interview the resident(s) to determine if there are any specific problems that need attention, and if there are any structures or property the owner wants to have disposed, stored, or left untouched. The contractor will conduct a thorough documentation of the property using drawings, digital photographs, and videotapes. Once documented, the owner is required to sign a property agreement which documents any special requests or considerations in cleaning up the yard, any contaminated yard areas that will not be cleaned up, provisions for structural concrete and fence restoration, and deviations from strict soil excavation and capping.

Step 3 (Excavation) - Each tract is excavated by the contractor(s), who will also complete documentation and provide depth confirmations.

Step 4 (Backfill) - After excavation of properties where full excavation to depth has been performed, the excavated area is backfilled and compacted. After excavation of properties with a vertical excavation limit, a permanent, permeable barrier/marker is placed in the excavated area. After placement of the barrier/marker, the excavation area is backfilled and compacted.

Step 5 (Restoration) - Restoration of the property, including landscaping, sod/seeding, fencing, and concrete (if needed) is conducted.

Step 6 (Final Inspection) - After restoration activities are complete, the EPA, PRP, or its agent (e.g., Corps of Engineers) will conduct a final inspection.

Step 7 (Closeout Form) - A property closeout form should be signed by the property owner, which documents the owner is satisfied with the remediation of the property. Any outstanding issues between the EPA and the homeowner that have not been fully resolved should be documented in the closeout form.

Step 8 (Clean Letter) - After the homeowner signs at property closeout form, the EPA issues a "clean" letter, which documents the property has been remediated. Any areas that are not cleaned up via the owner's request, such as gardens, should be noted in the "clean" letter. For properties where contamination is not completely removed, the clean letter should also document the presence of contamination at depth, and should describe the protective measures that were taken to prevent exposure to the remaining contamination (i.e., barriers/markers).

6.6 CLEANUP OF OTHER SOURCES OF LEAD

Lead in the environment can originate from many sources. In addition to soil, the main sources to consider when performing clean-up activities are interior and exterior LBP, lead-contaminated interior dust, drinking water, and occupational exposure resulting in subsequent contamination of homes. Generally, sources other than soil, exterior paint, dust, and tap water cannot be remediated by EPA in the course of residential lead cleanups.

Ultimately, the project managers should strive to address any unacceptable lead-exposure risks at the residence. Sampling and the establishment of clean-up mechanisms needed to take action, such as HUD grants for paint abatement, should be completed as early in the remedial process as possible. Even so, it may not be possible to address all sources of lead in the ideal sequence. When this occurs, other measures should be taken to minimize the potential for recontamination (i.e., to protect the remedy). For example, if deteriorating exterior LBP is present, it is recommended that it be removed prior to initiating any soil clean-up activities in the yard.

Due to transport of lead among media, the preferred sequence of lead clean-up activities at a residence with LBP and lead-contaminated soil would be to clean up the paint first, then the yard soil, and then the interior dust. Clean-up activities performed counter to this sequence increase the risk of recontamination. For example, performing a soil cleanup first at a residence with exterior paint problems increases the potential for recontamination of the soil from the exterior paint. Similarly, interior dust can be recontaminated by interior LBP. Exterior sources have been shown to cause recontamination of the interior when cleaned before community-wide yard cleanup is completed (EPA, 2000e). Accordingly, project managers should make every effort to coordinate the sequence of clean-up activities to prevent recontamination.

CERCLA and the NCP limit Superfund authority to address interior LBP (see Section 1.2) (EPA, 1990b). If a mechanism exists for addressing the paint, such as a HUD grant or a Supplemental Environmental Project (SEP), then the timing of the paint encapsulation or abatement activities may not coincide with the soil cleanup. Additionally, residents may be more reluctant to grant access for dust remediation since it is more intrusive. On the other hand, EPA actions taken to address lead in drinking water from site sources usually can be taken independently from any soil, dust, or paint cleanups, and should be done as soon as practical.

> Supplemental Environment Project (SEP) – Environmentally beneficial projects which a defendant/respondent agree to undertake in settlement of an enforcement action, but which the defendant/respondent is not otherwise legally required to perform.

6.6.1 Lead-Based Paint

The 1998 Clarification presents OSWER's policy with respect to remediation of interior paint, exterior paint, interior dust, and lead plumbing. Regarding interior LBP, the 1998 Clarification states:

> "EPA has limited legal authority to use Superfund to address exposure from interior lead-based paint. As a policy matter, OSWER recommends that such exposures not be addressed through actual abatement activities. However, EPA Regions should promote addressing interior paint risks through actions by others, such as HUD, local governments and health authorities, or individual homeowners as a component of an overall site management strategy. Any activities to clean up interior lead-based paint by potentially responsible parties (PRPs) or other parties should not result in an increase of the risk-based soil clean-up levels" (EPA, 1998a; Appendix B).

Regarding exterior LBP, the 1998 Clarification indicates that the Regions should avoid using the Superfund trust money for removing exterior LBP and soil contaminated from LBP. However, Superfund dollars may be used to respond to exterior LBP to prevent recontamination of soils that have been remediated, but only after determining that other funding sources are not available (EPA, 1998a; Appendix B). The 1998 Clarification states: "As with interior lead-based paint abatement, EPA Regions should promote remediation of exterior lead-based paint by others, such as PRPs, local governments, or individual homeowners. Clean-up activities of exterior paint conducted by PRPs or other parties should not result in an increase of the risk-based soil clean-up levels" (EPA, 1998a; Appendix B).

As a practical matter, project managers should inform each resident regarding the presence or absence of LBP in their home, and options for encapsulation and abatement. The local health agency and/or the state health agency should be informed regarding the availability of HUD grants for paint assessment and abatement. Additionally, regarding PRP-funded cleanups, if any penalties are being considered for non-compliance (Section 6.9), consideration should be given to allowing the PRPs to perform a SEP for paint assessment and abatement in lieu of some or all of the penalty amount.

6.6.2 Interior Dust

Lead-contaminated interior dust can be derived from multiple sources, including exterior soil, interior and exterior paint, homeowner hobbies, workplace, and other exterior sources; thus, it may be difficult to differentiate between sources of dust contamination. Household lead dust contamination may be a significant contributor to elevated blood lead levels, especially for younger children (under the age of three), and may need to be evaluated in determining risks and clean-up actions at residential lead sites. However, as pointed out previously, there are limitations on EPA's authority to abate these sources of contamination to the extent they are not related to releases or threatened releases to the environment (Appendix B).

Based on the 1998 Clarification, OSWER recommends that Superfund monies should generally not be used to take CERCLA response actions for addressing residential dust exposures due solely to interior paint or other interior sources. However, Superfund monies can be used to address interior dust if it can be shown to be derived from an exterior pollution source (e.g., air lead concentration caused by lead smelter, mining, or mineral processing). Dust mat sampling, which was done at the Bunker Hill Site in Idaho (EPA, 2000e), is one possible method of lead source identification; speciation, which is costly, is another method. (Dust mats are used to measure dust lead concentration and loading rates in residences and other structures.) Where interior dust is being addressed by other authorities, the recommendations presented here may be helpful to guide the dust cleanup.

If the lead in interior dust is solely derived from interior paint, EPA should promote addressing interior dust risks through the actions of others, such as HUD, state and local governments, PRPs, or individual homeowners, as a component of an overall site management strategy. The overall site strategy, as outlined below, should also consider the proper phasing/sequencing of actions to address the multiple sources of lead risks at residential lead sites, as discussed at the beginning of Section 6.6.

The baseline risk assessment should document the relative contributions of lead uptake from all relevant media including direct soil exposures and secondary exposures to soil in indoor dust. Replacement of defaults with a site-specific value for the interior dust concentration, or the soil-to-dust relationship (M_{sd}), should be justified through the use of high quality, compelling, site-specific data (EPA, 1994b, 1998c). Dust sampling is preferred for risk assessment and remedial decisions, but dust modeling may be needed to develop or refine soil action levels.

Lead-contaminated interior residential dust presents a significant exposure pathway that can readily be addressed. Consequently, significant health benefit is gained by removal of contaminated interior dust as early in clean-up activities as possible. However, exterior contamination sources present a threat of recontamination to interior of residences (EPA, 2000e; TerraGraphics, 2001). Therefore, any interior dust clean-up actions should be periodic throughout the project and should culminate in a final cleaning of all residences exceeding an action level after the exterior sources have been remediated. As a practical matter, risk management and reduction may need a phased strategy as recommended below:

Early-Phase Actions: Public awareness and health education efforts should be initiated immediately. Entry way dust mats should be provided to residents. HEPA-filter vacuum cleaners should be provided for use by residents. If warranted, a program to abate interior lead-contaminated dust in homes with acute levels should be initiated to provide temporary risk reduction.

Establish appropriate public health partnerships with state and local health departments, ATSDR, and HUD as early as practical.

Mid-Phase Actions: The source of the interior dust lead contamination should be identified. Monitoring of the changes in lead-contaminated dust (e.g., lead loading in dust, lead concentration in dust, exterior-to-interior lead transport) should be initiated. The public awareness/health education efforts and availability of HEPA-filter vacuum cleaners for use by residents should be continued. Assistance to remove and dispose of old carpets should be provided to residents after yard cleanup has occurred.

Final-Phase Actions: Once the exterior lead sources that were found to contribute to interior dust have been addressed, the final step should consider the active remediation of interior lead-contaminated dust. Actions may include: removal of carpeting, cleaning heat and ventilation ducts, wet wiping hard surfaces and soft surfaces (furniture, draperies, bedding, clothing, etc.). Most of these actions should be limited to living spaces. Areas such as attics, crawl spaces, and other non-living spaces need not be addressed unless they are shown to be a continued source of contamination to the living areas . It is important for dust remediation to be performed as the last phase in the site clean-up process to minimize the risk of recontamination.

6.6.3 Lead Plumbing/Tap Water

The 1998 Clarification states: “Generally CERCLA does not provide legal authority to respond to risks posed by lead plumbing within residential dwellings. It should be noted that the water utility is responsible for providing clean water to the residences. As with interior dust, OSWER recommends that EPA Regions coordinate with local agencies to establish a health education program to inform residents of the hazards associated with lead plumbing and how to protect themselves by regularly flushing, or preferably, replacing lead pipes. Soil clean-up levels should not be adjusted to account for possible remediation of lead plumbing” (EPA, 1998a; Appendix B).

With regard to tap water, it should be sampled, and lead levels in the purged sample in excess of the maximum contaminant level (MCL) established by the Safe Drinking Water Act should be addressed. In general, lead concentrations in the purged sample greater than a removal action level (RAL) of 30 : g/L should be addressed through TCRAs; concentrations between the MCL and RAL should be addressed through NTCRAs or long-term remedial actions. Actions that could be taken include provision of bottled

water, connection to a municipal water supply, tap filtration, and installation of deep wells (in remote areas and where shallow groundwater is contaminated). Regarding first run exceedance for lead, the homeowners should be notified that they may need to address a plumbing or corrosion problem, which is outside of the scope of Superfund.

6.7 PREVENTION OF RECONTAMINATION

Project managers should take steps to mitigate recontamination. During site closeout and five-year reviews, the project manager should also check for recontamination at levels which may threaten the remedy.

At many large-area lead sites, cleanup occurs over a long period of time and through multiple phases, throughout which the potential for recontamination exists. During each of these phases, windblown dust sources, vehicle tracking, flooding, and other mechanisms can recontaminate previously cleaned areas. Although best management practices (BMPs) should minimize the movement of contaminated material from each residence being cleaned, vehicle tracking of contamination from areas yet to be cleaned up can significantly raise concentrations of contaminants in cleaned areas. During the early phase, typically an emergency response action, cleanup is focused towards Tier 1 properties, and cleanup favors a "hop scotch" approach to address the worst risks first. This method of remediation can result in recontamination of clean properties. Confirmation samples should be collected in any areas that have been potentially recontaminated.

Another aspect of large-area lead sites is that complete cleanup of residential properties does not always take place for a variety of reasons (see Sections 6.2 and 6.4); instead a barrier or soil cover is put in place over contaminated soils. Flooding can pose a serious problem for these areas in that flood waters can erode away clean materials leaving subsurface contamination exposed, and entrained sediments bearing contamination may be left on top of newly remediated properties. Inadequate drainage of runoff can move lead into cleaned areas (e.g., lead particles on a crowned road with no curb and gutter may be rinsed onto adjacent residential properties with normal rainfall). Additionally, the activities of burrowing animals can bring contaminated soils to the surface.

Best Management Practice (BMP) – In general, BMPs are a combination of practices that are determined to be the most effective and practicable means of controlling point and nonpoint pollutants at levels compatible with environmental quality goals. In this document, BMPs specifically refer to measures taken during construction activities on properties where contamination has been left at depth to prevent the transfer of those contaminants to other media.

Recontamination of clean soil cover can be caused by ongoing homeowner projects, such as digging a hole through a clean barrier to install fence posts or a new tree or shrub, if preventative measures are not taken. Education and licensing of contractors who work on clean barriers/markers should generally be required (e.g., as part of a local ordinance) to ensure the longevity of the remedy. Also, at many sites (e.g., Bunker Hill), ICs have been most effective when linked to the "call before you dig" program typically operated by many counties to avoid disruption of utility service. In addition, large scale residential development projects that may raze old housing in favor of new will frequently recontaminate areas where lead-contaminated soil was left at depth, without appropriate BMPs in place. BMPs include silt fences, hay bales, etc., to limit movement of contamination off a project site, and stockpiling of contaminated soil on a tarp to prevent contamination of underlying soil (Figure 6-3). EPA provides guidance on the implementation of BMPs in construction activities at sites where contamination is present (EPA, 1997e). Best management practices typically add about 5 percent to project cost (TerraGraphics, 2000). Periodic inspections of residential areas should be performed by the local government to ensure that projects within the site are implementing BMPs.

Wind blown dust can pose a significant threat to the health of individuals at a site and can cause recontamination. Tailings impoundments that have dried can be large sources of windblown lead dust. Most tailings impoundments are large; a wind sweeping across the face of one can carry substantial amounts of contaminated dust and then deposit these particles on a downwind residential area, both causing increased exposure to contaminants, and recontaminating clean areas. Wind blown dust sources are typically a key issue to be addressed early in the sequencing of site activities to minimize this migration.

These are but a few examples of how recontamination can be an ongoing problem that needs to be considered at every site during each phase of cleanup. Although mechanisms vary from site to site, the types of response actions put in place and the sequence in which these actions take place can play a significant role in enhancing the permanence and effectiveness of a remedy.

A disposal area may be needed to dispose of contaminated soil from the site to support typical homeowner projects, as some municipal landfills may not accept contaminated soil. Without free or low cost disposal for contaminated soil available to each homeowner and renter, improper disposal is more likely, which would result in recontamination. In addition, a disposal area may be needed if certain materials at a site, such as carpets, fail TCLP and cannot be commingled with solid waste. It may even be appropriate for the remedy to provide free removal of contaminated soil and provision of clean soil to homeowners (but contractors may be required to pay for these services, or obtain material from approved sources) to encourage maximum compliance and further ensure the longevity of the remedy. The

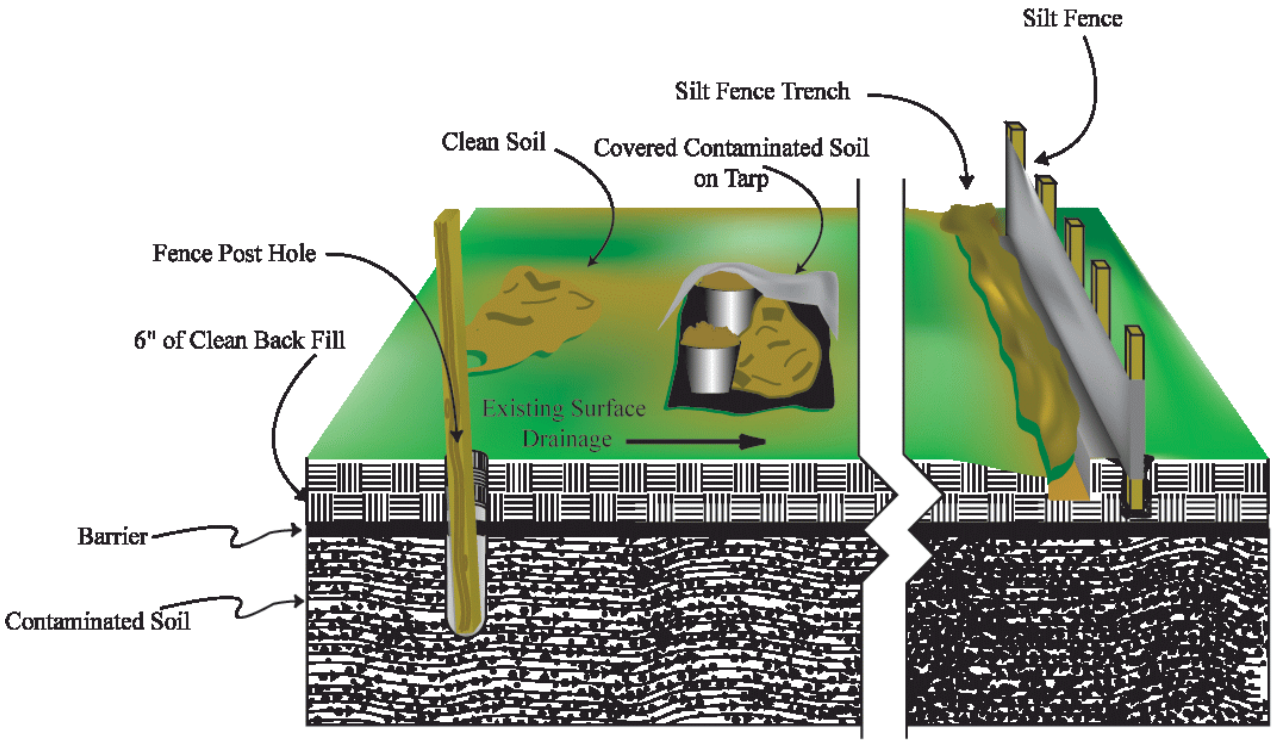


Figure 6-3. Implementing Best Management Practices (BMPs) during construction work. The best management practices (BMPs) shown in the above figure (e.g., a clean soil barrier) represent one component of the ICs which may be put in place by local ordinance to ensure the long-term protectiveness of the remedy and to prevent recontamination. The purpose of BMPs is to minimize the potential for accidental exposure of humans during construction and maintenance activities on sites where wastes have been left in place. The staging of contaminated soil on tarps and/or in small buckets, and the installation of silt fences downgradient of the construction area are examples of BMPs intended to prevent the migration of contaminated material from the construction site. Please refer to Section 6.7.3 for further explanation.

maximum concentration of lead (and perhaps other constituents) allowed in “clean” soil, and the required sampling frequency, should be specified in an IC.

Over the long term, cleanups may not be possible at every property at the same time. A trust fund should be established for the site for the cleanup of properties that are deferred for various reasons, which should be implemented by the local government. In this manner, changes in property ownership over time may be more closely monitored to determine when cleanup at deferred properties might be appropriate (see Section 6.9). Local implementation of the trust fund will ensure that cleanup of these properties occurs as soon as possible, further ensuring the protectiveness of the remedy, further ensuring the protectiveness of the remedy by minimizing the potential for recontamination to the extent possible.

6.7.1 Early Actions

Early response actions (including cleanups for sensitive subpopulations) can be an essential aspect of the response action at a site, as discussed above. These actions should be conducted simultaneously with source area control. The following are considerations that may reduce the potential for recontamination when scoping an early action.

- Seek permanence in selecting the clean-up alternative(s), if possible, such as complete removal to depth of soil contamination at properties where there is an acute risk.

- Consider cleanup of adjacent properties simultaneously that may threaten the permanence or effectiveness of the early action.

- Control fugitive dust sources, access, tracking, and erosion of contaminants to the extent possible.

- Perform HEPA street sweeping to minimize tracking of contaminants throughout a community.

- Evaluate the feasibility of conducting the cleanup of residential areas in their entirety during the early removal phase if contamination is widespread. If this is not possible, limit the early removal actions to immediate risks (Tier 1 and Tier 2 residential properties, including residences with elevated blood lead levels) in order to minimize the potential area where recontamination might occur.

- Provide informational fact sheets to homeowners on how to minimize recontamination on their property.

- Establish an IC to manage cleaned areas. This could involve local and state government agencies, and PRPs that are available to recommend best management practices for homeowner projects and provide education to the homeowner, as well as utility districts and companies likely to breach the barriers/markers put in place.

- Provide site plans or other documentation of areas that have been cleaned up, as well as information on areas that are still contaminated, to the local governmental entity responsible for the maintenance of the remedy, i.e., for monitoring ICs and for tracking properties over time.

- Establish a geographic information system (GIS) for monitoring ICs and properties.

6.7.2 Long-term Remedial Action

Some or all of the following measures may be useful to address the risk of recontamination during the remedial action (Tiers 2 and 3, if a tiered approach is used) and post-design phase:

- Evaluate the permanence and effectiveness of the various remedial actions under consideration. Consider the economic feasibility of complete contaminated soil removal to minimize reliance on ICs.

- Conduct a cost analysis comparing the cost of long term ICs to those of complete removal (EPA, 2000f). For example, property depreciation, tax base impact, additional procedures/cost of utility work, flooding complications/costs, and long term IC administration cost should be taken into account when comparing the cost of a partial removal of contaminants to a complete removal. Property depreciation, while possibly subtle for each property, may add up to substantial losses for the entire community in reference to a county tax base. Also, losses for an individual property over a lifetime of sales could add up to a significant cost. Following cleanup, increases in property valuation from source removal or drainage/infrastructure enhancements (and savings/in-kind services to municipalities) should be considered.

- Remedial action should strive to remediate the contamination in the community by segregable areas, such as a town, or a divisible segment of town. Each segregable area should be cleaned up as quickly as possible (e.g., within one construction season) to minimize recontamination of cleaned properties and to compound the protection to human health (EPA, 2000e). Each community should be cleaned up block by block within these segregable areas, utilizing BMPs to mitigate tracking of contaminants. Site experience suggests that cleanup of up to 800 properties per site per year is possible.

- Fugitive dust that may be a source for recontamination, and access to such sources should be controlled. Air monitoring along with depositional modeling may be necessary to determine if windblown dust presents a significant threat of recontamination. Significant sources of windblown dust should be controlled prior to or simultaneously with cleanup of adjacent residential areas. Consider HEPA street sweeping during remediation and immediately following completion of cleanup to minimize tracking of contaminants throughout a community.

- Complete removal of contaminants should be considered in flood prone areas or areas with a high groundwater level due to the inherent difficulty in maintaining a soil cover remedy in a flood prone area. Drainage-ways containing contamination within their 100-year floodplain, which are

not addressed in the remedy could also lead to remedy failure if the contaminants are eroded to other areas.

C Remediation of contaminated rights-of-way should occur within segregable areas simultaneously, if possible, or as close together in time as possible to minimize vehicle tracking and recontamination of driveways from the rights-of-way.

C Control measures for all remaining sources, such as mining waste piles surrounding the community, should be developed to ensure the remediated neighborhoods are kept clean. ICs should be established to ensure the control, or proper use and disposal of any wastes remaining on site.

C If the residential remedy includes replacement of soils, removal of deteriorating exterior LBP (e.g., by pressure washing) should be considered to minimize the soil recontamination potential.

• Other sources of residential property recontamination should also be considered. For example, homeowners may bring in contaminated soil for fill or other uses on their property.

C Establish permanent funding for ICs. Unless all contaminants are removed, some level of ICs may be necessary. Early establishment of a program is the key to success of a remedy that consists of a partial removal of contaminants.

6.7.3 Institutional Controls (ICs)

EPA defines ICs as administrative and/or legal mechanisms that: (1) help minimize the potential for human exposure to contamination, and (2) protect the integrity of the remedy. ICs accomplish these objectives by directly limiting land or resource use, and/or by providing information that modifies behavior. ICs are used throughout the remedy pipeline, including (1) when contamination is first discovered (i.e., prohibition of excavation of newly discovered soil contamination), (2) when the remedy is ongoing (i.e., restrictions on property use until clean-up levels are met), and (3) when hazardous substances, pollutants, or contaminants remain at the site above levels that allow for unlimited use and unrestricted exposure.

At sites where minimizing exposure is the primary purpose of the IC, it is EPA's policy that if a site cannot support "unlimited use and unrestricted exposure" (EPA, 2000f), ICs are generally required. The "unlimited use and unrestricted exposure" threshold is a site-specific determination similar to that of a five-year review. Essentially, if contamination could result in an unacceptable exposure, ICs would be

required. This is often the case at lead cleanups because residual contamination is frequently managed onsite. Note that the term "residential" is often used interchangeably with the "unlimited use and unrestricted exposure" threshold but these are not synonymous terms. For example, a lead cleanup where the top layer of soil has been removed and replaced can result in a residential use at a site that includes restrictions (e.g., restrictions on digging, requirements for elevated gardens, and an information/outreach program, etc.).

The second common purpose of an IC is to protect the integrity of a remedy. In the lead clean-up context this may mean using institutional controls to prevent penetration of a cap or damage to monitoring equipment. An important consideration in this context is what type of IC will provide the required remedy protection. For example, the primary concern for protecting a remedy in a lead clean-up scenario is typically uncontrolled excavation. For this reason it is important to select ICs that will be relevant to excavators. Examples of potentially effective ICs are local digging or drilling permits and "One-Call" or "Miss Utility" systems. Examples of potentially ineffective ICs are deed notices, because excavators seldom check land records prior to digging.

To better understand the correct IC approach, it is important to understand what tools are available. In general, there are four categories of ICs commonly used in cleanups: governmental controls, proprietary controls, enforcement and permit tools with IC components, and informational devices. The definitions provided below were taken in large part from the current EPA guidance (EPA, 2000f).

Governmental controls are usually implemented and enforced by a state or local government. Some of the more common examples include things like zoning restrictions, building/excavation permits, groundwater drilling and use permits, ordinances, or other provisions that restrict land or resource use at a site. These types of mechanisms are popular in remedies because the administrative processes are in place and are typically well understood within a particular jurisdiction. The greatest concern with this type of control is that it is often implemented, monitored, and enforced by an agency other than EPA or the state.

Proprietary controls are unique in that they have their basis in real property law and that they generally create legal property interests. An example of this type of control is an easement that provides access rights to a property so that an agency may inspect and monitor a cover system. A proprietary control may also be used to restrict certain activities on the property, such as excavating below a certain depth. These are powerful tools in that they can be made to "run-with-the-land" (i.e., effective if ownership changes), but they provide significant challenges because property interests are often transferred. EPA is limited by CERCLA §104(j) with regard to acquiring interests in real property. Prior to acquiring an interest in real property the state must provide an assurance that it will accept transfer of

that interest at completion of the remedial action. This requirement applies at both Fund-lead and enforcement-lead sites. Therefore, if a proprietary control involves the transfer of an interest in real property, EPA must obtain this assurance and find an appropriate entity to hold the interest following the remedial action. At Fund-lead sites this will most likely be the state. At enforcement sites, it may be the state, a PRP, or some other interested and qualified party. In addition, proprietary controls are based on state law, and EPA and many state environmental agencies have limited real estate or common law experience. This can complicate proprietary control enforcement.

Enforcement and permit tools with IC components under CERCLA Sections 104 and 106(a) include unilateral administrative orders (UAOs) and AOCs, which can be issued or negotiated to compel the land owner to limit certain site activities at both federal and private sites. In addition, CERCLA 122(d) authorizes the use of consent decrees at privately-owned sites. Enforcement devices are some of the more common ICs. The strength of these types of tools is that EPA or states can directly enforce them (rather than relying on a local agency for governmental controls or using real estate common law for proprietary controls). The major weakness is that they may be enforceable only against the signatory, recipient, or permitee (i.e., may not run with the land to bind future property owners).

Unilateral Administrative Order (UAO) – When EPA negotiates with a Potentially Responsible Party (PRP) to do cleanup work at a Superfund site, the agreement may be documented in an administrative order on consent (AOC). If the negotiations fail, EPA has the authority to compel the PRP to do the cleanup by issuing a unilateral administrative order (UAO). Administrative orders are issued under CERCLA sections 104 and 106.

Informational devices are types of devices that only provide information or notification that residual or capped contamination may remain on-site. These types of tools are common at lead cleanups to both provide notification of residual contamination and to provide information that may modify behavior to minimize the potential for unacceptable exposure. Examples include placing a property on a state contaminated properties registry, developing deed notices, and providing periodic lead-education advisories to residents. Due to the nature of informational devices and their non-enforceability, it is important to carefully consider the objective of this category of ICs. Informational devices are most likely to be used as a secondary "layer" to help ensure the overall reliability of other ICs.

There is typically an inverse relationship between the amount of cleanup and the degree of reliance on ICs (i.e., the more cleanup, the less reliance on ICs). EPA tends to focus on a number of considerations when evaluating the long-term viability and amount of redundancy required for ICs at a particular site. EPA guidance strongly advocates the use of ICs in "layers" and/or in "series" (EPA, 2000f). Layering ICs means using multiple ICs concurrently (e.g., a consent decree, deed notice, educational/informational devices and a covenant). Using ICs in series is appropriate when IC

mechanisms are removed or changed as site circumstances change, such as reduction in restrictions during the clean-up life-cycle. As illustrated in the descriptions of the different categories of ICs, there are inherent strengths and weaknesses with each type. The goal is to obtain the best mixture of ICs to manage the risk at a site over the long-term. There are many important factors to consider when determining how many ICs are required at a site. The following is not intended to be a comprehensive list, but rather illustrative of the site-specific nature of these types of decisions. A few common considerations include: (1) the type of enforcement mechanism used (consent decree, order, permit, ordinance); (2) who will enforce the mechanism (i.e., EPA, the state, local agency, third party, etc.); (3) who the intended IC will effect and how; (4) the level of sophistication of the party implementing the cleanup and those remaining on the property; (5) the expected property use (likelihood of redevelopment and/or resale); and (6) the degree of cooperation exhibited by the parties to the cleanup. Since ICs can impact future development at sites, it is important to work cooperatively to determine the appropriate mix of ICs. The objective is not to use as many layers of ICs as possible, but rather to strike a balance that gives the regulators the certainty that the site remedy will be protective over time while maximizing the site's future beneficial use.

At many large lead sites, GIS systems are used to track the cleanup status of properties located on the site. The tracking system facilitates the monitoring of ICs and the maintenance of the remedy. GIS systems can be operated by local governments, state governments or PRPs.

6.8 CLEAN-UP DOCUMENTATION

Upon confirmation that initial yard sampling indicates a given residential yard does not exceed the lead clean-up level for the site, or upon the completion of the cleanup of a residential yard, a letter ("clean" letter) should be sent to the property owner documenting that EPA considers the lead level in the yard to be below the level of human health concern. Prior to issuing a "clean" letter, a property closeout form should be signed by the property owner, which documents the owner is satisfied with the remediation of the property. Examples of property closeout forms are proved in Appendix G. Any areas that are not cleaned up via the owner's request, such as gardens, should be noted in the "clean" letter. If contamination is not cleaned up to depth, this fact, along with protections (i.e., barriers/markers) that are put in place, should be stated in the "clean" letter. The "clean" letter provides official documentation to the property owner for use in future property sales or transactions. Sample "clean" letters are provided in Appendix H.

6.9 ENFORCEMENT

The project manager should strive to characterize all residences within the identified zone of contamination, and achieve cleanup at all residences where lead concentrations exceed the clean-up level. At all residential clean-up sites, a percentage of homeowners typically will refuse to grant access to EPA for sampling and/or for cleanup. In order to meet remedial goals of protecting a community, all residences suspected of being located within a zone of contamination should be sampled. It is important to work with the landowner and be sensitive to a landowner's concerns regarding property access. The project manager should educate the landowner of the dangers that lead contamination may pose. If a landowner still refuses to grant access, the Region should consider issuing an access order for sampling (EPA, 1990c).

An owner of residential property on a Superfund site may be potentially liable under CERCLA § 107(a)(1). However, EPA, as an exercise of enforcement discretion, generally will not take CERCLA enforcement actions against an owner of residential property unless the residential homeowner's activities lead to a release or threat of release of hazardous substances resulting in the taking of a response action at a site. (See Policy Towards Owners of Residential Property at Superfund Sites (July 3, 1991)). Additionally, under CERCLA a residential property owner may qualify for protection from CERCLA liability as a contiguous property owner, bona fide prospective purchaser, or innocent landowner. Under both the statute and EPA's policy, a residential property owner is expected to cooperate with EPA and the person taking the response action. This obligation includes providing access and information as requested, agreeing to comply with land use restrictions relied on in connection with the remedy, and not impeding the effectiveness the effectiveness or integrity of institutional controls. (See CERCLA §§ 101(40)(B)-(H), 107(q)(1)(a), 101(35)(A)-(B)). The project manager should work to inform and educate an owner of EPA's expectations for cooperation in connection with the remedy. If necessary, to meet the commitments of the remedy, EPA should consider taking appropriate steps, such as issuing a UAO, to secure the cooperation of an uncooperative landowner.

If some properties are not addressed under site response actions (e.g., current homeowners with no young children or women of child-bearing age), then consideration should be given to establishing a trust fund (under state authority or local law), to be administered by a local government, for the cleanup of the property at a future date, when the property is transferred (e.g., by sale) to a new owner (see text box). Buyers of contaminated properties could make use of the fund to have the property cleaned up at their discretion.

Example Trust Fund – At the Bunker Hill Superfund Site, a number of property owners refused to have their residential yards cleaned up. Without any obvious need to cleanup the property right away, e.g. an unpaved, contaminated driveway that threatens to recontaminate the neighborhood or a child living at the residence or next door, the PRPs for the site were willing to give the State funds to set aside in an interest bearing account to clean up the properties in the future, when the property changes hands. Property status is then monitored by the local Health District as part of the institutional controls program. The State then manages the funds to ensure maximum interest accrual in an irrevocable trust and disbursement according to the limitations set up in the trust -- for residential property cleanup. Cleanup then occurs under State oversight at the time new owners buy the property thereby ensuring families with children that move into the community are protected.

In the case of rental properties, EPA should order access for cleanup by UAO to all owners of contaminated rental property who refuse access. To ensure the protection of occupants, enforcement of the UAO may be necessary to clean up all rental properties with contamination greater than the clean-up level.

7.0 FIVE-YEAR REVIEW

> Five-Year Review – Pursuant to section 121 of CERCLA and the NCP, remedial actions which result in any hazardous substances, pollutants, or contaminants remaining at the site above levels that allow for unlimited use and unrestricted exposure need to be reviewed every five years to ensure protection of human health and the environment.

CERCLA §121(c) requires an assessment of certain remedial actions every five years on sites where contamination has been left on site (EPA, 2000a). Guidance for conducting five-year reviews has been issued (EPA, 2001h). The purpose of a five-year review is to evaluate the performance of a remedy to determine if the remedy continues to be protective of human health and the environment. Typically, at large lead sites, such as mining and smelting sites, the volume and areal extent of contamination is such that total removal of all contamination above the health-based risk level is economically impractical. Contaminated wastes are generally left on site and covered with soil. The remedy for these types of sites typically includes some type of IC to address residual or encapsulated contamination. A five-year review can determine whether the remedy is stable (i.e., soil covers are undisturbed, and clean areas are not being recontaminated from sources remaining on the site). The review should also assess the ICs that were established for residual source control to determine their effectiveness in protecting human health. As described below, the five-year reviews at large lead sites may involve the collection and evaluation of substantial quantities of data and require significant up-front planning. Much of the following discussion may not apply to small sites.

At many sites, an exposure study has been performed prior to any clean-up activities to determine blood lead concentrations of children in the community. A follow-up exposure study of residents should be conducted during the five-year review to determine if the concentrations have decreased below levels of concern. If the blood lead concentrations have not decreased to acceptable levels, additional environmental studies and individualized, follow-up exposure investigations should be conducted to determine the pathways of exposure that may need to be addressed. Long-term exposure studies can be very useful in understanding exposure trends at a site. They also can be useful to ensure that no pathways of exposure have been missed and to help identify areas of the site that have been recontaminated. In this manner, the project manager can use health data as a means to "double check" the effectiveness of the remedy and to corroborate environmental data. However, blood lead data from limited sampling should not be used as the only metric for gauging the success of a remedy, even if it can be used to identify specific problems. The project manager should coordinate with ATSDR and the local health district with respect to planning and funding such a program.

The five-year review should include resampling at a percentage of each type of property that was remediated during the clean-up actions. A baseline level of resampling should be designed to achieve a

pre-specified level of statistical significance and power. This sampling should assess the potential for recontamination that may be occurring, and may help identify any pathways that may have been missed during remediation. Any sampling that indicates widespread or clusters of soil levels above clean backfill concentrations should be monitored over time to determine if an upward trend exists that may jeopardize the remedy.

Additionally, some level of house dust sampling should occur to determine if levels are rising or falling. House dust, being a primary exposure pathway, should be used as one indicator of remedy effectiveness and also used to detect the presence of recontamination. Lead concentrations in house dust levels often correlate to interior LBP, which is not usually addressed by Superfund (Appendix B). Therefore, interior paint sampling should also be conducted as a component of the risk assessment to aid in determining the source of the lead loading to dust.

At large lead sites, remedy protectiveness issues will often relate to the implementation and management of ICs and recontamination of areas previously cleaned. The five-year review should evaluate the effectiveness of the site ICs and recommend corrections to address any deficiencies that are identified. In order for a five-year review to be effective at sites where ICs are a component in ensuring the effectiveness of the remedy, there should be: (1) clear documentation of the specific type of ICs that were to be implemented, and (2) accurate and complete tracking of subsequent activities and changes in property use following completion of the Superfund remedy.

The following are possible deficiencies for several types of commonly-used ICs and other control measures taken to ensure the protectiveness of the remedy:

- HEPA vacuum loan program not being broadly used.
- Information on interior home cleaning not being widely distributed.
- Lack of access control along rights-of-way, and in unremediated areas.
- Inadequate decontamination of vehicles leaving areas of existing contamination.
- Erosion of unremediated areas onto remediated properties.
- Lack of or inadequate disposal area for snow (that contains contaminated soil).
- Lack of drainage infrastructure and maintenance by local entities.
- Uncontrolled utility excavation in areas with contamination at depth.
- Inadequate road maintenance in areas where contamination exists at depth.
- Inadequate disposal capacity to handle IC-generated wastes.
- Discontinuation of, or diminishing, health education program.
- Decrease of blood lead monitoring.
- Complicated/unfounded ICs and/or change in local government acceptance of ICs.

8.0 FEDERAL FACILITIES

The purpose of this section includes the following: (1) to provide direction to EPA federal facility project managers who oversee response actions involving lead contamination of soils from LBP in residential areas of federal facilities; (2) to build and elaborate on the joint March 1999 EPA and DOD Principles Memorandum (DOD/EPA, 1999a) and the December 1999 Lead-Based Paint Interim Field Guide (DOD/EPA, 1999b); (3) to address situations where the DOD service component will conduct the response actions and the regulatory agencies will provide oversight; and (4) to address the unique considerations that arise when the federal government transfers LBP-contaminated property that is subject to CERCLA §120(h) to non-federal parties (e.g., states, local governments, local reuse authorities [LRAs], and private entities, etc.).

While existing policy, guidance, and directives on lead contamination are applicable at federal facilities, property transfer issues present unique requirements that necessitate this section. This section applies to properties that will be transferred for residential use which are contaminated with lead due to LBP or to properties/parcels whose use would expose sensitive populations (e.g., infants, toddlers, small children, nursing mothers) to unacceptable exposure to lead after the properties are transferred to non-Federal entities.

Beginning in 1995, EPA and DOD began to address policy differences on the clean-up levels for lead in soils from LBP. In 1998, Sherri Goodman, then Deputy Under Secretary of Defense (Environmental Security) and Tim Fields, Assistant Administrator for OSWER, reached agreement on the management of LBP at residential and non-residential areas at BRAC properties. In March 1999, this agreement was formalized as the 'Principles Memorandum' (DOD/EPA, 1999a). The Principles Memorandum stated that for residential areas located on BRAC sites, Title X procedures provide an efficient, effective, and legally adequate framework for addressing LBP in residential areas, and that as a matter of policy, CERCLA/RCRA would apply in limited circumstances. EPA and DOD agreed that generally for residential areas that were being transferred, Title X regulations would apply and that CERCLA/RCRA would apply in limited circumstances. Residential real property is defined by Title X as real property on which there is situated one or more residential dwellings used or occupied, in whole or in part, as the home or residence of one or more persons. It is important to note that Title X defines residential property differently than the Handbook.

For federal property transfers subject to CERCLA where there is a concern about lead contamination to soils from LBP, EPA Regions, where they are involved, will need to make a determination whether the property meets the requirements of CERCLA §120(h)(3). This section of CERCLA outlines deed requirements for transferring property and requires covenants indicating that all

remedial actions have been taken at the site. Federal property contaminated with lead from LBP should be evaluated based on its use, or its intended reuse, before the property has been sold or transferred to another private entity. EPA's evaluation of the transfer should be based on an evaluation of lead contamination by either relying on existing and available information gathered through a combination of file searches and a review of existing data and/or a site risk assessment, which may require the collection and analysis of additional soil samples.

The soil sampling design should be specific to the site. The actual or suspected presence of lead contamination in soil does not necessarily require sampling. Factors to be considered before designing a sampling plan include, but are not limited to, the nature of the facility's operations, its operating records, the age of the buildings/structures under consideration, the maintenance schedule for the buildings/structure, visual inspection, and future use. Based on these factors, it may be reasonable to conclude that the potential risks posed by lead may be acceptable and no further evaluation is needed. It may also be important to consider the ultimate disposition of the property once it leaves federal control. For example, the structures may be scheduled to be demolished, so that the abatement of the hazard may be addressed in the demolition process and may negate the need to conduct clean-up activities.

The EPA project manager and, as appropriate, an EPA risk assessor should work with their federal, state, and local government counterparts to develop a sampling design, where required, that would be scientifically appropriate, minimize the cost of sampling, and provide the information required for risk management decisions. As appropriate, the local redevelopment or reuse authority should be consulted as well. Information from the sampling effort could result in different outcomes: a "no further action decision", a conclusion that more extensive sampling is necessary, or, in some cases, a response action. All of these potential outcomes should be discussed with the lead federal agency, and others as appropriate, prior to the initiation of sampling.

If there is insufficient knowledge to make a conclusion about the risk at the site or if the initial sample results indicate an unacceptable risk from lead, data may be collected by a focused sampling of an environmental media to develop an improved understanding of the risk that may be posed by the lead exposure. It may be appropriate to determine that after visual inspection and/or focused sampling, and after consultation with an EPA risk assessor, the lead from the area may not pose a significant risk that requires further evaluation. Risk evaluations should be based upon a number of factors including the reasonably anticipated future land use, exposure potential, ICs proposed or in place, and bioavailability. The Handbook user is encouraged to obtain detailed information on ICs for federal facilities in the document "Institutional Controls and Transfer of Real Property under CERCLA Section 120(h)3(A), (B), or (C)" (EPA, 2000g).

If the property has been used or will be reused as residential real property after transfer, the EPA project manager should verify that the lead federal agency has followed the Title X regulations and policies regarding sampling and risk assessment. As a guide to assist site managers in understanding Title X regulations and policies, EPA and DOD jointly issued a Field Guide (DOD/EPA, 1999b) that is used by EPA and DOD field personnel when assessing hazards due to LBP. The field guide contains information on performing a Title X paint inspection and risk assessment and outlines the requirements for abating soil contaminated by LBP

The Title X program, through the implementation of the new Title IV of TSCA, establishes certification programs and work practice standards to regulate LBP hazard evaluation and abatement in target housing and child-occupied facilities. There are two types of evaluations covered by Title X. The first evaluation is a paint inspection that includes a surface-by-surface inspection to determine the presence of LBP. All painted surfaces with distinct painting histories are sampled. Usually the paint inspection is done by a combination of portable XRF devices and paint chip sampling.

The second evaluation is a risk assessment to determine if LBP hazards exist. A risk assessment includes taking samples of all deteriorating paint, dust, and soil. The final report recommends methods to deal with all LBP hazards that were found, which could include interim controls or abatement. A comprehensive evaluation consists of a combination of a paint inspection and risk assessment. Paint inspections and risk assessment conducted in accordance with Title X must be performed by certified personnel. All results, whether positive or negative, must be disclosed at the time of sale or rental.

The final TSCA 403 regulation (EPA/HUD, 2001), defines a soil-lead hazard as bare soil on residential real property, or on property of a child-occupied facility, that contains concentrations of lead equal to or exceeding 400 ppm in the play area or an average of 1,200 ppm in the rest of the yard. EPA and DOD have agreed that as a matter of policy, for bare soil with lead concentration between 400 ppm and 1,200 ppm, the Service, in consultation with the EPA, has the option of abatement or interim controls. Based on the final HUD 1012/1013 regulations (24 CFR Part 35) (HUD, 2001), federal agencies can transfer the control and abatement requirements to the purchaser, but by law the federal agency is responsible for performing the LBP inspection and risk assessment and must assure that through contractual mechanisms, the purchaser has performed the abatement of the soil in accordance with Title X.

In cases where the EPA project manager makes a determination that actions taken to address LBP hazards are sufficient (following the requirements outlined in the Field Guide), EPA should agree with the federal agency on the transfer documents and the covenant that all remedial action necessary to protect human health and the environment with respect to any such substances remaining on the property has been taken before the date of such transfer . In the case of BRAC sites, the EPA project manager can agree on the Findings of Suitability to Transfer (FOST) or Findings of Suitability to Lease (FOSL) language, and/or the operating properly and successfully (OPS) determination as required by CERCLA. When an EPA project manager has unresolved questions as to whether actions at residential areas meet the requirements of CERCLA, she/he should raise these issues to the federal agency and provide an opportunity for response. In the case of BRAC sites, it is proper to highlight these concerns in EPA's comments on the FOST/FOSL. Efforts should be made to determine that the purchaser is fully aware that EPA has questions about the condition of the property.

Finding of Suitability to Transfer (FOST) – A process that has been established to identify and prepare property for transfer by deed. Such transfers are usually undertaken at a property where environmental response is not needed or has been taken. However, under certain conditions, new authority now permits earlier transfer. The FOST process also looks at the compatibility of an anticipated reuse with completed restoration activities and identifies restrictions necessary to protect human health and the environment.

Finding of Suitability to Lease (FOSL) – A process that has been established for leasing of property that cannot be transferred by deed because environmental restoration activities are still ongoing. The FOSL process also looks at the compatibility of a proposed reuse with ongoing restoration activities and identifies restrictions necessary to protect human health and the environment and prevent interference with the cleanup.

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APPENDIX A

TITLE X AND EPA'S TOXIC SUBSTANCES CONTROL ACT (TSCA) TITLE IV LEAD PROGRAM

TITLE X AND EPA'S TOXIC SUBSTANCES CONTROL ACT (TSCA) TITLE IV LEAD PROGRAM

Background

The Housing and Community Development Act of 1992 (PL102-550) contained Title X the "Residential Lead-Based Paint Hazard Reduction Act of 1992" (HUD, 1992). Even though this was a U.S. Department of Housing and Urban Development (HUD) authorization bill, it established a series of requirements for EPA. Title X includes a new Title IV of the Toxics Substances Control Act (TSCA). The sections that address EPA alone have section numbers in the four hundred (400) series, such as Section 403, Health Based Standards, whereas the HUD portions have numbers in the one thousand (1000) series, such as Section 1015, Task Force. There is one section, Section 1018, that Congress required both HUD and EPA to jointly issue a rule on disclosure.

Overview

Title X addresses LBP and LBP hazards and requires EPA and HUD to issue regulations to address those items. Title X's emphasis is on actual hazards such as deteriorating paint, lead in dust, or lead in soil versus potential hazards such as intact paint. Generally, Title X does not mandate inspections, risk assessments, abatements of LBP, or LBP hazards. The exceptions are HUD program related actions (Section 1012) or when a federal agency disposes of a property that will be used for residential purposes (Section 1013). However, if you choose to do an inspection, risk assessment, or abatement, Title X establishes certification requirements and work practice standards that must be followed. Title X requires disclosure at the time of sale or rental (Section 1018) and the provision of a brochure *Protect Your Family from Lead in Your Home* (EPA, 1999a), before rehabilitation (Section 406b). EPA may authorize state programs to operate in lieu of the federal program for the 400 series regulations but not Section 1018. See Appendix A for a full discussion of Title X.

Scope of Title X

Title X contains specific classes of structures that it regulates. The first category is "target housing", which is defined as "...any housing constructed prior to 1978 except housing for the elderly or persons with disabilities (unless any child who is less than 6 years of age resides or is expected to reside in such housing for the elderly or persons with disabilities) or any 0-bedroom dwelling."

The second category is "child occupied facilities", which are defined as "... a building or a portion of a building, constructed prior to 1978, visited regularly by the same child, 6 years of age or under, on at least two different days within any week (Sunday through Saturday period), provided that each day's visit lasts at least 3 hours and the combined weekly visit lasts at least 6 hours, and the combined annual visits last at least 60 hours. Child-occupied facilities may include, but are not limited to, day-care centers, preschools and kindergarten classrooms" (EPA, 2001a).

As of December 2001 target housing and child occupied facilities are the only classes of structures for which EPA has issued final regulations.

CERCLA 121(e)(1) exempts any response action conducted entirely on-site from having to obtain a federal, state, or local permit, where the action is carried out under §121. In general, on-site actions need to comply only with the substantive aspects of ARARs and not with the corresponding administrative requirements. Therefore, the administrative requirements laid out under TSCA 402 and 403 are not considered ARARs for actions conducted entirely on-site.

More Information

Section 405 requires EPA to establish a Hot Line and Clearing House for lead. This has been done and the National Lead Information Center's toll free number is 1-(800)-424-LEAD. Additionally the EPA web site at www.epa.gov/lead has all the rules, fact sheets, and guidance documents that the EPA Office of Pollution Prevention and Toxics has developed.

Description of the Sections of Title X

Title X Final Rules in Effect for ONLY Target Housing:

Section 1012. This section establishes the requirements for those who get assistance or mortgage insurance from HUD. The requirements are HUD program specific, but only pertain to those who are involved with a particular HUD program.

Section 1013. This section establishes the requirements for federal agencies that dispose of target housing that will be used for residential purposes.

Section 1018. Section 1018 requires that sellers and landlords disclose known LBP and LBP hazards and provide available reports to buyers and renters. Sellers and landlords must also provide a copy of *Protect Your Family from Lead in Your Home* (EPA, 1999a).

This is a joint rule between EPA and HUD. Section 1018 does not include "child occupied facilities"; EPA developed the concept of "child occupied facilities" under TSCA Title IV, the term is only in effect for TSCA four hundred (400) series rules.

TSCA Final Rules in Effect for ONLY Target Housing and Child Occupied Facilities:

Section 402/404 State Certification Programs establishes a nationally consistent federal Program for the certification of individuals and firms engaged in training, paint inspections, risk assessments, and certification of abatement workers, supervisors and training providers. There are two aspects of the program. States and tribes are encouraged to establish a program that as a whole, is at least as protective as EPA's federal program. The state programs can be more protective. When a state program is approved, it becomes the federal program in that state.

If the state or tribe does not establish an acceptable certification program, EPA operates the national program in that state. Much of the work is done in the EPA Regional Office. As of December 2001, 39 states, the District of Columbia, and 2 tribes have EPA authorized programs. Two states with large populations, which do not have authorized programs, are New York and Florida.

Section 403 establishes hazard standards for lead in paint, dust, and soil. Lead-based paint is a hazard if (1) it is deteriorated; (2) it is present on a friction surface that is subject to abrasion and the dust-lead levels on the nearest horizontal surface are equal to or greater than the applicable dust hazard standard; or (3) it is present on any chewable surface on which there is evidence of teeth marks. (Lead-based paint is statutorily defined as paint containing 1.0 milligram or more lead per square centimeter or 0.5% or more lead by weight.) Dust is a hazard if it contains 40 micrograms or more lead per square foot on floors or 250 micrograms or more lead per square foot on window sills. Soil is a hazard if it contains 400 parts per million or more in play areas or 1,200 parts per million or more in the rest of the yard.

This regulation also established the following clearance levels for interior dust: 40 micrograms lead per square foot for floors, 250 micrograms lead per square foot for window sills, and 400 micrograms lead per square foot for window troughs.

EPA's Section 403 rule was intended to prioritize risks as opposed to being inclusive of situations in which risks of concern exist. Per the rule preamble, *"The hazard standard in this TSCA rule was intended as a ''worst first'' level that will aid in setting priorities to address the greatest lead risks promptly at residential and child-occupied facilities affected by lead-based paint"* (EPA, 2001a). While identification of lead hazards (as defined under TSCA) is a necessary part of the facility reuse process, a minimal approach that would insure only that the letter of the hazard standards are met may not protect against some important risks.

Section 405 establishes standards of environmental sampling laboratories. The National Lead Laboratory Accreditation Program (NLLAP) is administered by the American Industrial Hygiene Association and the American Association for Laboratory Accreditation. All laboratory samples must be analyzed by an NLLAP accredited laboratory.

Section 406b requires that the pamphlet *Protect Your Family from Lead in Your Home* (EPA, 1999a) be distributed no more than 60 days before a renovation in the home.

TSCA Rules Being Developed

Section 402. Renovation and remodeling requirements for target housing and child occupied facilities are being drafted as a proposed rule. Requirements for bridges and structures constructed prior to 1978 are being drafted for re-proposal. Both of these could include training, certification, and work practice standards.

Lead-based Paint Debris. This rule was not required by Title X, but the need was clearly there to treat portions of the debris from lead-based activities differently than the RCRA requirements. There are two categories of waste discussed. First is the paint chips and dust, sludges and filtercakes, wash water and contaminated and decontaminated protective clothing equipment that would continue to be subject to all the requirements of RCRA. Second is the "lead-based paint architectural component debris", which would be exempt from the Toxicity Characteristics rule including Toxicity Characteristic Leaching Procedure (TCLP) testing for lead only. This would allow disposal of these components at construction-demolition (CD) landfills.

Although the Pb Debris Rule is still being developed, in the interim, **EPA has issued a Memorandum that "Regulatory Status of Waste Generated by Contractors and Residents from Lead-Based Paint Activities Conducted in Households" - signed July 31, 2000.** This memo clarifies the regulatory status of waste generated as a result of LBP activities (including abatement, renovation activities, and remodeling) in homes and other residences. This memo explains why LBP generated by contractors in households is "household waste" and thus excluded from the RCRA Subtitle C hazardous waste regulations. The household exclusion applies only to waste generated by either residents or contractors conducting LBP activities in residents. As a result, LBP waste from residences can be discarded in a municipal solid waste landfill or a municipal solid waste combustor.

APPENDIX B

1998 OSWER Directive 9200.4-27P ('Clarification')

9200.4-27
EPA/540/F-98/030
PB98-963244

OSWER Directive # 9200.4-27P

MEMORANDUM

SUBJECT: Clarification to the 1994 Revised Interim Soil Lead Guidance for CERCLA Sites and RCRA Corrective Action Facilities

FROM: Timothy Fields, Jr.
Acting Assistant Administrator

TO: Regional Administrators I-X

PURPOSE

This directive clarifies the existing 1994 Revised Interim Soil Lead Guidance for CERCLA Sites and RCRA Corrective Action Facilities, OSWER Directive 9355.4-12. Specifically, this directive clarifies OSWER's policy on (1) using EPA's Science Advisory Board (SAB) reviewed Integrated Exposure Uptake Biokinetic Model (IEUBK) and blood lead studies, (2) determining the geographic area to use in evaluating human exposure to lead contamination ("exposure units"), (3) addressing multimedia lead contamination and (4) determining appropriate response actions at lead sites. The purpose for clarifying the existing 1994 directive is to promote national consistency in decision-making at CERCLA and RCRA lead sites across the country.

BACKGROUND

OSWER Directive 9355.4-12, issued on July 14, 1994 established OSWER's current approach to addressing lead in soil at CERCLA and RCRA sites. The existing directive established a streamlined approach for determining protective levels for lead in soil at CERCLA sites and RCRA facilities as follows:

- It recommends a 400 ppm screening level for lead in soil at residential properties;
- It describes how to develop site-specific preliminary remediation goals (PRGs) at CERCLA sites and media cleanup standards at RCRA Corrective Action facilities for residential land use; and,
- It describes a strategy for management of lead contamination at CERCLA sites and RCRA Corrective Action facilities that have multiple sources of lead.

The existing interim directive provides direction regarding risk assessment and risk management approaches for addressing soil lead contaminated sites. The OSWER directive states that, " ... implementation of this guidance is expected to provide more consistent decisions across the country ..." However, since that directive was released, OSWER determined that clarification of the guidance is needed. Key areas being clarified by issuance of this directive include: (1) using the IEUBK model and blood lead studies, (2) determining exposure units to be considered in evaluating risk and developing risk management strategies, (3) addressing multimedia lead contamination and (4) determining appropriate response actions at residential lead sites. The existing directive provides the following guidance on these areas:

1. The OSWER directive recommends using the Integrated Exposure Uptake Biokinetic (IEUBK) Model for Lead in Children (Pub. # 9285.7-15-1, PB93-963510) for setting site-specific residential preliminary risk-based remediation goals (PRGs) at CERCLA sites and media cleanup standards (MCSs) at RCRA corrective actions Facilities. The directive states that the IEUBK model is the best tool currently available for predicting the potential blood lead levels of children exposed to lead in the environment. OSWER's directive also recommends the evaluation of blood lead data, where available, and states that well-conducted blood lead studies provide useful information to site managers. The directive however recommends that "... blood lead data not be used alone to assess risk from lead exposure or to develop soil lead cleanup levels."

2. The directive describes OSWER's risk reduction goal as "...generally, OSWER will attempt to limit exposure to soil lead levels such that a typical (or hypothetical) child or group of similarly exposed children would have an estimated risk of no more than 5% of exceeding a 10 ìg/dl blood lead level." The directive also states that "... EPA recommends that a soil lead concentration be determined so that a typical child or group of children exposed to lead at this level would have an estimated risk of no more than 5% of exceeding a blood lead of 10 ìg/dl." OSWER generally defines an exposure unit as a geographic area where exposures occur to the receptor of concern during the time of interest and believes that for a child or group of similarly exposed children, this is typically the individual residence and other areas where routine exposures are occurring.

3. The directive recommends that risk managers assess the contribution of multiple environmental sources of lead to overall lead exposure (e.g., consideration of the importance of soil lead levels relative to lead from drinking water, paint, and household dust) which promotes development of risk reduction strategies that address all sources that contribute significantly to exposure.

4. The OSWER directive states that the IEUBK model is not the only factor to be considered in establishing lead cleanup goals. Rather, the IEUBK model is the primary risk assessment tool available for evaluating lead risk and the results of the model are used to guide selection of appropriate risk management strategies for each site.

Since the OSWER directive was issued in 1994, there has been a trend toward a more consistent approach to managing risk at residential lead sites, however, OSWER was interested in identifying areas requiring additional clarification to facilitate more effective implementation of the directive. As a first step in the process, meetings were held with various EPA Regions, States and local governments to discuss how the directive has been implemented nationally at lead sites since 1994. By participating in these meetings and by reviewing the decisions that are being made across the country, OSWER believed that clarification of certain aspects of the 1994 directive would be useful.

All of the documents and guidance referenced in this directive are available through the National Technical Information Service (NTIS) at 703-605-6000 or could be downloaded electronically from: http//epa.gov/superfund/oerr/ini_prod/lead/prods.htm.

OBJECTIVE

At lead contaminated residential sites, OSWER seeks assurance that the health of the most susceptible population (children and women of child bearing age) is protected and promotes a program that proactively assesses and addresses risk. OSWER believes that predictive tools should be used to evaluate the risk of lead exposure, and that cleanup actions should be designed to address both current and potential future risk.

While health studies, surveys, and monitoring can be valuable in identifying current exposures and promoting improved public health, they are not definitive tools in evaluating potential risk from exposure to environmental contaminants. In the case of lead exposure, blood lead monitoring programs can be of critical importance in identifying individuals experiencing potential negative health outcomes and

directing education and intervention resources to address those risks. However, CERCLA §121(b) requires EPA to select cleanup approaches that are protective of human health and the environment and that utilize permanent solutions to the maximum extent practicable. To comply with the requirements set forth in CERCLA §121(b), OSWER will generally require selection of cleanup programs that are proactive in mitigating risk and that do not simply rely on biological monitoring programs to determine if an exposure has already occurred.

To meet these objectives, OSWER will seek actions that limit exposure to soil lead levels such that a typical child or group of similarly exposed children would have an estimated risk of no more than 5% of exceeding a 10 ìg/dl blood lead level. If lead is predicted to pose a risk to the susceptible population, OSWER recommends that actions be taken to significantly minimize or eliminate this exposure to lead.

The principles laid out in the **four attached fact sheets** (Appendix) support OSWER's goals by encouraging appropriate assessment and response actions at CERCLA and RCRA lead sites across the country.

This clarification directive emphasizes the following key messages regarding the four areas and encourages the users of this directive, be they EPA Regions, States, or other stakeholders, to adopt these principles in assessing and managing CERCLA and RCRA lead sites across the country. The critical elements of the attached papers are as follows:

*I. Using Blood Lead Studies and IEUBK Model at Lead Site*s:

OSWER emphasizes the use of the IEUBK Model for estimating risks for childhood lead exposure from a number of sources, such as soils, dust, air, water, and other sources to predict blood lead levels in children 6 months to 84 (7 years) months old. The 1994 directive also recommended evaluation of available blood lead data and stated that data from a well-conducted blood lead study of children could provide useful information to site managers. In summary, OSWER's clarification policy on the appropriate use of the IEUBK and blood lead studies is that:

- OSWER recommends that the IEUBK model be used as the primary tool to generate risk-based soil cleanup levels at lead sites for current or future residential land use. If Regions propose an alternative method for generating cleanup levels, they are required to submit their approach to the national Lead Sites Consultation Group (LSCG)[1] for review and comment;

- Response actions can be taken using IEUBK predictions alone; blood lead studies are not required; and

- Blood lead studies and surveys are useful tools at lead sites and can be used to identify key site-specific exposure pathways and to direct health professionals to individuals needing immediate assistance in minimizing lead exposure; however, OSWER recommends that blood lead studies not be used for establishing long-term remedial or non-time-critical removal cleanup levels at lead sites.

II. Determining Exposure and Remediation Units at Lead Sites

[1]The Lead Sites Consultation Group (LSCG) is comprised of senior management representatives from the Waste Management Divisions in all 10 EPA regions along with senior representatives from the Office of Emergency and Remedial Response in EPA headquarters. The LSCG is supported by EPA's Technical Review Workgroup (TRW) for lead and the national Lead Sites Workgroup (LSW). The TRW consists of key scientific experts in lead risk assessment from various EPA Regions, labs and headquarters. The LSW is comprised of senior Regional Project Managers from various Regions and key representatives from headquarters who are experienced in addressing lead threats at Superfund sites.

OSWER recommends that cleanup levels at lead sites be designed to reduce risk to a typical or individual child receiving exposures at the residence to meet Agency guidelines *(i.e*., no greaterthan a 5% chance of exceeding a 10 ：g/dl blood lead level for a full-time child resident). Therefore, it is recommended that risk assessments conducted at lead-contaminated residential sites use the individual residence as the primary exposure unit of concern. This does not mean that a risk assessment should be conducted for every yard, rather that the soil lead contamination data from yards and other residential media (for example, interior dust and drinking water) should be input into the IEUBK model to provide a preliminary remediation goal (PRG) for the residential setting. When applicable, potential exposure to accessible site-related lead sources outside the residential setting should also be evaluated to understand how these other potential exposures contribute to the overall risk to children, and to suggest appropriate cleanup measures for those areas.

III. Addressing Multimedia Contamination at Lead Sites

EPA generally has limited legal authority to use Superfund to address exposure from **interior lead-based paint**. As a policy matter, OSWER recommends that such exposures not be addressed through actual abatement activities. However, EPA Regions should promote addressing interior paint risks through actions by others (*e.g.*, potentially responsible parties (PRPs), other government programs, etc.) as a component of an overall site management strategy. Because of other competing demands on the Superfund Trust Fund, OSWER recommends that EPA Regions avoid using the Superfund Trust Fund for removing **exterior lead-based paint** and soil contaminated from lead-based paint. Superfund dollars *may* however be used in limited circumstances to remediate exterior lead-based paint in order to protect the overall site remedy (*i.e.*, to avoid re-contamination of soils that have been remediated) but generally only after determining that other funding sources are unavailable. As with interior lead-based paint abatement, EPA Regions should promote remediation of exterior lead-based paint by others, such as PRPs, local governments or individual homeowners.

IV. Determining Appropriate Response Actions at Lead Sites

In selecting site management strategies, it is OSWER's preference to seek early risk reduction with a combination of engineering controls (actions which permanently remove or treat contaminants, or create reliable barriers to mitigate the risk of exposure) and non-engineering response actions. All potential lead sources should be identified in site assessment activities. Non-engineering response actions, such as education and health intervention programs, should be considered an integral part of early risk reduction efforts because of their potential to provide immediate health benefits. In addition, engineering controls should be implemented early at sites presenting the greatest risk to children and other susceptible subpopulations.

As a given project progresses, OSWER's goal should be to reduce the reliance on education and intervention programs to mitigate risk. The goal should be cleanup strategies that move away from reliance on long-term changes in community behavior to be protective since behavioral changes may be difficult to maintain over time. The actual remedy selected at each CERCLA site must be determined by application of the National Oil and Hazardous Substances Pollution Contingency Plan (NCP) (55 FR 8666- 8865, March 8, 1990) remedy selection criteria to site-specific circumstances. This approach also recognizes the NCP preference for permanent remedies and emphasizes selection of engineering over non-engineering remedies for long-term response actions.

This directive clarifies OSWER's policy on four key issue areas addressed in the 1994 OSWER soil lead directive in order to promote a nationally consistent decision-making process for assessing and managing risks associated with lead contaminated sites across the country. The policy presented in these specific issue areas supersedes all existing OSWER policy and directives on these subjects. No other aspects of the existing 1994 directive are affected.

IMPLEMENTATION

The principles laid out in this directive (which includes the four attached factsheets) are meant to apply to all residential lead sites currently being evaluated through the CERCLA Remedial Investigation/Feasibility Study process and all future CERCLA Sites and RCRA Corrective Action Facilities contaminated with lead. The Regions will be required to submit their rationale for deviating from the policies laid out in this directive to the Lead Sites Consultation Group. This directive does not apply to previous remedy selection decisions.

Attachments

cc: Waste Management Policy Managers (Regions I-X)
Stephen Luftig, OERR
Elizabeth Cotsworth, OSW
James Woolford, FFRRO
Barry Breen, OSRE
Larry Reed, OERR
Tom Sheckells, OERR
Murray Newton, OERR
Betsy Shaw, OERR
John Cunningham, OERR
Paul Nadeau, OERR
Bruce Means, OERR
Earl Salo, OGC

NOTICE: This document provides guidance to EPA staff. The document does not, however, substitute for EPA's statutes or regulations, nor is it a regulation itself. Thus it cannot impose legally-binding requirements on EPA, states, or the regulated community, and may not apply to a particular situation based upon the circumstances. EPA may change this guidance in the future, as appropriate.

Factsheet: Using the IEUBK Model and Blood Lead Studies at Residential Lead Sites

Question: What is OSWER's policy on using the IEUBK model and blood lead studies in conducting risk assessments and setting cleanup standards at residential lead contamination sites?

Answer: OSWER's policy on using the IEUBK model and blood lead studies in conducting risk assessment and setting cleanup standards is as follows:

A. Use of the IEUBK Model:

1. The IEUBK model is a good predictor of potential long-term blood lead levels for children in residential settings. OSWER recommends that the IEUBK model be used as the primary tool to generate risk-based soil cleanup levels at lead sites for current or future residential land use. If Regions propose an alternative method for generating cleanup levels, they are required to submit their approach to the National Lead Sites Consultation Group (LSCG) for review and comment.

2. Blood lead distributions predicted by the IEUBK model illustrate a plausible range of variability in children's physiology, behavior, and household conditions.

3. Response actions can be taken, and remedial goals developed, using IEUBK predictions alone.

B. Use of Blood Lead Studies/Data:

1. Blood lead studies, surveys, and monitoring are useful tools at lead sites and can be used to help identify key site-specific exposure pathways and direct health professionals to individuals needing immediate assistance in minimizing lead exposure.

2. The utility of blood lead testing results and studies depends on how representative the information is of the population being evaluated, the design of the data collection, and the quality of the laboratory analysis. To this end, OSWER recommends that EPA Regions consult with ATSDR or CDC to assess or design studies according to their intended use.

3. Many blood lead screening, monitoring, or testing programs differ from blood lead studies in that they do not attempt to identify risk factors for childhood exposure to lead sources. Although these programs may be extremely beneficial in identifying children with elevated blood lead levels and identifying candidates for referral to medical professionals for evaluation, they may not provide an accurate representation of community-wide exposure.

4. Well-designed blood lead studies may be used to identify site specific factors and pathways to be considered in applying the IEUBK model at residential lead sites. However, OSWER recommends that blood lead studies not be used to determine future long-term risk where exposure conditions are expected to change over time; rather, they should be considered a snapshot of ongoing exposure under a specific set of circumstances (including community awareness and education) at a specific time. Long-term studies may be helpful in understanding exposure trends within a community and evaluating the effectiveness of cleanup strategies over time.

C. IEUBK and Blood Lead Studies/Data:

1. Blood lead data and IEUBK model predictions are expected to show a general concordance for most sites. However, some deviations between measured and predicted levels are expected. On some occasions, declines in blood lead levels have been observed in association with lead exposure-reduction and health education. However, long-term cleanup goals should be protective

in the absence of changes in community behavior as there is little evidence of the sustained effectiveness of these education/intervention programs over long periods of time.

2. Where actual blood lead data varies significantly from IEUBK Model predictions, the model parameters should not automatically be changed. In such a case, the issue should be raised to the Lead Technical Review Workgroup (TRW) to further identify the source of those differences. Site work need not be put on hold while the issue is being reviewed by the TRW; the site manager should review other elements of the lead directive and the "Removal Actions at Lead Sites" guidance to determine appropriate interim actions to be taken at the site.

The Regions will be required to submit their rationale for deviating from the policies laid out in this factsheet to the Lead Sites Consultation Group.

Factsheet: Determining Exposure and Remediation Units at Residential Lead Sites

Question: How does OSWER define an exposure unit, and subsequently apply this definition in conducting risk assessment and risk management activities at residential lead sites?

Answer: OSWER recognizes that defining and characterizing exposure unit(s) for a site is critically important in undertaking risk assessment activities and in designing protective cleanup strategies. An **exposure unit** is defined as a geographic area where exposures occur to the receptor of concern during the time of interest and that for a child, or group of similarly exposed children, this is typically the individual residence and other areas where chronic or ongoing exposures are occurring.

Various approaches to characterizing and managing risks by exposure units have been examined by OSWER. OSWER recognizes that lead ingestion can also cause adverse health effects in adults and fetuses but believes that by adequately limiting lead exposures to young children at residential sites, these other receptors will generally be likewise protected from adverse health impacts.

EPA's goal is to protect human health and the environment under current and future exposure scenarios. At lead sites, OSWER wants to assure that children's health is protected and promotes a program that proactively assesses risks rather than relying on biological monitoring to determine if an exposure has already occurred. OSWER emphasizes actions be taken at lead sites that will minimize or eliminate exposure of children to environmental lead contamination.

To achieve the above stated goal, OSWER recommends characterizing **exposure units as exposure potential at the individual residence as the primary unit of concern for evaluating potential risk at lead contaminated residential sites**. This recognizes that there are children whose domain and activities occur principally within the confines of a particular residential property. For determining exposure potential (and ultimately developing protective cleanup levels) at the individual home, OSWER recommends the scenario to be evaluated (through use of the IEUBK Model) would be a young child in full-time residence. This approach helps achieve OSWER's recommended health protection goal that an individual child or group of similarly exposed children would have <5% chance of exceeding a blood lead concentration of 10 : g/dl. In designing community wide cleanup strategies, it is essential that non-residential areas (*e.g.*, parks, day care facilities, playgrounds, etc.), where lead exposure may occur, also be characterized with respect to their contribution to soil-lead exposure, and appropriate cleanup actions implemented.

OSWER recommends that risk management decisions for response to residential lead contamination sites focus on reducing risk at residences, but also recommends that response strategies be developed for other site locations (exposure units) where children receive exposure. Flexibility in determining appropriate response actions that provide protection at the individual residence should be considered in context of the NCP remedy selection criteria. The lead exposure issues are complex and OSWER recommends that EPA Regions try to communicate clearly the risk characterization and risk management decisions to the site residents. Affected communities must clearly understand the context of risk management decisions, how these decisions affect the health of their children, and how cleanup actions will influence the future growth and development of the community.

The Regions will be required to submit their rationale for deviating from the policies laid out in this factsheet to the Lead Sites Consultation Group.

Factsheet: Addressing Multimedia Contamination at Residential Lead Sites

Question: What is OSWER's policy on addressing multimedia contamination at residential lead sites?

Answer: OSWER recognizes that several sources of lead-contamination, including soil, ground water, airborne particulates, lead plumbing, interior dust, and interior and exterior lead-based paint may be present at Superfund sites where children are at risk or have documented lead exposure. These lead sources may contribute to elevated blood lead levels and may need to be evaluated in determining risks and cleanup actions at residential lead sites. However, there are limitations on the Agency's statutory authority under CERCLA to abate some of these sources, such as indoor lead-based paint and lead plumbing because CERCLA responses may be taken only to releases or threatened releases into the environment (CERCLA §104 (a)(3) and (4)).

When EPA's resources, or authority to respond or to expend monies under Superfund is limited, OSWER recommends that EPA Regions identify and coordinate to the greatest extent possible with other authorities and funding sources (*e.g.*, other federal agencies and state or local programs). EPA Regions should coordinate with these other authorities to design a comprehensive, cost-effective response strategy that addresses as many sources of lead as practicable. These strategies should include actions to respond to lead-based paint, interior dust, and lead plumbing, as well as ground water sources and lead-contaminated soil.

Although OSWER will encourage that EPA Regions fully cooperate in the development of a comprehensive site management strategy, OSWER realizes that complete active cleanup of these other sources may be difficult to complete due to limited funding available to other authorities. Since complete cleanups of these sources is not guaranteed, and at most sites may be unlikely, OSWER recommends that the soil cleanup levels not be compromised. In other words, the soil cleanup levels should be calculated with the IEUBK model using existing pre-response action site specific data. This is due to the fact that soil cleanup levels at residential lead sites are generally established to protect individuals, from excess exposures to soils, and house dust attributable to those soils, and are not attributable to exposure to other sources such as interior lead paint which should be managed on a residence specific basis. Remediation of non-soil lead sources to mitigate overall lead exposure at individual residences should therefore not be used to modify site-wide soil lead cleanup levels.

The recommendations provided below represent OSWER's policy on addressing lead-contaminated media and/or sources for which EPA has limited or no authority to remediate.

Interior Paint: EPA has limited legal authority to use Superfund to address exposure from interior lead-based paint. As a policy matter, OSWER recommends that such exposures not be addressed through actual abatement activities. However, EPA Regions should promote addressing interior paint risks through actions by others, such as HUD, local governments, or individual home owners as a component of an overall site management strategy. Any activities to clean up interior lead-based paint by PRPs or other parties should not result in an increase of the risk-based soil cleanup levels.

Exterior Paint: Because of other competing demands on the Superfund Trust Fund, OSWER recommends that EPA Regions avoid using the Superfund Trust Fund for removing exterior lead-based paint and soil contaminated from lead-based paint. Superfund dollars *may* be used to respond to exterior lead-based paint for protecting the overall site remedy (*i.e.*, to prevent re-contamination of soils that have been remediated) but only after determining that other funding sources are unavailable. Where other sources of funding are not available, EPA may utilize the CERCLA monies to remediate exterior lead-based paint on homes/buildings, around which soil contaminated by other sources has been cleaned up to prevent recontamination of the soil. The Superfund should not be used to remediate exterior lead-based paint where no soil cleanup has occurred. As with interior lead-based paint abatement, EPA Regions

should promote remediation of exterior lead-based paint by others, such as PRPs, local governments or individual homeowners. Cleanup activities of exterior paint conducted by PRPs or other parties should not result in an increase of the risk-based soil cleanup levels.

Interior Dust: Lead contaminated interior dust can be derived from several sources, including interior paint, home owner hobbies, exterior soil, and other exterior sources. In many cases, it may be difficult to differentiate the source(s) for the lead contamination in the dust. In general, EPA Regions should refrain from using the Superfund Trust Fund to remediate interior dust. Because of the multi-source aspects of interior dust contamination, potential for recontamination, and the need for a continuing effort to manage interior dust exposure, OSWER recommends the use of an aggressive health education program to address interior dust exposure. Such programs, administered through the local health department (or other local agency), should be implemented in conjunction with actions to control the dust source. At a minimum, the program should include blood lead monitoring, and personal hygiene and good housekeeping education for the residents. OSWER believes that EPA Regions can also support the program by providing HEPA vacuums to the health agency for use in thoroughly cleaning home interiors.

Lead Plumbing: Generally CERCLA does not provide for legal authority to respond to risks posed by lead plumbing within residential dwellings. It should be noted that the water purveyor is responsible for providing clean water to the residences. As with interior dust, OSWER recommends that EPA Regions coordinate with local agencies to establish a health education program to inform residents of the hazards associated with lead plumbing and how to protect themselves by regularly flushing, or preferably, replacing lead pipes. Soil cleanup levels should not be adjusted to account for possible remediation of lead plumbing.

Factsheet: Determining Appropriate Response Actions at Residential Lead Sites

Question: What is OSWER's position on the appropriate use of engineering and non-engineering response actions in developing risk management strategies for lead sites?

Answer: One goal emphasized in the recent third round of Superfund Reforms is for EPA to take a consistent approach in selecting and implementing both long- and short-term response actions at lead sites in all regions. One obstacle to achieving this consistency has been differing degrees of reliance on non-engineering response actions in reducing risk.

Site management strategies at lead sites typically include a range of response actions. Alternatives range from engineering controls that permanently remove or treat the contaminant source to non-engineering response actions, such as educational programs and land use restrictions. This continuum represents the range of response options available to risk managers. This position paper clarifies the relationship between engineering and non-engineering response actions in developing site management strategies.

In selecting site management strategies, OSWER's policy will be to seek early risk reduction with a combination of engineering controls (actions which permanently remove or treat contaminants, or which create reliable barriers to mitigate the risk of exposure) and non-engineering response actions. All potential lead sources should be identified in site assessment activities. Non-engineering response actions, such as education and health intervention programs, should be considered an integral part of early risk reduction efforts due to their potential to provide immediate health benefits.[2] In addition, engineering controls should be implemented early at sites presenting the greatest risk to children and other susceptible subpopulations. Community concerns should receive a high priority in site decision-making; local support is vital to the success of health intervention and education programs.

As the project progresses, OSWER's goal should be to reduce reliance on education and intervention programs to mitigate risk. The goal should be cleanup strategies that move away from reliance on long-term changes in community behavior to be protective; behavioral changes may be difficult to maintain over time. The actual remedy selected at each site must be determined by application of the NCP remedy selection criteria to site-specific circumstances. However, this approach recognizes the NCP preference for permanent remedies and emphasizes the use of engineering controls for long-term response actions. This approach also recognizes that well-designed health intervention and education programs, when combined with deed restrictions and/or other institutional controls, may be appropriate for reducing future exposure potential and may supplement engineering controls.

In instances where Regions believe that the use of engineering controls is impracticable, and education, health intervention, or institutional controls are proposed as the sole remedy, Regions will be required to consult with the LSCG.

[2]The actual effectiveness of health intervention and educational programs in reducing risk continues to be a subject of discussion. Anecdotal information suggests that such programs can provide short-term benefits in some populations. Rigorous statistical studies demonstrating the benefits of educational programs in preventing lead exposure are lacking. It is generally recognized that not all segments of the population will be influenced by such programs, and that long-term benefits are less certain. Local support for such programs is critical. The active (and long-term) participation of local and state public health agencies is needed in implementing institutional controls, including health intervention and education programs; without local implementation of such programs their success is uncertain. Additional research on the effectiveness of these programs is critical to consideration of their use in future cleanups.

APPENDIX C

Contacts and Software for Sampling Design

Table C-1
Contacts and Software for Sample Planning Design

Topic		Contact(s)
Sampling plan design/ Systematic Planning	General support	EPA HQ Quality Staff Phone: (202) 564-6830 FAX: (202) 565-2441 E-mail: quality@epa.gov
	Dynamic Field Activities	Internet: http://www.epa.gov/superfund/programs/dfa/index.htm
Software	DEFT: Data Quality Objectives Decision Error Feasibility Trials	E-mail: quality@epa.gov Internet: http://www.ornl.gov/doe_oro/dqo/resdqo.htm
	FIELDS: Fully Integrated Environmental Decision Support	Internet: http://www.epa.gov/region5fields/static/pages/index.html
	Geo-EAS: Geostatistical Environmental Assessment Software	E-mail: englund.evan@epa.gov Internet: http://www.ai-geostats.org/
	SADA: Spatial Analysis Decision Assistance	E-mail: sada@tiem.utk.edu Internet: http://www.tiem.utk.edu/~sada/
	VSP: Visual Sample Plan	E-mail: nell.cliff@pnl.gov Internet: http://dqo.pnl.gov/vsp/

APPENDIX D

Examples of Property Access Agreement Forms

CONSENT FOR ACCESS TO PROPERTY FOR SAMPLING

Name: _____ Daytime Phone Number: _____

Address(es) of Property(ies): _____

_____

I consent to officers, employees, and authorized representatives of the United States Environmental Protection Agency (EPA) entering and having access to my property for the purpose of taking [DESCRIBE NUMBER OF SAMPLING LOCATIONS AND DEPTHS] which are necessary to implement the cleanup of lead contamination in the soil.

This written permission is given by me voluntarily with knowledge of my right to refuse and without threats or promises of any kind. I understand that EPA or authorized representatives of EPA will contact me at least one week in advance before the soil samples are collected. This agreement is only for the purpose of soil sampling and no other work.

_____
Date

☐ I grant access to my property

☐ I do not grant access to my property

_____
Signature

_____
Signature

☐ I would also like EPA to have a lead expert contact me to schedule a free inspection to identify potential lead hazards in my home and provide safety tips.

United States Environmental Protection Agency Region 6
1445 Ross Avenue, Suite 1200
Dallas, Texas 75202-2733

CONSENT FOR ENTRY AND ACCESS TO PROPERTY FOR SAMPLING

Description of property (including address) for which consent to access is granted:

Example: XXXX Street, Texarkana, Arkansas, more particularly described as a lot measuring approximately 3,000 square feet, including a two-room wood structure of approximately 300 square feet

Name of Signatory: _____

Address: _____
_____
_____ Phone: (_____)_____

Relationship to property (e.g., owner, lessee, agent or employee of owner, etc.):

I HEREBY CONSENT to officers, employees and parties authorized by the U.S. Environmental Protection Agency (EPA), entering and having continued access to the property described above at reasonable times for the following purposes (List the activities to be undertaken on the property):
Example:

- Sample collection including: (1) the gathering of soil from the outside area of the property; (2) drawing water from the tap; and (3) vacuuming the inside area of any inhabitable structure in order to collect dust.

- Taking photographs to record the sampling process.

I realize that these actions are undertaken pursuant to EPA's response and enforcement responsibilities under the Comprehensive Environmental Response, Compensation, and Liability Act (CERCLA), 42 U.S.C. Sections 9601-9675. This written permission is given by me voluntarily with the knowledge of my right to refuse and without threats or promises of any kind.

This agreement expires on: _____
(Date)

I HEREBY WARRANT that I have authority to make this access agreement.

_____ _____
Date Signature

_____
Print name

CONSENT FOR ACCESS TO PROPERTY FOR SAMPLING AND TO TAKE RESPONSE ACTION

Name: _____ Daytime Phone Number: _____

Address(es) of Property(ies): _____

_____

I consent to officers, employees, and authorized representatives of the United States Environmental Protection Agency (EPA) entering and having access to my property for the purpose of sampling and taking a response action including: (1) preparing for and excavation of soil from my property; (2) backfilling the excavated area(s) with clean soil and/or backfill; and (3) restoring any grass or other vegetation or structures to their pre-excavation state. These activities are necessary to implement the cleanup of lead contamination in the soil.

This written permission is given by me voluntarily with knowledge of my right to refuse and without threats or promises of any kind. I understand that EPA or authorized representatives of EPA will contact me approximately two weeks in advance before the removal of soil begins, to discuss the steps involved in the excavation and removal program and all measures EPA will take to restore my yard. I also understand that if there is any damage to structures such as sidewalks that is caused by the work conducted by EPA or authorized representatives of EPA, then EPA or authorized representatives of EPA shall repair such damage.

_____
Date

☐ I grant
access to my property

☐ I do not grant
access to my property

_____
Signature

_____
Signature

XXXX TRIBE OF OKLAHOMA

PROPERTY ACCESS CONSENT AGREEMENT
FOR SAMPLING AND TO TAKE RESPONSE ACTION

The Property which is the subject of this agreement is described as follows:

NE 1/4 SE 1/4, Section 6, Township 28 North, Range 24 East, Xxxx County, Oklahoma otherwise described as Beaver Springs Park and Tribal Office which includes the Pow Wow grounds (hereinafter the Property).

THIS ____ DAY OF _____, 1999, by authority of the Xxxx Tribal Business Committee, permission is hereby granted to officers, employees and parties authorized by the United States Environmental Protection Agency (EPA) entering and having continued access to the Property until 4:30 pm (CST) on _____, to conduct the following work (hereinafter the work):

(1) To perform necessary response actions (e.g., excavation of contaminated soil, backfilling with clean soil or gravel, and sodding or seeding) to address lead and other metals from mining waste contamination on the above-described lands in accordance with the EPA Record of Decision issued August 27, 1997;

(2) To take necessary samples of environmental media to identify lead and other metals that may be a threat to public health or welfare or the environment.

Nothing contained in this permit shall operate to delay or prevent a termination of Federal trust responsibilities with respect to the Property by the issuance of a fee patent or otherwise during the term of the work; however, such termination shall not serve to terminate the work. The Xxxx Tribal Business Committee shall notify EPA of any change in status or ownership of the Property.

The Xxxx Tribal Business Committee realizes that the work will be undertaken pursuant to EPA's Superfund authority under the Comprehensive Environmental Response, Compensation, and Liability Act (CERCLA), 42 U.S.C. Sections 9601-9675.

This written permission is given by the Xxxx Tribal Business Committee voluntarily with the knowledge of its right to refuse and without threats or promises of any kind.

The Xxxx Tribal Business Committee is the property owner or a responsible representative of the property owner and I, Xx Xxxx, as Chairman of that Committee, warrant that I have authority to make this access agreement.

_____ _____

Xx Xxxx
Xxxx Tribal Chairman
Xxxx Tribe of Oklahoma

Date

_____ _____

U.S. Environmental Protection Agency

Date

CONSENT FOR ACCESS TO PROPERTY TO TAKE RESPONSE ACTION

Name: _____ Daytime Phone Number: _____

Address(es) of Property(ies): _____

_____

I consent to officers, employees, and authorized representatives of the United States Environmental Protection Agency (EPA) entering and having access to my property for the purpose of taking a response action including: (1) preparing for and excavation of soil from my property; (2) backfilling the excavated area(s) with clean soil and/or backfill; and (3) restoring any grass or other vegetation or structures to their pre-excavation state. These activities are necessary to implement the cleanup of lead contamination in the soil.

This written permission is given by me voluntarily with knowledge of my right to refuse and without threats or promises of any kind. I understand that EPA or authorized representatives of EPA will contact me approximately two weeks in advance before the removal of soil begins, to discuss the steps involved in the excavation and removal program and all measures EPA will take to restore my yard. I also understand that if there is any damage to structures such as sidewalks that is caused by the work conducted by EPA or authorized representatives of EPA, then EPA or authorized representatives of EPA shall repair such damage.

_____
Date

☐ I grant
access to my property

☐ I do not grant
access to my property

_____
Signature

_____
Signature

APPENDIX E

Example of Dust Abatement Access Form

CONSENT FOR ACCESS TO PROPERTY

Name: _____ Daytime Phone Number: _____

Address(es) of Property(ies): _____

_____

I hereby consent to grant officers, employees, contractors, sub-contractors and authorized representatives of the United States Environmental Protection Agency (EPA) access to the interior of my home and/or property for the purpose of interior dust abatement. The home dust abatement program being offered at this time consists of vacuuming floors and walls with a special vacuuming system. This system is portable and compact and easy to use. A team of bonded representatives will be providing the service at no charge to the homeowner.

Videotaping of the interior of the residence will be necessary to provide backup documentation in the event of any claims. It will be necessary that someone remain at the residence for one or two days while it is being vacuumed. This lead abatement program is offered only to homeowners who have or will grant access to their property for the remediation of in their yards. These activities are necessary to interrupt the movement of lead through soil dust, house dust, and paint dust.

If you want the process completed in your home and prefer to do it yourself, please note in the appropriate space and arrangements will be made to schedule the loan of a HEPA-VAC unit to you.

This written permission is given voluntarily with the knowledge of its right to refuse and without threats or promises of any kind. I understand that , if any damage to my property results from these activities or any work conducted by the USEPA or its authorized representatives, then the USEPA or its authorized representatives shall repair or replace such damage.

_____

Date

‘ I grant access to my property for Representatives of the EPA to video and vacuum.
‘ I wish to make arrangements to vacuum myself.
‘ I do not grant access to my property.

_____

Signature

Please return as soon as possible for scheduling of work. If you should have any questions please contact [LOCAL CONTACT NAME] at [PHONE NUMBER].

APPENDIX F

Example of Property Inspection Checklist

TAR CREEK PROJECT
PROPERTY HOME INSPECTION CHECKLIST

Address _____ Date _____

Property Group Number _____

Home Interior Access (check one, see comments):
☐ Approved by Property Owner ☐ Denied by Property Owner

Property (Yard) Access (check one, see comments):
☐ Approved by Property Owner ☐ Denied by Property Owner

	OK	NA	PROBLEM/CONDITION
YARD AREA			
1. Lawn Area			
A. Location of Flower/Plant Boxes			
B. Soil (grade) next to house			
C. Shrubbery			
D. Trees			
E. Low areas near house (that could cause ponding of water)			
F. Other: _____			
2. Utility			
A. Water Meter			
B. Gas Meter			
C. Sewer Lines			
D. Other: _____			
3. Driveway			
A. Concrete cracked, damaged			
B. Blacktop cracked, damaged			
C. Uneven Settling			
D. Other: _____			

	OK	NA	PROBLEM/CONDITION
YARD AREA (cont.)			
4. Streetwalk & Walkways			
A. Concrete cracked, eroded			
B. Tripping hazards			
C. Tree roots cracking, lifting slab			
D. Sections missing			
E. Other _____			
5. Garage			
A. Settlement cracks in walls			
B. Concrete floor slab cracked, damaged			
C. Door jambs damaged, rotted			
D. Door hard to open, close			
E. Other: _____			
6. Swimming Pool (Above Ground)			
A. Leakage			
B. Visible damage			
C. Other: _____			
7. Swimming Pool (Below Ground)			
A. Leakage			
B. Visible damage			
C. Other _____			
8. Storm Cellar			
A. Damaged			
B. Indication of Flooding			
C. Other: _____			

	OK	NA	PROBLEM/CONDITION
YARD AREA (cont.)			
9. Electrical Service			
A. Damaged circuit breaker panel box			
B. Wiring hanging outside			
C. Damaged electric meter			
D. Other:_____			
EXTERIOR AREA			
10. 9 Brick 9 Siding			
A. Brick bulging, spalling, cracking			
B. Mortar loose, needs repointing			
C. Lintel needs repair			
D. Stucco bulging, cracking			
E. Siding dented, damaged			
F. Finish wearing off siding			
G. Siding loose, not level, missing			
H. Siding rotted, termites			
I. Composite shingles worn, broken, missing			
J. Windows damaged			
K. Other:_____			
11. Roofing			
A. Age of covering			
B. Shingles worn, damaged, patched			
C. Brick chimney broken, leaning			
D. Joint open between chimney & exterior wall			
E. Need flashing at chimney, vents, walls			

	OK	NA	PROBLEM/CONDITION
EXTERIOR AREA (cont.)			
F. Parapet wall leaning			
G. Roof sagging			
H. Metal flashing damaged, missing			
I. Other: _____			
12. Gutters & Leaders **9 Yes 9 No**			
A. Copper discolored, greenish, damaged			
B. Galvanized rusted, patched			
C. Fascia board rotted, damaged, patched			
D. Drain onto foundation wall			
E. Need to divert water from wall			
F. Soffit venting 9 Yes 9 No			
G. Concrete slab cracked, deteriorated			
H. Concrete slab/splash block need			
I. Other: _____			
13. Entrance Steps			
A. Concrete cracked			
B. Brick cracked, mortar loose			
C. Structurally sound			
D. Handrail			
E. Other: _____			
14. Exterior Doors			
A. Damaged			
B. Opens/closes freely			
C. Weatherstripping			
D. Trim rotted, missing			

	OK	NA	PROBLEM/CONDITION
EXTERIOR AREA (cont.)			
E. Jambs rotted, damaged			
F. Frame separation from walls			
G. Other:_____			
INTERIOR AREA			
15. Windows			
A. Trim/sills rotted			
B. Broken glass			
C. Open freely			
E. Frame separation from walls			
F. Other: _____			
16. Kitchen			
A. Cracked walls, ceiling			
B. Loose nails, tape on drywall			
C. Soft, springy floors			
D. Wood, tiles on floor damaged			
E. Faucet leaks			
F. Doors don't close			
G. Cabinets don't close			
H. Moisture in cabinets			
I. Walls have moisture damage			
J. Other: _____			
17. Interior Rooms			
A. Cracked walls, ceiling			
B. Loose nails, tape on drywall			
C. Soft, springy floor			
D. Carpeting water damaged			
E. Water stains near windows			

	OK	NA	PROBLEM/CONDITION
INTERIOR AREA (cont.)			
F. Mold/mildew on walls			
G. Other:_____			
18. Toilet Facility			
A. Cracked tile, plaster on walls			
B. Cracked plaster on ceilings			
C. Loose tiles on walls, floors			
D. Loose nails, tape on drywall			
E. Toilet cracked			
F. Water leaks at closet flange			
G. Grout missing around tub			
H. Shower pan damaged, missing			
I. Shower door damaged, missing			
J. Need new shower door			
K. Water stains on ceiling below bathroom			
L. Hot water heater tank corroded			
M. Water stains on floor around hot water heater			
N. Moisture present around hot water heater			
O. Other:_____			
19. Interior Doors			
A. Open freely			
B. Frame separation from walls			
C. Other:_____			
20. Attic			
A. Only if visual indicator			
B. Other:_____			

	OK	NA	PROBLEM/CONDITION
INTERIOR AREA (cont.)			
21. Foundation			
A. Minor cracks			
B. Settlement cracks at corners, walls			
C. Wall bulging inward			
D. Seepage into basement/cellar			
E. Mortar deteriorating			
F. Other: _____			
22. Basement or Cellar			
A. Seepage, water stains on floor/wall			
B. Sump pump installed			
C. Water pipe leaks			
D. Sewer pipe leaks			
E. Other: _____			
FOUNDATION AREA			
23. Foundation (Slab on Grade)			
A. Settlement cracks			
B. Joint separation			
C. Spalding			
D. Other: _____			
24. Foundation (Elevated Slab w/Crawl Space)			
A. Concrete support integrity			
B. Evidence of moisture or visible moisture in crawl space			
C. Evidence of water accumulation (e.g., water stains)			

	OK	NA	PROBLEM/CONDITION
FOUNDATION AREA (cont.)			
D. Sagging joist/support girders			
E. Fungus growth evident			
F. Sump pump evident			
G. Vents present			
H. Vapor barriers			
I. Pier settlement			
J. Uneven subgrade			
K. Insect damage			
L. Sill plate damaged			
M. Subfloor damaged, loose			
N. Need subfloor			
O. Other:_____			
25. Plumbing (Raised Floors Only)			
A. Pipe insulation crumbling, missing			
B. Need to insulate pipes			
C. Water pipes leaking			
D. Sewer pipes leaking			
E. Water pipe condition			
F. Other: _____			
26. Plumbing			
A. Water pipe conditions			
B. Sewage pipe conditions			
C. Pipes leaking			
D. Pipe insulation			
E. Corrosion on drain lines			
F. Other: _____			

	OK	NA	PROBLEM/CONDITION
FOUNDATION AREA (cont.)			
27. Other Area			
A. _____			
B. _____			
C. _____			
D. _____			

COMMENTS: _____

_____

_____

Topo Survey Requested 9 Yes 9 No

_____ _____

Inspector Signature Date

APPENDIX G

Examples of Property Closeout Forms

USEPA REMEDIATION AGREEMENT FORM

Name: Sam's Restaurant

Address: 5000 Main St.

Madison, IL 62060

Phone: 000-123-4567

This form documents the completion of remedial activity performed on my property. My signature will designate that I am satisfied with the restoration of my property, and that no items are in question, now, or at any time in the future, except those items listed below, if any.

Comments: 100% satisfied.

Restoration items in question:

1. NONE
2.
3.
4.
5.
6.
7.

Chloe Irish	Chloe Irish	01/24/98
Resident Signature	Printed Name	Date
Brad W. Bradley	Brad W. Bradley	04/13/98
USEPA Representative Signature	Printed Name	Date

RESIDENTIAL REMEDIATION INSPECTION/AGREEMENT FORM

Name Sara O'Mara

Address 777 East Ave, Whoville, IN 45123

Phone 000-987-6543

This form documents the completion of remedial activities performed on my property. My signature will designate that I am satisfied with the restoration of my property, and that no items are in question, now, or at any time in the future, except those items listed below, if any.

Comments

Restoration Items in Question:

1. Roll netting on sod to be trimmed off.
2. Stone left side, more stone to be added, taper from building.
3. At double doors back left corner, add rock up to lip to allow vehicle to get in.
4. Also add rock at back of building in middle in front of concrete ledge.
5. Also add rock in open parking area & grade the tops off of the high spots.
6. Check outside of fence on 7th Street, clean up dirt clods rolling under sod & fence.
7.

Property Inspection Date 12/04/98

Lawn Care Instructions Reviewed/Delivered 12/04/98

Sara O'Mara	Sara O'Mara	12/09/98
Resident Signature	Printed Name	Date
Brad W. Bradley	Brad W. Bradley	02/12/99
USEPA Representative Signature	Printed Name	Date

APPENDIX H

Examples of Clean Letters

EPA LOGO AND ADDRESS

Date

Name
Address
City, State Zip

Dear :

The U.S. Environmental Protection Agency (EPA) has completed the cleanup of the lead contamination in your yard located at [ADDRESS, CITY, STATE], in connection with the [SITE NAME] site in [CITY, STATE] (the Site). By way of this letter, U.S. EPA is certifying that your yard has been cleaned up to less than [CLEAN-UP LEVEL] parts per million lead, the level which U.S. EPA considers protective of children's health at the Site.

Thank you for your cooperation in this clean-up effort. It has been our pleasure to work with you. If you have any questions concerning this letter or need further information, please contact me at [PROJECT MANAGER'S PHONE NUMBER].

Sincerely,

[PROJECT MANAGER NAME]
Remedial Project Manager

EPA LOGO AND ADDRESS

Date

Name
Address
City, State Zip

Dear :

The United States Environmental Protection Agency (U.S. EPA) has sampled your yard located at [ADDRESS, CITY, STATE] for lead. The results of this sampling, which are enclosed with this letter, indicate that your yard contains less than [CLEAN-UP LEVEL] per million lead, the level which U.S. EPA considers protective of children's health at the [SITE NAME, CITY, STATE]. Thus, U.S. EPA will not need to perform soil clean-up activities in your yard.

If you have any questions concerning this letter or the enclosure, please contact me at [PROJECT MANAGER'S PHONE NUMBER].

Sincerely,

PROJECT MANAGER NAME
Remedial Project Manager

Enclosure

ENCLOSURE

Analytical results for [ADDRESS]
in parts per million (ppm) of lead:

Depth Zone (inches)	Yards		OR Quadrant			
	Front	Back	1	2	3	4
0 to 1	ppm	ppm	ppm	ppm	ppm	ppm
1 to 6	ppm	ppm	ppm	ppm	ppm	ppm
6 to 12	ppm	ppm	ppm	ppm	ppm	ppm
18 to 24	ppm	ppm	ppm	ppm	ppm	ppm
Deeper Zones (if applicable)	ppm	ppm	ppm	ppm	ppm	ppm
Drip Zone Composite	ppm	ppm	ppm	ppm	ppm	ppm

Mr. John Smith
123 N. Main
Joplin, Missouri 64108

Dear Mr. Smith,

This letter serves as written notification that a lead-contaminated soil clean-up action was performed under authority of the Comprehensive Environmental Response, Compensation, and Liability Act of 1980 as amended by the Superfund Amendments and reauthorization Act of 1986 on property you have an interest in at the Jasper County, National Priorities Listed Superfund site. Our records show that your property located at **123 N. Main** was included in this action. The clean-up action conducted by the U.S. Environmental Protection Agency (EPA) and the U.S. Army Corps of Engineers (COE) addressed residences with soil lead levels over 800 ppm, day care facilities, and residences with children under six years of age with blood lead levels over 15 g/dL.

Briefly, the primary objective of the clean-up action on your property was to remove highly lead-contaminated near-surface yard soils that were located at your residence. In some cases trees, shrubs, flowers, and other vegetation were left in place. As a result a small amount of lead-contaminated soils may be left near the surface on your property. This small amount of contamination should not cause a health threat under normal circumstances. In the future if additional landscaping, or planting requiring excavation below six inches are done, care should be exercised to minimize recontamination.

The excavation criteria for the project was as follows:

A) From the surface to 12 inches, excavation continued until 500 ppm or less lead levels concentrations were achieved;

B) If the residual lead concentrations at a depth of one foot exceeded 1,500 ppm a “marker barrier” was placed at that depth. The marker barrier used was the temporary orange plastic construction-type fence. This material is permeable, and will allow water and plant roots to pass through it. Only a small number of properties required the installation of the barrier. The primary purpose of this marker barrier is to inhibit and alert individuals excavating in these areas in future years.

In general, all areas of the yard that exceeded 500 mg/kg lead at the surface were removed. Soil brought in to backfill the excavation contained less than 240 mg/kg lead.

IF YOU HAVE PLANS TO DO ANY EXCAVATION WORK AT YOUR PROPERTY AND YOU ENCOUNTER THE ORANGE BARRIER PLEASE CONTACT YOUR LOCAL HEALTH DEPARTMENT, THE MISSOURI DEPARTMENT OF NATURAL RESOURCES, OR THE EPA FOR GUIDANCE.

Please save this document for your permanent records. In the event you sell or transfer the property to someone you can show the next owner that a lead cleanup was performed. If you require more specific information concerning the excavation on your property, please feel free to contact me at (xxx) xxx-xxxx.

Sincerely,

(Project Manager)

OSWER Directive 9360.4-10
EPA 540/R-95/141
PB96-963207
December 1995

SUPERFUND PROGRAM

REPRESENTATIVE SAMPLING GUIDANCE

VOLUME 1: SOIL

Interim Final

Environmental Response Team

Office of Emergency and Remedial Response
Office of Solid Waste and Emergency Response
U.S. Environmental Protection Agency
Washington, DC 20460

Notice

This document has been reviewed in accordance with U.S. Environmental Protection Agency policy and approved for publication. Mention of trade names or commercial products does not constitute endorsement or recommendation for use.

The policies and procedures established in this document are intended solely for the guidance of government personnel, for use in the Superfund Program. They are not intended, and cannot be relied upon, to create any rights, substantive or procedural, enforceable by any party in litigation with the United States. The Agency reserves the right to act at variance with these policies and procedures and to change them at any time without public notice.

For more information on Soil Sampling and Surface Geophysics procedures, refer to the *Compendium of ERT Soil Sampling and Surface Geophysics Procedures*, OSWER directive 9360.4-02, EPA/540/P-91/006. Topics covered in this compendium include Sampling Equipment Decontamination, Soil Sampling, Soil Gas Sampling, and General Surface Geophysics. The compendium describes procedures for collecting representative soil samples and provides a quick means of waste site evaluation. It also addresses the general procedures used to acquire surface geophysical data.

Questions, comments, and recommendations are welcomed regarding the *Superfund Program Representative Sampling Guidance, Volume 1 -- Soil*. Send remarks to:

Mr. William A. Coakley
Chair, Representative Sampling Committee
U.S. EPA - ERT
Raritan Depot - Building 18, MS-101
2890 Woodbridge Avenue
Edison, NJ 08837-3679

Acknowledgments

This document was prepared by the U.S. EPA Committee on Representative Sampling for the Removal Program, under the direction of Mr. William A. Coakley, the Removal Program QA Coordinator of the Environmental Response Team, Emergency Response Division. Additional support was provided by the following EPA Workgroup and under U.S. EPA contract # 68-WO-0036 and U.S. EPA contract # 68-03-3482.

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1.0 INTRODUCTION

1.1 OBJECTIVE AND SCOPE

This is the first volume in a series of guidance documents that assist Superfund Program Site Managers, On-Scene Coordinators (OSCs), Remedial Project Managers (RPMs), and other field staff in obtaining representative samples at Superfund sites. The objective of representative sampling is to ensure that a sample or a group of samples accurately characterizes site conditions. This document specifically addresses representative sampling for soil. The information presented here is valid throughout the Superfund program, but focuses on the objectives of early action activities and emergency responses. Topics covered in the document include: assessing available information; selecting an appropriate sampling approach; selecting and utilizing geophysical, analytical screening, and sampling equipment; utilizing proper sample preparation techniques; incorporating suitable types and numbers of Quality Assurance/Quality Control (QA/QC) samples; and interpreting and presenting the analytical and geophysical data.

In the Superfund program, representative sample data collected during emergency responses or early actions may form the basis of remedial response. Longer, more complex responses require a variety of sampling objectives, including identifying threat, delineating sources and extent of contamination, and confirming the achievement of clean-up standards. Many important and potentially costly decisions are based on the sampling data, making it very important that OSCs and field personnel understand how accurately the sampling data characterize the actual site conditions. In keeping with this strategy, this document emphasizes analytical screening and geophysical techniques as cost effective approaches to characterize the site and to select sampling locations.

1.2 Conceptual Site Model

A conceptual site model is a useful tool for selecting sampling locations. It helps ensure that sources, pathways, and receptors throughout the site have been considered before sampling locations are chosen. The conceptual model assists the Site Manager in evaluating the interaction of different site features. Risk assessors use conceptual models to help plan for risk assessment activities. Frequently, a conceptual model is created as a site map (see Figure 1) or it may be developed as a flow diagram which describes potential migration of contaminants to site receptors (see Appendix A).

A conceptual model follows contaminants from their sources, to pathways (e.g., air, surface water), and eventually to the assessment endpoints. Consider the following when creating a conceptual model:

- The state(s) of each contaminant and its potential mobility
- Site topographical features
- Meteorological conditions (e.g., wind direction/speed, average precipitation, temperature, humidity)
- Human/wildlife activities on or near the site

The conceptual site model on the next page is an example created for this document. The model assists in identifying the following site characteristics:

Potential Sources:

Site (waste pile); drum dump; agricultural activities

Potential Exposure Pathway (Soil):

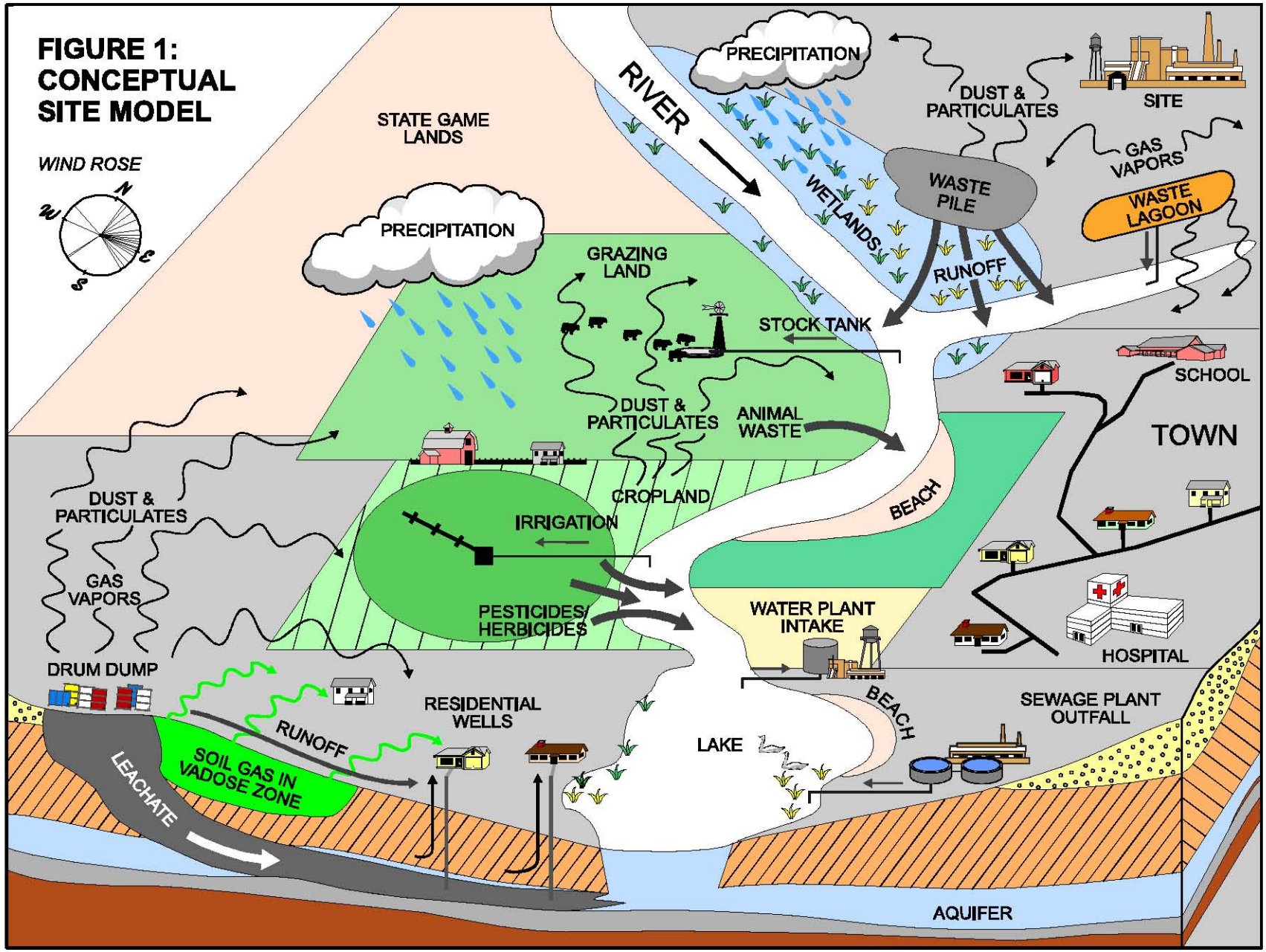
Leachate from the waste pile or drum dump; contaminated soil from direct contact with the waste pile or drum dump; agricultural activities such as pesticide application onto cropland

NOTE: Soil is described as an *exposure* pathway rather than a *migration* pathway because, unlike other media (e.g., air), contact between contaminated soil and a receptor is initiated by the receptor.

Potential Exposure Routes:

Ingestion -- Soil particles from the waste pile, drum dump or area of agricultural activity

Absorption/direct contact -- Soil near the waste pile, drum dump or area of agricultural activity



Potential Receptors of Concern (and associated potential exposure routes):

Human Population

Residents/Trespassers:

Leachate into soil from the drum dump; direct contact with soil contaminated by pesticides or other agricultural activities in the cropland

Workers/Trespassers:

Leachate into soil from the waste pile; contaminated soil associated with the waste pile or agricultural activities in the cropland

Biota

Endangered/threatened species or human food chain organisms, if suspected to be in contact with an area of potentially contaminated soil

Preliminary site information may provide the identification of the contaminant(s) of concern and the level(s) of the contamination. A sampling plan should be developed based upon the selected receptors of concern and the suspected sources and pathways. The model may assist in the selection of on-site and off-site sampling locations.

1.3 REPRESENTATIVE SAMPLING OBJECTIVES

Representative sampling applies to all phases of a Superfund response action. Representative sampling objectives for soil include:

1. Establishing threat to public health or welfare or to the environment;
2. Locating and identifying potential sources of contamination;
3. Defining the extent of contamination;
4. Determining treatment and disposal options; and
5. Documenting the attainment of clean-up goals.

These objectives are discussed in detail in Section 2.5.

1.4 REPRESENTATIVE SAMPLING

Representative soil sampling ensures that a sample or group of samples accurately reflects the concentration of the contaminant(s) of concern at a given time and location. Analytical results from representative samples reflect the variation in pollutant presence and concentration throughout a site.

This document concentrates on the variables that are introduced in the field -- namely, those that relate to the site-specific conditions, the sampling design approach, and the techniques for collection and preparation of samples. The following variables affect the representativeness of samples and subsequent measurements:

- Geological variability -- Regional and local variability in the mineralogy of rocks and soils, the buffering capacity of soils, lithologic permeability, and in the sorptive capacity of the vadose zone.
- Contaminant concentration variability -- Variations in the contaminant concentrations throughout the site.
- Collection and preparation variability -- Deviations in analytical results attributable to bias introduced during sample collection, preparation, and transportation (for analysis).
- Analytical variability -- Deviations in analytical results attributable to the manner in which the sample was stored, prepared, and analyzed by the on-site or off-site laboratory. Although analytical variability cannot be corrected through representative sampling, it can falsely lead to the conclusion that error is due to sample collection and handling procedures.

1.5 EXAMPLE SITE

An example site, presented at the end of each chapter, illustrates the development of a representative soil sampling plan that meets Superfund Program objectives for early actions or emergency responses.

2.0 SAMPLING DESIGN

2.1 INTRODUCTION

The following procedures are recommended for developing a sound sampling design. Many steps can be performed simultaneously, and the sequence is not rigid.

- Review existing historical site information;
- Perform a site reconnaissance;
- Evaluate potential migration pathways and receptors;
- Determine the sampling objectives;
- Establish the data quality objectives;
- Utilize screening techniques;
- Select parameters for which to be analyzed;
- Select an appropriate sampling approach; and
- Determine the locations to be sampled.

Real-time analytical screening techniques can be used throughout the removal action. The results can be used to modify the site sampling plan as the extent of contamination becomes known.

2.2 HISTORICAL DATA REVIEW

Unless the site is considered a classic emergency, every effort should be made to first thoroughly review relevant site information. An historical data review examines past and present site operations and disposal practices, providing an overview of known and potential site contamination and other site hazards. Sources of information include federal, state and local officials and files (e.g., site inspection reports and legal actions), deed or title records, current and former facility employees, potentially responsible parties, local residents, and facility records or files. For any previous sampling efforts, obtain information regarding sample locations (on maps, if possible), matrices, methods of collection and analysis, and relevant contaminant concentrations. Assess the reliability and usefulness of existing analytical data. Even data which are not substantiated by documentation or QA/QC controls may still be useful.

Collect information that describes any specific chemical processes used on site, as well as descriptions of raw materials used, products and wastes, and waste storage and disposal practices. Whenever possible, obtain site maps, facility blueprints, and historical aerial photographs, detailing past and present storage, process, and waste disposal locations. The local Agricultural Extension Agent, a Soil Conservation Service (SCS) representative, has information on soil types and drainage patterns. County property and tax records, and United States Geological Survey (USGS) topographic maps are also useful sources of site and regional information.

2.3 SITE RECONNAISSANCE

A site reconnaissance, conducted either prior to or in conjunction with sampling, is invaluable to assess site conditions, to evaluate areas of potential contamination, to evaluate potential hazards associated with sampling, and to develop a sampling plan. During the reconnaissance, fill data gaps left from the historical review by:

- Interviewing local residents, and present or past employees about site-related activities;
- Researching facility files or records (where records are made accessible by owner/operator);
- Performing a site entry, utilizing appropriate personal protective equipment and instrumentation. Observe and photo-document the site; note site access routes; map process and waste disposal areas such as landfills, lagoons, and effluent pipes; inventory site wastes; and map potential transport routes such as ponds, streams, and irrigation ditches. Note topographic and structural features, dead animals and dead or stressed vegetation, potential safety hazards, and visible label information from drums, tanks, or other containers found on the site.

2.4 MIGRATION PATHWAYS AND RECEPTORS

The historical review and site visit are the initial steps in defining the source areas of contamination which could pose a threat to human health and the environment. This section addresses how to delineate the spread of contamination away from the source areas. Included are pollutant migration pathways and

the routes by which persons or the environment may be exposed to the on-site chemical wastes.

2.4.1 Migration Pathways and Transport Mechanisms

Migration pathways are routes by which contaminants have moved or may be moved away from a contamination source. Pollutant migration pathways may include man-made pathways, surface drainage, vadose zone transport, and wind dispersion. Human activity (such as foot or vehicular traffic) also transports contaminants away from a source area. These five transport mechanisms are described below.

- Man-made pathways -- A site located in an urban setting has the following man-made pathways which can aid contaminant migration: storm and sanitary sewers, drainage culverts, sumps and sedimentation basins, French drain systems, and underground utility lines.

- Surface drainage -- Contaminants can be adsorbed onto sediments, suspended independently in the water column, or dissolved in surface water runoff and be rapidly carried into drainage ditches, streams, rivers, ponds, lakes, and wetlands. Consider prior surface drainage routes; historical aerial photographs can be invaluable for delineation of past surface drainage patterns. An historical aerial photograph search can be requested through the EPA Regional Remote Sensing Coordinator.

- Vadose zone transport -- Vadose zone transport is the vertical or horizontal movement of water and of soluble and insoluble contaminants within the unsaturated zone of the soil profile. Contaminants from a surface source or a leaking underground storage tank can percolate through the vadose zone and be adsorbed onto subsurface soil or reach groundwater.

- Wind dispersion -- Contaminants deposited over or adsorbed onto soil may migrate from a waste site as airborne particulates. Depending on the particle-size distribution and associated settling rates, these particulates may be deposited downwind or remain suspended, resulting in contamination of surface soils and/or exposure of nearby populations.

- Human and animal activity -- Foot and vehicular traffic of facility workers, response personnel, and trespassers can move contaminants away from a source. Animal burrowing, grazing, and migration can also contribute to contaminant migration.

2.4.2 Receptors

Once the migration pathways have been determined, identify all receptors (i.e., potentially affected human and environmental populations) along these pathways. Human receptors include on-site and nearby residents and workers. Note the attractiveness and accessibility of site wastes (including contaminated soil) to children and other nearby residents. Environmental receptors include Federal- or state-designated endangered or threatened species, habitats for these species, wetlands, and other Federal- and state-designated wilderness, critical, and natural areas.

2.5 SOIL REPRESENTATIVE SAMPLING OBJECTIVES

Collect samples if any of the following sampling objectives in the scope of the project are not fulfilled by existing data.

1. Establishing Threat to Public Health or Welfare or to the Environment -- The Comprehensive Environmental Response, Compensation and Liability Act of 1980 (CERCLA) and the National Contingency Plan (NCP) establish the funding mechanism and authority which allow the OSC to activate a Federal removal action. The OSC must establish (often with sampling) that the site poses a threat to public health or welfare or to the environment.

2. Locating and Identifying Potential Sources of Contamination -- Sample to identify the locations and sources of contamination. Use the results to formulate removal priorities, containment and clean-up strategies, and cost projections.

3. Defining the Extent of Contamination -- Where appropriate, sample to assess horizontal and vertical extent of contaminant concentrations. Use the results to determine the site boundaries (i.e., extent of contamination), define clean areas, estimate volume of contaminated soil, establish a clearly defined removal approach, and assess removal costs and timeframe.

4. Determining Treatment and Disposal Options -- Sample to characterize soil for in situ or other on-site treatment, or excavation and off-site treatment or disposal.

5. Documenting the Attainment of Clean-up Goals -- During or following a site cleanup, sample to determine whether the goals were achieved, and to delineate areas requiring further treatment or excavation when appropriate.

2.6 DATA QUALITY OBJECTIVES

Data quality objectives (DQOs) state the level of uncertainty that is acceptable from data collection activities. DQOs also define the data quality necessary to make a certain decision. Consider the following when establishing DQOs for a particular project:

- Decision(s) to be made or question(s) to be answered;
- Why environmental data are needed and how the results will be used;
- Time and resource constraints on data collection;
- Descriptions of the environmental data to be collected;
- Applicable model or data interpretation method used to arrive at a conclusion;
- Detection limits for analytes of concern; and
- Sampling and analytical error.

In addition to these considerations, the quality assurance components of precision, accuracy (bias), completeness, representativeness, and comparability should also be considered. Quality assurance components are defined as follows:

- Precision -- measurement of variability in the data collection process.
- Accuracy (bias) -- measurement of bias in the analytical process. The term "bias" throughout this document refers to the QA/QC accuracy component.
- Completeness -- percentage of sampling measurements which are judged to be valid.
- Representativeness -- degree to which sample data accurately and precisely represent the characteristics of the site contaminants and their concentrations.
- Comparability -- evaluation of the similarity of conditions (e.g., sample depth, sample homogeneity) under which separate sets of data are produced.

Quality assurance/quality control (QA/QC) objectives are discussed further in Chapter 5.

2.7 ANALYTICAL SCREENING AND GEOPHYSICAL TECHNIQUES

There are two primary types of analytical data which can be generated during sampling: laboratory analytical data and analytical screening data. Analytical screening techniques (e.g., using a photoionization detector (PID), portable X-ray fluorescence (XRF) unit, and hazard categorization kits) provide real-time or direct reading capabilities. These screening methods can narrow the possible groups or classes of chemicals for laboratory analysis and are effective and economical for gathering large amounts of site data. Once an area is identified using screening techniques, a subset of samples can be sent for laboratory analysis to substantiate the screening results. Under a limited sampling budget, analytical screening (with laboratory confirmation) will generally result in more analytical data from a site than will sampling for off-site laboratory analysis alone. To minimize the potential for false negatives (not detecting on-site contamination), use only those analytical screening methods which provide detection limits below applicable action levels. It should be noted, that some analytical screening methods which do not achieve detection limits below site action levels can still detect grossly contaminated areas, and can be useful for some sampling events.

Geophysical techniques may also be utilized during a removal action to help depict locations of any potential buried drums or tanks, buried waste, and disturbed areas. Geophysical techniques include ground penetrating radar (GPR), magnetometry, electromagnetic conductivity (EM) and resistivity surveys.

2.8 PARAMETERS FOR ANALYSIS

If the historical data review yields little information about the types of waste on site, use applicable screening methods to narrow the parameters for analysis by ruling out the presence of high concentrations of certain contaminants. If the screening results are inconclusive, send a subset of samples from the areas of concern for a full chemical

characterization by an off-site laboratory. It is advised that samples from known or suspected source areas be sent to the laboratory for a full chemical characterization so that all contaminants of concern can be identified (even at low detection levels), and future sampling and analysis can then focus on those substances.

Away from source areas, select a limited number of indicator parameters (e.g., lead, PAHs) for analysis based on the suspected contaminants of concern. This will result in significant cost savings over a full chemical characterization of each sample. Utilize EPA-approved methodologies and sample preparation, where possible, for all requested off-site laboratory analyses.

2.9 SAMPLING APPROACHES

Selecting sampling locations for screening or laboratory analysis entails choosing the most appropriate sampling approach. Representative sampling approaches include **judgmental, random, stratified random, systematic grid, systematic random, search, and transect sampling**. A representative sampling plan may combine two or more of these approaches. Each approach is defined below.

2.9.1 Judgmental Sampling

Judgmental sampling is the subjective selection of sampling locations at a site, based on historical information, visual inspection, and on best professional judgment of the sampling team. Use judgmental sampling to identify the contaminants present at areas having the highest concentrations (i.e., worst-case conditions). Judgmental sampling has no randomization associated with the sampling strategy, precluding any statistical interpretation of the sampling results.

2.9.2 Random Sampling

Random sampling is the arbitrary collection of samples within defined boundaries of the area of concern. Choose random sample locations using a random selection procedure (e.g., using a random number table). Refer to U.S. EPA, 1984a, for a random number table. The arbitrary selection of sampling points requires each sampling point to be selected independent of the location of all other points, and results in all locations within the area of concern having an equal chance of being selected. Randomization is necessary in order to make probability or confidence statements about the sampling results. The key to interpreting these probability statements is the assumption that the site is homogeneous with respect to the parameters being monitored. The higher the degree of heterogeneity, the less the random sampling approach will adequately characterize true conditions at the site. Because hazardous waste sites are very rarely homogeneous, other statistical sampling approaches (discussed below) provide ways to subdivide the site into more homogeneous areas. These sampling approaches may be more appropriate for removal activities than random sampling. Refer to U.S. EPA, February 1989, pages 5-3 to 5-5 for guidelines on selecting sample coordinates for random sampling. Figure 2 illustrates a random sampling approach.

2.9.3 Stratified Random Sampling

Stratified random sampling often relies on historical information and prior analytical results (or screening data) to divide the sampling area into smaller areas called strata. Each strata is more homogeneous than the site is as a whole. Strata can be defined based on various factors, including: sampling depth, contaminant concentration levels, and contaminant source areas. Place sample locations within each of these strata using random selection procedures. Stratified random sampling imparts some control upon the sampling scheme but still allows for random sampling within each stratum. Different sampling approaches may also be selected to address the different strata at the site. Stratified random sampling is a useful and flexible design for estimating the pollutant concentration within each depth interval or area of concern. Figure 3 illustrates a stratified random sampling approach where strata are defined based on depth. In this example, soil coring devices are used to collect samples from given depths at randomly selected locations within the strata.

Figure 2: Random Sampling

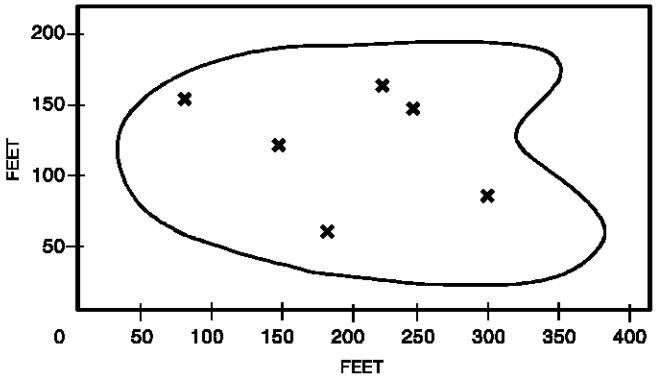


Figure 3: Stratified Random Sampling

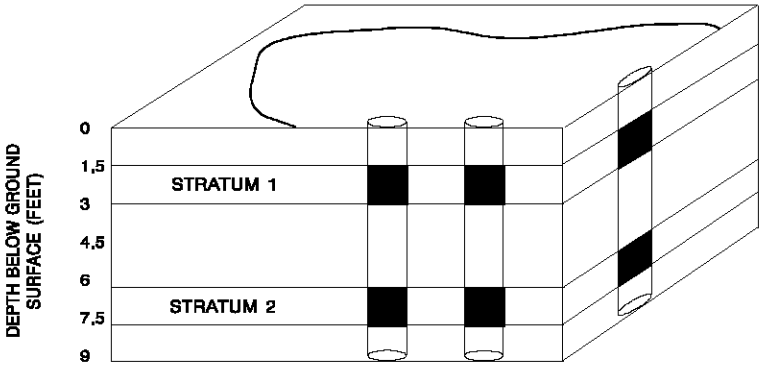
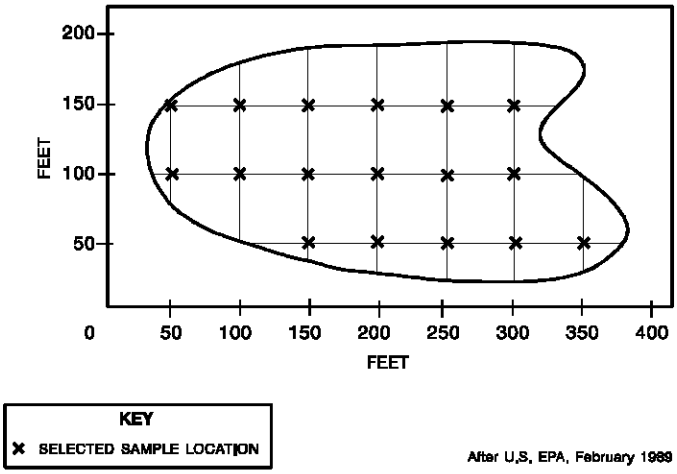


Figure 4: Systematic Grid Sampling



2.9.4 Systematic Grid Sampling

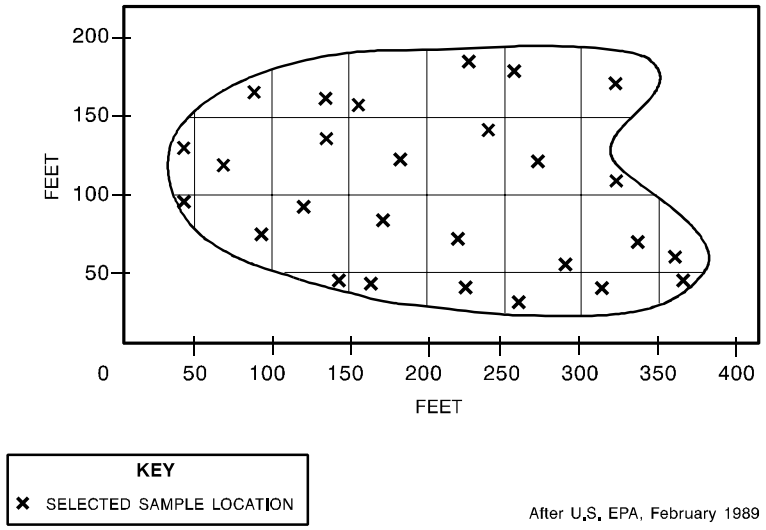
Systematic grid sampling involves subdividing the area of concern by using a square or triangular grid and collecting samples from the nodes (intersections of the grid lines). Select the origin and direction for placement of the grid using an initial random point. From that point, construct a coordinate axis and grid over the whole site. The distance between sampling locations in the systematic grid is determined by the size of the area to be sampled and the number of samples to be collected.

Systematic grid sampling is often used to delineate the extent of contamination and to define contaminant concentration gradients. Refer to U.S. EPA February 1989, pages 5-5 to 5-12, for guidelines on selection of sample coordinates for systematic grid sampling. Figure 4 illustrates a systematic grid sampling approach.

2.9.5 Systematic Random Sampling

Systematic random sampling is a useful and flexible design for estimating the average pollutant concentration within grid cells. Subdivide the area of concern using a square or triangular grid (as described in Section 2.9.4) then collect samples from within each cell using random selection procedures. Systematic random sampling allows for the isolation of cells that may require additional sampling and analysis. Figure 5 illustrates a systematic random sampling approach.

Figure 5: Systematic Random Sampling



After U.S. EPA, February 1989

2.9.6 Search Sampling

Search sampling utilizes either a systematic grid or systematic random sampling approach to search for areas where contaminants exceed applicable clean-up standards **(hot spots)**. The number of samples and the grid spacing are determined on the basis of the acceptable level of error (i.e., the chance of missing a hot spot). Search sampling requires that assumptions be made about the size, shape, and depth of the hot spots. As illustrated in Figure 6, the smaller and/or narrower the hot spots are, the smaller the grid spacing must be in order to locate them. Also, the smaller the acceptable error of missing hot spots is, the smaller the grid spacing must be. This, in effect, means collecting more samples.

Once grid spacing has been selected, the probability of locating a hot spot can be determined. Using a systematic grid approach, Table 1 lists approximate probabilities of missing an elliptical hot spot based on the grid method chosen as well as the dimensions of the hot spot. The lengths of the long and short axes (L and S) are represented as a percentage of the grid spacing chosen. The triangular grid method consistently shows lower probabilities of missing a hot spot in comparison to the block grid method. Table 1 can be used in two ways. If the acceptable probability of missing a hot spot is known, then the size of the hot spot which can be located at that probability level can be determined. Conversely, if the approximate size of the hot spot is known, the probability of locating it can be determined.

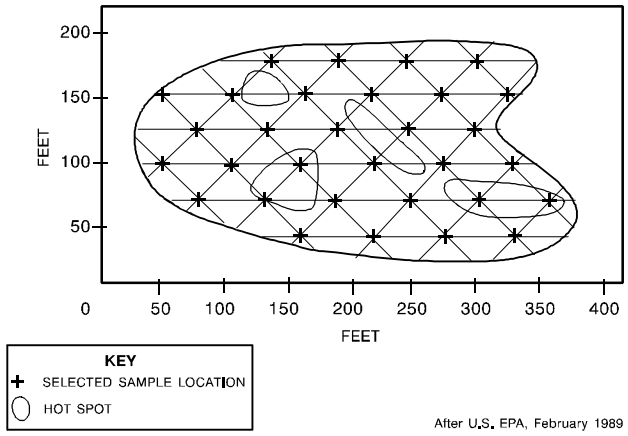
For example, suppose the block grid method is chosen with a grid spacing of 25 feet. The OSC is willing to accept a 10% chance of missing an elliptical hot spot. Using Table 1, there would be a 90% probability of locating an elliptical hot spot with L equal to 90% of the grid spacing chosen and S equal to 40% of the grid spacing chosen. Therefore the smallest elliptical hot spot which can be located would have a long axis L = 0.90 × 25ft. = 22.5 ft. and a short axis S = 0.40 × 25ft. = 10 ft.

Similarly, if the approximate size of the hot spot being searched for is known, then the probability of missing that hot spot can be determined. For example, if a triangular grid method was chosen with a 25 foot grid spacing and the approximate shape of the hot spot is known, and L is approximately 15 feet or 60% of the grid spacing, and S is approximately 10 feet or 40% of the grid spacing, then there is approximately a 15% chance of missing a hot spot of this size and shape.

2.9.7 Transect Sampling

Transect sampling involves establishing one or more transect lines across the surface of a site. Collect samples at regular intervals along the transect lines at the surface and/or at one or more given depths. The length of the transect line and the number of samples to be collected determine the spacing between sampling points along the transect. Multiple transect lines may be parallel or non-parallel to one another. If the lines are parallel, the sampling objective is sim-

Figure 6: Search Sampling



After U.S. EPA, February 1989

Table 1: Probability of Missing an Elliptical Hot Spot

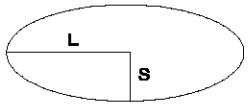
LENGTH OF SHORT AXIS AS A PERCENTAGE OF GRID SPACING

LENGTH OF LONG AXIS AS A PERCENTAGE OF GRID SPACING

	10%	20%	30%	40%	50%	60%	70%	80%	90%	100%
10%	0.97 / 0.95									
20%	0.95 / 0.92	0.88 / 0.85								
30%	0.92 / 0.87	0.83 / 0.78	0.72 / 0.66							
40%	0.88 / 0.85	0.75 / 0.71	0.65 / 0.55	0.50 / 0.41						
50%	0.85 / 0.82	0.69 / 0.63	0.54 / 0.44	0.38 / 0.27	0.21 / 0.08					
60%	0.80 / 0.80	0.62 / 0.58	0.45 / 0.35	0.27 / 0.15	0.12 / 0.03	0.06 / 0.0				
70%	0.77 / 0.77	0.56 / 0.54	0.38 / 0.29	0.18 / 0.12	0.07 / 0.01	0.03 / 0.0	0.0 / 0.0			
80%	0.75 / 0.75	0.54 / 0.50	0.32 / 0.23	0.12 / 0.08	0.05 / 0.0	0.0 / 0.0	0.0 / 0.0	0.0 / 0.0		
90%	0.72 / 0.72	0.51 / 0.45	0.30 / 0.21	0.10 / 0.06	0.03 / 0.0	0.0 / 0.0	0.0 / 0.0	0.0 / 0.0	0.0 / 0.0	
100%	0.70 / 0.66	0.45 / 0.37	0.24 / 0.18	0.08 / 0.04	0.01 / 0.0	0.0 / 0.0	0.0 / 0.0	0.0 / 0.0	0.0 / 0.0	0.0 / 0.0

From tables in Gilbert, 1987

L=length of long side
S=length of short side



BLOCK GRID 0.97 / 0.95 TRIANGULAR GRID

ilar to systematic grid sampling. A primary benefit of transect sampling over systematic grid sampling is the ease of establishing and relocating individual transect lines versus an entire grid. Transect sampling is often used to delineate the extent of contamination and to define contaminant concentration gradients. It is also used, to a lesser extent, in compositing sampling schemes. For example, a transect sampling approach might be used to characterize a linear feature such as a drainage ditch. A transect line is run down the center of the ditch, along its full length. Sample aliquots are collected at regular intervals along the transect line and are then composited. Figure 7 illustrates transect sampling.

Table 2 summarizes the various representative sampling approaches and ranks the approaches from most to least suitable, based on the sampling objective. Table 2 is intended to provide general guidelines, but it cannot cover all site-specific conditions encountered.

2.10 SAMPLING LOCATIONS

Once a sampling approach has been selected, the next step is to select sampling locations. For statistical (non-judgmental) sampling, careful placement of each sampling point is important to achieve representativeness.

Factors such as the difficulty in collecting a sample at a given point, the presence of vegetation, or discoloration of the soil could bias a statistical sampling plan.

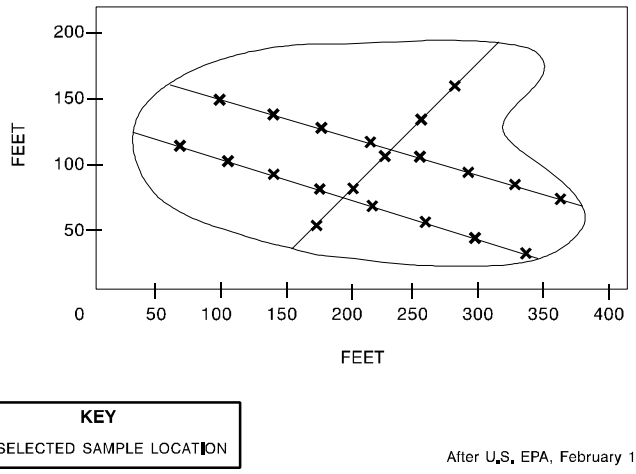
Sampling points may be located with a variety of methods. A relatively simple method for locating random points consists of using either a compass and a measuring tape, or pacing, to locate samplingpoints with respect to a permanent landmark, such as a survey marker. Then plot sampling coordinates on a map and mark the actual sampling points for future reference. Where the sampling design demands a greater degree of precision, locate each sample point by means of a survey. After sample collection, mark each sample point with a permanent stake so that the survey team can identify all the locations.

2.11 EXAMPLE SITE

2.11.1 Background Information

The ABC Plating Site is located in Carroll County, Pennsylvania, approximately 1.5 miles north of the town of Jonesville (Figure 8). The site covers approximately 4 acres, and operated as an electroplating facility from 1947 to 1982. During its years of operation, the company plated automobile and airplane parts with chromium, nickel, and copper. Cyanide solutions were used in the plating process. ABC Plating deposited electroplating wastes into two shallow surface settling lagoons in the northwest sector of the site. The county environmental health department was attempting to enforce cleanup by the site owner, when, in early 1982, a fire on site destroyed most of the process building. The owner then abandoned the facility and could not be located by enforcement and legal authorities. The county contacted EPA for an assessment of the site for a possible response.

Figure 7: Transect Sampling



After U.S. EPA, February 1989

Table 2: Representative Sampling Approach Comparison

	SAMPLING APPROACH						
SAMPLING OBJECTIVE	JUDGEMENTAL	RANDOM	STRATIFIED RANDOM	SYSTEMATIC GRID	SYSTEMATIC RANDOM	SEARCH	TRANSECT
ESTABLISH THREAT	1	4	3	2 [a]	3	3	2
IDENTIFY SOURCES	1	4	2	2 [a]	3	2	3
DELINEATE EXTENT OF CONTAMINATION	4	3	3	1 [b]	1	1	1
EVALUATE TREATMENT & DISPOSAL OPTIONS	3	3	1	2	2	4	2
CONFIRM CLEANUP	4	1 [c]	3	1 [b]	1	1	1 [d]

1 - PREFERRED APPROACH
2 - ACCEPTABLE APPROACH
3 - MODERATELY ACCEPTABLE APPROACH
4 - LEAST ACCEPTABLE APPROACH
a - SHOULD BE USED WITH FIELD ANALYTICAL SCREENING
b - PREFERRED ONLY WHERE KNOWN TRENDS ARE PRESENT
c - ALLOWS FOR STATISTICAL SUPPOFT OF CLEANUP VERIFICATION IF SAMPLING OVER ENTIRE SITE
d - MAY BE EFFECTIVE WITH COMPOSTING TECHNIQUE IF SITE IS PRESUMED TO BE CLEAN

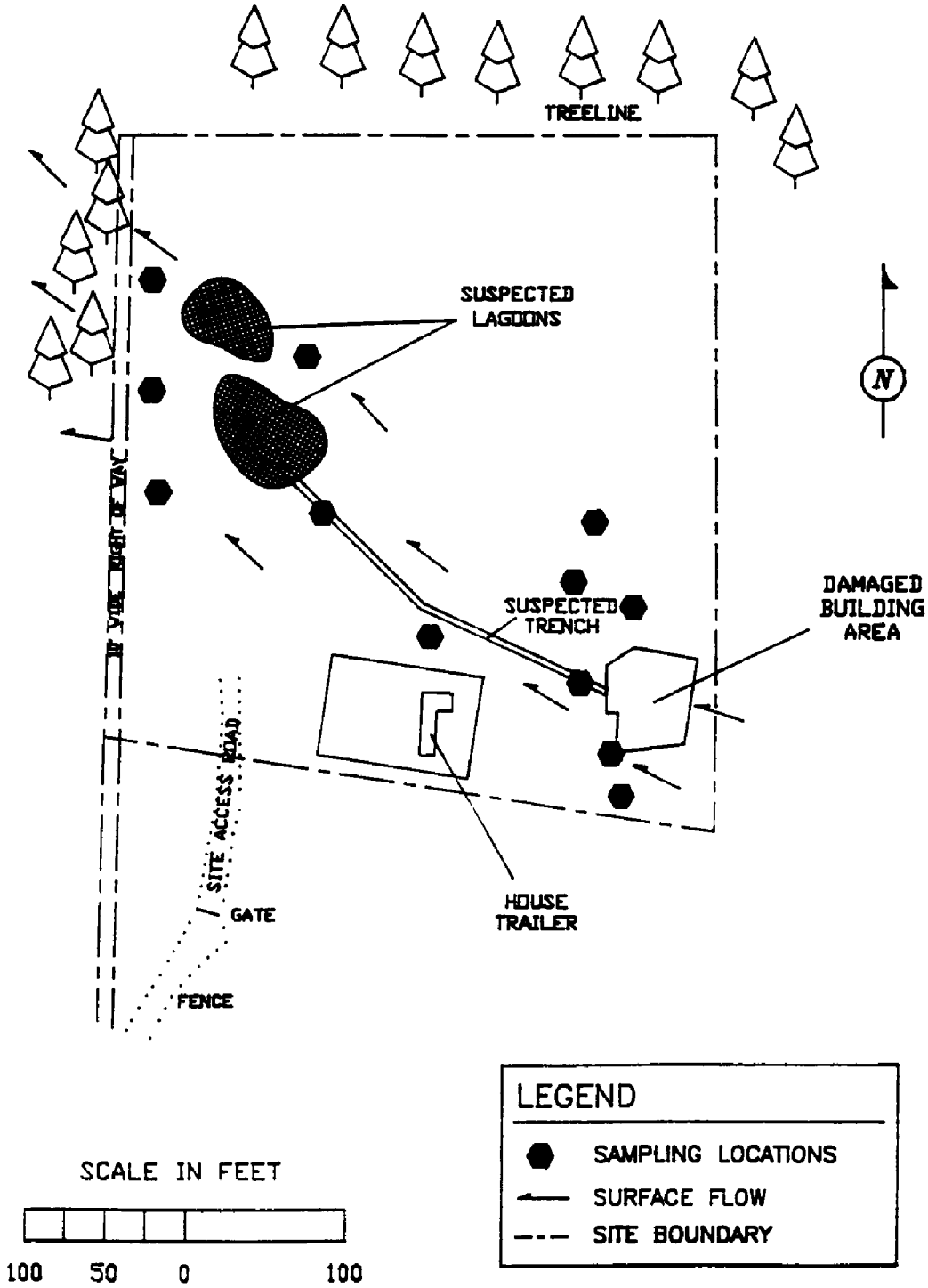
2.11.2 Historical Data Review and Site Reconnaissance

The EPA On-Scene Coordinator (OSC) reviewed the county site file, finding that in 1974, the owner was cited for violating the Clean Streams Act and for storing and treating industrial waste without a permit. The owner was ordered to file a site closure plan and to remediate the storage lagoons. The owner, however, continued operations and was then ordered to begin remediation in 90 days or be issued a cease and desist order. Soon after, a follow-up inspection revealed that the lagoons had been backfilled without removing the waste.

The OSC and response contractor arrived on site to interview local officials, fire department officers, neighboring residents (including a past facility employee), and county representatives, regarding site operating practices and other site details. A past employee sketched facility process features on a map which was obtained from the county (Figure 8). The features included two settling lagoons and a feeder trench which transported plating wastes from the process building to the lagoons. The OSC obtained copies of aerial photographs of the site area from the district office of the U.S. Soil Conservation Service. The county also provided the OSC with copies of all historical site and violation reports.

The OSC and response contractor made a site entry utilizing appropriate personal protective equipment and instrumentation. They observed 12 vats, likely containing plating solutions, on a concrete pad where the original facility building once stood.

Figure 8: Site Sketch and Phase I Soil Sampling Locations
ABC Plating Site



Measurements of pH ranged from 1 to 11. In addition, 50 drums and numerous smaller containers (some on the concrete pad, others sitting directly on the ground) were leaking and bulging, due to the fire. The response contractor noted many areas of stained soil, which indicated container leakage, poor waste handling practices, and possible illegal dumping of wastes.

2.11.3 Identification of Migration Pathways, Transport Mechanisms and Receptors

During the site entry, the OSC noted that several areas were devoid of vegetation, threatening wind erosion which could transport heavy metal- and cyanide-contaminated soil particulates off site. These particulates could be deposited on residential property downwind or be inhaled by nearby residents.

Erosion gullies located on site indicated soil erosion and fluvial transport due to storms. Surface drainage sloped towards the northwest. The response contractor observed stressed and discolored vegetation immediately off site, along the surface drainage route. Surface drainage of heavy metals and cyanide was a direct contact hazard to local residents. Further downgradient, runoff enters an intermittent tributary of Little Creek. Little Creek in turn feeds Barker Reservoir, the primary water supply for the City of Jonesville and neighboring communities, which are located 2.5 miles downgradient of the site. The site entry team observed that the site was not secure and there were signs of trespass (confirming a neighbor's claim that children play at the facility). These activities could lead to direct contact with cyanide and heavy metal contaminants, in addition to the potential for chemical burns from direct contact with strong acids and bases.

Information obtained from the historical data review and site reconnaissance was used to create a site-specific conceptual model. Sources (e.g., vats, drums), pathways (e.g., gullies) and potential receptors (e.g., local residents) were detailed on a map to assist the selection of sampling approaches, objectives, and locations.

2.11.4 Sampling Objectives

The OSC selected three specific sampling objectives, as follows:

- Phase 1 -- Determine whether a threat to public health, welfare, and the environment exists. Identify sources of contamination to support an immediate CERCLA-funded activation for containment of contaminants and security fencing.

- Phase 2 -- Define the extent of contamination at the site and adjacent residential properties. Estimate the volume of contaminated soil and the associated removal costs.

- Phase 3 -- After excavation (or treatment), document the attainment of clean-up goals. Assess that cleanup was completed to the selected level.

2.11.5 Selection of Sampling Approaches

The OSC selected a judgmental sampling approach for Phase 1. Judgmental sampling supports the Action Memorandum process by best defining on site contaminants in the worst-case scenario in order to evaluate the threat to human health, welfare, and the environment. Threat is typically established using a relatively small number of samples (less than 20) collected from source areas, or suspected contaminated areas based on the historical data review and site reconnaissance. For this site, containerized wastes were screened to categorize the contents and to establish a worst- case waste volume, while soil samples were collected to demonstrate whether a release had already occurred.

For Phase 2, a stratified systematic grid design was selected to define the extent of contamination. The grid can accommodate analytical screening and geophysical surveys and allow for contaminated soil excavation on a cell-by-cell basis. Based on search sampling conducted at similar sites, the hot spots being searched for were assumed to be elliptical in shape and 45 feet by 20 feet in size. Under these assumptions, a block grid, with a 50 foot grid spacing, was selected. This grid size ensured a no more than 10% probability of missing a hot spot (see Table 1). The grid was extended to adjacent residential properties when contaminated soil was identified at grid points near the boundary of the site.

Phase 3 utilized a systematic grid sampling approach to confirm the attainment of clean-up goals. Following cleanup, analytical screening was conducted on excavated soil areas using a

transportable X-ray fluorescence (XRF) unit mounted in a trailer (mobile laboratory instrument). Based on the results, each area was documented as clean, or was excavated to additional depth, as necessary.

2.11.6 Analytical Screening, Geophysical Techniques, and Sampling Locations

During Phase 1 operations, containerized wastes were screened using hazard categorization techniques to identify the presence of acids, bases, oxidizers, and flammable substances. Following this procedure, photoionization detector (PID) and flame ionization detector (FID) instruments, a radiation meter, and a cyanide monitor were used to detect the presence of volatile organic compounds, radioactive substances, and cyanide, respectively, in the containerized wastes. Phase 1 screening indicated the presence of strong acids and bases and the absence of volatile organic compounds. The response contractor collected a total of 12 surface soil samples (0-3 inches) during this phase and sent them to a laboratory for analysis. The soil sampling locations included stained soil areas, erosion channels and soil adjacent to leaking containers. Background samples were not collected during Phase 1 because they were unnecessary for activating funding. Phase 1 sampling locations are shown in Figure 8. Based on Phase 1 analytical results, consultation with a Regional EPA toxicologist and with the Agency for Toxic Substances and Disease Registry (ATSDR), an action level of 100 ppm for chromium was selected for cleanup.

During Phase 2 sampling activities, the OSC used a transportable XRF unit installed in an on-site trailer to screen samples for total chromium in order to limit the number of samples to be sent for off-site laboratory analysis. The transportable XRF (rather than a portable unit) was selected for analytical screening to accommodate the 100 ppm action level for chromium. Sampling was performed at all grid nodes at the surface (0-4 inches) and subsurface (36-40 inches) (Figure 9). The 36-40 inch depth was selected based on information obtained from county reports and local interviews which indicated the lagoon wastes were approximately 3 feet below ground surface. The samples were homogenized and sieved (discussed in Chapter 4), then screened for chromium using the XRF. The surface and subsurface samples from areas downgradient of the original facility (21 grid nodes) and three upgradient (background) locations were sent for off-site laboratory analysis following XRF screening. The analytical results from these samples allowed for site-specific calibration of the XRF unit. Once grid nodes with a contamination level greater than the selected action level were located, composite samples were collected from each adjoining cell. Surface aliquots were collected and then composited, sieved, thoroughly homogenized, and screened using the XRF to pinpoint contaminated cells. Additionally, four subsurface aliquots were collected at the same locations as the surface aliquots. They were also composited, sieved, thoroughly homogenized, and screened using the XRF. Figure 10 illustrates a Phase 2 sampling grid cell diagram. Based on the XRF data, each adjoining cell was either identified as clean (below action level), or designated for excavation (at or above action level).

For Phase 3 sampling, cleanup was confirmed by collecting and compositing four aliquots from the surface of each grid cell excavated during Phase 2. The surface composites were then screened (as in Phase 2), using the transportable XRF. Ten percent of the screened samples were also sent to an off-site laboratory for confirmatory sampling. Based on the Phase 3 screening and sampling results, each cell was documented as clean, or, excavated to additional depth, as necessary.

During Phase 2, the OSC conducted ground penetrating radar (GPR) and electromagnetic conductivity (EM) geophysical surveys to help delineate the buried trench and lagoon areas along with any other waste burial areas. The GPR survey was run along the north-south grid axis across the suspected locations of the trench and lagoons. Several structural discontinuities, defining possible disturbed areas, were detected. One anomaly corresponded with the suspected location and orientation of the feeder trench. Several discontinuities were identified in the suspected lagoon areas; however, the data did not conclusively pinpoint precise locations. This could be due to a disturbance of that area during the backfilling process by the PRP. The GPR survey is illustrated in Figure 11.

Figure 9: Soil Sampling and SRF Screening Locations
ABC Plating Site

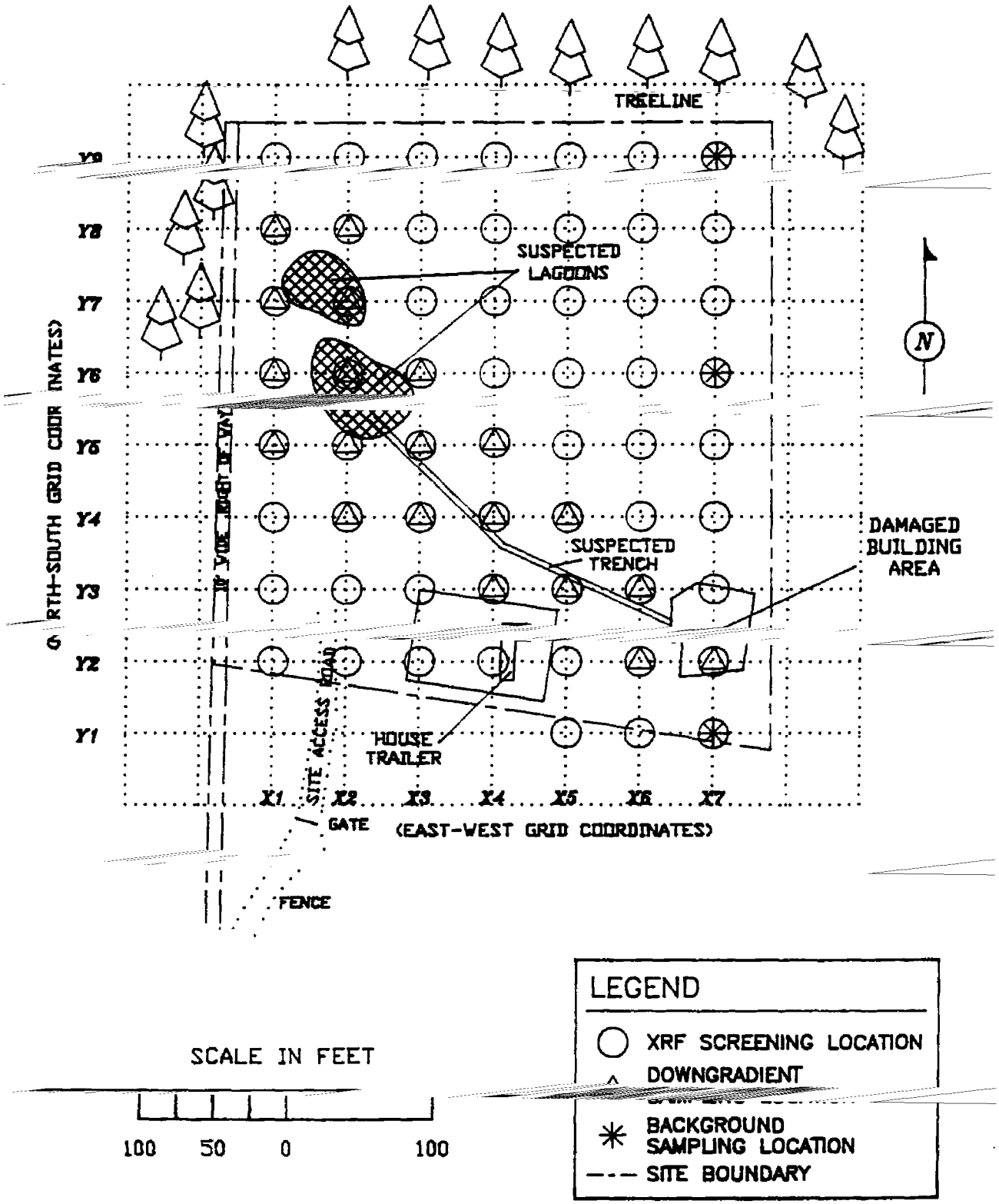
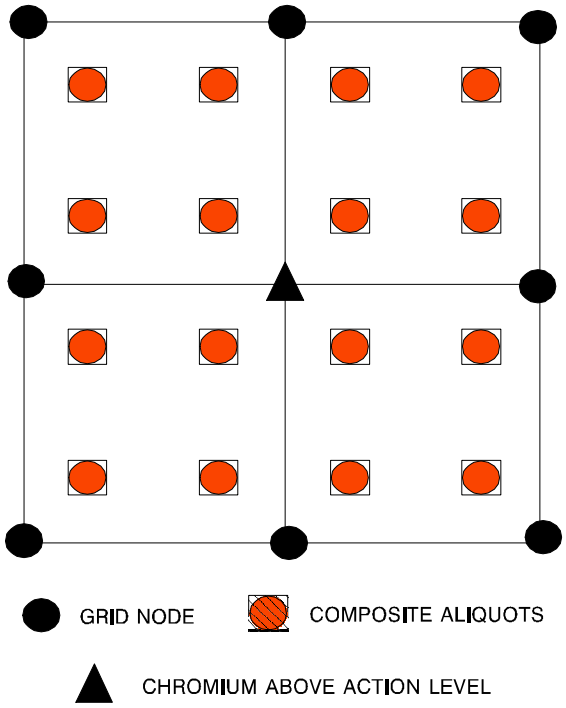


Figure 10: Phase 2 Sampling Grid Cell Diagram*



* Surface samples should be taken over a minimum area of one square foot. Sampling areas for depth sampling are limited by the diameter of the sampling equipment (e.g., auger, split spoon, or coring devices).

For the comprehensive EM survey, the original 50 foot grid spacing was decreased to 25 feet along the north-south grid axis. The EM survey was run along the north-south axes and readings were obtained at the established grid nodes. The EM survey was utilized throughout the site to detect the presence of buried metal objects (e.g., buried pipe leading to the lagoons), and potential subsurface contaminant plumes. The EM survey identified several high conductivity anomalies: the suspected feeder trench location, part of the lagoon area, and a small area west of the process building (Figure 12), which could have been an illegal waste dumping area. Several areas of interference were encountered due to the presence of large metal objects at the surface (a dumpster, surface vats and a junk car).

2.11.7 Parameters for Analysis

During Phase 1 sampling activities, full priority pollutant metals and total cyanide analyses were conducted on all samples. Since Phase 1 samples were collected from the areas of highest suspected contaminant concentration (i.e., sources and drainage pathways), Phase 2 samples were run for total chromium and cyanide, the only analytes detected during the Phase 1 analyses. During Phase 3, the samples sent to the laboratory for definitive analysis were analyzed for total chromium and cyanide. Throughout the removal, it was not possible to screen soils on site for cyanide, therefore the OSC requested laboratory cyanide analysis on the 10% confirmatory samples.

Figure 11: GPR Survey Results
ABC Plating Site

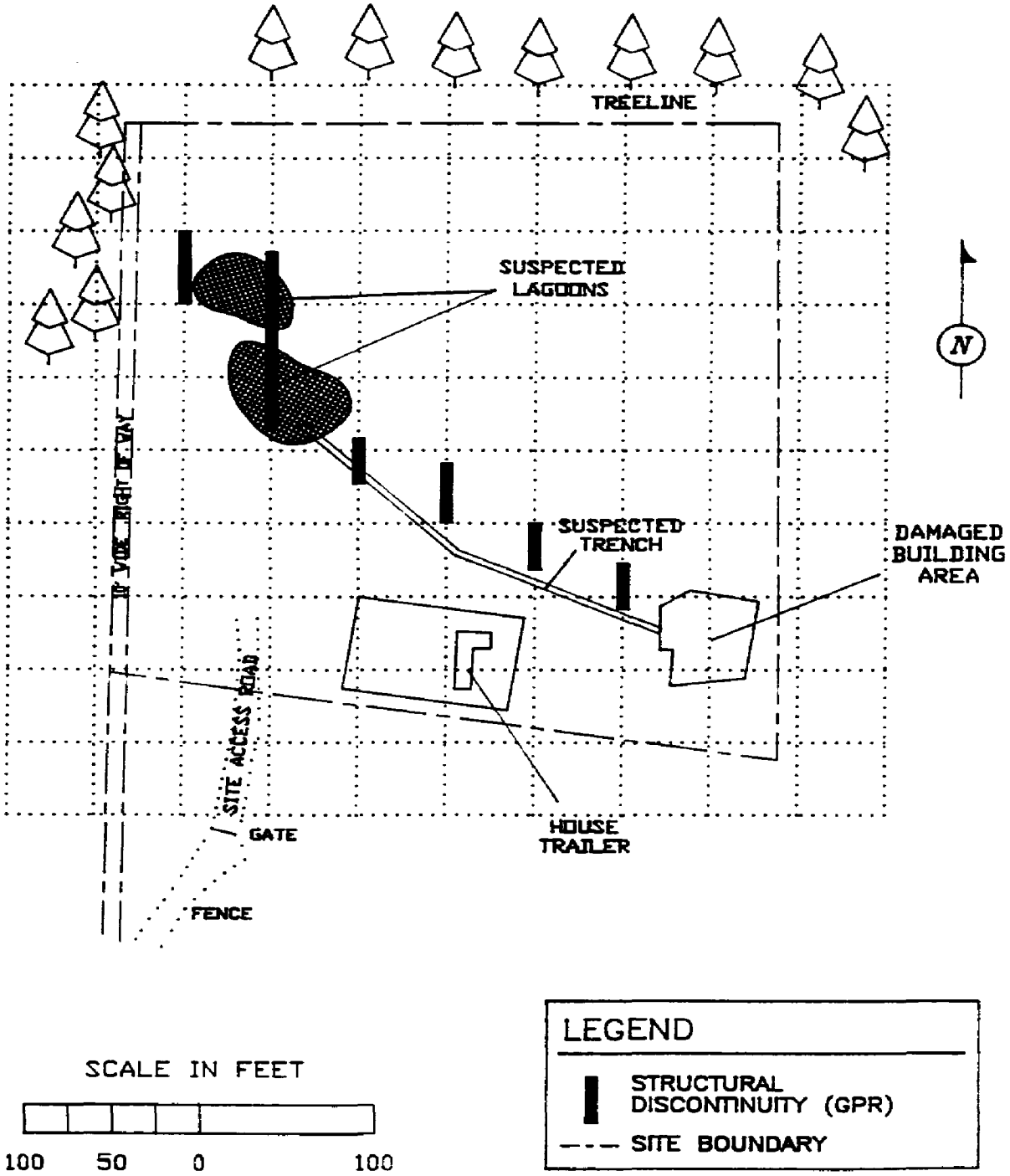
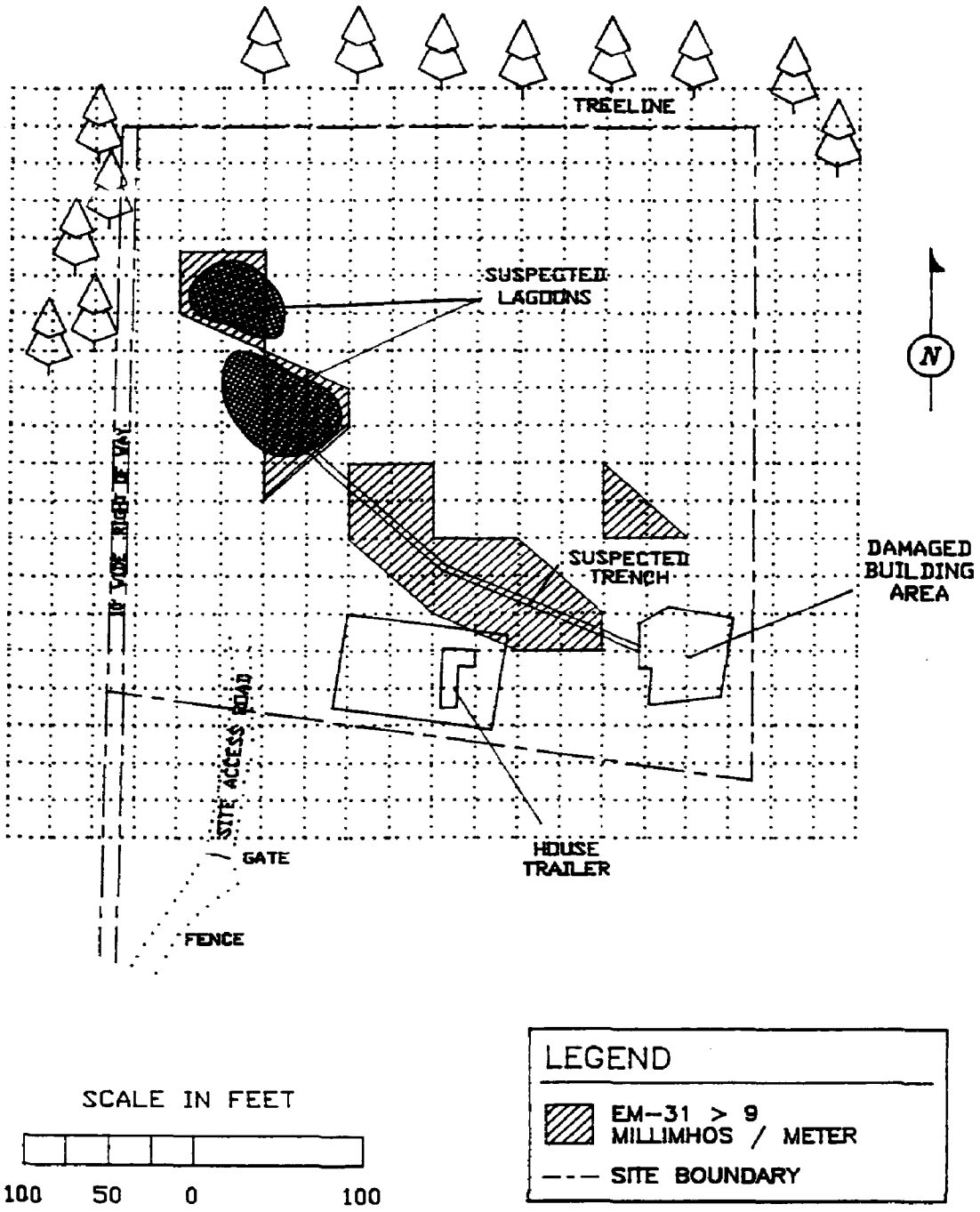


Figure 12: EM-31 Survey Results
ABC Plating Site



3.0 EQUIPMENT

3.1 INTRODUCTION

Sample collection requires an understanding of the capabilities of the sampling equipment, since using inappropriate equipment may result in biased samples. This chapter provides information for selecting sampling and screening equipment.

3.2 ANALYTICAL SCREENING EQUIPMENT

Analytical screening methods provide on-site measurements of contaminants of concern, limiting the number of samples which need to be sent to an off-site laboratory for time-consuming and often costly analysis. Screening techniques can also evaluate soil samples for indications that soil contamination exists (e.g., X-ray fluorescence (XRF) for target metals or soil gas survey for identification of buried wastes or other subsurface contamination). All screening equipment and methods described in this section are **portable** (the equipment is hand-held, and generally no external power is necessary). Examples are photoionization detectors (PID), flame ionization detectors (FID), and some XRF devices.

Screening generally provides analytical data of suitable quality for site characterization, monitoring during response activities, and on-site health and safety decisions. The methods presented here can provide rapid, cost-effective, real-time data; however, results are often not compound-specific and not quantitative.

When selecting one screening method over another, consider relative cost, sample analysis time, potential interferences or instrument limitations, detection limit, QA/QC requirements, level of training required for operation, equipment availability, and data bias. Also consider which elements, compounds, or classes of compounds the screening instrument is designed to analyze. As discussed in Section 2.7, the screening method selected should be sensitive enough to minimize the potential for false negatives. When collecting samples for on-site analysis (e.g., XRF), evaluate the detection limits and bias of the screening method by sending a minimum of 10% of the samples to an off-site laboratory for confirmation. Table 3 summarizes the advantages and disadvantages of selected portable screening equipment.

3.3 GEOPHYSICAL EQUIPMENT

Geophysical techniques can be used in conjunction with analytical screening to help delineate areas of subsurface contamination, including buried drums and tanks. Geophysical data can be obtained relatively rapidly, often without disturbing the site. Geophysical techniques suitable for emergency or removal activities include: ground penetrating radar (GPR), magnetometry, electromagnetic conductivity (EM) and resistivity. Specific advantages and disadvantages associated with geophysical equipment are summarized in Table 4. See also EPA ERT Standard Operating Procedure (SOP) #2159, General Surface Geophysics (U.S. EPA, January 1991).

3.4 SELECTING SAMPLING EQUIPMENT

The mechanical method by which a sampling tool collects the sample may impact representativeness. For example, if the sampling objective is to determine the concentrations of contaminants at each soil horizon interface, using a hand auger would be inappropriate: the augering technique would disrupt and mix soil horizons, making the precise horizon interface difficult to determine. Depth of sampling is another factor to consider in the proper selection of sampling equipment. A trowel, for example, is suitable for unconsolidated surface soils, but may be a poor choice for sampling at 12 inches, due to changes in soil consistency with depth.

All sampling devices should be of sufficient quality not to contribute contamination to samples (e.g., painted surfaces which could chip off into the sample). In addition, the sampling equipment should be either easily decontaminated, or cost-effective if considered to be expendable. Consider ease of use when selecting sampling equipment.

Table 3: Portable Field Analytical Screening Equipment

Equipment	Application to Sampling Design	Advantages and Disadvantages
X-ray fluorescence (portable)	Detects heavy metals in soils	Rapid sample analysis; may be used in situ; requires trained operator; potential matrix interferences; may be used with a generic or site-specific calibration model; detection limit may exceed action level; detects to ppm level; detection limit should be calculated on a site-specific basis.
Flame ionization detector (FID)	Semi-quantitatively detects VOCs in soils	Immediate results; can be used in GC mode to identity specific organic compounds; detects VOCs only; detects to ppm level.
Photoionization detector (PID)	Detects total concentration of VOCs and some non-volativle organics and inorganics in soils	Immediate results; easy to use; non-compound specific; results affected by high ambient humidity and electrical sources such as radios; does not respond to methane; detects to ppm level.
Field test kits	Detects specific elements, compounds, or compound classes in soils	Rapid results; easy to use; low cost; limited number of kit types available; kits may be customized to user needs; semi-quantitative; interferences by other analytes is common; colorimetric interpretation is needed; detection level dependent upon type of kit used; can be prone to error.
Radiation detector	Detects the presence of selected forms of radiation in soils or other waste materials	Easy to use; low cost; probes for one or a combination of alpha, beta or gamma forms of radiation; unit and detection limits vary greatly; detailed site surveys are time intensive and require experienced personnel to interpret results.

Sources: U.S. EPA, September 1988a; U.S. EPA, December 1987; U.S. EPA 1987.

Table 4: Geophysical Equipment

Equipment	Application to Sampling Design	Advantages and Disadvantages
Ground penetrating radar (GPR)	Detects reflection anomalies caused by lithology changes buried objects; varying depths of investigation, 15 to 30 feet, are possible.	Capable of high resolution; generates continuous measurement profile; can survey large area quickly; site specific; best results are achieved in dry, sandy soils; clay-rich and water saturated soils produce poor reflections and limit depth of penetration; data interpretation requires a trained geophysicist.
Magnetometer	Detects presence and areal extent of ferromagnetic material in subsurface soils, including buried metal containers. Single 55-gallon drums can be identified at depths up to 10 feet and large massed of drums up to 30 feet or more.	Quick and easy to operate; good initial survey instrument; readings are often affected by nearby man-made steel structures (including above-ground fences, buildings, and vehicles); data interpretation may require geophysicist.
Electromagnetic conductivity meter (EM)	Detects electrical conductivity changes in subsurface geologic lithology, pore fluids, and buried objects. Depth of investigation varies from 9 feet to 180 feet depending on instrument used, coil spacing, and coil configuration.	Rapid data collection; can delineate inorganic and large-scale organic contamination in subsurface fluids; sensitive to man-made structures (including buried cables, above-ground steel structures and electrical power lines); survey planning and data interpretation may require geophysicist.
Wadi	Detects electrical conductivity changes in surface and sub-surface materials utilizing existing very low frequency (VLF) radio waves.	Utilizes existing long-distance communication VLF radio waves (10-30 Khz range); no need to induce electrical field; directional problems can be overcome with portable transmitters.
Resistivity meter	Detects electrical resistivity variations in subsurface materials (e.g., lithology, pore fluids, buried pipelines and drums). Vertical resolution to depths of 100 feet are possible.	Detects lateral and vertical variations; instrument requires direct ground contact, making it relatively labor intensive; sensitive to outside interference; data interpretation requires a trained geophysicist.

Sources: Benson, et. al. 1988; NJDEP, 1988.

Complicated sampling procedures usually require increased training and introduce a greater likelihood of procedural errors. Standard operating procedures help to avoid such errors. Sample volume is another selection concern. Specific advantages and disadvantages of soil sampling equipment are given in Table 5. Refer also to EPA ERT SOP #2012, Soil Sampling (in U.S. EPA, January 1991) for guidance on using various types of soil sampling equipment.

3.5 EXAMPLE SITE

3.5.1 Selection of Sampling Equipment

Dedicated plastic scoops were used for Phase 1 soil sampling. For Phase 2, the OSC used bucket augers for both surface and subsurface soil sampling because of their ease of use, good vertical depth range, and uniform surface sampling volume. Standard operating procedures were followed to promote proper sample collection, handling, and decontamination. From the bucket auger, each sample was placed into a dedicated plastic pan and mixed using a dedicated plastic scoop. Samples were further prepared for XRF screening and laboratory analysis (Section 4.8).

3.5.2 Selection of Analytical Screening Equipment

Phase 1 sampling identified the sources and types of on-site contaminants in order to establish a threat. Hazard categorization techniques, organic vapor detecting instruments, and radiation and cyanide monitors were utilized to tentatively identify containerized liquid wastestreams in order to select initial judgmental soil sampling locations. During Phase 2 sampling, a portable XRF unit was used to determine the extent of contamination and to identify additional hot spots. Samples to be sent for laboratory analysis were then placed into sampling jars (as discussed in Section 4.8). Samples collected from upgradient grid nodes for XRF screening only were stored on site for later treatment/disposal. For Phase 3, the XRF was used to confirm whether contaminated areas identified during Phase 2 were sufficiently excavated.

3.5.3 Selection of Geophysical Equipment

The GPR instrument delineated buried trench and lagoon boundaries. The EM meter detected subsurface conductivity changes due to buried metal containers and contaminants. The EM-31 (a shallower-surveying instrument than the EM-34) was selected because expected contaminant depth was less than 10 feet and because of the instrument's maneuverability and ease of use.

Table 5: Soil Sampling Equipment

Equipment	Applicability	Advantages and Disadvantages
Trier	Soft surface soil	Inexpensive; easy to use and decontaminate; difficult to use in stony, dry, or sandy soil.
Scoop or trowel	Soft surface soil	Inexpensive, easy to use and decontaminate; trowels with painted surfaces should be avoided.
Tulip bulb planter	Soft soil, 0-6 in.	Easy to use and decontaminate; uniform diameter and sample volume; preserves soil core (suitable for VOA and undisturbed sample collection); limited depth capability; not useful for hard soils.
Soil coring device	Soft soil, 0-24 in.	Relatively easy to use; preserves soil core (suitable for VOA and undisturbed sample collection); limited depth capability; can be difficult to decontaminate.
Thin-wall tube sampler	Soft soil, 0-10 ft.	Easy to use; preserves soil core (suitable for VOA and undisturbed sample collection); may be used in conjunction with bucket auger; acetate sleeve may be used to help maintain integrity of VOA samples, easy to decontaminate; can be difficult to remove cores from sampler.
Split spoon sampler	Soil, 0 in.-bedrock	Excellent depth range; preserves soil core (suitable for VOA and undisturbed sample collection); acetate sleeve may be used to help maintain integrity of VOA samples; useful for hard soils; often used in conjunction with drill rig for obtaining deep cores.
Shelby tube sampler	Soft soil, 0 in.-bedrock	Excellent depth range; preserves soil core (suitable for VOA and undisturbed sample collection); tube may be used to ship sample to lab undisturbed; may be used in conjunction with drill rig for obtaining deep cores and for permeability testing; not durable in rocky soils.
Bucket auger	Soft soil, 3 in.-10 ft.	Easy to use; good depth range; uniform diameter and sample volume; acetate sleeve may be used to help maintain integrity of VOA samples; may disrupt and mix soil horizons greater than 6 inches in thickness.
Hand-operated power auger	Soil, 6 in.-15 ft.	Good depth range; generally used in conjunction with bucket auger for sample collection; destroys soil core (unsuitable for VOA and undisturbed sample collection); requires 2 or more equipment operators; can be difficult to decontaminate; requires gasoline-powered engine (potential for cross-contamination).

Sources: NJDEP, 1988; U.S. EPA, January 1991.

4.0 SAMPLE COLLECTION AND PREPARATION

4.1 INTRODUCTION

In addition to sampling equipment, sample collection includes sample quantity and sample volume. Sample preparation refers to all aspects of sample handling after collection, until the sample is received by the laboratory. Sample preparation for soils may include, but is not limited to:

- removing extraneous material;
- sieving samples;
- homogenizing samples;
- splitting samples;
- compositing samples; and
- final preparation.

Sample preparation depends on the sampling objectives and analyses to be performed. Proper sample preparation and handling help to maintain sample integrity. Improper handling can result in a sample becoming unsuitable for the type of analysis required. For example, homogenizing, sieving, and compositing samples all result in a loss of volatile constituents and are therefore inappropriate when volatile contaminants are the concern.

4.2 SAMPLE COLLECTION

How a sample is collected can affect its representativeness. The greater the number of samples collected from a site and the larger the volume of each sample, the more representative the analytical results will be. However, sampling activities are often limited by sampling budgets and project schedules. The following sections provide guidelines on appropriate sample numbers and volumes.

4.2.1 Sample Number

The number of samples needed will vary according to the particular sampling approach that is being used. For example, in grid sampling, one sample is generally collected at each grid node, regardless of grid size. As discussed in Section 2.11.6, once contaminated grid node samples are located, adjoining grid cells can be sampled more thoroughly to define areas of contamination. Four aliquots from each grid cell, situated equidistant from the sides of each cell and each other (as illustrated in Figure 10), are recommended for grid cells measuring up to 100 x 100 feet. One additional aliquot may be collected from the center of each cell, making a total of five aliquots per cell. For grid sizes greater than 100 feet x 100 feet, nine aliquots, situated equidistant from the sides of each cell and each other (as illustrated in Figure 13), are recommended. Depending on budget and other considerations, grid cell aliquots can be analyzed as separate samples or composited into one or more samples per cell.

4.2.2 Sample Volume

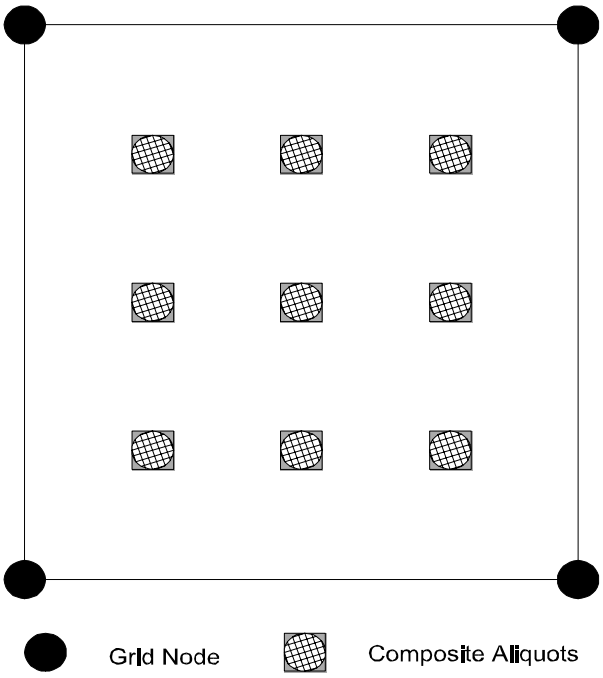
Both sample depth and area are considerations in determining appropriate sample volume. Depending on the analytes being investigated, samples are collected at the surface (0-3 in.), extended surface (0-6 in.), and/or at one-foot depth intervals. Non-water soluble contaminants such as dioxin and PCBs are often encountered within the first six inches of soil. Water-soluble contaminants such as metals, acids, ketones, and alcohols will be encountered at deeper depths in most soils except clays. Contaminants in solution, such as PCPs in diesel fuel and pesticides in solvents, can penetrate to great depths (e.g., down to bedrock), depending on soil type.

For surface samples, collect soil over a surface area of one square foot per sample. A square cardboard template measuring 12 in. x 12 in., or a round template with a 12 in. diameter can be used to mark sampling areas. For subsurface samples, one of several coring devices may be used (see Table 5). Using a coring device results in a smaller diameter sampling area than a surface template, and therefore somewhat lessens the representativeness of the sample.

4.3 REMOVING EXTRANEOUS MATERIAL

Identify and discard materials in a sample which are not relevant or vital for characterizing the sample or the site, since their presence may introduce an error in the sampling or analytical procedures. Examples of extraneous material in soil samples include pieces glass, twigs or leaves. However, not all non-soil material is extraneous. For example, when sampling at a junkyard, lead-contaminated battery casing pieces should not be removed from a sample if the casing composes more than 10% of the sample composition. For a sample to be representative, it must also incorporate the lead from the casing. Collect samples

Figure 13: Phase 2 Sampling Grid Cell Diagram (Grid Sizes > 100 x 100 ft.)



of any material thought to be a potential source of contamination for a laboratory extraction procedure. Discuss any special analytical requirements for extraneous materials with project management, geologists, and chemists and notify the laboratory of any special sample handling requirements.

4.4 SIEVING SAMPLES

Sieving is the process of physically sorting a sample to obtain uniform particle sizes, using sieve screens of predetermined size. For example, the sampler may wish to sieve a certain number of samples to determine if particle size is related to contaminant distribution. Sieving is generally only conducted when preparing soil samples for XRF screening. For this purpose, a 20-mesh screen size is recommended.

Be aware of the intent of the sampling episode, when deciding whether to sieve a sample prior to analysis. Prior to sieving, samples may need to be oven-dried. Discarding non-soil or non-sieved materials, as well as the sieving process itself, can result in physical and chemical losses. Sieving is not recommended where volatile compounds are of concern. Analyze the discarded materials, or a fraction thereof, to determine their contribution to the contamination of the site being investigated.

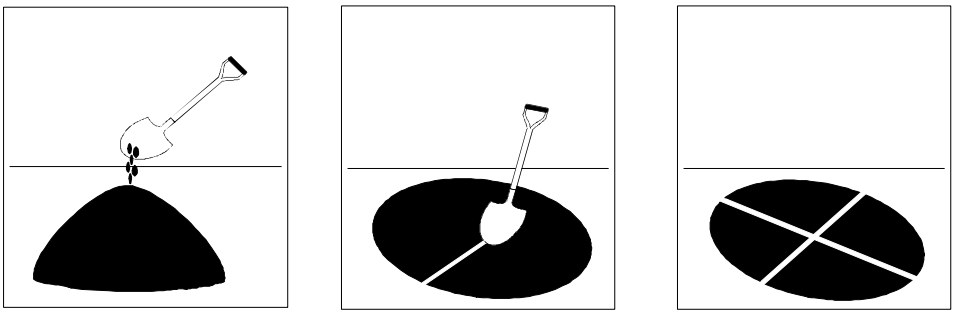
4.5 HOMOGENIZING SAMPLES

Homogenization is the mixing or blending of a soil sample in an attempt to provide uniform distribution of contaminants. (Do not homogenize samples for volatile compound analysis). Ideally, proper homogenization ensures that portions of the containerized samples are equal or identical in composition and are representative of the total soil sample collected. Incomplete homogenization will increase sampling error. All samples to be composited or split should be homogenized after all aliquots have been combined. Manually homogenize samples using a stainless steel spoon or scoop and a stainless steel bucket, or use a disposable scoop and pan. Quarter and split the sample as illustrated in Figure 14, repeating each step a minimum of 5 times until the sample is visually homogenized. Samples can also be homogenized using a mechanically-operated stirring device as depicted in ASTM standard D422-63.

4.6 SPLITTING SAMPLES

Splitting samples after collection and field preparation into two or more equivalent parts is performed when two or more portions of the same sample need to be analyzed separately. Split samples are most often collected in enforcement actions to compare sample results obtained by EPA with those obtained by the potentially responsible party (PRP). Split samples also provide a measure of the sample variability, and a measure of the analytical and extraction errors. Before splitting, follow homogenization techniques outlined above. Fill two sample collection jars simultaneously with alternate spoonfuls (or scoopfuls) of homogenized sample. To simultaneously homogenize and split a sample, **quarter** (as illustrated in Figure 14) or **mechanically** split the sample using a riffle sample splitter. The latter two techniques are described in detail in ASTM Standard C702-87.

Figure 14: Quartering to Homogenize and Split Samples



Step 1:

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

Step 2:

- Flatten cone
- Divide sample into quarters

Step 3: (not shown)

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

4.7 COMPOSITING SAMPLES

Compositing is the process of physically combining and homogenizing several individual soil aliquots. Compositing samples provides an average concentration of contaminants over a certain number of sampling points, which reduces both the number of required lab analyses and the sample variability. Compositing can be a useful technique, but must always be implemented with caution. Compositing is not recommended where volatile compounds are of concern.

Specify the method of selecting the aliquots that are composited and the compositing factor in the sampling plan. The compositing factor is the number of aliquots to be composited into one sample (e.g., 3 to 1; 10 to 1). Determine this factor by evaluating detection limits for parameters of interest and comparing them with the selected action level for that parameter. Compositing also requires that each discrete aliquot be the same in terms of volume or weight, and that the aliquots be thoroughly homogenized. Since compositing dilutes high concentration aliquots, the applicable detection limits should be reduced accordingly. If the composite value is to be compared to a selected action level, then the action level must be divided by the number of aliquots that make up the composite in order to determine the appropriate detection limit (e.g., if the action level for a particular substance is 50 ppb, an action level of 10 ppb should be used when analyzing a 5-aliquot composite). The detection level need not be reduced if the composite area is assumed to be homogeneous in concentration (for example, stack emission plume deposits of particulate contamination across an area, or roadside spraying of waste oils).

4.8 FINAL PREPARATION

Select sample containers on the basis of compatibility with the material being sampled, resistance to breakage, and volume. For soil sampling, use wide-mouth glass containers with Teflon-lined lids. Appropriate sample volumes and containers will vary according to the parameter being analyzed. Keep low and medium concentration soil samples to be analyzed for organic constituents at 4EC. Actual sample volumes, appropriate containers, and holding times are specified in the *QA/QC Guidance for Removal Activities* (U.S. EPA, April 1990), in 40 CFR 136, and in the Compendium of ERT Soil Sampling and Surface Geophysics (U.S. EPA, January 1991). Package all samples in compliance with Department of Transportation (DOT) or International Air Transport Association (IATA) requirements.

It is sometimes possible to ship samples to the laboratory directly in the sampling equipment. For example, the ends of a Shelby tube can be sealed with caps, taped, and sent to the laboratory for analysis. To help maintain the integrity of VOA samples, collect soil cores using acetate sleeves and send the sleeves to the laboratory. To ensure the integrity of the sample after delivery to the laboratory, make laboratory sample preparation procedures part of all laboratory bid contracts.

4.9 EXAMPLE SITE

After placing each sample in a dedicated pan and mixing (as discussed in Section 3.5.1), plant matter, stones, and broken glass were removed. Soil samples were oven-dried (at 104E C) and sieved using a 20-mesh screen in preparation for XRF analysis. Samples were then homogenized and split using the quartering technique. Opposite quarters were remixed and quartering was repeated five times to ensure thorough homogenization. A portion of each sample was placed into XRF analysis cups for screening. The remainder of each sample was placed into 8-ounce, wide-mouth glass jars with Teflon-lined lids and sent to a laboratory for inorganic analysis. The samples were packaged in compliance with IATA requirements. Chain-of-custody paperwork was prepared for the samples. Laboratory paperwork was completed as appropriate and the samples were shipped to the predesignated laboratories for analysis.

5.0 QUALITY ASSURANCE/QUALITY CONTROL EVALUATION

5.1 INTRODUCTION

The goal of representative sampling is to collect samples which yield analytical results that accurately depict site conditions during a given time frame. The goal of quality assurance/quality control (QA/QC) is to identify and implement correct methodologies which limit the introduction of error into the sampling and analytical procedures, ultimately affecting the analytical data.

QA/QC samples evaluate the degree of site variation, whether samples were cross-contaminated during sampling and sample handling procedures, or if a discrepancy in sample results is due to laboratory handling and analysis procedures. The QA/QC sample results are used to assess the quality of the analytical results of waste and environmental samples collected from a site.

5.2 DATA CATEGORIES

EPA has established a process of data quality objectives (DQOs) which ensure that the precision, accuracy, representativeness, and quality of environmental data are appropriate for their intended application. Superfund DQO guidance defines two broad categories of analytical data: *screening* and *definitive*.

Screening data are generated by rapid, less precise methods of analysis with less rigorous sample preparation. Sample preparation steps may be restricted to simple procedures such as dilution with a solvent, rather than elaborate extraction/digestion and cleanup. At least 10 percent of the screening data are confirmed using the analytical methods and QA/QC procedures and criteria associated with definitive data. Screening data without associated confirmation data are not considered to be data of known quality. To be acceptable, screening data must include the following: chain of custody, initial and continuing calibration, analyte identification, and analyte quantification. Streamlined QC requirements are the defining characteristic of screening data.

Definitive data are generated using rigorous analytical methods (e.g., approved EPA reference methods). These data are analyte-specific, with confirmation of analyte identity and concentration. Methods produce tangible raw data (e.g., chromatograms, spectra, digital values) in the form of paper printouts or computer-generated electronic files. Data may be generated at the site or at an off-site location, as long as the QA/QC requirements are satisfied. For the data to be definitive, either analytical or total measurement error must be determined. QC measures for definitive data contain all of the elements associated with screening data, but also may include trip, method, and rinsate blanks; matrix spikes; performance evaluation samples; and replicate analyses for error determination.

For further information on these QA/QC objectives, please refer to EPA's *Quality Assurance/Quality Control Guidance for Removal Activities* or EPA's *Data Quality Objectives Process for Superfund*.

5.3 SOURCES OF ERROR

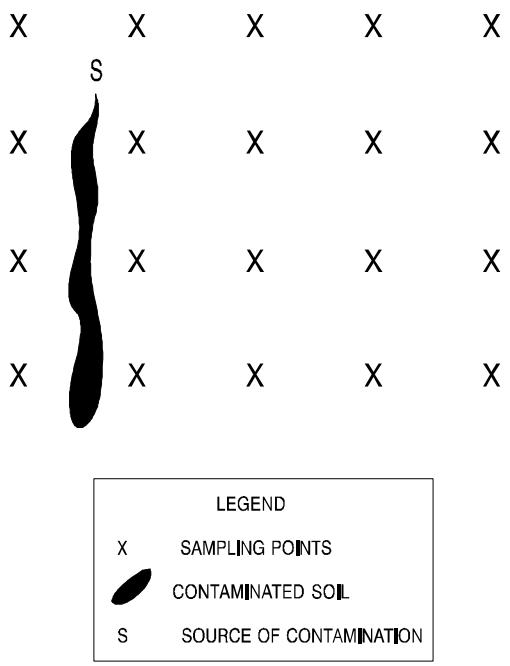
Identifying and quantifying the error or variation in sampling and laboratory analysis can be difficult. However, it is important to limit their effect(s) on the data. Four potential sources of error are:

- sampling design;
- sampling methodology;
- sample heterogeneity; and
- analytical procedures.

5.3.1 Sampling Design

Site variation includes the variation both in the types and in the concentration levels of contaminants throughout a site. Representative sampling should accurately identify and define this variation. However, error can be introduced by the selection of a sampling design which "misses" site variation. For example, a sampling grid with relatively large distances between sampling points or a biased sampling approach (i.e., judgmental sampling) may allow significant contaminant trends to go unidentified, as illustrated in Figure 15.

Figure 15: Sampling Error Due to Sampling Design



5.3.2 Sampling Methodology

Error can be introduced by the sampling methodology and sample handling procedures, as in cross-contamination from inappropriate use of sample collection equipment, unclean sample containers, improper sampling equipment decontamination and shipment procedures, and other factors. Standardized procedures for collecting, handling, and shipping samples allow for easier identification of the source(s) of error, and can limit error associated with sampling methodology. The use of standard operating procedures ensures that all sampling tasks for a given matrix and analyte will be performed in the same manner, regardless of the individual sampling team, date, or location of sampling activity. Trip blanks, field blanks, replicate samples, and rinsate blanks are used to identify error due to sampling methodology and sample handling procedures.

5.3.3 Sample Heterogeneity

Sample heterogeneity is a potential source of error. Unlike water, soil is rarely a homogeneous medium and it exhibits variable properties with lateral distance and with depth. This heterogeneity may also be present in the sample container unless the sample was homogenized in the field or in the laboratory. The laboratory uses only a small aliquot of the sample for analysis; if the sample is not properly homogenized, the analysis may not be truly representative of the sample and of the corresponding site. Thoroughly homogenizing samples, therefore, can limit error associated with sample heterogeneity.

5.3.4 Analytical Procedures

Error which may originate in analytical procedures includes cross-contamination, inefficient extraction, and inappropriate methodology. Matrix spike samples, replicate samples, performance evaluation samples, and associated quality assurance evaluation of recovery, precision, and bias, can be used to distinguish analytical error from error introduced during sampling activities.

5.4 QA/QC SAMPLES

This section briefly describes the types and uses of QA/QC samples that are collected in the field, or prepared for or by the laboratory. QA/QC samples are analyzed in addition to field samples and provide information on the variability and usability of environmental sample results. They assist in identifying the origin of analytical discrepancies to help determine how the analytical results should be used. They are used mostly to validate analytical results. Field replicate, collocated, background, and rinsate blank samples are the most commonly collected field QA/QC samples. Performance evaluation, matrix spike, and matrix spike duplicate samples, either prepared for or by the laboratory, provide additional measures of control for the data generated. QA/QC results may suggest the need for modifying sample collection, preparation, handling, or analytical procedures if the resultant data do not meet site-specific quality assurance objectives. Refer to data validation procedures in U.S. EPA, April 1990, for guidelines on utilizing QA/QC analytical results. The following paragraphs briefly describe each type of QA/QC sample.

5.4.1 Field Replicates

Field replicates are field samples obtained from one location, homogenized, divided into separate containers and treated as separate samples throughout the remaining sample handling and analytical processes. These samples are used to assess error associated with sample heterogeneity, sample methodology and analytical procedures. Use field replicates when determining total error for critical samples with contamination concentrations near the action level. For statistical analysis to be valid in such a case, a minimum of eight replicate samples would be required.

5.4.2 Collocated Samples

Collocated samples are collected adjacent to the routine field sample to determine local variability of the soil and contamination at the site. Typically, collocated samples are collected about one-half to three feet away from the selected sample location. Analytical results from collocated samples can be used to assess site variation, but only in the immediate sampling area. Due to the non-homogeneous nature of soil at sites, collocated samples should not be used to assess variability across a site and are not recommended for assessing error. Determine the applicability of collocated samples on a site-by-site basis. Collecting many samples (more than 50 samples/acre), is sufficient to demonstrate site variation.

5.4.3 Background Samples

Background samples are collected upgradient of the area(s) of contamination (either on or off site) where there is little or no chance of migration of the contaminants of concern. Background samples determine the natural composition of the soil (especially important in areas with high concentrations of naturally-occurring metals) and are considered "clean" samples. They provide a basis for comparison of contaminant concentration levels with samples collected on site. At least one background soil sample should be collected; however, more are warranted when site-specific factors such as natural variability of local soil, multiple on-site contaminant source areas, and presence of off-site facilities potentially contributing to soil contamination exist. Background samples may be collected for all QA objectives, in order to evaluate potential error associated with sampling design, sampling methodology, and analytical procedures.

5.4.4 Rinsate Blanks

Rinsate blanks are samples obtained by running analyte-free water over decontaminated sampling equipment to test for residual contamination. The blank is placed in sample containers for handling, shipment, and analysis identical to the samples collected that day. A rinsate blank is used to assess cross-contamination brought about by improper decontamination procedures. Where dedicated sampling equipment is not utilized, collect one rinsate blank, per type of sampling device, per day.

5.4.5 Performance Evaluation Samples

Performance evaluation (PE) samples evaluate the overall bias of the analytical laboratory and detect any error in the analytical method used. These samples are usually prepared by a third party, using a quantity of analyte(s) which is known to the preparer but unknown to the laboratory, and always undergo certification analysis. The analyte(s) used to prepare the PE sample is the same as the analyte(s) of concern. Laboratory procedural error is evaluated by the percentage of analyte identified in the PE sample (percent recovery). Even though they are not available for every single analyte, analysis of PE samples is required to obtain definitive data.

5.4.6 Matrix Spike Samples

Matrix spike and matrix spike duplicate samples (MS/MSDs) are environmental samples that are spiked in the laboratory with a known concentration of a target analyte(s) to verify percent recoveries. MS/MSDs are primarily used to check sample matrix interferences. They can also be used to monitor laboratory performance. However, a dataset of at least three or more results is necessary to distinguish between laboratory performance and matrix interference.

MS/MSDs can also monitor method performance. Again, a dataset is helpful to assess whether a method is performing properly. Generally, interference and poor method performance go together.

MS/MSDs can also evaluate error due to laboratory bias and precision (when four or more pairs are analyzed). Analyze one MS/MSD pair to assess bias for every 20 soil samples. Use the average percent recovery for the pair. To assess precision, analyze at least 8 matrix spike replicates from the same sample, determine the standard deviation and the coefficient of variation. See pages 9 - 10 of the *QA/QC Guidance*

for Removal Activities (U.S. EPA, April 1990) for procedures on calculating analytical error. MS/MSDs are optional when the goal is to obtain screening data and required to obtain definitive data as one of several methods to determine analytical error.

5.4.7 Field Blanks

Field blanks are samples prepared in the field using certified clean sand or soil and are then submitted to the laboratory for analysis. A field blank is used to evaluate contamination error associated with sampling methodology and laboratory procedures. If available, submit field blanks at a rate of one per day.

5.4.8 Trip Blanks

Trip blanks are samples prepared prior to going into the field. Trip blanks consist of certified clean sand or soil and are handled, transported, and analyzed in the same manner as the other volatile organic samples acquired that day. Trip blanks are used to evaluate error associated with sampling methodology and analytical procedures by determining if any contamination was introduced into samples during sampling, sample handling and shipment, and/or during laboratory handling and analysis. If available, utilize trip blanks for volatile organic analyses.

5.5 EVALUATION OF ANALYTICAL ERROR

The percentage and types of QA/QC samples needed to help identify the error and confidence in the data is based on the sampling objectives and the corresponding QA/QC objectives. The acceptable level of error is determined by the intended use of the data and the sampling objectives, including such factors as: the degree of threat to public health, welfare, or the environment; selected action levels; litigation concerns; and budgetary constraints.

The use of replicate samples is one method to evaluate error. To evaluate the total error of samples with contaminant concentrations near the selected action level, prepare and analyze a minimum of eight replicates of the same sample. Analytical data from replicate samples can also be used for a quick check on errors associated with sample heterogeneity, sample methodology and analytical procedures. Differing analytical results from two or more replicate samples could indicate improper sample preparation (e.g., incomplete homogenization), or that contamination was introduced during sample collection, preparation, handling, shipment, or analysis.

It may be desirable to try to quantify confidence; however, quantification or analytical data correction is not always possible. A 95% confidence level (i.e., 5% acceptable error) should be adequate for most sampling activities. Experience will provide the best determination of whether to use a higher (e.g., 99%) or lower (e.g., 90%) level of confidence. It must be recognized that the use of confidence levels is based on the assumption that a sample is homogeneous. See also Section 6.8 for information on total error.

5.6 CORRELATION BETWEEN SCREENING RESULTS AND DEFINITIVE RESULTS

One cost-effective approach for delineating the extent of site contamination is to correlate inexpensive screening data and other field measurements (e.g., XRF, soil-gas measurements) with laboratory results. The relationship between the two methods can then be described by a regression analysis and used to predict laboratory results based on screening measurements. In this manner, cost-effective screening results may be used in addition to, or in lieu of, off-site laboratory sample analysis.

Statistical regression involves developing a model (equation) that relates two or more variables at an acceptable level of correlation. When screening techniques, such as XRF, are used along with laboratory methods (e.g., atomic absorption (AA)), a regression equation can be used to predict a laboratory value based on the results of the screening device. The model can also be used to place confidence limits around predictions. Additional discussion of correlation and regression can be found in most introductory statistics textbooks. A simple regression equation (e.g., linear) can be developed on many calculators or computer databases; however, a statistician should be consulted to check the accuracy of more complex models.

Evaluation of the accuracy of a model in part relies on statistical correlation. Statistical correlation involves computing an index called the correlation coefficient (r) that indicates the degree and nature of the relationship between two or more sets of values. The correlation coefficient ranges from !1.0 (a perfect inverse or negative relationship), through 0 (no relationship), to +1.0 (a perfect direct, or positive, relationship). The square of the correlation coefficient, called the coefficient of determination, or simply R^2, is an estimate of the proportion of variance

in one variable (the dependent variable) that can be accounted for by the independent variables. The R^2 value that is acceptable depends on the sampling objectives and intended data uses. As a rule of thumb, statistical relationships should have an R^2 value of at least 0.6 to determine a reliable model; however, for health or risk assessment purposes, the acceptable R^2 value may be made more stringent (e.g., 0.8). Analytical calibration regressions have an R^2 value of 0.98 or better.

Once a reliable regression equation has been derived, the screening data can be used to predict laboratory results. These predicted values can then be located on a base map and contoured (mapping methods are described in Chapter 6). These maps can be examined to evaluate the estimated extent of contamination and the adequacy of the sampling program.

5.7 EXAMPLE SITE

The screening of containerized liquid wastes was performed to quickly obtain data indicating general chemical class. Definitive analysis was run on 10% of the samples in order to verify screening results. The definitive analyses provided were analyte and concentration specific. Recoveries of matrix spike and matrix spike duplicate samples indicated no matrix interferences. Dedicated equipment was used during Phase 1 sampling, making rinsate blanks unnecessary. Phase 2 screening was performed using XRF. During Phase 2, samples were collected at 30% of the nodes screened with the XRF. These samples were sent for laboratory AA analysis. A correlation was established by plotting the Phase 2 AA and XRF data. This allowed the XRF data from the other 70% of the nodes to be used to evaluate the chromium levels across the site.

For Phase 2 and 3 sampling, 10% of the data were confirmed by running replicate analyses to obtain an estimate of precision. The results indicated good correlation. Matrix spikes and matrix spike duplicate samples indicated no matrix interferences. During Phase 2, the OSC included performance evaluation (PE) samples for metals to evaluate the overall laboratory bias. The laboratory achieved 92% recovery, which was within the acceptable control limits.

During Phases 2 and 3, a rinsate blank was collected each day. Following the decontamination of the bucket augers, analyte-free water was poured over the augers and the rinsate was placed into 1-liter polyethylene bottles and preserved. The rinsate blanks were analyzed for total metals and cyanide to determine the effectiveness of the decontamination procedures and the potential for cross-contamination. All rinsate blank samples were "clean", indicating sufficient decontamination procedures.

The correlation analysis run on Phase 2 laboratory (AA) data and corresponding XRF values resulted in r values of 0.97 for both surface and subsurface data, which indicated a strong relationship between the AA and XRF data. Following the correlation analyses, regression analyses were run and equations to predict laboratory values based on the XRF data were developed. The resulting equation for the surface data was: AA = 0.87 (XRF) + 10.16. The resulting regression equation for the subsurface data was: AA = 0.94 (XRF) + 0.30.

6.0 DATA PRESENTATION AND ANALYSIS

6.1 INTRODUCTION

Data presentation and analysis techniques are performed with analytical, geophysical, or screening results. The techniques discussed below can be used to compare analytical values, to evaluate numerical distribution of data, to determine and illustrate the location of hot spots and the extent of contamination across a site, and to assess the need for removal of contaminated soil with concentrations at or near the action level. The appropriate methods to present and analyze sample data depend on the sampling objectives, the number of samples collected, the sampling approaches used, and a variety of other considerations.

6.2 DATA POSTING

Data posting involves placement of sample values on a site basemap. Data posting is useful for displaying the spatial distribution of sample values to visually depict extent of contamination and to locate hot spots. Data posting requires each sample to have a specific location (e.g., X and Y coordinates). Ideally, the sample coordinates would be surveyed values to facilitate placement on a scaled map.

6.3 GEOLOGIC GRAPHICS

Geologic graphics include cross-sections and fence diagrams, which are two- and three-dimensional depictions, respectively, of soils and strata to a given depth beneath the site. These types of graphics are useful for posting subsurface analytical data as well as for interpreting subsurface geology and contaminant migration.

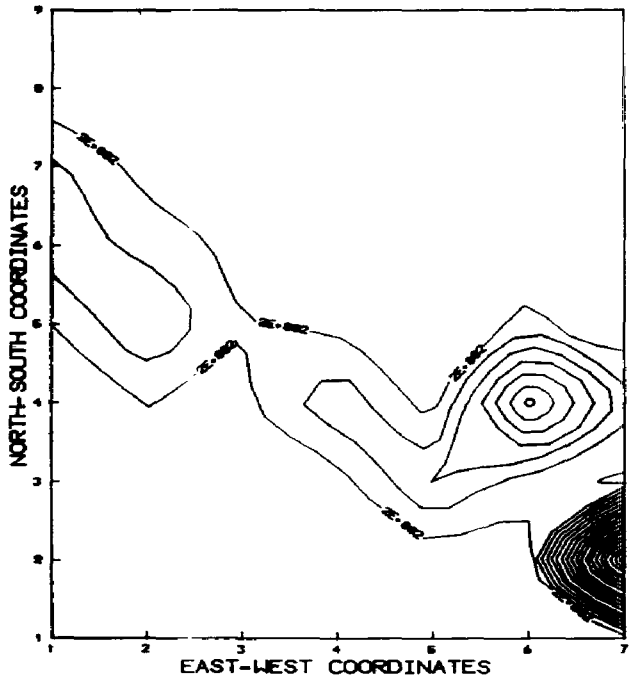
6.4 CONTOUR MAPPING

Contour maps are useful for depicting contaminant concentration values throughout a site. Contour mapping requires an accurate, to-scale basemap of the site. After data posting sample values on the basemap, insert contour lines (or isopleths) at a specified contour interval, interpolating values between sample points. Contour lines can be drawn manually or be generated by computer using contouring software. Although the software makes the contouring process easier, computer programs have a limitation: they may interpolate between all data points, attempting to fit a contour interval to the full range of data values. This can result in a contour map that does not accurately represent general site contaminant trends. Typical emergency or early action sites have low concentration/non-detect areas and hot spots. Computer contouring programs may represent these features as in Figure 16 which illustrates a site that has a 4000 mg/kg hot spot. Because there is a large difference in concentration between the hot spot and the surrounding area, the computer contouring program used a contour interval that eliminated most of the subtle site features and general trends. However, if that same hot spot concentration value is posted at a reduced value, then the contouring program can select a more appropriate contour interval to better illustrate the general site trends. Figure 17 depicts the same site as in Figure 16, but the hot spot concentration value has been arbitrarily posted at 1400 mg/kg. The map was recontoured and the contouring program selected a contour interval that resulted in a map which enhanced the subtle detail and general site contaminant trends.

6.5 STATISTICAL GRAPHICS

The distribution or spread of the data set is important in determining which statistical techniques to use. Common statistical analyses such as the t-test relies on normally distributed data. The histogram is a statistical bar graph which displays the distribution of a data set. A normally distributed data set takes the shape of a bell curve, with the mean and median close together about halfway between the maximum and minimum values. A probability plot depicts cumulative percent against the concentration of the contaminant of concern. A normally distributed data set, when plotted as a probability plot, would appear as a straight line. Use a histogram or probability plot to see trends and anomalies in the data prior to conducting more rigorous forms of statistical analysis.

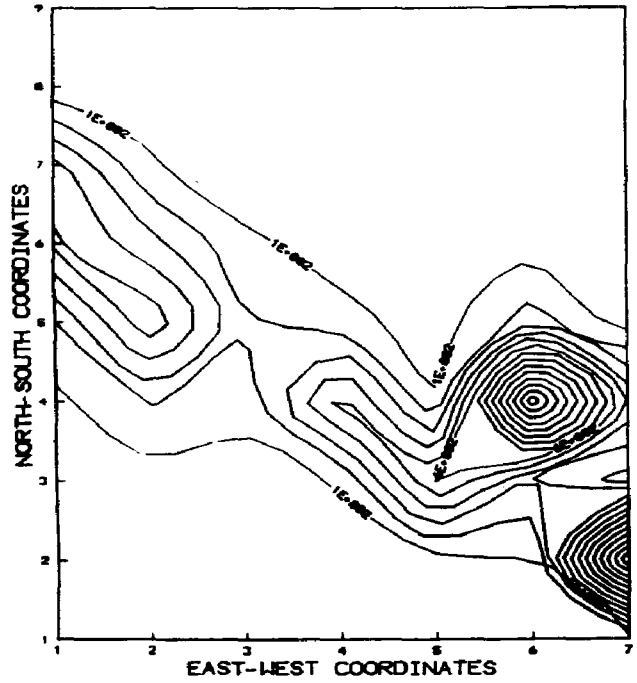
Figure 16: Computer-Generated Contour Map (4000 mg/kg Hot Spot)
ABC Plating Site



Total Chromium Concentration
Units = mg/kg
Contour Interval = 100 mg/kg

Includes 4000 mg/kg Hot Spot

Figure 17: Computer-Generated Contour Map (1400 mg/kg Hot Spot)
ABC Plating Site



Total Chromium Concentration
Units = mg/kg
Contour Interval = 100 mg/kg

Includes 1400 mg/kg Hot Spot*

* 1400 mg/kg hot spot is substituted for 4000 mg/kg hot spot

6.6 GEOSTATISTICS

Geostatistical methods are useful for data analysis and presentation. The characteristic feature of geostatistics is the use of variograms to quantify and model the spatial relationship between values at different sampling locations and for interpolating (e.g., kriging) estimated values across a site. The geostatistical analysis can be broken down into two phases. First, a model is developed that describes the spatial relationship between sample locations on the basis of a plot of spatial variance versus the distance between pairs of samples. This plot is called a variogram. Second, the spatial relationship modeled by the variogram is used to compute a weighted-average interpolation of the data. The result of geostatistical mapping by data interpolation is a contour map that represents estimates of values across a site, and maps depicting potential error in the estimates. The error maps are useful for deciding if additional samples are needed and for calculating best or worst-case scenarios for site cleanup. More information on geostatistics can be found in U.S. EPA, September 1988b and U.S. EPA, 1990. Geo-EAS and GEOPACK, geostatistical environmental assessment software packages developed by U.S. EPA, can greatly assist with geostatistical analysis methods.

6.7 RECOMMENDED DATA INTERPRETATION METHODS

The data interpretation method chosen depends on project-specific considerations, such as the number of sampling locations and their associated range in values. A site depicting extremely low data values (e.g., non-detects) with significantly higher values (e.g., 5,000 ppm) from neighboring hot spots, with little or no concentration gradient in-between, does not lend itself to contouring and geostatistics, specifically the development of variograms. However, data posting would be useful at such a site to illustrate hot spot and clean areas. Conversely, geostatistics and contour mapping, as well as data posting, can be applied to site data with a wide distribution of values (i.e., depicting a "bell shaped" curve) with beneficial results.

6.8 UTILIZATION OF DATA

When conducting search sampling to determine the locations of hot spots (as discussed in Section 2.9), analyze the data using one of the methods discussed in this chapter. For each node that is determined to be close to or above the action level, the following procedure is recommended.

Investigate all neighboring grid cells to determine which areas must be excavated and/or treated. From each grid cell, take a composite sample consisting of four or more aliquots, using the procedure described in Section 2.11.6. Grid cells with contaminant concentrations significantly above the action level (e.g., 20%) should be marked for removal. Grid cells with contaminant concentrations significantly less than the action level should be designated as clean. For grid cells with contaminant concentrations close to the action level, it is recommended that additional sampling be done within that grid cell to determine whether it is truly a hot spot, or whether the analytical result is due to sampling and/or analytical procedural error. If additional sampling is to be performed, one of the following methods should be considered:

- Collect a minimum of four grab samples within the grid cell in question. Use these samples to develop a 95% confidence interval around the mean concentration. If the action level falls within or below this confidence interval, then consider removal/treatment of the soil within that grid cell. More information on confidence intervals and standard deviation can be found in Gilbert, 1987.

- Collect additional composite samples from the grid cells in question using the technique discussed in Section 2.11.6. From these additional samples, determine the need for removal/treatment.

These two practical approaches help to determine the total error associated with collecting a sample from a non-homogeneous site. Total error includes design error, sampling error, non-homogeneous sampling error, and analytical error.

If additional sampling is being considered, weigh the cost-effectiveness of collecting the additional samples versus removing the soil from the areas in question. This decision must be made on a site-by-site basis.

After removal/treatment of the contaminated soil, re-investigate the grid cells to verify cleanup below the action level. Each grid cell that had soil removed must either be composite sampled again, or have multiple grab samples collected with a 95% confidence interval set up again. Again, this decision must be made on a site-by-site basis. The methodology should be repeated until all grid cells are determined to have soil concentrations below the action level.

6.9 EXAMPLE SITE

The Phase 2 XRF/atomic absorption (AA) data were examined to determine the appropriate data interpretation method to use. A histogram was generated to illustrate the distribution of the data as depicted in Figure 18. The histogram showed an uneven distribution of the data with most values less than 50 (approximately 4 on the LN scale of the histogram). Also, the presence of a single data point of 4000 (8 on the LN scale) was shown on the histogram. The data were initially posted as illustrated in Figures 19 and 20. Data posting was performed manually to give the OSC a quick depiction of the general site contamination trends. A contour mapping program was used to generate contours based on the posted data. Figure 16 illustrates the results of contouring with the 4000 mg/kg hot spot included. This contour map exaggerated the hot spot while eliminating the subtle site features and contaminant trends. Figure 17 depicts the same site data with the hot spot arbitrarily reduced to 1400 mg/kg. The resulting contour map enhanced more of the subtle site features and trends while reducing the effects of the hot spot.

AA concentrations predicted by the regression equations were kriged and contoured using Geo-EAS (Figures 21 and 22). Both the kriged contours and the data posting showed the same general site contaminant trends. However, data posting gave a more representative depiction of actual levels of contamination and the OSC used data posting for decision-making.

For each node with chromium concentrations close to or above the 100 ppm action level, the adjacent grid cells were further investigated. Composite samples consisting of four aliquots of soil were taken from within each grid cell in question and analyzed. If the soil concentration level was significantly below 100 ppm of chromium, the cell was designated as clean. Each cell that had a soil concentration level well above the action level was marked for removal/treatment. Any cells having soil concentrations close to the action level were sampled further using the compositing method to better quantify the actual contaminant concentration. Since the surrounding area is residential, on-site landfilling was not considered a viable treatment option. To expedite treatment/disposal, all excavated soil from contaminated cells was stockpiled on site until treatment/disposal could be accomplished under a fixed-price contract. The stockpile, placed in the area of the most highly contaminated grid cells (where the lagoons were located), was covered until treatment/disposal could be arranged. Cleanup was verified with composite sampling in the excavated cells. Results of the composite sampling were compared with the action level to verify cleanup. All action levels were met. The excavation pits were filled with stone and clean soil, covered with topsoil, graded and seeded.

Figure 18: Histogram of Surface Chromium Concentrations
ABC Plating Site

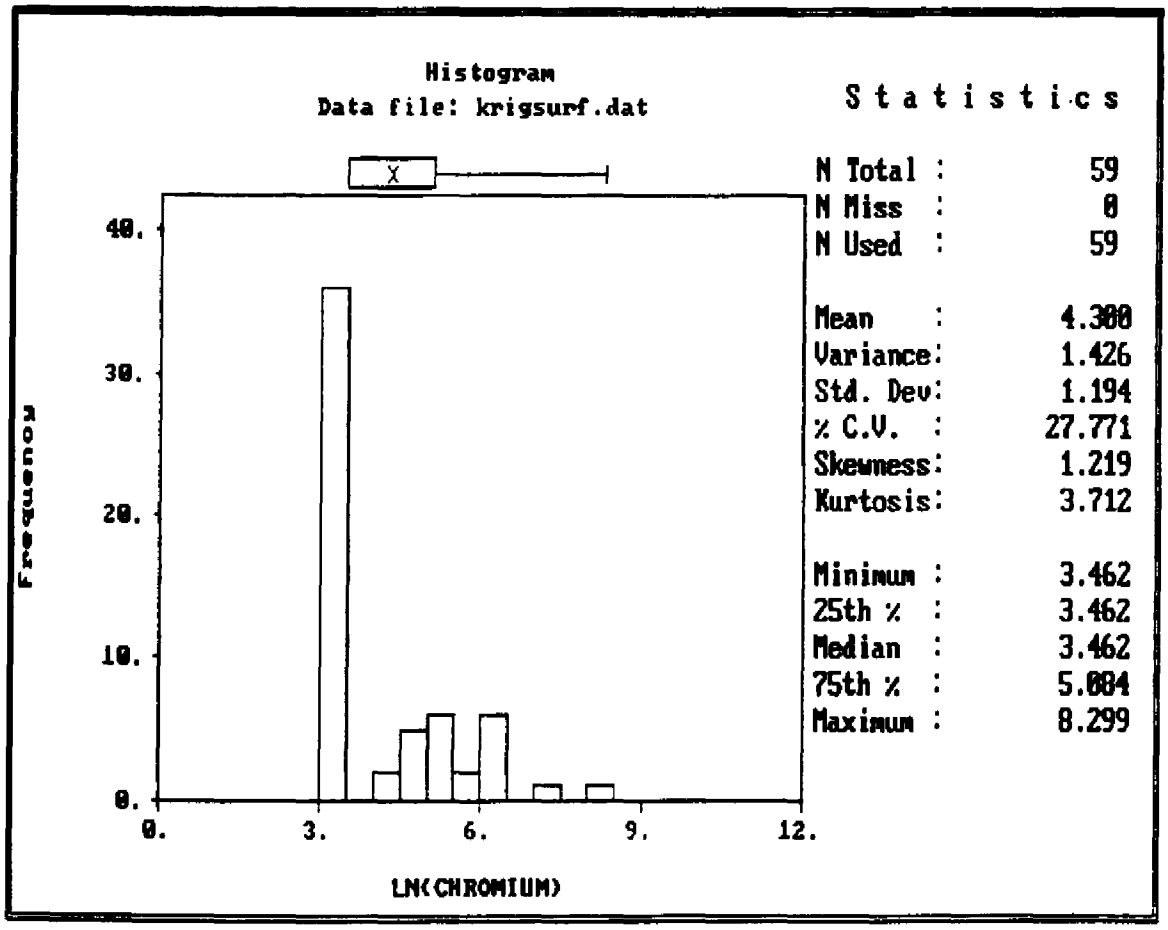


Figure 19: Phase 2 Surface Data Posting for Chromium
ABC Plating Site

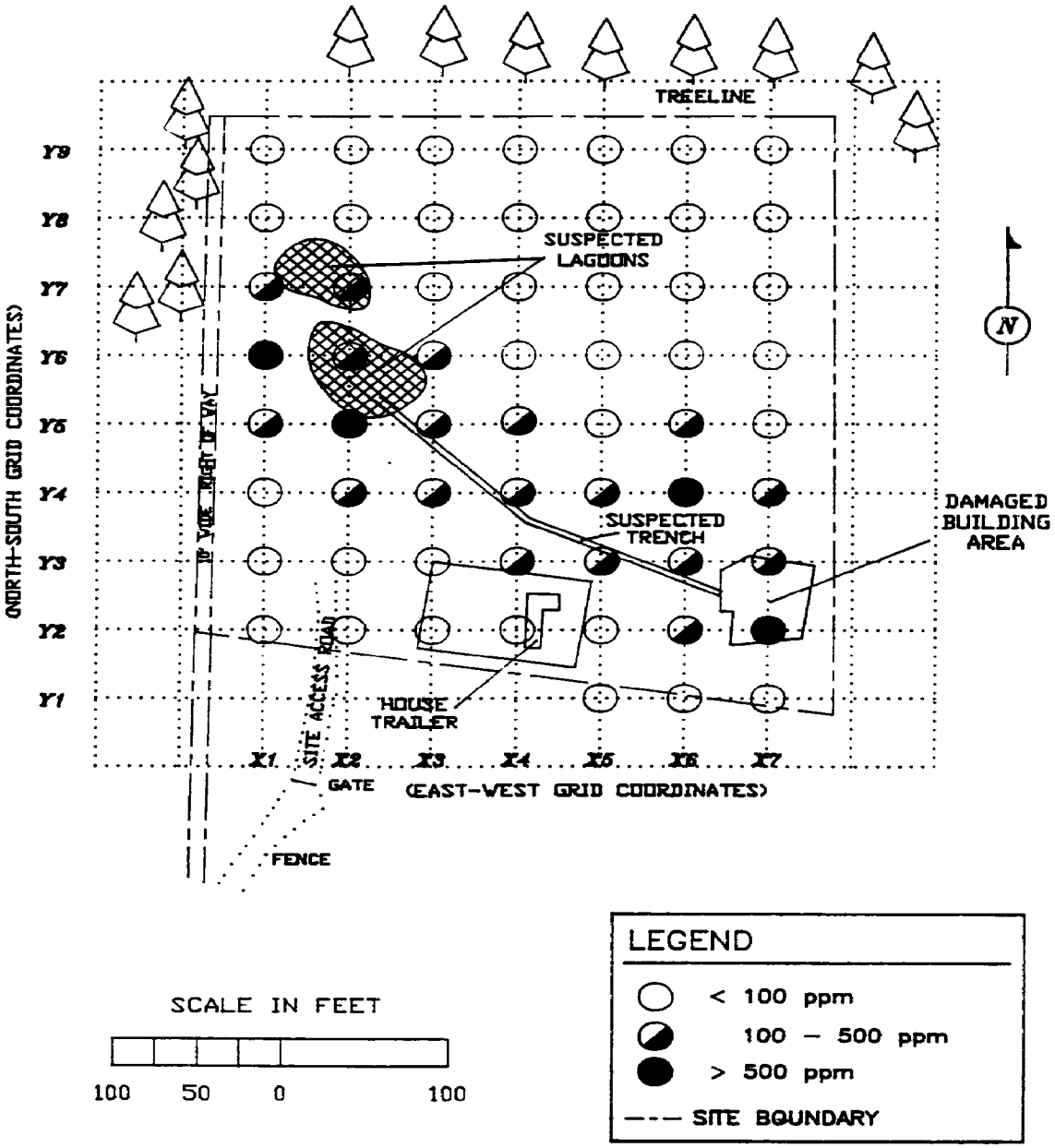


Figure 20: Phase 2 Subsurface Data Posting for Chromium
ABC Plating Site

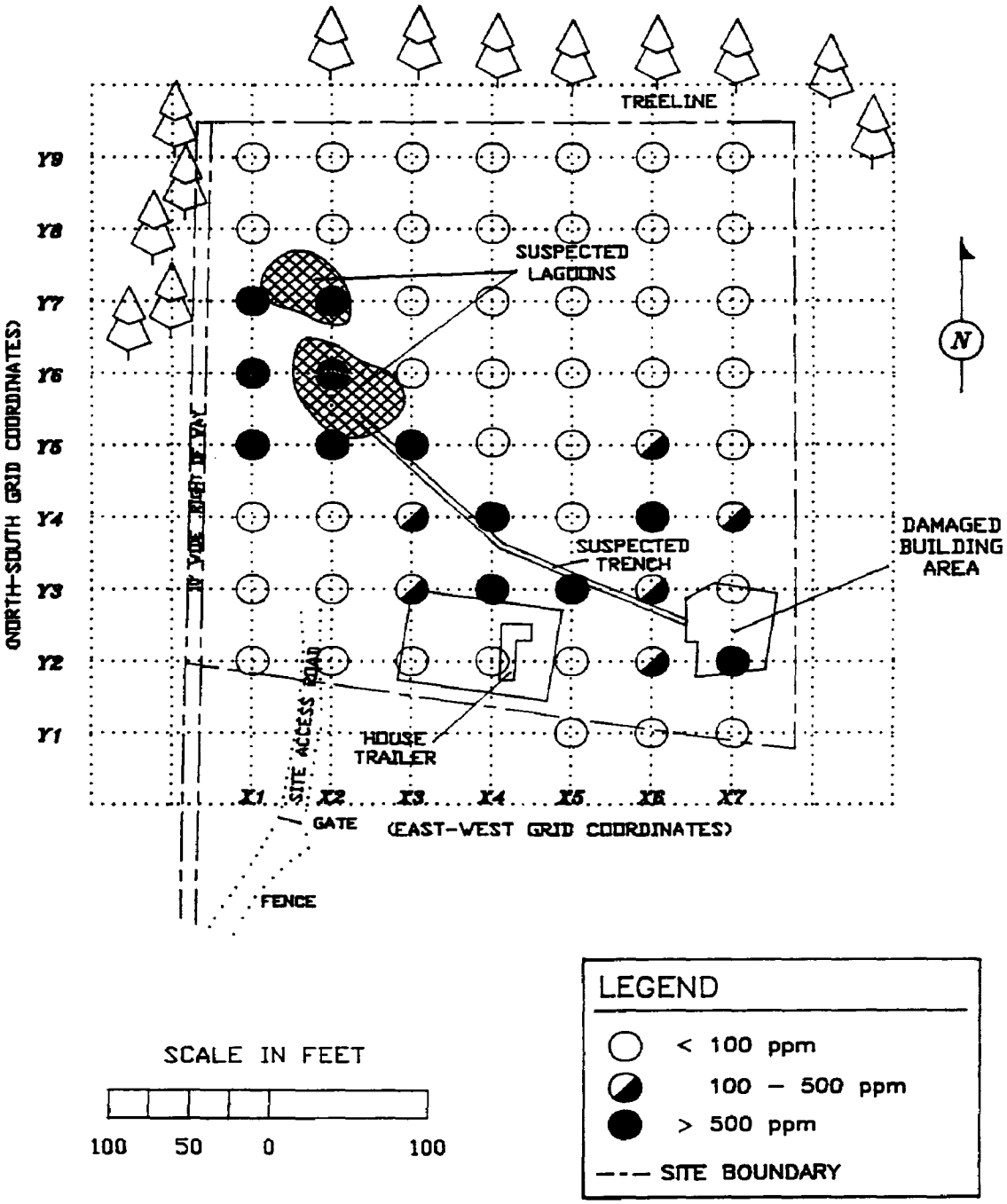


Figure 21: Contour Map of Surface Chromium Data (ppm)
ABC Plating Site

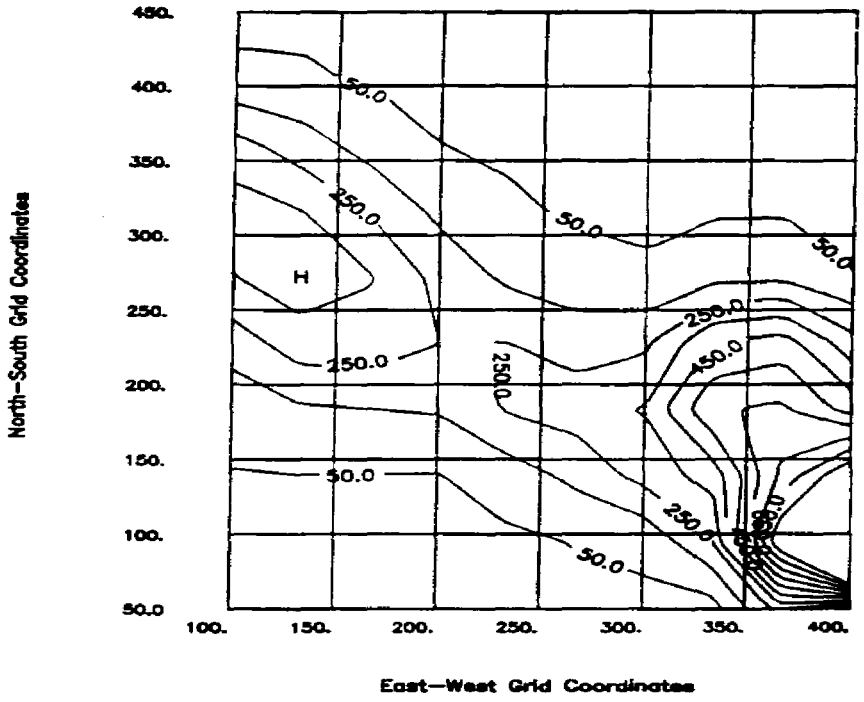
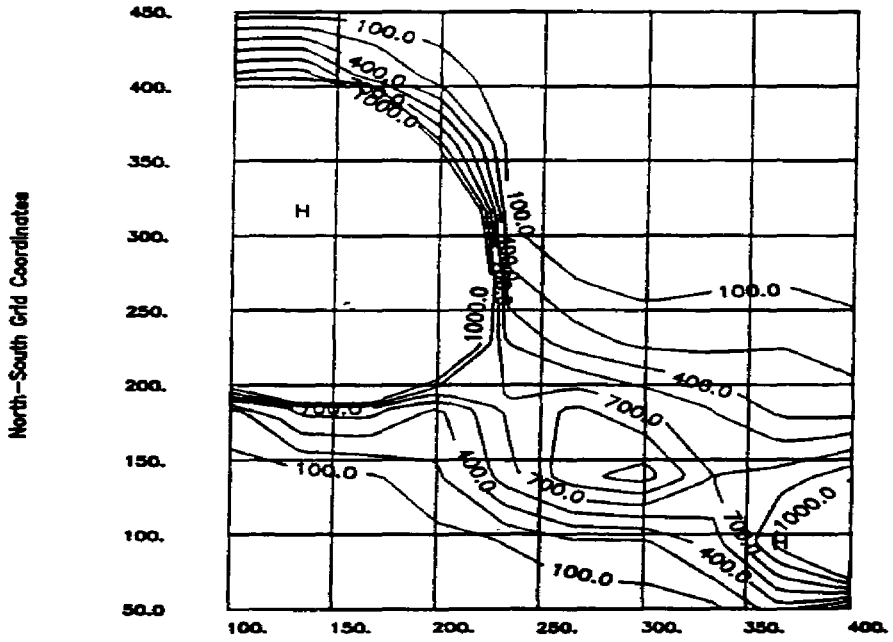


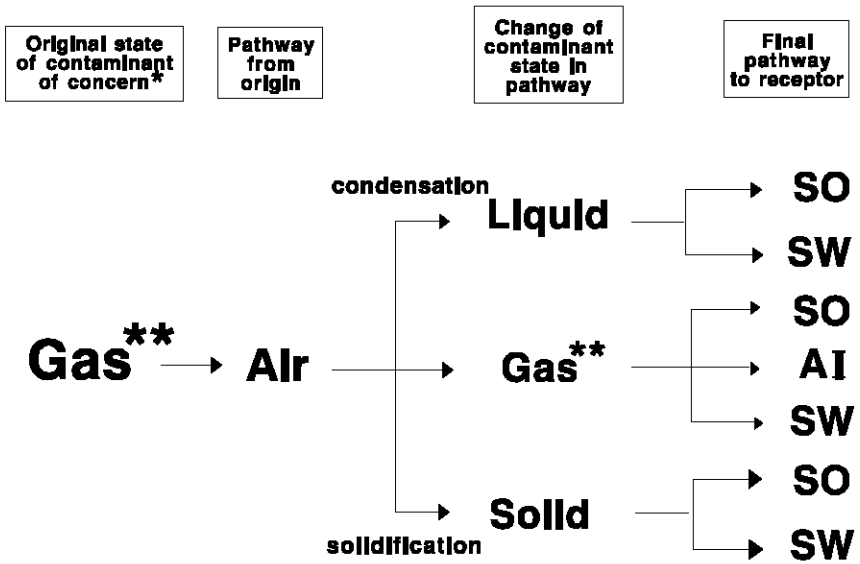
Figure 22: Contour Map of Subsurface Chromium Data (ppm)
ABC Plating Site



APPENDIX A -- EXAMPLE OF FLOW DIAGRAM FOR CONCEPTUAL SITE MODEL

Figure A-1

Migration Routes of a Gas Contaminant from Origin to Receptor



Receptor		
Human	Ecological Threat	
	Terrestrial	Aquatic
G,D	G,D	N/A
G,D	G,D	G,D
I,D	I,D	N/A
I,D	I,D	N/A
G,D	I,D	G,D
G,D	G,D	N/A
G,D	G,D	G,D

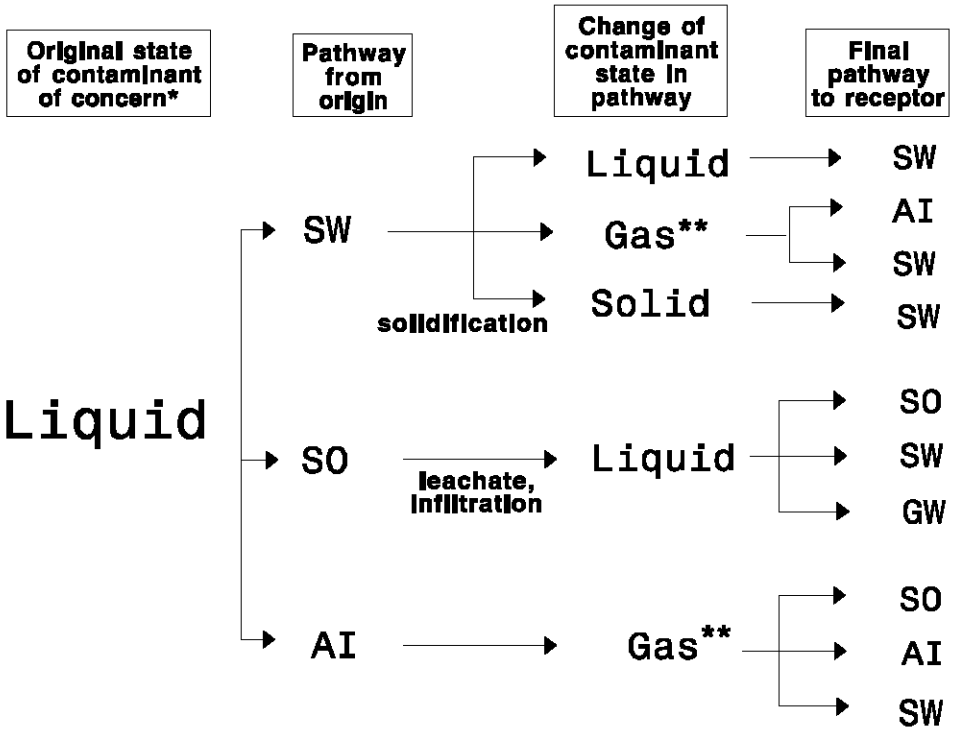
* May be a transformation product
** Includes vapors

Receptor Key
D = Dermal Contact
I = Inhalation
G = Ingestion
N/A = Not Applicable

Pathway Key
AI = Air
SO = Soil
SW = Surface Water (including sediments)
GW = Ground Water

Figure A-2

Migration Routes of a Liquid Contaminant from Origin to Receptor



Receptor		
Human	Ecological Threat	
	Terrestrial	Aquatic
G,D	G,D	G,D
I,D	I,D	N/A
G,D	G,D	G,A
G,D	G,D	G,D
G,D	G,D	N/A
G,D	G,D	G,D
G,D	N/A	N/A
G,D	G,D	N/A
I,D	I,D	N/A
G,D	G,D	G,D

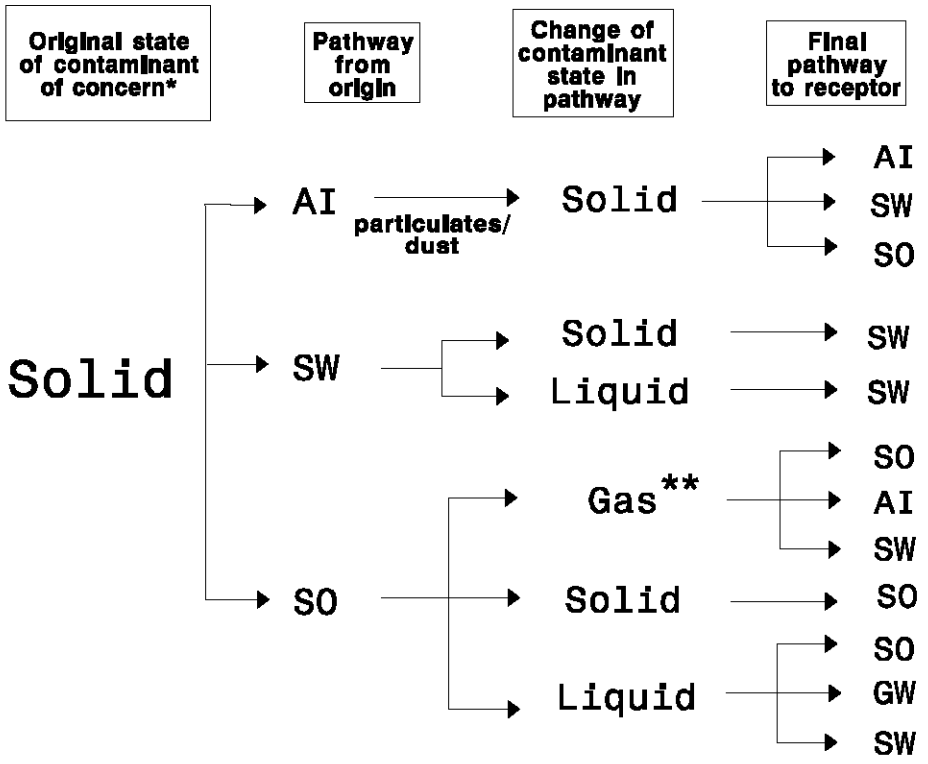
* May be a transformation product
** Includes vapors

Receptor Key
D = Dermal Contact
I = Inhalation
G = Ingestion
N/A = Not Applicable

Pathway Key
AI = Air
SO = Soil
SW = Surface Water (including sediments)
GW = Ground Water

Figure A-3

Migration Routes of a Solid Contaminant from Origin to Receptor



Receptor		
Human	Ecological Threat	
	Terrestrial	Aquatic
I,D	I,D	N/A
G,D	G,D	G,D
G,D	G,D	N/A
G,D	G,D	G,D
G,D	G,D	G,D
G,D	G,D	N/A
I,D	I,D	N/A
G,D	G,D	G,D
G,D	G,D	N/A
G,D	G,D	N/A
G,D	N/A	N/A
G,D	G,D	G,D

* May be a transformation product
** Includes vapors

Receptor Key
D = Dermal Contact
I = Inhalation
G = Ingestion
N/A = Not Applicable

Pathway Key
AI = Air
SO = Soil
SW = Surface Water (including sediments)
GW = Ground Water

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APPENDIX C

DETECTION LIMIT DETERMINATION LOG

DETECTION LIMIT DETERMINATION LOG
OMAHA LEAD SITE - OMAHA, NEBRASKA

Date: _____ **XRF Manufacturer:** _____

Technician: _____ **Model Number:** _____

Company: _____ **Serial Number:** _____

Precision-Based Method Detection Limit & Method Quantitation Limit

Standard Concentration:	
Readings:	
Standard Deviation:	
Precision-Based Method Detection Limit:	
Method Quantitation Limit:	

Note: The $^{+}$/- error listed must be less than 20% of the reading.

APPENDIX D

DAILY CALIBRATION LOG

DAILY CALIBRATION LOG
OMAHA LEAD SITE - OMAHA, NEBRASKA

Date: _____ **XRF Manufacturer:** _____

Technician: _____ **Model Number:** _____

Company: _____ **Serial Number:** _____

Energy Calibration Check

Time	Reading	Pass/Fail

Notes: - Checks need to be made at the following times: Beginning of work day, end of work day, any time the unit's power is turned off (battery change, etc.), or if the technician feels that drift is occurring.

- Pass/Fail - Readings after the beginning of the work day must be within 20% of the intial daily value.

Instrument Blank Check

Time	Reading	Pass/Fail

Notes: - Readings will be taken at 30 nominal seconds and blank checks will be done every 25 readings.

- Readings must less than the method quantitation limit.

Precision Measurement Check

Readings:							
Std Conc.:							
Std Deviation:							
Mean Conc.:							
RSD:							
Pass/Fail:							

Notes: - Precision calibration must be done prior to beginning work each day.

- The RSD must be less than 20% to pass.
- The $^{+}$/- error listed must be less than 20% of the reading.

Field Calibration Check

Initial		Mid-Day		Final		Additional (if necessary)	
Std. Conc.:		**Std. Conc.:**		**Std. Conc.:**		**Std. Conc.:**	
Readings:		**Readings:**		**Readings:**		**Readings:**	
Average:		**Average:**		**Average:**		**Average:**	
%:		**%:**		**%:**		**%:**	
Pass/Fail		**Pass/Fail**		**Pass/Fail**		**Pass/Fail**	

Notes: - Readings must be within 20% of the standard concentration.

- The $^{+}$/- error listed must be less than 20% of the reading.

APPENDIX E

REGION 6 HUMAN HEALTH MEDIUM-SPECIFIC SCREENING LEVELS

Key : i=IRIS p= PPRTV n=NCEA h=HEAST o=Other DOCUMENTS r=ROUTE EXTRAPOLATION

Key : C=CANCER N=NONCANCER sat=SOIL SATURATION max=CEILING LIMIT

Region 6 Human Health Medium-Specific Screening Levels 2008

TOXICITY INFORMATION

EENING LEVELS

Contaminants	SFo 1/(mg/kg-d)	K E Y	Mutagenic	CANCER CLASS	RfDo (mg/kg-d)	K E Y	SFi 1/(mg/kg-d)	K E Y	RfDi (mg/kg-d)	K E Y	RfC (mg/m3)	K E Y	MCL (ug/l)	CAS No.	Residential Soil (mg/kg)	K E Y	Industrial Indoor Worker w/o Dermal (mg/kg)	K E Y	Industrial-Outdoor Worker Soil (mg/kg)	K E Y	Ambient Air (ug/m^3)	K E Y	Residential Water (ug/l)	K E Y	DAF 1 (mg/kg)
Acetaldehyde							7.7E-03 i		2.6E-03 i		9.0E-03 i			75-07-0	**1.1E+01**	C	**2.3E+01**	C	**2.6E+01**	C	**8.7E-01**	C	**1.7E+00**	C	
Acetochlor					2.0E-02 i				2.0E-02 r					34256-82-1	**1.2E+03**	N	**4.1E+04**	N	**1.4E+04**	N	**7.3E+01**	N	**7.3E+02**	N	
Acetone					9.0E-01 i				9.0E-01 r					67-64-1	**1.4E+04**	N	**5.6E+04**	N	**6.0E+04**	N	**3.3E+03**	N	**5.5E+03**	N	**8.0E-01**
Acetonitrile									1.7E-02 i		6.0E-02 i			75-05-8	**1.5E+03**	N	**2.0E+03**	N	**2.3E+03**	N	**6.2E+01**	N	**1.2E+02**	N	
Acetophenone					1.0E-01 i				1.0E-01 r					98-86-2	**1.7E+03**	sat	**1.7E+03**	sat	**1.7E+03**	sat	**3.7E+02**	N	**6.1E+02**	N	
Acrolein					5.0E-04 i				5.7E-06 i		2.0E-05 i			107-02-8	**2.4E-01**	N	**3.4E-01**	N	**3.7E-01**	N	**2.1E-02**	N	**4.2E-02**	N	
Acrylamide	4.6E+00 i			B2	2.0E-04 i		4.6E+00 i		2.0E-04 r					79-06-1	**1.1E-01**	C	**1.3E+00**	C	**4.2E-01**	C	**1.5E-03**	C	**1.5E-02**	C	
Acrylic acid					5.0E-01 i				2.9E-04 i		1.0E-03 i			79-10-7	**3.0E+04**	N	**1.0E+05**	max	**1.0E+05**	max	**1.0E+00**	N	**1.8E+04**	N	
Acrylonitrile	5.4E-01 i			B1	1.0E-03 h		2.4E-01 i		5.7E-04 i		2.0E-03 i			107-13-1	**2.1E-01**	C	**5.2E-01**	C	**5.5E-01**	C	**2.8E-02**	C	**3.9E-02**	C	
Alachlor	8.1E-02 h				1.0E-02 i		8.0E-02 r		1.0E-02 r				2.0E+00	15972-60-8	**6.0E+00**	C	**7.1E+01**	C	**2.4E+01**	C	**8.4E-02**	C	**8.4E-01**	C	
Alar					1.5E-01 i				1.5E-01 r					1596-84-5	**9.2E+03**	N	**1.0E+05**	max	**1.0E+05**	max	**5.5E+02**	N	**5.5E+03**	N	
Aldicarb					1.0E-03 i				1.0E-03 r				7.0E+00	116-06-3	**6.1E+01**	N	**2.0E+03**	N	**6.8E+02**	N	**3.7E+00**	N	**3.7E+01**	N	
Aldicarb sulfone					1.0E-03 i				1.0E-03 r				7.0E+00	1646-88-4	**6.1E+01**	N	**2.0E+03**	N	**6.8E+02**	N	**3.7E+00**	N	**3.7E+01**	N	
Aldrin	1.7E+01 i			B2	3.0E-05 i		1.7E+01 i		3.0E-05 r					309-00-2	**2.9E-02**	C	**3.4E-01**	C	**1.1E-01**	C	**3.9E-04**	C	**4.0E-03**	C	**2.0E-02**
Allyl chloride					5.0E-02 h				2.9E-04 i		1.0E-03 i			107-05-1	**3.0E+03**	N	**9.7E+04**	N	**3.4E+04**	N	**1.0E+00**	N	**1.8E+03**	N	
Aluminum					1.0E+00 p				1.4E-03 p		5.0E-03 p			7429-90-5	**7.7E+04**	N	**1.0E+05**	max	**1.0E+05**	max	**5.2E+00**	N	**3.7E+04**	N	
Amdro					3.0E-04 i				3.0E-04 r					67485-29-4	**1.8E+01**	N	**6.1E+02**	N	**2.1E+02**	N	**1.1E+00**	N	**1.1E+01**	N	
4-Aminopyridine					2.0E-05 h				2.0E-05 r					504-24-5	**1.2E+00**	N	**4.1E+01**	N	**1.4E+01**	N	**7.3E-02**	N	**7.3E-01**	N	
Ammonia									2.9E-02 i		1.0E-01 i			7664-41-7	**1.0E+05**	max					**1.0E+02**	N	**2.1E+02**	N	
Aniline	5.7E-03 i			B2	7.0E-03 p		5.7E-03 r		2.9E-04 i		1.0E-03 i			62-53-3	**8.5E+01**	C	**1.0E+03**	C	**3.4E+02**	C	**1.0E+00**	N	**1.2E+01**	C	
Antimony and compounds					4.0E-04 i								6.0E+00	7440-36-0	**3.1E+01**	N	**8.2E+02**	N	**4.5E+02**	N			**1.5E+01**	N	**3.0E-01**
Antimony pentoxide					5.0E-04 h									1314-60-9	**3.9E+01**	N	**1.0E+03**	N	**5.7E+02**	N			**1.8E+01**	N	
Antimony tetroxide					4.0E-04 h									1332-81-6	**3.1E+01**	N	**8.2E+02**	N	**4.5E+02**	N			**1.5E+01**	N	
Antimony trioxide					4.0E-04 h				5.7E-05 i		2.0E-04 i			1309-64-4	**3.1E+01**	N	**8.2E+02**	N	**4.5E+02**	N	**2.1E-01**	N	**1.5E+01**	N	
Arsenic (noncancer endpoint)					3.0E-04 i								1.0E+01	7440-38-2	**2.2E+01**	N	**6.1E+02**	N	**2.8E+02**	N					
Arsenic (cancer endpoint)	1.5E+00 i			A	3.0E-04 i		1.5E+01 i						1.0E+01	7440-38-2	**3.9E-01**	C	**3.8E+00**	C	**1.8E+00**	C	**4.5E-04**	C	**4.5E-02**	C	**1.0E+00**
Arsine									1.4E-05 i		5.0E-05 i			7784-42-1							**5.2E-02**	N	**1.0E-01**	N	
Assure					9.0E-03 i				9.0E-03 r					76578-14-8	**5.5E+02**	N	**1.8E+04**	N	**6.2E+03**	N	**3.3E+01**	N	**3.3E+02**	N	
Atrazine	2.2E-01 h				3.5E-02 h		2.2E-01 r		3.5E-02 h				3.0E+00	1912-24-9	**2.2E+00**	C	**2.6E+01**	C	**8.6E+00**	C	**3.1E-02**	C	**3.0E-01**	C	
Azobenzene	1.1E-01 i			B2			1.1E-01 i							103-33-3	**4.4E+00**	C	**5.2E+01**	C	**1.7E+01**	C	**6.2E-02**	C	**6.1E-01**	C	
Barium and compounds					2.0E-01 i				2.0E-01 r				2.0E+03	7440-39-3	**1.6E+04**	N	**1.0E+05**	max	**1.0E+05**	max	**7.3E+02**	N	**7.3E+03**	N	**8.2E+01**
Baygon					4.0E-03 i				4.0E-03 r					114-26-1	**2.4E+02**	N	**8.2E+03**	N	**2.7E+03**	N	**1.5E+01**	N	**1.5E+02**	N	
Baythroid					2.5E-02 i				2.5E-02 r					68359-37-5	**1.5E+03**	N	**5.1E+04**	N	**1.7E+04**	N	**9.1E+01**	N	**9.1E+02**	N	
Bentazon					3.0E-02 i				3.0E-02 r					25057-89-0	**1.8E+03**	N	**6.1E+04**	N	**2.1E+04**	N	**1.1E+02**	N	**1.1E+03**	N	
Benzaldehyde					1.0E-01 i				1.0E-01 r					100-52-7	**6.1E+03**	N	**1.0E+05**	max	**6.8E+04**	N	**3.7E+02**	N	**3.7E+03**	N	
Benzene	5.5E-02 i			A	4.0E-03 i		2.7E-02 i		8.6E-03 i		3.0E-02 i		5.0E+00	71-43-2	**6.6E-01**	C	**1.5E+00**	C	**1.6E+00**	C	**2.5E-01**	C	**3.5E-01**	C	**2.0E-03**
Benzidine	2.3E+02 i	y		A	3.0E-03 i		2.3E+02 i		3.0E-03 r					92-87-5	**5.0E-04**	C	**2.5E-02**	C	**8.3E-03**	C	**9.4E-06**	C	**9.4E-05**	C	
Benzoic acid					4.0E+00 i				4.0E+00 i					65-85-0	**1.0E+05**	max	**1.0E+05**	max	**1.0E+05**	max	**1.5E+04**	N	**1.5E+05**	N	**2.0E+01**
Benzyl alcohol					3.0E-01 h				3.0E-01 r					100-51-6	**1.8E+04**	N	**1.0E+05**	max	**1.0E+05**	max	**1.1E+03**	N	**1.1E+04**	N	
Benzyl chloride	1.7E-01 i			B2			1.7E-01 r							100-44-7	**8.9E-01**	C	**2.3E+00**	C	**2.4E+00**	C	**4.0E-02**	C	**6.6E-02**	C	
Beryllium and compounds				B1	2.0E-03 i		8.4E+00 i		5.7E-06 i		2.0E-05 i		4.0E+00	7440-41-7	**1.6E+02**	N	**2.2E+03**	C	**2.2E+03**	N	**8.0E-04**	C	**7.3E+01**	N	**3.0E+00**
1,1-Biphenyl					5.0E-02 i				5.0E-02 r					92-52-4	**3.0E+03**	N	**3.0E+04**	N	**2.6E+04**	N	**1.8E+02**	N	**3.0E+02**	N	
Bis(2-chloroethyl)ether	1.1E+00 i			B2			1.2E+00 i							111-44-4	**2.1E-01**	C	**6.2E-01**	C	**6.2E-01**	C	**5.8E-03**	C	**9.8E-03**	C	**2.0E-05**
Bis(2-chloro-1-methylethyl) ether	7.0E-02 h				4.0E-02 i		3.5E-02 h		4.0E-02 r					108-60-1	**2.9E+00**	C	**8.1E+00**	C	**8.2E+00**	C	**1.9E-01**	C	**2.7E-01**	C	
Bis(chloromethyl)ether	2.2E+02 i			A			2.2E+02 i							542-88-1	**1.9E-04**	C	**4.4E-04**	C	**4.8E-04**	C	**3.1E-05**	C	**5.2E-05**	C	
Bis(2-ethylhexyl)phthalate (DEHP)	1.4E-02 i			B2	2.0E-02 i		1.4E-02 r		2.0E-02 r				6.0E+00	117-81-7	**3.5E+01**	C	**4.1E+02**	C	**1.4E+02**	C	**4.8E-01**	C	**4.8E+00**	C	**1.8E+02**
Boron					2.0E-01 i				5.7E-03 h					7440-42-8	**1.6E+04**	N	**1.0E+05**	max	**1.0E+05**	max	**2.1E+01**	N	**7.3E+03**	N	
Boron trifluoride									2.0E-04 h					7637-07-2	**1.0E+05**	max	**1.0E+05**	max	**1.0E+05**	max	**7.3E-01**	N			
Bromobenzene					2.0E-02 p				3.3E-03 p		1.2E-02 p			108-86-1	**7.3E+01**	N	**1.1E+02**	N	**1.2E+02**	N	**1.2E+01**	N	**2.3E+01**	N	
Bromodichloromethane	6.2E-02 i			B2	2.0E-02 i		6.2E-02 r		2.0E-02 r					75-27-4	**1.0E+00**	C	**2.4E+00**	C	**2.6E+00**	C	**1.1E-01**	C	**1.8E-01**	C	**3.0E-02**
Bromoform (tribromomethane)	7.9E-03 i			B2	2.0E-02 i		3.9E-03 i		2.0E-02 r					75-25-2	**6.2E+01**	C	**7.2E+02**	C	**2.4E+02**	C	**1.7E+00**	C	**8.5E+00**	C	**4.0E-02**
Bromomethane					1.4E-03 i				1.4E-03 i		5.0E-03 i			74-83-9	**8.7E+00**	N	**1.3E+01**	N	**1.5E+01**	N	**5.2E+00**	N	**8.7E+00**	N	**1.0E-02**
Bromophos					5.0E-03 h				5.0E-03 r					2104-96-3	**3.1E+02**	N	**1.0E+04**	N	**3.4E+03**	N	**1.8E+01**	N	**1.8E+02**	N	
Bromoxynil					2.0E-02 i				2.0E-02 r					1689-84-5	**1.2E+03**	N	**4.1E+04**	N	**1.4E+04**	N	**7.3E+01**	N	**7.3E+02**	N	
1,3-Butadiene				B2			1.1E-01 i		5.7E-04 i		2.0E-03 i			106-99-0	**6.2E-02**	C	**1.3E-01**	C	**1.5E-01**	C	**6.4E-02**	C	**1.3E-01**	C	
1-Butanol					1.0E-01 i				1.0E-01 r					71-36-3	**6.1E+03**	N	**1.0E+05**	max	**6.8E+04**	N	**3.7E+02**	N	**3.7E+03**	N	**9.0E-01**
Butylate					5.0E-02 i				5.0E-02 r					2008-41-5	**3.1E+03**	N	**1.0E+05**	max	**3.4E+04**	N	**1.8E+02**	N	**1.8E+03**	N	
n-Butylbenzene					1.0E-02 n				1.0E-02 r					104-51-8	**1.4E+02**	N	**2.4E+02**	sat	**2.4E+02**	sat	**3.7E+01**	N	**6.1E+01**	N	
sec-Butylbenzene					1.0E-02 n				1.0E-02 r					135-98-8	**1.1E+02**	N	**2.2E+02**	sat	**2.2E+02**	sat	**3.7E+01**	N	**6.1E+01**	N	
tert-Butylbenzene					1.0E-02 n				1.0E-02 r					98-06-6	**1.3E+02**	N	**3.9E+02**	sat	**3.9E+02**	sat	**3.7E+01**	N	**6.1E+01**	N	
Butyl benzyl phthalate					2.0E-01 i				2.0E-01 r					85-68-7	**2.4E+02**	sat	**2.4E+02**	sat	**2.4E+02**	sat	**7.3E+02**	N	**7.3E+03**	N	**8.1E+02**
Cadmium and compounds					5.0E-04 i		6.3E+00 i		x				5.0E+00	7440-43-9	**3.9E+01**	N	**1.0E+03**	N	**5.6E+02**	N	**1.1E-03**	C	**1.8E+01**	N	**4.0E-01**
Caprolactam					5.0E-01 i				5.0E-01 r					105-60-2	**3.1E+04**	N	**1.0E+05**	max	**1.0E+05**	max	**1.8E+03**	N	**1.8E+04**	N	
Captan	3.5E-03 h				1.3E-01 i		3.5E-03 r		1.3E-01 r					133-06-2	**1.4E+02**	C	**1.6E+03**	C	**5.5E+02**	C	**1.9E+00**	C	**1.9E+01**	C	
Carbaryl					1.0E-01 i				1.0E-01 r					63-25-2	**6.1E+03**	N	**1.0E+05**	max	**6.8E+04**	N	**3.7E+02**	N	**3.7E+03**	N	
Carbazole	2.0E-02 h						2.0E-02 r							86-74-8	**2.4E+01**	C	**2.9E+02**	C	**9.6E+01**	C	**3.4E-01**	C	**3.4E+00**	C	**3.0E-02**
Carbofuran					5.0E-03 i				5.0E-03 r				4.0E+01	1563-66-2	**3.1E+02**	N	**1.0E+04**	N	**3.4E+03**	N	**1.8E+01**	N	**1.8E+02**	N	
Carbon disulfide					1.0E-01 i				2.0E-01 i		7.0E-01 i			75-15-0	**7.2E+02**	sat	**7.2E+02**	sat	**7.2E+02**	sat	**7.3E+02**	N	**1.0E+03**	N	**2.0E+00**
Carbon tetrachloride	1.3E-01 i			B2	7.0E-04 i		5.3E-02 i		x				5.0E+00	56-23-5	**2.4E-01**	C	**5.3E-01**	C	**5.8E-01**	C	**1.3E-01**	C	**1.7E-01**	C	**3.0E-03**
Carbosulfan					1.0E-02 i				1.0E-02 r					55285-14-8	**6.1E+02**	N	**2.0E+04**	N	**6.8E+03**	N	**3.7E+01**	N	**3.7E+02**	N	
Chloral					1.0E-01 i									302-17-0	**6.1E+03**	N	**1.0E+05**	max	**6.8E+04**	N			**3.7E+03**	N	

Key : i=IRIS p= PPRTV n=NCEA h=HEAST o=Other DOCUMENTS r=ROUTE EXTRAPOLATION

Key : C=CANCER N=NONCANCER sat=SOIL SATURATION max=CEILING LIMIT

Region 6 Human Health Medium-Specific Screening Levels 2008

Contaminants	TOXICITY INFORMATION														EENING LEVELS											
	SFo 1/(mg/kg-d)	K E Y	Mutagenic	CANCER CLASS	RfDo (mg/kg-d)	K E Y	SFi 1/(mg/kg-d)	K E Y	RfDi (mg/kg-d)	K E Y	RfC (mg/m3)	K E Y	MCL (ug/l)	CAS No.	Residential Soil (mg/kg)	K E Y	Industrial Indoor Worker w/o Dermal (mg/kg)	K E Y	Industrial-Outdoor Worker Soil (mg/kg)	K E Y	Ambient Air (ug/m^3)	K E Y	Residential Water (ug/l)	K E Y	DAF 1 (mg/kg)
Chloranil	4.0E-01 h						4.0E-01 r							118-75-2	**1.2E+00**	C	**1.4E+01**	C	**4.8E+00**	C	**1.7E-02**	C	**1.7E-01**	C	
Chlordane	3.5E-01 i			B2	5.0E-04 i		3.5E-01 i		2.0E-04 i		7.0E-04 i		2.0E+00	57-74-9	**1.6E+00**	C	**1.6E+01**	C	**7.2E+00**	C	**1.9E-02**	C	**1.9E-01**	C	**5.0E-01**
Chlorine					1.0E-01 i									7782-50-5	**7.8E+03**	N	**1.0E+05**	max	**1.0E+05**	max			**3.7E+03**	N	
Chlorine dioxide									5.7E-05 i		2.0E-04 i			10049-04-4							**2.1E-01**	N	**4.2E-01**	N	
Chloroacetic acid					2.0E-03 h				2.0E-03 r					79-11-8	**1.2E+02**	N	**4.1E+03**	N	**1.4E+03**	N	**7.3E+00**	N	**7.3E+01**	N	
4-Chloroaniline					4.0E-03 i				4.0E-03 r					106-47-8	**2.4E+02**	N	**8.2E+03**	N	**2.7E+03**	N	**1.5E+01**	N	**1.5E+02**	N	**3.0E-02**
Chlorobenzene					2.0E-02 i				1.4E-02 p		5.0E-02 p		1.0E+02	108-90-7	**2.7E+02**	N	**4.6E+02**	N	**5.0E+02**	N	**5.2E+01**	N	**9.1E+01**	N	**7.0E-02**
Chlorobenzilate	2.7E-01 h				2.0E-02 i		2.7E-01 h		2.0E-02 r					510-15-6	**1.8E+00**	C	**2.1E+01**	C	**7.1E+00**	C	**2.5E-02**	C	**2.5E-01**	C	
p-Chlorobenzoic acid					2.0E-01 h				2.0E-01 r					74-11-3	**1.2E+04**	N	**1.0E+05**	max	**1.0E+05**	max	**7.3E+02**	N	**7.3E+03**	N	
4-Chlorobenzotrifluoride					3.0E-03 p				8.6E-02		3.0E-01 p			98-56-6	**1.8E+02**	N	**6.1E+03**	N	**2.1E+03**	N	**3.1E+02**	N	**1.1E+02**	N	
2-Chloro-1,3-butadiene					2.0E-02 h				2.0E-03 h					126-99-8	**3.6E+00**	N	**1.2E+01**	N	**1.3E+01**	N	**7.3E+00**	N	**1.4E+01**	N	
1-Chlorobutane					4.0E-02 p				4.0E-02 r					109-69-3	**7.1E+01**	N	**2.4E+02**	N	**2.6E+02**	N	**1.5E+02**	N	**2.4E+02**	N	
1-Chloro-1,1-difluoroethane					1.4E+01 r				1.4E+01 i		5.0E+01 i			75-68-3	**3.4E+02**	sat	**3.4E+02**	sat	**3.4E+02**	sat	**5.2E+04**	N	**8.7E+04**	N	
Chlorodifluoromethane					1.4E+01 r				1.4E+01 i		5.0E+01 i			75-45-6	**3.4E+02**	sat	**3.4E+02**	sat	**3.4E+02**	sat	**5.1E+04**	N	**8.5E+04**	N	
Chloroform				B2	1.0E-02 i		8.1E-02 i		1.3E-02 p		4.5E-02 p			67-66-3	**2.5E-01**	C	**5.2E-01**	C	**5.8E-01**	C	**8.4E-02**	C	**1.7E-01**	C	**3.0E-02**
Chloromethane				D					2.6E-02 i		9.0E-02 i			74-87-3	**1.1E+02**	N	**1.6E+02**	N	**1.7E+02**	N	**9.4E+01**	N	**1.9E+02**	N	
4-Chloro-2-methylaniline	5.8E-01 h						5.8E-01 r							95-69-2	**8.4E-01**	C	**9.9E+00**	C	**3.3E+00**	C	**1.2E-02**	C	**1.2E-01**	C	
beta-Chloronaphthalene					8.0E-02 i				8.0E-02 r					91-58-7	**3.9E+03**	N	**2.7E+04**	N	**2.6E+04**	N	**2.9E+02**	N	**4.9E+02**	N	
o-Chloronitrobenzene	9.7E-03 p				1.0E-03 p		9.7E-03 r		2.0E-05 p		7.0E-05 p			88-73-3	**9.0E+00**	N	**1.4E+01**	N	**1.6E+01**	N	**7.3E-02**	N	**1.5E-01**	N	
p-Chloronitrobenzene	6.7E-03 p				1.0E-03 p		6.7E-03 r		1.7E-04 p		6.0E-03 p			100-00-5	**5.4E+01**	C	**1.0E+02**	N	**1.1E+02**	N	**6.2E-01**	N	**1.2E+00**	N	
2-Chlorophenol					5.0E-03 i				5.0E-03 r					95-57-8	**6.4E+01**	N	**2.4E+02**	N	**2.6E+02**	N	**1.8E+01**	N	**3.0E+01**	N	**2.0E-01**
2-Chloropropane														75-29-6	**1.1E+03**	sat	**1.1E+03**	sat	**1.1E+03**	sat					
o-Chlorotoluene					2.0E-02 i				2.0E-02 r					95-49-8	**1.6E+02**	N	**5.1E+02**	sat	**5.1E+02**	sat	**7.3E+01**	N	**1.2E+02**	N	
Chlorpyrifos					3.0E-03 i				3.0E-03 r					2921-88-2	**1.8E+02**	N	**6.1E+03**	N	**2.1E+03**	N	**1.1E+01**	N	**1.1E+02**	N	
Chlorpyrifos-methyl					1.0E-02 h				1.0E-02 r					5598-13-0	**6.1E+02**	N	**2.0E+04**	N	**6.8E+03**	N	**3.7E+01**	N	**3.7E+02**	N	
Chromium III					1.5E+00 i								1.0E+02	16065-83-1	**1.0E+05**	max	**1.0E+05**	max	**1.0E+05**	max			**5.5E+04**	N	
Total Chromium (1/6 ratio Cr VI/Cr III)							4.2E+01 i						1.0E+02	7440-47-3	**2.1E+02**	C	**4.5E+02**	C	**5.0E+02**	C	**1.6E-04**	C			**2.0E+00**
Chromium VI				A	3.0E-03 i		2.9E+02 i		2.9E-05 i				1.0E+02	18540-29-9	**3.0E+01**	C	**6.4E+01**	C	**7.1E+01**	C	**2.3E-05**	C	**1.1E+02**	N	**2.0E+00**
Cobalt					2.0E-02 p		9.8E+00 p		5.7E-06 p					7440-48-4	**9.0E+02**	C	**1.9E+03**	C	**2.1E+03**	C	**6.9E-04**	C	**7.3E+02**	N	
Coke Oven Emissions		y		A			2.2E+00 i							8007-45-2	**1.3E+03**	C	**8.7E+03**	C	**9.6E+03**	C	**9.9E-04**	C			
Copper and compounds					3.7E-02 h								1.3E+03	7440-50-8	**2.9E+03**	N	**7.6E+04**	N	**4.2E+04**	N			**1.4E+03**	N	
Crotonaldehyde	1.9E+00 h						x							123-73-9	**3.4E-01**	C	**3.0E+00**	C	**1.7E+00**	C			**3.5E-02**	C	
Cumene (isopropylbenzene)					1.0E-01 i				1.1E-01 i		4.0E-01 i			98-82-8	**3.7E+02**	N	**5.2E+02**	N	**5.8E+02**	N	**4.0E+02**	N	**6.6E+02**	N	
Cyanazine	8.4E-01 h				2.0E-03 h		8.4E-01 r		2.0E-03 r					21725-46-2	**5.8E-01**	C	**6.8E+00**	C	**2.3E+00**	C	**8.0E-03**	C	**8.0E-02**	C	
Cyanides														n/a											
Barium cyanid [e]					1.0E-01 h									542-62-1	**6.1E+03**	N	**1.0E+05**	max	**6.8E+04**	N			**3.7E+03**	N	
Calcium cyanide					4.0E-02 i									592-01-8	**2.4E+03**	N	**8.2E+04**	N	**2.7E+04**	N			**1.5E+03**	N	
Copper cyanid [e]					5.0E-03 i									544-92-3	**3.1E+02**	N	**1.0E+04**	N	**3.4E+03**	N			**1.8E+02**	N	
Cyanogen					4.0E-02 i									460-19-5	**3.1E+03**	N	**8.2E+04**	N	**4.5E+04**	N			**1.5E+03**	N	
Cyanogen bromide					9.0E-02 i									506-68-3	**7.0E+03**	N	**1.0E+05**	max	**1.0E+05**	max			**3.3E+03**	N	
Cyanogen chlorid [e]					5.0E-02 i									506-77-4	**3.9E+03**	N	**1.0E+05**	max	**5.7E+04**	N			**1.8E+03**	N	
Free cyanid [e]					2.0E-02 i								2.0E+02	57-12-5	**1.2E+03**	N	**4.1E+04**	N	**1.4E+04**	N			**7.3E+02**	N	**2.0E+00**
Hydrogen cyanid [e]					2.0E-02 i				8.6E-04 i		3.0E-03			74-90-8	**2.5E+01**	N	**3.5E+01**	N	**3.9E+01**	N	**3.1E+00**	N	**6.2E+00**	N	
Potassium cyanid [e]					5.0E-02 i									151-50-8	**3.1E+03**	N	**1.0E+05**	max	**3.4E+04**	N			**1.8E+03**	N	
Potassium silver cyanid [e]					2.0E-01 i									506-61-6	**1.2E+04**	N	**1.0E+05**	max	**1.0E+05**	max			**7.3E+03**	N	
Silver cyanide					1.0E-01 i									506-64-9	**6.1E+03**	N	**1.0E+05**	max	**6.8E+04**	N			**3.7E+03**	N	
Sodium cyanid [e]					4.0E-02 i									143-33-9	**2.4E+03**	N	**8.2E+04**	N	**2.7E+04**	N			**1.5E+03**	N	
Zinc cyanid [e]					5.0E-02 i									557-21-1	**3.1E+03**	N	**1.0E+05**	max	**3.4E+04**	N			**1.8E+03**	N	
Cyclohexane									1.7E+00 i		6.0E+00			110-82-7	**1.4E+02**	sat	**1.4E+02**	sat	**1.4E+02**	sat	**6.3E+03**	N	**1.3E+04**	N	
Cyclohexanone					5.0E+00 i				5.0E+00 r					108-94-1	**1.0E+05**	max	**1.0E+05**	max	**1.0E+05**	max	**1.8E+04**	N	**1.8E+05**	N	
Cyhalothrin/Karate					5.0E-03 i				5.0E-03 r					68085-85-8	**3.1E+02**	N	**1.0E+04**	N	**3.4E+03**	N	**1.8E+01**	N	**1.8E+02**	N	
Cypermethrin					1.0E-02 i				1.0E-02 r					52315-07-8	**6.1E+02**	N	**2.0E+04**	N	**6.8E+03**	N	**3.7E+01**	N	**3.7E+02**	N	
Dacthal					1.0E-02 i				1.0E-02 r					1861-32-1	**6.1E+02**	N	**2.0E+04**	N	**6.8E+03**	N	**3.7E+01**	N	**3.7E+02**	N	
Dalapon					3.0E-02 i				3.0E-02 r				2.0E+02	75-99-0	**1.8E+03**	N	**6.1E+04**	N	**2.1E+04**	N	**1.1E+02**	N	**1.1E+03**	N	
DDD	2.4E-01 i			B2			2.4E-01 r							72-54-8	**2.4E+00**	C	**2.4E+01**	C	**1.1E+01**	C	**2.8E-02**	C	**2.8E-01**	C	**8.0E-01**
DDE	3.4E-01 i			B2			3.4E-01 r							72-55-9	**1.7E+00**	C	**1.7E+01**	C	**7.8E+00**	C	**2.0E-02**	C	**2.0E-01**	C	**3.0E+00**
DDT	3.4E-01 i			B2	5.0E-04 i		3.4E-01 i		5.0E-04 r					50-29-3	**1.7E+00**	C	**1.7E+01**	C	**7.8E+00**	C	**2.0E-02**	C	**2.0E-01**	C	**2.0E+00**
Diazinon					9.0E-04 h				9.0E-04 r					333-41-5	**5.5E+01**	N	**1.8E+03**	N	**6.2E+02**	N	**3.3E+00**	N	**3.3E+01**	N	
Dibenzofuran					2.0E-03 n				2.0E-03 r					132-64-9	**1.5E+02**	N	**2.5E+03**	N	**1.7E+03**	N	**7.3E+00**	N	**1.2E+01**	N	
1,4-Dibromobenzene					1.0E-02 i				1.0E-02 r					106-37-6	**6.1E+02**	N	**2.0E+04**	N	**6.8E+03**	N	**3.7E+01**	N	**3.7E+02**	N	
Dibromochloromethane	8.4E-02 i			C	2.0E-02 i		8.4E-02 r		2.0E-02 r					124-48-1	**1.0E+00**	C	**2.4E+00**	C	**2.6E+00**	C	**8.0E-02**	C	**1.3E-01**	C	**2.0E-02**
1,2-Dibromo-3-chloropropane	8.0E-01 p	y			2.0E-04 p		2.1E+01 p		5.7E-05 i		2.0E-04 i			96-12-8	**2.6E-03**	C	**1.8E-02**	C	**2.0E-02**	C	**1.0E-04**	C	**2.0E-04**	C	
1,2-Dibromoethane	2.0E+00 i			likely	9.0E-03 i		2.0E+00 i		2.6E-03 i					106-93-4	**2.8E-02**	C	**6.5E-02**	C	**7.0E-02**	C	**3.4E-03**	C	**5.6E-03**	C	
Dibutyl phthalate					1.0E-01 i				1.0E-01 r					84-74-2	**6.1E+03**	N	**1.0E+05**	max	**6.8E+04**	N	**3.7E+02**	N	**3.7E+03**	N	**2.7E+02**
Dicamba					3.0E-02 i				3.0E-02 r					1918-00-9	**1.8E+03**	N	**6.1E+04**	N	**2.1E+04**	N	**1.1E+02**	N	**1.1E+03**	N	
1,2-Dichlorobenzene					9.0E-02 i				6.9E-03 n		2.4E-02 n		6.0E+02	95-50-1	**2.8E+02**	N	**3.7E+02**	sat	**3.7E+02**	sat	**2.5E+01**	N	**4.9E+01**	N	**9.0E-01**
1,3-Dichlorobenzene					3.0E-03 n				2.3E-03 n		8.0E-03 n			541-73-1	**6.9E+01**	N	**1.3E+02**	N	**1.4E+02**	N	**8.3E+00**	N	**1.4E+01**	N	
1,4-Dichlorobenzene	2.4E-02 h				3.0E-02 n		2.4E-02 r		2.3E-01 i		8.0E-01 i		7.5E+01	106-46-7	**3.2E+00**	C	**7.5E+00**	C	**8.1E+00**	C	**2.8E-01**	C	**4.7E-01**	C	**1.0E-01**
3,3-Dichlorobenzidine	4.5E-01 i			B2			4.5E-01 r							91-94-1	**1.1E+00**	C	**1.3E+01**	C	**4.3E+00**	C	**1.5E-02**	C	**1.5E-01**	C	**3.0E-04**
1,4-Dichloro-2-butene	9.3E+00 r						9.3E+00 h							764-41-0	**7.9E-03**	C	**1.8E-02**	C	**2.0E-02**	C	**7.2E-04**	C	**1.2E-03**	C	
Dichlorodifluoromethane					2.0E-01 i				5.7E-02 h					75-71-8	**9.4E+01**	N	**3.1E+02**	N	**3.4E+02**	sat	**2.1E+02**	N	**3.9E+02**	N	
1,1-Dichloroethane					2.0E-01 p				2.0E-01 r					75-34-3	**8.5E+02**	N	**2.3E+03**	sat	**2.3E+03**	sat	**7.3E+02**	N	**1.2E+03**	N	**1.0E+00**

Region 6 Human Health Medium-Specific Screening Levels 2008

Contaminants	TOXICITY INFORMATION														EENING LEVELS													
	SFo 1/(mg/kg-d)	K E Y	Mutagenic	CANCER CLASS	RfDo (mg/kg-d)	K E Y	SFi 1/(mg/kg-d)	K E Y	RfDi (mg/kg-d)	K E Y	RfC (mg/m3)	K E Y	MCL (ug/l)	CAS No.	Residential Soil (mg/kg)	K E Y	Industrial Indoor Worker w/o Dermal (mg/kg)	K E Y	Industrial-Outdoor Worker Soil (mg/kg)	K E Y	Ambient Air (ug/m^3)	K E Y	Residential Water (ug/l)	K E Y	DAF 1 (mg/kg)
1,2-Dichloroethane (EDC)	9.1E-02 i		B2	2.0E-02 n		9.1E-02 i		1.4E-03 n				5.0E+00	107-06-2	**3.5E-01**	C	**7.7E-01**	C	**8.4E-01**	C	**7.4E-02**	C	**1.2E-01**	C	**1.0E-03**
1,1-Dichloroethylene				5.0E-02 i				5.7E-02 i		2.0E-01 i		7.0E+00	75-35-4	**2.8E+02**	N	**4.3E+02**	N	**4.7E+02**	N	**2.1E+02**	N	**3.4E+02**	N	**3.0E-03**
1,2-Dichloroethylene (cis)				1.0E-02 p				1.0E-02 r				7.0E+01	156-59-2	**4.3E+01**	N	**1.5E+02**	N	**1.6E+02**	N	**3.7E+01**	N	**6.1E+01**	N	**2.0E-02**
1,2-Dichloroethylene (trans)				2.0E-02 i				1.7E-02 p		6.0E-02 p		1.0E+02	156-60-5	**1.2E+02**	N	**1.8E+02**	N	**2.0E+02**	N	**6.3E+01**	N	**1.1E+02**	N	**3.0E-02**
2,4-Dichlorophenol				3.0E-03 i				3.0E-03 r					120-83-2	**1.8E+02**	N	**6.1E+03**	N	**2.1E+03**	N	**1.1E+01**	N	**1.1E+02**	N	**5.0E-02**
4-(2,4-Dichlorophenoxy)butyric Acid (2,4-DB)				8.0E-03 i				8.0E-03 r					94-82-6	**4.9E+02**	N	**1.6E+04**	N	**5.5E+03**	N	**2.9E+01**	N	**2.9E+02**	N	
2,4-Dichlorophenoxyacetic Acid (2,4-D)				1.0E-02 i				1.0E-02 r				7.0E+01	94-75-7	**6.9E+02**	N	**2.0E+04**	N	**8.5E+03**	N	**3.7E+01**	N	**3.7E+02**	N	
1,2-Dichloropropane	6.8E-02 h			1.1E-03 r		6.8E-02 r		1.1E-03 i		4.0E-03 i		5.0E+00	78-87-5	**3.5E-01**	C	**7.7E-01**	C	**8.5E-01**	C	**9.9E-02**	C	**1.6E-01**	C	**1.0E-03**
1,3-Dichloropropane				2.0E-02 p				2.0E-02 r					142-28-9	**1.1E+02**	N	**3.7E+02**	N	**4.1E+02**	N	**7.3E+01**	N	**1.2E+02**	N	**1.0E-03**
1,3-Dichloropropene	1.0E-01 i		B2	3.0E-02 i		1.4E-02 i		5.7E-03 i		2.0E-02 i			542-75-6	**7.0E-01**	C	**1.6E+00**	C	**1.7E+00**	C	**4.8E-01**	C	**4.0E-01**	C	**2.0E-04**
2,3-Dichloropropanol				3.0E-03 i				3.0E-03 r					616-23-9	**1.8E+02**	N	**6.1E+03**	N	**2.1E+03**	N	**1.1E+01**	N	**1.1E+02**	N	
Dichlorvos	2.9E-01 i		B2	5.0E-04 i		2.9E-01 r		1.4E-04 i		5.0E-04 i			62-73-7	**1.7E+00**	C	**2.0E+01**	C	**6.6E+00**	C	**2.3E-02**	C	**2.3E-01**	C	
Dicofol		x				r							115-32-2											
Dicyclopentadiene				8.0E-03 p				2.0E-03 p		7.0E-03 p			77-73-6	**4.2E+01**	N	**6.2E+01**	N	**6.9E+01**	N	**7.3E+00**	N	**1.4E+01**	N	
Dieldrin	1.6E+01 i		B2	5.0E-05 i		1.6E+01 i		5.0E-05 r					60-57-1	**3.0E-02**	C	**3.6E-01**	C	**1.2E-01**	C	**4.2E-04**	C	**4.2E-03**	C	**2.0E-04**
Diethylene glycol, monobutyl ether				1.0E-02 p				5.7E-03 p					112-34-5	**6.1E+02**	N	**2.0E+04**	N	**6.8E+03**	N	**2.1E+01**	N	**3.7E+02**	N	
Diethylene glycol, monoethyl ether				6.0E-02 p				8.6E-04 p					111-90-0	**3.7E+03**	N	**1.0E+05**	max	**4.1E+04**	N	**3.1E+00**	N	**2.2E+03**	N	
Di(2-ethylhexyl)adipate	1.2E-03 i		C	6.0E-01 i		1.2E-03 r		6.0E-01 r				4.0E+02	103-23-1	**4.1E+02**	C	**4.8E+03**	C	**1.6E+03**	C	**5.6E+00**	C	**5.6E+01**	C	
Diethyl phthalate				8.0E-01 i				8.0E-01 r					84-66-2	**4.9E+04**	N	**1.0E+05**	max	**1.0E+05**	max	**2.9E+03**	N	**2.9E+04**	N	
Diethylstilbestrol	4.7E+03 h					4.7E+03 r							56-53-1	**1.0E-04**	C	**1.2E-03**	C	**4.1E-04**	C	**1.4E-06**	C	**1.4E-05**	C	
Difenzoquat (Avenge)				8.0E-02 i				8.0E-02 r					43222-48-6	**4.9E+03**	N	**1.0E+05**	max	**5.5E+04**	N	**2.9E+02**	N	**2.9E+03**	N	
1,1-Difluoroethane				1.1E+01 r				1.1E+01 i		4.0E+01 i			75-37-6	**1.0E+05**	max	**1.0E+05**	max	**1.0E+05**	max	**4.2E+04**	N	**6.9E+04**	N	
Diisopropyl methylphosphonate				8.0E-02 i				8.0E-02 r					1445-75-6	**4.9E+03**	N	**1.0E+05**	max	**5.5E+04**	N	**2.9E+02**	N	**2.9E+03**	N	
3,3'-Dimethoxybenzidine	1.4E-02 h					1.4E-02 r							119-90-4	**3.5E+01**	C	**4.1E+02**	C	**1.4E+02**	C	**4.8E-01**	C	**4.8E+00**	C	
Dimethylamine					r				x				124-40-3	**1.0E+05**	max	**1.0E+05**	max	**1.0E+05**	max					
N-N-Dimethylaniline				2.0E-03 i				2.0E-03 r					121-69-7	**1.2E+02**	N	**4.1E+03**	N	**1.4E+03**	N	**7.3E+00**	N	**7.3E+01**	N	
2,4-Dimethylaniline	7.5E-01 h					7.5E-01 r							95-68-1	**6.5E-01**	C	**7.6E+00**	C	**2.6E+00**	C	**9.0E-03**	C	**9.0E-02**	C	
2,4-Dimethylaniline hydrochloride	5.8E-01 h					5.8E-01 r							21436-96-4	**8.4E-01**	C	**9.9E+00**	C	**3.3E+00**	C	**1.2E-02**	C	**1.2E-01**	C	
3,3'-Dimethylbenzidine	2.3E+00 p					2.3E+00 r							119-93-7	**2.1E-01**	C	**2.5E+00**	C	**8.3E-01**	C	**2.9E-03**	C	**2.9E-02**	C	
1,1-Dimethylhydrazine		x				x							57-14-7											
1,2-Dimethylhydrazine		x				x							540-73-8											
Dimethylphenethylamine				1.0E-03 n				1.0E-03 r					122-09-8	**6.1E+01**	N	**2.0E+03**	N	**6.8E+02**	N	**3.7E+00**	N	**3.7E+01**	N	
2,4-Dimethylphenol				2.0E-02 i				2.0E-02 r					105-67-9	**1.2E+03**	N	**4.1E+04**	N	**1.4E+04**	N	**7.3E+01**	N	**7.3E+02**	N	**4.0E-01**
2,6-Dimethylphenol				6.0E-04 i				6.0E-04 r					576-26-1	**3.7E+01**	N	**1.2E+03**	N	**4.1E+02**	N	**2.2E+00**	N	**2.2E+01**	N	
3,4-Dimethylphenol				1.0E-03 i				1.0E-03 r					95-65-8	**6.1E+01**	N	**2.0E+03**	N	**6.8E+02**	N	**3.7E+00**	N	**3.7E+01**	N	
Dimethyl phthalate				1.0E+01 h				1.0E+01 r					131-11-3	**1.0E+05**	max	**1.0E+05**	max	**1.0E+05**	max	**3.7E+04**	N	**3.7E+05**	N	
4,6-Dinitro-o-cyclohexyl phenol				2.0E-03 i				2.0E-03 r					131-89-5	**1.2E+02**	N	**4.1E+03**	N	**1.4E+03**	N	**7.3E+00**	N	**7.3E+01**	N	
1,2-Dinitrobenzene				1.0E-04 p				1.0E-04 r					528-29-0	**6.1E+00**	N	**2.0E+02**	N	**6.8E+01**	N	**3.7E-01**	N	**3.7E+00**	N	
1,3-Dinitrobenzene				1.0E-04 i				1.0E-04 r					99-65-0	**6.1E+00**	N	**2.0E+02**	N	**6.8E+01**	N	**3.7E-01**	N	**3.7E+00**	N	
1,4-Dinitrobenzene				1.0E-04 p				1.0E-04 r					100-25-4	**6.1E+00**	N	**2.0E+02**	N	**6.8E+01**	N	**3.7E-01**	N	**3.7E+00**	N	
2,4-Dinitrophenol				2.0E-03 i				2.0E-03 r					51-28-5	**1.2E+02**	N	**4.1E+03**	N	**1.4E+03**	N	**7.3E+00**	N	**7.3E+01**	N	**1.0E-02**
Dinitrotoluene mixture	6.8E-01 i		B2			6.8E-01 r							25321-14-6	**7.2E-01**	C	**8.4E+00**	C	**2.8E+00**	C	**9.9E-03**	C	**9.9E-02**	C	**4.0E-05**
2,4-Dinitrotoluene				2.0E-03 i				2.0E-03 r					121-14-2	**1.2E+02**	N	**4.1E+03**	N	**1.4E+03**	N	**7.3E+00**	N	**7.3E+01**	N	**4.0E-05**
2,6-Dinitrotoluene				1.0E-03 p				1.0E-03 r					606-20-2	**6.1E+01**	N	**2.0E+03**	N	**6.8E+02**	N	**3.7E+00**	N	**3.7E+01**	N	**3.0E-05**
Dinoseb				1.0E-03 i				1.0E-03 r				7.0E+00	88-85-7	**6.1E+01**	N	**2.0E+03**	N	**6.8E+02**	N	**3.7E+00**	N	**3.7E+01**	N	
di-n-Octyl phthalate													117-84-0											
1,4-Dioxane	1.1E-02 i		B2			1.1E-02 r							123-91-1	**4.4E+01**	C	**5.2E+02**	C	**1.7E+02**	C	**6.1E-01**	C	**6.1E+00**	C	
Dioxin (2,3,7,8-TCDD)	1.5E+05 h					1.5E+05 h							1746-01-6	**3.9E-06**	C	**3.8E-05**	C	**1.8E-05**	C	**4.5E-08**	C	**4.5E-07**	C	
Diphenylamine				2.5E-02 i				2.5E-02 r					122-39-4	**1.5E+03**	N	**5.1E+04**	N	**1.7E+04**	N	**9.1E+01**	N	**9.1E+02**	N	
1,2-Diphenylhydrazine	8.0E-01 i		B2			7.7E-01 i							122-66-7	**6.1E-01**	C	**7.2E+00**	C	**2.4E+00**	C	**8.7E-03**	C	**8.4E-02**	C	
Diphenyl sulfone				3.0E-03 p				3.0E-03 r					127-63-9	**1.8E+02**	N	**6.1E+03**	N	**2.1E+03**	N	**1.1E+01**	N	**1.1E+02**	N	
Diquat				2.2E-03 i				2.2E-03 r				2.0E+01	85-00-7	**1.3E+02**	N	**4.5E+03**	N	**1.5E+03**	N	**8.0E+00**	N	**8.0E+01**	N	
Disulfoton				4.0E-05 i				4.0E-05 r					298-04-4	**2.4E+00**	N	**8.2E+01**	N	**2.7E+01**	N	**1.5E-01**	N	**1.5E+00**	N	
1,4-Dithiane				1.0E-02 i				1.0E-02 r					505-29-3	**6.1E+02**	N	**2.0E+04**	N	**6.8E+03**	N	**3.7E+01**	N	**3.7E+02**	N	
Diuron				2.0E-03 i				2.0E-03 r					330-54-1	**1.2E+02**	N	**4.1E+03**	N	**1.4E+03**	N	**7.3E+00**	N	**7.3E+01**	N	
Endosulfan				6.0E-03 i				6.0E-03 r					115-29-7	**3.7E+02**	N	**1.2E+04**	N	**4.1E+03**	N	**2.2E+01**	N	**2.2E+02**	N	**9.0E-01**
Endothall				2.0E-02 i				2.0E-02 r				1.0E+02	145-73-3	**1.2E+03**	N	**4.1E+04**	N	**1.4E+04**	N	**7.3E+01**	N	**7.3E+02**	N	
Endrin				3.0E-04 i				3.0E-04 r				2.0E+00	72-20-8	**1.8E+01**	N	**6.1E+02**	N	**2.1E+02**	N	**1.1E+00**	N	**1.1E+01**	N	**5.0E-02**
Epichlorohydrin	9.9E-03 i		B2	6.0E-03 p		4.2E-03 i		2.9E-04 i		1.0E-03 i			106-89-8	**1.8E+01**	N	**2.9E+01**	N	**2.9E+01**	N	**1.0E+00**	N	**2.1E+00**	N	
Ethion				5.0E-04 i				5.0E-04 r					563-12-2	**3.1E+01**	N	**1.0E+03**	N	**3.4E+02**	N	**1.8E+00**	N	**1.8E+01**	N	
2-Ethoxyethanol				4.0E-01 h				5.7E-02 i		2.0E-01 i			110-80-5	**2.4E+04**	N	**1.0E+05**	max	**1.0E+05**	max	**2.1E+02**	N	**1.5E+04**	N	
2-Ethoxyethanol acetate				3.0E-01 h				3.0E-01 r					111-15-9	**1.8E+04**	N	**1.0E+05**	max	**1.0E+05**	max	**1.1E+03**	N	**1.1E+04**	N	
Ethyl acetate				9.0E-01 i				9.0E-01 r					141-78-6	**1.9E+04**	N	**3.7E+04**	sat	**3.7E+04**	sat	**3.3E+03**	N	**5.5E+03**	N	
Ethylbenzene				1.0E-01 i				2.9E-01 i		1.0E+00 i		7.0E+02	100-41-4	**2.3E+02**	sat	**2.3E+02**	sat	**2.3E+02**	sat	**1.1E+03**	N	**1.3E+03**	N	**7.0E-01**
Ethyl chloride	2.9E-03 n			4.0E-01 n		2.9E-03 r		2.9E+00 i		1.0E+01 i			75-00-3	**3.0E+00**	C	**6.5E+00**	C	**7.2E+00**	C	**2.3E+00**	C	**3.9E+00**	C	
Ethylene diamine				9.0E-02 p				9.0E-02 r					107-15-3	**5.5E+03**	N	**1.0E+05**	max	**6.2E+04**	N	**3.3E+02**	N	**3.3E+03**	N	
Ethylene glycol				2.0E+00 i				2.0E+00 r					107-21-1	**1.0E+05**	max	**1.0E+05**	max	**1.0E+05**	max	**7.3E+03**	N	**7.3E+04**	N	
Ethylene glycol, monobutyl ether				5.0E-01 i				3.7E+00		1.3E+01 i			111-76-2	**3.1E+04**	N	**1.0E+05**	max	**1.0E+05**	max	**1.4E+04**	N	**1.8E+04**	N	
Ethylene oxide	1.0E+00 h					3.5E-01 h							75-21-8	**1.4E-01**	C	**3.6E-01**	C	**3.8E-01**	C	**1.9E-02**	C	**2.4E-02**	C	
Ethylene thiourea (ETU)	1.1E-01 h			8.0E-05 i		1.1E-01 r		8.0E-05 r					96-45-7	**4.4E+00**	C	**5.2E+01**	C	**1.7E+01**	C	**6.1E-02**	C	**6.1E-01**	C	
Ethyl ether				2.0E-01 i				2.0E-01 r					60-29-7	**1.8E+03**	sat	**1.8E+03**	sat	**1.8E+03**	sat	**7.3E+02**	N	**1.2E+03**	N	
Ethyl methacrylate				9.0E-02 h				9.0E-02 r					97-63-2	**1.4E+02**	sat	**1.4E+02**	sat	**1.4E+02**	sat	**3.3E+02**	N	**5.5E+02**	N	

Key : i=IRIS p= PPRTV n=NCEA h=HEAST o=Other DOCUMENTS r=ROUTE EXTRAPOLATION

Key : C=CANCER N=NONCANCER sat=SOIL SATURATION max=CEILING LIMIT

Region 6 Human Health Medium-Specific Screening Levels 2008	TOXICITY INFORMATION													EENING LEVELS												
Contaminants	SFo 1/(mg/kg-d)	K E Y	Mutagenic	CANCER CLASS	RfDo (mg/kg-d)	K E Y	SFi 1/(mg/kg-d)	K E Y	RfDi (mg/kg-d)	K E Y	RfC (mg/m3)	K E Y	MCL (ug/l)	CAS No.	Residential Soil (mg/kg)	K E Y	Industrial Indoor Worker w/o Dermal (mg/kg)	K E Y	Industrial-Outdoor Worker Soil (mg/kg)	K E Y	Ambient Air (ug/m^3)	K E Y	Residential Water (ug/l)	K E Y	DAF 1 (mg/kg)
Fenamiphos					2.5E-04	i			2.5E-04	r				22224-92-6	**1.5E+01**	N	**5.1E+02**	N	**1.7E+02**	N	**9.1E-01**	N	**9.1E+00**	N	
Fluometuron					1.3E-02	i			1.3E-02	r				2164-17-2	**7.9E+02**	N	**2.7E+04**	N	**8.9E+03**	N	**4.7E+01**	N	**4.7E+02**	N	
Fluoride					6.0E-02	i							4.0E+03	16984-48-8	**3.7E+03**	N	**1.0E+05**	max	**4.1E+04**	N			**2.2E+03**	N	
Fomesafen	1.9E-01	i		C			1.9E-01	r						72178-02-0	**2.6E+00**	C	**3.0E+01**	C	**1.0E+01**	C	**3.5E-02**	C	**3.5E-01**	C	
Fonofos					2.0E-03	i			2.0E-03	r				944-22-9	**1.2E+02**	N	**4.1E+03**	N	**1.4E+03**	N	**7.3E+00**	N	**7.3E+01**	N	
Formaldehyde	4.60E-02	r			1.5E-01	i	4.6E-02	i	1.5E-01	r				50-00-0	**1.1E+01**	C	**1.2E+02**	C	**4.2E+01**	C	**1.5E-01**	C	**1.5E+00**	C	
Formic Acid					2.0E+00	h			8.6E-04	p				64-18-6	**1.0E+05**	max	**1.0E+05**	max	**1.0E+05**	max	**3.1E+00**	N	**7.3E+04**	N	
Furan					1.0E-03	i			1.0E-03	r				110-00-9	**2.5E+00**	N	**8.6E+00**	N	**9.5E+00**	N	**3.7E+00**	N	**6.1E+00**	N	
Furazolidone	3.8E+00	h					3.8E+00	r						67-45-8	**1.3E-01**	C	**1.5E+00**	C	**5.0E-01**	C	**1.8E-03**	C	**1.8E-02**	C	
Furfural					3.0E-03	i			1.4E-02	h				98-01-1	**1.8E+02**	N	**6.1E+03**	N	**2.1E+03**	N	**5.2E+01**	N	**1.1E+02**	N	
Glycidaldehyde					4.0E-04	i			2.9E-04	h				765-34-4	**2.4E+01**	N	**8.2E+02**	N	**2.7E+02**	N	**1.0E+00**	N	**1.5E+01**	N	
Glyphosate					1.0E-01	i			1.0E-01	r			7.0E+02	1071-83-6	**6.1E+03**	N	**1.0E+05**	max	**6.8E+04**	N	**3.7E+02**	N	**3.7E+03**	N	
HMX					5.0E-02	i			5.0E-02	r				2691-41-0	**3.1E+03**	N	**1.0E+05**	max	**3.4E+04**	N	**1.8E+02**	N	**1.8E+03**	N	
Heptachlor	4.5E+00	i		B2	5.0E-04	i	4.6E+00	i	5.0E-04	r			1.0E-01	76-44-8	**1.1E-01**	C	**1.3E+00**	C	**4.3E-01**	C	**1.5E-03**	C	**1.5E-02**	C	**1.0E+00**
Heptachlor epoxide	9.1E+00	i		B2	1.3E-05	i	9.1E+00	i	1.3E-05	r			2.0E-01	1024-57-3	**5.3E-02**	C	**6.3E-01**	C	**2.1E-01**	C	**7.4E-04**	C	**7.4E-03**	C	**3.0E-02**
Hexabromobenzene					2.0E-03	i			2.0E-03	r				87-82-1	**1.2E+02**	N	**4.1E+03**	N	**1.4E+03**	N	**7.3E+00**	N	**7.3E+01**	N	
Hexachlorobenzene	1.6E+00	i		B2	8.0E-04	i	1.6E+00	i	8.0E-04	r			1.0E+00	118-74-1	**3.0E-01**	C	**3.6E+00**	C	**1.2E+00**	C	**4.2E-03**	C	**4.2E-02**	C	**1.0E-01**
Hexachlorobutadiene	7.8E-02	i		C	1.0E-03	p	7.7E-02	i						87-68-3	**6.2E+00**	C	**7.3E+01**	C	**2.5E+01**	C	**8.7E-02**	C	**8.6E-01**	C	**1.0E-01**
HCH (alpha)	6.3E+00	i		B2			6.3E+00	i						319-84-6	**9.0E-02**	C	**9.1E-01**	C	**4.0E-01**	C	**1.1E-03**	C	**1.1E-02**	C	**3.0E-05**
HCH (beta)	1.8E+00	i		C			1.8E+00	i						319-85-7	**3.2E-01**	C	**3.2E+00**	C	**1.4E+00**	C	**3.7E-03**	C	**3.7E-02**	C	**1.0E-04**
HCH (gamma) Lindane	1.3E+00	h			3.0E-04	i	1.3E+00	r	3.0E-04	r			2.0E-01	58-89-9	**4.4E-01**	C	**4.4E+00**	C	**1.9E+00**	C	**5.2E-03**	C	**5.2E-02**	C	**5.0E-04**
HCH-technical	1.8E+00	i		B2			1.8E+00	i						608-73-1	**3.2E-01**	C	**3.2E+00**	C	**1.4E+00**	C	**3.8E-03**	C	**3.7E-02**	C	**1.0E-04**
Hexachlorocyclopentadiene					6.0E-03	i			5.7E-05	i	2.0E-04	i	5.0E+01	77-47-4	**3.7E+02**	N	**1.2E+04**	N	**4.1E+03**	N	**2.1E-01**	N	**2.2E+02**	N	**2.0E+01**
Hexachlorodibenzo-p-dioxin mixture (HxCDD)	6.2E+03	i		B2			4.6E+03	i						19408-74-3	**7.8E-05**	C	**9.2E-04**	C	**3.1E-04**	C	**1.5E-06**	C	**1.1E-05**	C	
Hexachloroethane	1.4E-02	i		C	1.0E-03	i	1.4E-02	i	1.0E-03	r				67-72-1	**3.5E+01**	C	**4.1E+02**	C	**1.4E+02**	C	**4.8E-01**	C	**4.8E+00**	C	**2.0E-02**
Hexachlorophene					3.0E-04	i			3.0E-04	r				70-30-4	**1.8E+01**	N	**6.1E+02**	N	**2.1E+02**	N	**1.1E+00**	N	**1.1E+01**	N	
Hexahydro-1,3,5-trinitro-1,3,5-triazine	1.1E-01	i		C	3.0E-03	i	1.1E-01	r	3.0E-03	r				121-82-4	**4.4E+00**	C	**5.2E+01**	C	**1.7E+01**	C	**6.1E-02**	C	**6.1E-01**	C	
1,6-Hexamethylene diisocyanate					2.9E-06	r			2.9E-06	i	1.0E-05	i		822-06-0	**1.7E-01**	N	**5.8E+00**	N	**2.0E+00**	N	**1.0E-02**	N	**1.0E-01**	N	
n-Hexane					1.1E+01	p			2.0E-01	i	7.0E-01	i		110-54-3	**1.1E+02**	sat	**1.1E+02**	sat	**1.1E+02**	sat	**7.3E+02**	N	**1.5E+03**	N	
Hexazinone					3.3E-02	i			3.3E-02	r				51235-04-2	**2.0E+03**	N	**6.7E+04**	N	**2.3E+04**	N	**1.2E+02**	N	**1.2E+03**	N	
Hydrazine, hydrazine sulfate	3.0E+00	i		B2			1.7E+01	i						302-01-2	**1.6E-01**	C	**1.9E+00**	C	**6.4E-01**	C	**3.9E-04**	C	**2.2E-02**	C	
Hydrogen chloride									5.7E-03	i	2.0E-02	i		7647-01-0	**1.0E+05**	max	**1.0E+05**	max	**1.0E+05**	max	**2.1E+01**	N			
Hydrogen sulfide					3.0E-03	i			5.7E-04	i	2.0E-03	i		7783-06-4	**1.8E+02**	N	**6.1E+03**	N	**2.1E+03**	N	**2.1E+00**	N	**1.1E+02**	N	
p-Hydroquinone	5.6E-02	p			4.0E-02	p	5.6E-02	r	4.0E-02	r				123-31-9	**8.7E+00**	C	**1.0E+02**	C	**3.4E+01**	C	**1.2E-01**	C	**1.2E+00**	C	
Iron					7.0E-01	p								7439-89-6	**5.5E+04**	N	**1.0E+05**	max	**1.0E+05**	max			**2.6E+04**	N	
Isobutanol					3.0E-01	i			3.0E-01	r				78-83-1	**1.3E+04**	N	**4.0E+04**	sat	**4.0E+04**	sat	**1.1E+03**	N	**1.8E+03**	N	
Isophorone	9.5E-04	i		C	2.0E-01	i	9.5E-04	r	2.0E-01	r				78-59-1	**5.1E+02**	C	**6.0E+03**	C	**2.0E+03**	C	**7.1E+00**	C	**7.1E+01**	C	**3.0E-02**
Isopropalin					1.5E-02	i			1.5E-02	r				33820-53-0	**9.2E+02**	N	**3.1E+04**	N	**1.0E+04**	N	**5.5E+01**	N	**5.5E+02**	N	
Isopropyl methyl phosphonic acid					1.0E-01	i			1.0E-01	r				1832-54-8	**6.1E+03**	N	**1.0E+05**	max	**6.8E+04**	N	**3.7E+02**	N	**3.7E+03**	N	
Kepone	8.0E+00	p			2.0E-04	p								143-50-0	**6.1E-02**	C	**7.2E-01**	C	**2.4E-01**	C			**8.4E-03**	C	
Lead	**Screening Levels Based on EPA Models, IEUBK (1994) and TRW (1996), Tap Water # = MC**												1.5E+01	7439-92-1	**4.0E+02**		**8.0E+02**		**8.0E+02**				**1.5E+01**		
Lead (tetraethyl)					1.0E-07	i								78-00-2	**6.1E-03**	N	**2.0E-01**	N	**6.8E-02**	N			**3.7E-03**	N	
Lithium						x								7439-93-2											
Malathion					2.0E-02	i			2.0E-02	r				121-75-5	**1.2E+03**	N	**4.1E+04**	N	**1.4E+04**	N	**7.3E+01**	N	**7.3E+02**	N	
Maleic anhydride					1.0E-01	i			1.0E-01	r				108-31-6	**6.1E+03**	N	**1.0E+05**	max	**6.8E+04**	N	**3.7E+02**	N	**3.7E+03**	N	
Manganese and compounds					4.7E-02	i			1.4E-05	i	5.0E-05	i		7439-96-5	**3.5E+03**	N	**4.7E+04**	N	**3.5E+04**	N	**5.1E-02**	N	**1.7E+03**	N	
Mephosfolan					9.0E-05	h			9.0E-05	r				950-10-7	**5.5E+00**	N	**1.8E+02**	N	**6.2E+01**	N	**3.3E-01**	N	**3.3E+00**	N	
Mepiquat					3.0E-02	i			3.0E-02	r				24307-26-4	**1.8E+03**	N	**6.1E+04**	N	**2.1E+04**	N	**1.1E+02**	N	**1.1E+03**	N	
2-Mercaptobenzothiazole	2.9E-02	n			1.0E-01	n	2.9E-02	r	1.0E-01	r				149-30-4	**1.7E+01**	C	**2.0E+02**	C	**6.6E+01**	C	**2.3E-01**	C	**2.3E+00**	C	
Mercury and compounds					3.0E-04	i							2.0E+00	7487-94-7	**2.3E+01**	N	**6.1E+02**	N	**3.4E+02**	N			**1.1E+01**	N	
Mercury (elemental)									8.6E-05	i	3.0E-04	i		7439-97-6							**3.1E-01**	N	**6.3E-01**	N	**1.0E-01**
Mercury (methyl)					1.0E-04	i								22967-92-6	**6.1E+00**	N	**2.0E+02**	N	**6.8E+01**	N			**3.7E+00**	N	
Methacrylonitrile					1.0E-04	i			2.0E-04	h				126-98-7	**2.1E+00**	N	**8.8E+00**	N	**9.3E+00**	N	**7.3E-01**	N	**1.0E+00**	N	
Methanol					5.0E-01	i			5.0E-01	r				67-56-1	**3.1E+04**	N	**1.0E+05**	max	**1.0E+05**	max	**1.8E+03**	N	**1.8E+04**	N	
Methidathion					1.0E-03	i			1.0E-03	r				950-37-8	**6.1E+01**	N	**2.0E+03**	N	**6.8E+02**	N	**3.7E+00**	N	**3.7E+01**	N	
Methoxychlor					5.0E-03	i			5.0E-03	r			4.0E+01	72-43-5	**3.1E+02**	N	**1.0E+04**	N	**3.4E+03**	N	**1.8E+01**	N	**1.8E+02**	N	**8.0E+00**
Methyl acetate					1.0E+00	h			1.0E+00	r				79-20-9	**2.2E+04**	N	**9.6E+04**	N	**1.0E+05**	max	**3.7E+03**	N	**6.1E+03**	N	
Methyl acrylate					3.0E-02	h			3.0E-02	r				96-33-3	**7.0E+01**	N	**2.3E+02**	N	**2.6E+02**	N	**1.1E+02**	N	**1.8E+02**	N	
2-Methylaniline (o-toluidine)	2.4E-01	h					2.4E-01	r						95-53-4	**2.0E+00**	C	**2.4E+01**	C	**8.0E+00**	C	**2.8E-02**	C	**2.8E-01**	C	
2-Methyl-4-chlorophenoxyacetic acid					5.0E-04	i			5.0E-04	r				94-74-6	**3.1E+01**	N	**1.0E+03**	N	**3.4E+02**	N	**1.8E+00**	N	**1.8E+01**	N	
4-(2-Methyl-4-chlorophenoxy) butyric acid (MCPB)					1.0E-02	i			1.0E-02	r				94-81-5	**6.1E+02**	N	**2.0E+04**	N	**6.8E+03**	N	**3.7E+01**	N	**3.7E+02**	N	
2-(2-Methyl-4-chlorophenoxy) propionic acid					1.0E-03	i			1.0E-03	r				93-65-2	**6.1E+01**	N	**2.0E+03**	N	**6.8E+02**	N	**3.7E+00**	N	**3.7E+01**	N	
2-(2-Methyl-1,4-chlorophenoxy) propionic acid (MCPP)					1.0E-03	i			1.0E-03	r				16484-77-8	**6.1E+01**	N	**2.0E+03**	N	**6.8E+02**	N	**3.7E+00**	N	**3.7E+01**	N	
Methylcyclohexane					8.6E-01	r			8.6E-01	h				108-87-2	**1.4E+02**	sat	**1.4E+02**	sat	**1.4E+02**	sat	**3.1E+03**	N	**5.2E+03**	N	
4,4'-Methylene bis(2-chloroaniline)	1.0E-01	p	y		2.0E-03	p	1.3E-01	h	7.0E-04	r				101-14-4	**1.2E+00**	C	**5.7E+01**	C	**1.9E+01**	C	**1.7E-02**	C	**2.2E-01**	C	
4,4'-Methylene bis(N,N'-dimethyl)aniline	4.6E-02	i		B2			4.6E-02	r						101-61-1	**1.1E+01**	C	**1.2E+02**	C	**4.2E+01**	C	**1.5E-01**	C	**1.5E+00**	C	
Methylene bromide					1.0E-02	h			1.0E-02	r				74-95-3	**1.4E+02**	N	**5.5E+02**	N	**5.9E+02**	N	**3.7E+01**	N	**6.1E+01**	N	
Methylene chloride	7.5E-03	i		B2	6.0E-02	i	1.6E-03	i	8.6E-01	h			5.0E+00	75-09-2	**8.9E+00**	C	**2.1E+01**	C	**2.2E+01**	C	**4.1E+00**	C	**4.3E+00**	C	**1.0E-03**
4,4'-Methylenediphenyl isocyanate					1.7E-04	r			1.7E-04	i	6.0E-04	i		101-68-8	**1.0E+01**	N	**3.5E+02**	N	**1.2E+02**	N	**6.2E-01**	N	**6.2E+00**	N	
Methyl ethyl ketone					6.0E-01	i			1.4E+00	i	5.0E+00	i		78-93-3	**3.2E+04**	N	**3.4E+04**	sat	**3.4E+04**	sat	**5.2E+03**	N	**7.1E+03**	N	
Methyl hydrazine	1.1E+00	h					1.1E+00	r						60-34-4	**4.4E-01**	C	**5.2E+00**	C	**1.7E+00**	C	**6.1E-03**	C	**6.1E-02**	C	
Methyl isobutyl ketone					8.0E-02	h			8.6E-01	i	3.0E+00	i		108-10-1	**5.8E+03**	N	**1.7E+04**	sat	**1.7E+04**	sat	**3.1E+03**	N	**2.0E+03**	N	

Region 6 Human Health Medium-Specific Screening Levels 2008

Contaminants	SFo 1/(mg/kg-d)	K E Y	Mutagenic	CANCER CLASS	RfDo (mg/kg-d)	K E Y	SFi 1/(mg/kg-d)	K E Y	RfDi (mg/kg-d)	K E Y	RfC (mg/m3)	K E Y	MCL (ug/l)	CAS No.	Residential Soil (mg/kg)	K E Y	Industrial Indoor Worker w/o Dermal (mg/kg)	K E Y	Industrial-Outdoor Worker Soil (mg/kg)	K E Y	Ambient Air (ug/m^3)	K E Y	Residential Water (ug/l)	K E Y	DAF 1 (mg/kg)
Methyl mercaptan					5.7E-04 r				5.7E-04 n		2.0E-03 n			74-93-1	3.5E+01	N	1.2E+03	N	3.9E+02	N	2.1E+00	N	2.1E+01	N	
Methyl methacrylate					1.4E+00 i				2.0E-01 i		7.0E-01 i			80-62-6	2.7E+03	sat	2.7E+03	sat	2.7E+03	sat			7.3E+02	N	1.4E+03	N	
2-Methyl-5-nitroaniline	3.3E-02 h						3.3E-02 r							99-55-8	1.5E+01	C	1.7E+02	C	5.8E+01	C	2.0E-01	C	2.0E+00	C	
Methyl parathion					2.5E-04 i				2.5E-04 r					298-00-0	1.5E+01	N	5.1E+02	N	1.7E+02	N	9.1E-01	N	9.1E+00	N	
2-Methylphenol					5.0E-02 i				r					95-48-7	3.1E+03	N	1.0E+05	max	3.4E+04	N			1.8E+03	N	8.0E-01
3-Methylphenol					5.0E-02 i				r					108-39-4	3.1E+03	N	1.0E+05	max	3.4E+04	N			1.8E+03	N	
4-Methylphenol					5.0E-03 h				5.0E-03 r					106-44-5	3.1E+02	N	1.0E+04	N	3.4E+03	N	1.8E+01	N	1.8E+02	N	
Methyl phosphonic acid					2.0E-02 p				2.0E-02 r					993-13-5	1.2E+03	N	4.1E+04	N	1.4E+04	N	7.3E+01	N	7.3E+02	N	
Methyl styrene (mixture)					6.0E-03 h				1.1E-02 h					25013-15-4	1.3E+02	N	5.6E+02	N	6.0E+02	N	4.2E+01	N	6.0E+01	N	
Methyl styrene (alpha)					7.0E-02 h				7.0E-02 r					98-83-9	6.8E+02	sat	6.8E+02	sat	6.8E+02	sat	2.6E+02	N	4.3E+02	N	
Methyl tertbutyl ether (MTBE)	1.8E-03 o				8.6E-01 r		9.1E-04 o		8.6E-01 i		3.0E+00 i			1634-04-4	3.2E+01	C	7.2E+01	C	7.9E+01	C	7.4E+00	C	1.1E+01	C	
Metolaclor (Dual)					1.5E-01 i				1.5E-01 r					51218-45-2	9.2E+03	N	1.0E+05	max	1.0E+05	max	5.5E+02	N	5.5E+03	N	
Mirex	1.8E+00 h				2.0E-04 i		1.8E+00 r		2.0E-04 r					2385-85-5	2.7E-01	C	3.2E+00	C	1.1E+00	C	3.7E-03	C	3.7E-02	C	
Molybdenum					5.0E-03 i									7439-98-7	3.9E+02	N	1.0E+04	N	5.7E+03	N			1.8E+02	N	
Monochloramine					1.0E-01 h				1.0E-01 h					10599-90-3	6.1E+03	N	1.0E+05	max	6.8E+04	N	3.7E+02	N	3.7E+03	N	
Naled					2.0E-03 i				2.0E-03 r					300-76-5	1.2E+02	N	4.1E+03	N	1.4E+03	N	7.3E+00	N	7.3E+01	N	
Nickel and compounds					2.0E-02 i									7440-02-0	1.6E+03	N	4.1E+04	N	2.3E+04	N			7.3E+02	N	7.0E+00
Nickel refinery dust				A			8.4E-01 i							n/a	1.1E+04	C	2.2E+04	C	2.5E+04	C	8.0E-03	C			
Nickel subsulfide				A			1.7E+00 i							12035-72-2	5.2E+03	C	1.1E+04	C	1.2E+04	C	4.0E-03	C			
Nitrate	Tap Water Screening Level Based on Infant NOAEL (see IRIS)												1.0E+04	14797-55-8									1.0E+04		
Nitric Oxide					x									10102-43-9											
Nitrite	Tap Water Screening Level Based on Infant NOAEL (see IRIS)												1.0E+03	14797-65-0									1.0E+03		
2-Nitroaniline					3.0E-03 p				2.9E-05 p					88-74-4	1.8E+02	N	5.9E+03	N	2.0E+03	N	1.0E-01	N	1.1E+02	N	
Nitrobenzene					5.0E-04 i				5.7E-04 h					98-95-3	2.0E+01	N	1.1E+02	N	1.1E+02	N	2.1E+00	N	3.4E+00	N	7.0E-03
Nitrofurantoin					7.0E-02 h				7.0E-02 r					67-20-9	4.3E+03	N	1.0E+05	max	4.8E+04	N	2.6E+02	N	2.6E+03	N	
Nitrofurazone	1.5E+00 h						9.4E+00 h							59-87-0	3.2E-01	C	3.8E+00	C	1.3E+00	C	7.2E-04	C	4.5E-02	C	
Nitrogen dioxide					x									10102-44-0											
Nitroglycerin	1.7E-02 p				1.0E-04 p		1.7E-02 r							10102-44-0	6.1E+00	N	2.0E+02	N	6.8E+01	N	4.0E-01	C	3.7E+00	N	
4-Nitrophenol					8.0E-03 n				8.0E-03 r					100-02-7	4.9E+02	N	1.6E+04	N	5.5E+03	N	2.9E+01	N	2.9E+02	N	
2-Nitropropane	9.4E+00 r				5.7E-03 r		9.4E+00 h		5.7E-03 i		2.0E-02 i			79-46-9	6.8E-02	C	6.1E-01	C	3.4E-01	C	7.2E-04	C	1.2E-03	C	
N-Nitrosodi-n-butylamine	5.4E+00 i			B2			5.6E+00 i							924-16-3	2.4E-02	C	6.2E-02	C	6.5E-02	C	1.2E-03	C	2.0E-03	C	
N-Nitrosodiethanolamine	2.8E+00 i			B2			2.8E+00 r							1116-54-7	1.7E-01	C	2.0E+00	C	6.8E-01	C	2.4E-03	C	2.4E-02	C	
N-Nitrosodiethylamine	1.5E+02 i	y		B2			1.5E+02 i							55-18-5	7.7E-04	C	3.8E-02	C	1.3E-02	C	1.4E-05	C	1.4E-04	C	
N-Nitrosodimethylamine	5.1E+01 i	y		B2	8.0E-06 p		4.9E+01 i							62-75-9	2.3E-03	C	1.1E-01	C	3.8E-02	C	4.4E-05	C	4.2E-04	C	
N-Nitrosodiphenylamine	4.9E-03 i			B2			4.9E-03 r							86-30-6	9.9E+01	C	1.2E+03	C	3.9E+02	C	1.4E+00	C	1.4E+01	C	6.0E-02
N-Nitroso di-n-propylamine	7.0E+00 i			B2			7.0E+00 r							621-64-7	6.9E-02	C	8.2E-01	C	2.7E-01	C	9.6E-04	C	9.6E-03	C	2.0E-06
N-Nitroso-N-methylethylamine	2.2E+01 i			B2			2.2E+01 r							10595-95-6	2.2E-02	C	2.6E-01	C	8.7E-02	C	3.1E-04	C	3.1E-03	C	
N-Nitrosopyrrolidine	2.1E+00 i			B2			2.1E+00 i							930-55-2	2.3E-01	C	2.7E+00	C	9.1E-01	C	3.1E-03	C	3.2E-02	C	
m-Nitrotoluene					2.0E-02 p				2.0E-02 r					99-08-1	1.6E+03	N	4.1E+04	N	2.3E+04	N	7.3E+01	N	1.2E+02	N	
o-Nitrotoluene	2.3E-01 p				1.0E-02 h									88-72-2	2.8E+00	C	2.5E+01	C	1.4E+01	C			2.9E-01	C	
p-Nitrotoluene	1.6E-02 p				4.0E-03 p									99-99-0	4.0E+01	C	3.6E+02	C	2.0E+02	C			4.2E+00	C	
NuStar					7.0E-04 i				7.0E-04 r					85509-19-9	4.3E+01	N	1.4E+03	N	4.8E+02	N	2.6E+00	N	2.6E+01	N	
Octahydro-1357-tetranitro-1357- tetrazocine (HMX)					5.0E-02 i				5.0E-02 r					2691-41-0	3.1E+03	N	1.0E+05	max	3.4E+04	N	1.8E+02	N	1.8E+03	N	
Oryzalin					5.0E-02 i				5.0E-02 r					19044-88-3	3.1E+03	N	1.0E+05	max	3.4E+04	N	1.8E+02	N	1.8E+03	N	
Oxadiazon					5.0E-03 i				5.0E-03 r					19666-30-9	3.1E+02	N	1.0E+04	N	3.4E+03	N	1.8E+01	N	1.8E+02	N	
Oxamyl					2.5E-02 i				2.5E-02 r				2.0E+02	23135-22-0	1.5E+03	N	5.1E+04	N	1.7E+04	N	9.1E+01	N	9.1E+02	N	
Oxyfluorfen					3.0E-03 i				3.0E-03 r					42874-03-3	1.8E+02	N	6.1E+03	N	2.1E+03	N	1.1E+01	N	1.1E+02	N	
Paraquat					4.5E-03 i				4.5E-03 r					4685-14-7	2.7E+02	N	9.2E+03	N	3.1E+03	N	1.6E+01	N	1.6E+02	N	
Parathion					6.0E-03 h				6.0E-03 r					56-38-2	3.7E+02	N	1.2E+04	N	4.1E+03	N	2.2E+01	N	2.2E+02	N	
Pentachlorobenzene					8.0E-04 i				8.0E-04 r					608-93-5	4.9E+01	N	1.6E+03	N	5.5E+02	N	2.9E+00	N	2.9E+01	N	
Pentachloronitrobenzene	2.6E-01 h				3.0E-03 i		2.6E-01 r		3.0E-03 r					82-68-8	1.9E+00	C	2.2E+01	C	7.4E+00	C	2.6E-02	C	2.6E-01	C	
Pentachlorophenol	1.2E-01 i			B2	3.0E-02 i		1.2E-01 r		3.0E-02 r				1.0E+00	87-86-5	3.0E+00	C	4.8E+01	C	1.0E+01	C	5.6E-02	C	5.6E-01	C	1.0E-03
Perchlorate					7.0E-04 i									7601-90-3	5.5E+01	N	1.4E+03	N	7.9E+02	N			<24.5	N	
Permethrin					5.0E-02 i				5.0E-02 r					52645-53-1	3.1E+03	N	1.0E+05	max	3.4E+04	N	1.8E+02	N	1.8E+03	N	
Phenol					3.0E-01 i									108-95-2	1.8E+04	N	1.0E+05	max	1.0E+05	max			1.1E+04	N	5.0E+00
Phenothiazine					2.0E-03 n				2.0E-03 r					92-84-2	1.2E+02	N	4.1E+03	N	1.4E+03	N	7.3E+00	N	7.3E+01	N	
m-Phenylenediamine					6.0E-03 i				6.0E-03 r					108-45-2	3.7E+02	N	1.2E+04	N	4.1E+03	N	2.2E+01	N	2.2E+02	N	
p-Phenylenediamine					1.9E-01 h				1.9E-01 r					106-50-3	1.2E+04	N	1.0E+05	max	1.0E+05	max	6.9E+02	N	6.9E+03	N	
Phenylmercuric acetate					8.0E-05 i				8.0E-05 r					62-38-4	4.9E+00	N	1.6E+02	N	5.5E+01	N	2.9E-01	N	2.9E+00	N	
2-Phenylphenol	1.9E-03 h						1.9E-03 r							90-43-7	2.5E+02	C	2.9E+03	C	9.9E+02	C	3.5E+00	C	3.5E+01	C	
Phosphine					3.0E-04 h				8.6E-05 i		3.0E-04 i			7803-51-2	1.8E+01	N	6.1E+02	N	2.1E+02	N	3.1E-01	N	1.1E+01	N	
Phosphoric acid									2.9E-03 i		1.0E-02 i			7664-38-2							1.0E+01	N	2.1E+01	N	
Phosphorus (white)					2.0E-05 i									7723-14-0	1.6E+00	N	4.1E+01	N	2.3E+01	N			7.3E-01	N	
p-Phthalic acid					1.0E+00 h				1.0E+00 r					100-21-0	6.1E+04	N	1.0E+05	max	1.0E+05	max	3.7E+03	N	3.7E+04	N	
Phthalic anhydride					2.0E+00 i				3.4E-02 h					85-44-9	1.0E+05	max	1.0E+05	max	1.0E+05	max	1.2E+02	N	7.3E+04	N	
Polybrominated biphenyls	8.9E+00 h				7.0E-06 h		8.9E+00 r		7.0E-06 r						5.5E-02	C	6.4E-01	C	2.2E-01	C	7.6E-04	C	7.6E-03	C	
Polychlorinated biphenyls (PCBs)	2.0E+00 i			B2			2.0E+00 r						5.0E-01	1336-36-3	2.2E-01	C	2.9E+00	C	8.3E-01	C	3.4E-03	C	3.4E-02	C	
Aroclor 1016	7.0E-02 i			B2	7.0E-05 i		7.0E-02 i		7.0E-05 r					12674-11-2	3.9E+00	N	8.2E+01	C	2.4E+01	C	9.6E-02	C	9.6E-01	C	
Aroclor 1221	2.0E+00 i			B2			2.0E+00 i							11104-28-2	2.2E-01	C	2.9E+00	C	8.3E-01	C	3.4E-03	C	3.4E-02	C	
Aroclor 1232	2.0E+00 i			B2			2.0E+00 i							11141-16-5	2.2E-01	C	2.9E+00	C	8.3E-01	C	3.4E-03	C	3.4E-02	C	
Aroclor 1242	2.0E+00 i			B2			2.0E+00 i							53469-21-9	2.2E-01	C	2.9E+00	C	8.3E-01	C	3.4E-03	C	3.4E-02	C	
Aroclor 1248	2.0E+00 i			B2			2.0E+00 i							12672-29-6	2.2E-01	C	2.9E+00	C	8.3E-01	C	3.4E-03	C	3.4E-02	C	

Key : i=IRIS p= PPRTV n=NCEA h=HEAST o=Other DOCUMENTS r=ROUTE EXTRAPOLATION

Key : C=CANCER N=NONCANCER sat=SOIL SATURATION max=CEILING LIMIT

Region 6 Human Health Medium-Specific Screening Levels 2008

Contaminants	SFo 1/(mg/kg-d)	K E Y	Mutagenic	CANCER CLASS	RfDo (mg/kg-d)	K E Y	SFi 1/(mg/kg-d)	K E Y	RfDi (mg/kg-d)	K E Y	RfC (mg/m3)	K E Y	MCL (ug/l)	CAS No.	Residential Soil (mg/kg)	K E Y	Industrial Indoor Worker w/o Dermal (mg/kg)	K E Y	Industrial-Outdoor Worker Soil (mg/kg)	K E Y	Ambient Air (ug/m^3)	K E Y	Residential Water (ug/l)	K E Y	DAF 1 (mg/kg)
Aroclor 1254	2.0E+00 i			B2	2.0E-05 i		2.0E+00 i		2.0E-05 r					11097-69-1	2.2E-01	C	2.9E+00	C	8.3E-01	C	3.4E-03	C	3.4E-02	C	
Aroclor 1260	2.0E+00 i			B2			2.0E+00 i							11096-82-5	2.2E-01	C	2.9E+00	C	8.3E-01	C	3.4E-03	C	3.4E-02	C	
Polynuclear aromatic hydrocarbons																									
Acenaphthen [e]					6.0E-02 i				6.0E-02 r					83-32-9	3.7E+03	N	3.8E+04	N	3.3E+04	N	2.2E+02	N	3.7E+02	N	2.9E+01
Anthracen [e]					3.0E-01 i				3.0E-01 r					120-12-7	2.2E+04	N	1.0E+05	max	1.0E+05	max	1.1E+03	N	1.8E+03	N	5.9E+02
Benz[a]anthracen [e]	7.3E-01 n	y					3.1E-01 n							56-55-3	1.5E-01	C	7.8E+00	C	2.3E+00	C	6.9E-03	C	2.9E-02	C	8.0E-02
Benzo[b]fluoranthen [e]	7.3E-01 n	y		B2			3.1E-01 n							205-99-2	1.5E-01	C	7.8E+00	C	2.3E+00	C	6.9E-03	C	2.9E-02	C	2.0E-01
Benzo[k] fluoranthene	7.3E-02 n	y		B2			3.1E-02 n							207-08-9	1.5E+00	C	7.8E+01	C	2.3E+01	C	6.9E-02	C	2.9E-01	C	2.0E+00
Benzo[a]pyren [e]	7.3E+00 i	y		B2			3.1E+00 n						2.0E-01	50-32-8	1.5E-02	C	7.8E-01	C	2.3E-01	C	6.9E-04	C	2.9E-03	C	4.0E-01
Chrysen [e]	7.3E-03 n	y		B2			3.1E-03 n							218-01-9	1.5E+01	C	7.8E+02	C	2.3E+02	C	6.9E-01	C	2.9E+00	C	8.0E+00
Dibenz[ah]anthracen [e]	7.3E+00 n	y		B2			3.1E+00 n							53-70-3	1.5E-02	C	7.8E-01	C	2.3E-01	C	6.9E-04	C	2.9E-03	C	8.0E-02
Fluoranthen [e]				D	4.0E-02 i				4.0E-02 r					206-44-0	2.3E+03	N	8.2E+04	N	2.4E+04	N	1.5E+02	N	1.5E+03	N	2.1E+02
Fluorene				D	4.0E-02 i				4.0E-02 r					86-73-7	2.6E+03	N	3.3E+04	N	2.6E+04	N	1.5E+02	N	2.4E+02	N	2.8E+01
Indeno[1,2,3-cd]pyrene	7.3E-01 n	y		B2			3.1E-01 n							193-39-5	1.5E-01	C	7.8E+00	C	2.3E+00	C	6.9E-03	C	2.9E-02	C	7.0E-01
Naphthalen [e]				C	2.0E-02 i				8.6E-04 i		3.0E-03 i			91-20-3	1.2E+02	N	1.9E+02	N	2.1E+02	N	3.1E+00	N	6.2E+00	N	4.0E+00
Pyrene				D	3.0E-02 i				3.0E-02 r					129-00-0	2.3E+03	N	5.4E+04	N	3.2E+04	N	1.1E+02	N	1.8E+02	N	2.1E+02
Prometon					1.5E-02 i				1.5E-02 r					1610-18-0	9.2E+02	N	3.1E+04	N	1.0E+04	N	5.5E+01	N	5.5E+02	N	
Prometryn					4.0E-03 i				4.0E-03 r					7287-19-6	2.4E+02	N	8.2E+03	N	2.7E+03	N	1.5E+01	N	1.5E+02	N	
Propachlor					1.3E-02 i				1.3E-02 r					1918-16-7	7.9E+02	N	2.7E+04	N	8.9E+03	N	4.7E+01	N	4.7E+02	N	
Propanil					5.0E-03 i				5.0E-03 r					709-98-8	3.1E+02	N	1.0E+04	N	3.4E+03	N	1.8E+01	N	1.8E+02	N	
Propargite					2.0E-02 i				2.0E-02 r					2312-35-8	1.2E+03	N	4.1E+04	N	1.4E+04	N	7.3E+01	N	7.3E+02	N	
Propargyl alcohol					2.0E-03 i				2.0E-03 r					107-19-7	1.2E+02	N	4.1E+03	N	1.4E+03	N	7.3E+00	N	7.3E+01	N	
Propazine					2.0E-02 i				2.0E-02 r					139-40-2	1.2E+03	N	4.1E+04	N	1.4E+04	N	7.3E+01	N	7.3E+02	N	
Propiconazole					1.3E-02 i				1.3E-02 r					60207-90-1	7.9E+02	N	2.7E+04	N	8.9E+03	N	4.7E+01	N	4.7E+02	N	
n-Propylbenzene					1.0E-02 n				1.0E-02 r					103-65-1	1.4E+02	N	2.4E+02	sat	2.4E+02	sat	3.7E+01	N	6.1E+01	N	
Propylene glycol					5.0E-01 p				8.6E-04 p					57-55-6	3.0E+04	N	1.0E+05	max	1.0E+05	max	3.1E+00	N	1.8E+04	N	
Propylene glycol, monoethyl ether					7.0E-01 h									111-35-3	4.3E+04	N	1.0E+05	max	1.0E+05	max			2.6E+04	N	
Propylene glycol, monomethyl ether					7.0E-01 h				5.7E-01 i		2.0E+00 i			107-98-2	4.3E+04	N	1.0E+05	max	1.0E+05	max	2.1E+03	N	2.6E+04	N	
Propylene oxide	2.4E-01 i			B2	8.6E-03 r		1.3E-02 i		8.6E-03 i		3.0E-02 i			75-56-9	1.9E+00	C	9.1E+00	C	7.3E+00	C	5.2E-01	C	2.2E-01	C	
Pursuit					2.5E-01 i				2.5E-01 r					81335-77-5	1.5E+04	N	1.0E+05	max	1.0E+05	max	9.1E+02	N	9.1E+03	N	
Pyridine					1.0E-03 i				1.0E-03 r					110-86-1	6.1E+01	N	2.0E+03	N	6.8E+02	N	3.7E+00	N	3.7E+01	N	
Quinoline	3.0E+00 i			likely			1.2E+01 r							91-22-5	1.6E-01	C	1.9E+00	C	6.4E-01	C	5.6E-04	C	2.2E-02	C	
RDX (Cyclonite)	1.1E-01 i			C	3.0E-03 i		1.1E-01 r		3.0E-03 r					121-82-4	4.4E+00	C	5.2E+01	C	1.7E+01	C	6.1E-02	C	6.1E-01	C	
Resmethrin					3.0E-02 i				3.0E-02 r					10453-86-8	1.8E+03	N	6.1E+04	N	2.1E+04	N	1.1E+02	N	1.1E+03	N	
Ronnel					5.0E-02 h				5.0E-02 r					299-84-3	3.1E+03	N	1.0E+05	max	3.4E+04	N	1.8E+02	N	1.8E+03	N	
Rotenone					4.0E-03 i				4.0E-03 r					83-79-4	2.4E+02	N	8.2E+03	N	2.7E+03	N	1.5E+01	N	1.5E+02	N	
Selenious Acid					5.0E-03 i									7783-00-8	3.1E+02	N	1.0E+04	N	3.4E+03	N			1.8E+02	N	
Selenium					5.0E-03 i								5.0E+01	7782-49-2	3.9E+02	N	1.0E+04	N	5.7E+03	N			1.8E+02	N	3.0E-01
Silver and compounds					5.0E-03 i									7440-22-4	3.9E+02	N	1.0E+04	N	5.7E+03	N			1.8E+02	N	2.0E+00
Simazine	1.2E-01 h				5.0E-03 i		1.2E-01 r		2.0E-03 r				4.0E+00	122-34-9	4.1E+00	C	4.8E+01	C	1.6E+01	C	5.6E-02	C	5.6E-01	C	
Sodium azide					4.0E-03 i				4.0E-03 r					26628-22-8	2.4E+02	N	8.2E+03	N	2.7E+03	N	1.5E+01	N	1.5E+02	N	
Sodium diethyldithiocarbamate	2.7E-01 h				3.0E-02 i		2.7E-01 r		3.0E-02 r					148-18-5	1.8E+00	C	2.1E+01	C	7.1E+00	C	2.5E-02	C	2.5E-01	C	
Sodium fluoroacetate					2.0E-05 i				2.0E-05 r					62-74-8	1.2E+00	N	4.1E+01	N	1.4E+01	N	7.3E-02	N	7.3E-01	N	
Sodium metavanadate					1.0E-03 h				1.0E-03 r					13718-26-8	6.1E+01	N	2.0E+03	N	6.8E+02	N	3.7E+00	N	3.7E+01	N	
Strontium, stable					6.0E-01 i									7440-24-6	4.7E+04	N	1.0E+05	max	1.0E+05	max			2.2E+04	N	
Strychnine					3.0E-04 i				3.0E-04 r					57-24-9	1.8E+01	N	6.1E+02	N	2.1E+02	N	1.1E+00	N	1.1E+01	N	
Styrene					2.0E-01 i				2.9E-01 i		1.0E+00 i		1.0E+02	100-42-5	1.7E+03	sat	1.7E+03	sat	1.7E+03	sat	1.1E+03	N	1.6E+03	N	2.0E-01
2,3,7,8-TCDD (dioxin)	1.5E+05 h						1.5E+05 h						3.0E-05	1746-01-6	3.9E-06	C	3.8E-05	C	1.8E-05	C	4.5E-08	C	4.5E-07	C	
1,2,4,5-Tetrachlorobenzene					3.0E-04 i				3.0E-04 r					95-94-3	1.8E+01	N	6.1E+02	N	2.1E+02	N	1.1E+00	N	1.1E+01	N	
1,1,1,2-Tetrachloroethane	2.6E-02 i			C	3.0E-02 i		2.6E-02 i		3.0E-02 r					630-20-6	3.0E+00	C	7.1E+00	C	7.6E+00	C	2.6E-01	C	4.3E-01	C	
1,1,2,2-Tetrachloroethane	2.0E-01 i			C	6.0E-02 p		2.0E-01 i		6.0E-02 r					79-34-5	3.8E-01	C	9.0E-01	C	9.7E-01	C	3.3E-02	C	5.5E-02	C	2.0E-04
Tetrachloroethylene (PCE)	5.4E-01 o				1.0E-02 i		2.1E-02 o		1.7E-01		6.0E-01		5.0E+00	127-18-4	5.5E-01	C	1.8E+00	C	1.7E+00	C	3.3E-01	C	1.0E-01	C	3.0E-03
2,3,4,6-Tetrachlorophenol					3.0E-02 i				3.0E-02 r					58-90-2	1.8E+03	N	6.1E+04	N	2.1E+04	N	1.1E+02	N	1.1E+03	N	
p,a,a,a-Tetrachlorotoluene	2.0E+01 h						2.0E+01 r							5216-25-1	2.4E-02	C	2.9E-01	C	9.6E-02	C	3.4E-04	C	3.4E-03	C	
Tetrachlorovinphos	2.4E-02 h				3.0E-02 i		2.4E-02 r		3.0E-02 r					961-11-5	2.0E+01	C	2.4E+02	C	8.0E+01	C	2.8E-01	C	2.8E+00	C	
Tetrahydrofuran	7.6E-03 n				2.0E-01 n		6.8E-03 n		8.6E-02 n		3.0E-01 n			109-99-9	9.0E+00	C	2.2E+01	C	2.3E+01	C	9.9E-01	C	1.6E+00	C	
Thallic oxide					7.0E-05 h									1314-32-5	5.5E+00	N	1.4E+02	N	7.9E+01	N			2.6E+00	N	
Thallium					7.0E-05 i								2.0E+00		5.5E+00		1.4E+02		7.9E+01				2.6E+00	N	4.0E-01
Thallium acetate					9.0E-05 i								2.0E+00	563-68-8	7.0E+00	N	1.8E+02	N	1.0E+02	N			3.3E+00	N	4.0E-01
Thallium carbonate					8.0E-05 i								2.0E+00	6533-73-9	6.3E+00	N	1.6E+02	N	9.1E+01	N			2.9E+00	N	4.0E-01
Thallium chloride					8.0E-05 i								2.0E+00	7791-12-0	6.3E+00	N	1.6E+02	N	9.1E+01	N			2.9E+00	N	4.0E-01
Thallium nitrate					9.0E-05 i								2.0E+00	10102-45-1	7.0E+00	N	1.8E+02	N	1.0E+02	N			3.3E+00	N	4.0E-01
Thallium selenite						x							2.0E+00	12039-52-0											4.0E-01
Thallium sulfate					8.0E-05 i								2.0E+00	7446-18-6	6.3E+00	N	1.6E+02	N	9.1E+01	N			2.9E+00	N	4.0E-01
Thiobencarb					1.0E-02 i				1.0E-02 r					28249-77-6	6.1E+02	N	2.0E+04	N	6.8E+03	N	3.7E+01	N	3.7E+02	N	
Thiocyanate					2.0E-04 p									N/A	1.2E+01	N	4.1E+02	N	1.4E+02	N			7.3E+00	N	
Tin and compounds					6.0E-01 h									n/a	4.7E+04	N	1.0E+05	max	1.0E+05	max			2.2E+04	N	
Toluene					8.0E-02 i				1.4E+00 i		5.0E+00 i		1.0E+03	108-88-3	5.2E+02	sat	5.2E+02	sat	5.2E+02	sat	5.2E+03	N	2.3E+03	N	6.0E-01
Toluene-2,4-diamine	3.2E+00 h						3.2E+00 r							95-80-7	1.5E-01	C	1.8E+00	C	6.0E-01	C	2.1E-03	C	2.1E-02	C	
Toluene-2,5-diamine					6.0E-01 h				6.0E-01 r					95-70-5	3.7E+04	N	1.0E+05	max	1.0E+05	max	2.2E+03	N	2.2E+04	N	
Toluene-2,6-diamine					3.0E-02 p				3.0E-02 r					823-40-5	1.8E+03	N	6.1E+04	N	2.1E+04	N	1.1E+02	N	1.1E+03	N	
p-Toluidine	1.9E-01 h						1.9E-01 r							106-49-0	2.6E+00	C	3.0E+01	C	1.0E+01	C	3.5E-02	C	3.5E-01	C	

Key : i=IRIS p= PPRTV n=NCEA h=HEAST o=Other DOCUMENTS r=ROUTE EXTRAPOLATION

Key : C=CANCER N=NONCANCER sat=SOIL SATURATION max=CEILING LIMIT

Region 6 Human Health Medium-Specific Screening Levels 2008

Contaminants	TOXICITY INFORMATION																EENING LEVELS											
	SFo	K E	Mutagenic	CANCER	RfDo	K E	SFi	K E	RfDi	K E	RfC	K E	MCL	CAS No.	Residential	K E	Industrial Indoor Worker w/o Dermal	K E	Industrial-Outdoor Worker	K E	Ambient Air	K E	Residential Water	K E	DAF 1
	1/(mg/kg-d)	Y		CLASS	(mg/kg-d)	Y	1/(mg/kg-d)	Y	(mg/kg-d)	Y	(mg/m3)	Y	(ug/l)		Soil (mg/kg)	Y	(mg/kg)	Y	Soil (mg/kg)	Y	(ug/m^3)	Y	(ug/l)	Y	(mg/kg)
Toxaphene	1.1E+00	i		B2			1.1E+00	i					3.0E+00	8001-35-2	**4.4E-01**	C	**5.2E+00**	C	**1.7E+00**	C	**6.0E-03**	C	**6.1E-02**	C	**2.0E+00**
1,2,4-Tribromobenzene					5.0E-03	i			5.0E-03	r				615-54-3	**3.1E+02**	N	**1.0E+04**	N	**3.4E+03**	N	**1.8E+01**	N	**1.8E+02**	N	
Tributyltin oxide (TBTO)					3.0E-04	i								56-35-9	**1.8E+01**	N	**6.1E+02**	N	**2.1E+02**	N			**1.1E+01**	N	
2,4,6-Trichloroaniline	3.4E-02	h					3.4E-02	r						634-93-5	**1.4E+01**	C	**1.7E+02**	C	**5.6E+01**	C	**2.0E-01**	C	**2.0E+00**	C	
1,2,4-Trichlorobenzene					1.0E-02	i			1.1E-03	p	4.0E-03	p	7.0E+01	120-82-1	**1.4E+02**	N	**2.4E+02**	N	**2.6E+02**	N	**4.2E+00**	N	**8.2E+00**	N	**3.0E-01**
1,1,1-Trichloroethane					2.0E+00	i			1.4E+00		5.0E+00	i	2.0E+02	71-55-6	**1.4E+03**	sat	**1.4E+03**	sat	**1.4E+03**	sat	**5.2E+03**	N	**9.1E+03**	N	**1.0E-01**
1,1,2-Trichloroethane	5.7E-02	i		C	4.0E-03	i	5.6E-02	i	4.0E-03	r			5.0E+00	79-00-5	**8.4E-01**	C	**1.9E+00**	C	**2.1E+00**	C	**1.2E-01**	C	**2.0E-01**	C	**9.0E-04**
Trichloroethylene (TCE)	4.0E-01	n			3.0E-04	n	4.0E-01	n	1.1E-02	n	4.0E-02	n	5.0E+00	79-01-6	**4.3E-02**	C	**9.2E-02**	C	**1.0E-01**	C	**1.7E-02**	C	**2.8E-02**	C	**3.0E-03**
Trichlorofluoromethane					3.0E-01	i			2.0E-01	h				75-69-4	**3.9E+02**	N	**1.3E+03**	N	**1.4E+03**	N	**7.3E+02**	N	**1.3E+03**	N	
2,4,5-Trichlorophenol					1.0E-01	i			1.0E-01	r				95-95-4	**6.1E+03**	N	**1.0E+05**	max	**6.8E+04**	N	**3.7E+02**	N	**3.7E+03**	N	**1.4E+01**
2,4,6-Trichlorophenol	1.1E-02	i		B2	1.0E-03	p	1.1E-02	i						88-06-2	**4.4E+01**	C	**5.2E+02**	C	**1.7E+02**	C	**6.2E-01**	C	**6.1E+00**	C	**8.0E-03**
2,4,5-Trichlorophenoxyacetic Acid					1.0E-02	i			1.0E-02	r				93-76-5	**6.1E+02**	N	**2.0E+04**	N	**6.8E+03**	N	**3.7E+01**	N	**3.7E+02**	N	
2-(2,4,5-Trichlorophenoxy) propionic acid					8.0E-03	i			8.0E-03	r				93-72-1	**4.9E+02**	N	**1.6E+04**	N	**5.5E+03**	N	**2.9E+01**	N	**2.9E+02**	N	
1,1,2-Trichloropropane					5.0E-03	i			5.0E-03	r				598-77-6	**1.5E+01**	N	**5.1E+01**	N	**5.7E+01**	N	**1.8E+01**	N	**3.0E+01**	N	
1,2,3-Trichloropropane	2.0E+00	n			6.0E-03	i		r	1.4E-03	r	5.0E-03	n		96-18-4	**3.2E-01**	C	**2.9E+00**	C	**1.6E+00**	C	**5.2E+00**	N	**3.4E-02**	C	
1,2,3-Trichloropropene					1.0E-02	p			2.9E-04	p	1.0E-03	p		96-19-5	**1.6E+00**	N	**2.2E+00**	N	**2.5E+00**	N	**1.0E+00**	N	**2.1E+00**	N	
1,1,2-Trichloro-1,2,2-trifluoroethane					3.0E+01	i			8.6E+00	h				76-13-1	**5.6E+03**	sat	**5.6E+03**	sat	**5.6E+03**	sat	**3.1E+04**	N	**5.9E+04**	N	
Triethylamine					2.0E-03	r			2.0E-03	i	7.0E-03	i		121-44-8	**4.6E+01**	N	**8.8E+01**	N	**9.6E+01**	N	**7.3E+00**	N	**1.2E+01**	N	
1,2,4-Trimethylbenzene						p			2.0E-03	p	7.0E-03	p		95-63-6	**5.7E+00**	sat	**2.0E+02**	N	**2.2E+02**	N	**7.3E+00**	N	**1.5E+01**	N	
1,3,5-Trimethylbenzene					5.0E-02	p			1.7E-03	p				108-67-8	**2.1E+01**	N	**7.0E+01**	N	**7.8E+01**	N	**6.2E+00**	N	**1.2E+01**	N	
Trimethyl phosphate	3.7E-02	h					3.7E-02	r						512-56-1	**1.3E+01**	C	**1.5E+02**	C	**5.2E+01**	C	**1.8E-01**	C	**1.8E+00**	C	
1,3,5-Trinitrobenzene					3.0E-02	i			3.0E-02	r				99-35-4	**1.8E+03**	N	**6.1E+04**	N	**2.1E+04**	N	**1.1E+02**	N	**1.1E+03**	N	
Trinitrophenylmethylnitramine					4.0E-03	p			4.0E-03	r				479-45-8	**2.4E+02**	N	**8.2E+03**	N	**2.7E+03**	N	**1.5E+01**	N	**1.5E+02**	N	
2,4,6-Trinitrotoluene	3.0E-02	i		C	5.0E-04	i	3.0E-02	r	5.0E-04	r				118-96-7	**1.6E+01**	C	**1.9E+02**	C	**6.4E+01**	C	**2.2E-01**	C	**2.2E+00**	C	
Vanadium					5.0E-03	i								7440-62-2	**3.9E+02**	N	**1.0E+04**	N	**5.7E+03**	N			**1.8E+02**	N	**3.0E+02**
Vanadium pentoxide					9.0E-03	i								1314-62-1	**7.0E+02**	N	**1.8E+04**	N	**1.0E+04**	N			**3.3E+02**	N	**3.0E+02**
Vinclozolin					2.5E-02	i			2.5E-02	r				50471-44-8	**1.5E+03**	N	**5.1E+04**	N	**1.7E+04**	N	**9.1E+01**	N	**9.1E+02**	N	
Vinyl acetate					1.0E+00	h			5.7E-02	i	2.0E-01	i		108-05-4	**9.9E+02**	N	**1.4E+03**	N	**1.6E+03**	N	**2.1E+02**	N	**4.1E+02**	N	**8.0E+00**
Vinyl bromide	1.1E-01	r			8.6E-04	r	1.1E-01	h	8.6E-04	i	3.0E-03	i		593-60-2	**1.9E-01**	C	**4.2E-01**	C	**4.7E-01**	C	**6.1E-02**	C	**1.0E-01**	C	
Vinyl chloride	7.2E-01	l	pecial cas	A	3.0E-03	i	1.5E-02	i	2.9E-02	i	1.0E-01	i	2.0E+00	75-01-4	**4.3E-02**	C	**8.6E-01**	C	**8.6E-01**	C	**1.6E-01**	C	**1.5E-02**	C	**7.0E-04**
Warfarin					3.0E-04	i			3.0E-04	r				81-81-2	**1.8E+01**	N	**6.1E+02**	N	**2.1E+02**	N	**1.1E+00**	N	**1.1E+01**	N	
m-Xylene					2.0E+00	i			2.9E-02	i	1.0E-01	i		108-38-3	**2.1E+02**	sat	**2.1E+02**	sat	**2.1E+02**	sat	**1.0E+02**	N	**2.1E+02**	N	**1.0E+01**
o-Xylene					2.0E+00	i				x				95-47-6	**2.8E+02**	sat	**2.8E+02**	sat	**2.8E+02**	sat			**7.3E+04**	N	**9.0E+00**
p-Xylene														106-42-3	**3.7E+02**	sat	**3.7E+02**	sat	**3.7E+02**	sat					**1.0E+01**
Xylenes					2.0E-01	i			2.9E-02	i	1.0E-01	i	1.0E+04	1330-20-7	**2.1E+02**	sat	**2.1E+02**	sat	**2.1E+02**	sat	**1.0E+02**	N	**2.0E+02**	N	**1.0E+01**
Zinc					3.0E-01	i								7440-66-6	**2.3E+04**	N	**1.0E+05**	max	**1.0E+05**	max			**1.1E+04**	N	**6.2E+02**
Zinc phosphide					3.0E-04	i								1314-84-7	**2.3E+01**	N	**6.1E+02**	N	**3.4E+02**	N			**1.1E+01**	N	
Zineb					5.0E-02	i			5.0E-02	r				12122-67-7	**3.1E+03**	N	**1.0E+05**	max	**3.4E+04**	N	**1.8E+02**	N	**1.8E+03**	N	

APPENDIX F

EXAMPLE FIELD SHEETS

Field Sheet

Site Name:				City:			
Project Manager:				Activity/ASR #:		Date:	
No. Of Samples	Matrix	Location	Purpose	Depth or other Descriptor	Requested Analysis	Sampling Method	Analytical Method/SOP

Sample Collection Field Sheet

US EPA Region 7
Kansas City, KS

ASR Number: **Sample Number:** **QC Code:** **Matrix:** **Tag ID:**

Project ID No.: **EPA Project Manager:**

Project Desc:

City: **State:**

Program:

Location Desc:
_____

External Sample Number: _____

Expected Concentration (Circle One): **Low** **Medium** **High** **Date:** **Time(24hr):**

Latitude: ___ ___ ___ **Sample Collection:** **Start** ___/___/___ ____:____
Longitude: ___ ___ ___ **End** ___/___/___ ____:____

Field Measurements:

Parameter	**Value**	**Units**
Conductance, Specific	_____	umhos/cm
pH	_____	SU

Laboratory Analyses:

Container	**Preservative**	**Holding**	**Time**	**Analysis Name**

Sample Comments:

Sample Collected By: _____

APPENDIX G

DAILY QUALITY CONTROL REPORT

DAILY QUALITY CONTROL REPORT

Project Manager: _____

Project: _____

Date: _____

S	M	T	W	TH	F	S

Weather	Bright Sun	Clear	Overcast	Rain	Snow
Temp	To 32	32-50	50-70	70-85	>85
Wind	Still	Moderate	High	Gusty	
Humidity	Dry	Moderate	Humid		

Personnel on Site: _____

Contractors on Site: _____

Visitors on Site: _____

Work Performed: _____

Project: _____ **Date:** _____

Quality Control Activities (including field calibration and duplicate samples collected): _____

Problems Encountered/Corrective Actions Taken: _____

Downtime/Standby: _____

Health and Safety Activities: _____

Special Notes: _____

By: _____ **Date:** _____

APPENDIX H

EXAMPLE CHAIN-OF-CUSTODY FORM

SAMPLE CHAIN-OF-CUSTODY

Project Name: _____ **Project Location:** _____

Activity Number: _____

Project Manager: _____

Samplers: _____

Sample Date	Time	Sample Identification	Preservative	No. of Containers	Type of Containers	Analysis

Remarks/Additional or Special Analyses: _____

Signatures	Date	Time	Mode of Shipment	Reason for change of custody
Relinquished By:				
Received By:				
Relinquished By:				
Received in Laboratory By:				